

**Eerste gefaseerd
bodemsaneringsproject :
Negende tussentijds
verslag
bodemverzorgingswerken
3M Belgium bvba
Canadastraat 11 te 2070
Zwijndrecht**

Periode augustus 2017 – juli 2020

24 september 2021

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Eerste gefaseerd bodemsaneringsproject : Negende tussentijds verslag bodemsaneringswerken 3M Belgium bvba Canadastraat 11 te 2070 Zwijndrecht

Periode augustus 2017 – juli 2020



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Akroniemen en afkortingen

BTEX	Benzeen, tolueen, ethelbenzeen en xylenen
d.s.	Droge stof
MO	Minerale olie
m-mv	Meter minus maaiveld
OVAM	Openbare Vlaamse Afvalstoffenmaatschappij
PFHS	Perfluorhexaansulfonzuur
PFOA	Perfluor-n-octaanzuur
PFOS	Perfluoroctaansulfonzuur
PFOSA	Perfluor-1-octaansulfonamide
PFBS	Perfluorbutaansulfonzuur
P&T	Pump & Treat
WWTP	Bedrijfswaterzuiveringsinstallatie 3M (“waste water treatment plant”)

1. INLEIDING

Environmental Resources Management - ERM nv (ERM) is aangesteld door 3M Belgium bvba (3M) voor de milieukundige begeleiding van de bodemsaneringswerken op het terrein van 3M gelegen aan de Canadastraat 11 te Zwijndrecht.

Op de site zijn als gevolg van voormalige activiteiten historische verontreinigingen met fluorchemicaliën (FC's) en kwik in het vaste deel van de aarde en FC's en aromatische verbindingen (voornamelijk xylenen) in het grondwater vastgesteld. In oktober 2008 is door Arcadis een eerste gefaseerd bodemsaneringsproject (BSP, Ref. 11/003460) opgesteld, waarin de saneringsaanpak voor deze verontreinigingen ter hoogte van de site bepaald is. Dit BSP is conform verklaard door de OVAM op 9 februari 2009 (ref. BB-O-DS/2009500832).

De saneringswerken zoals opgenomen in het 1^e gefaseerd BSP zijn in uitvoering waarbij momenteel verontreinigd grondwater ter hoogte van het productiegebied (gebouw 16) en het afvalwaterzuiveringsstation (WWTP) opgepompt en gezuiverd wordt (P&T); de verontreinigingssituatie wordt in deze zones periodiek gemonitord. Daarnaast worden het natuurgebied Blokkersdijk en de 2^e aquifer eveneens periodiek gemonitord om de potentiële migratie van FC's van het 3M terrein op te volgen.

De organofluorimpact ter hoogte van de zuidelijke perceelsgrens en de Palingbeek wordt momenteel door middel van een grond- en oppervlaktewatermonitoring opgevolgd. Na afronding van de Oosterweelwerken op Linkeroever is voorzien om het beschrijvend bodemonderzoek met focus op grondwater te actualiseren, waarin de nieuwe omgevingssituatie weerspiegeld zal worden. Deze actualisatie zal de basis zijn voor het uitwerken van een tweede gefaseerde saneringsaanpak voor de grondwaterverontreiniging.

De saneringswerken uitgevoerd tussen juli 2009 en juli 2017 zijn beschreven in de acht voorgaande tussentijdse verslagen.

Het voorliggend 9^{de} tussentijds rapport (TTR9) geeft een overzicht van de bodemsaneringswerken en de monitoringsresultaten van het grondwater en het oppervlaktewater, uitgevoerd in de periode van 1 augustus 2017 tot 31 juli 2020 ⁽¹⁾.

Het rapport bestaat uit de volgende hoofdstukken:

- Hoofdstuk 2 – Projectinformatie;
- Hoofdstuk 3 – Algemene beschrijving saneringswerken;
- Hoofdstuk 4 – Grondwateronttrekking bronzone;
- Hoofdstuk 5 – Resultaten uitgevoerde monitoring en conclusies;
- Hoofdstuk 6 – Voorziene werken in volgende jaar van sanering;
- Hoofdstuk 7 – Schadegeval;
- Hoofdstuk 8 – Tweede fase bodemsaneringsproject; en
- Hoofdstuk 9 – Conclusies

De tussentijdse rapportering heeft tot doel de OVAM op een globale wijze op de hoogte te houden van de vooruitgang van de bodemsaneringswerken conform het goedgekeurde bodemsaneringsproject.

⁽¹⁾ De monitoring van juli 2020 is omwille van praktische redenen uitgesteld geweest tot 4-6 augustus 2020. De resultaten van deze staalname zijn wel mee opgenomen in het rapport, maar om de consistentie met vorige rapporten te behouden, zal gesproken worden over de periode van 1 augustus 2017 tot 31 juli 2020.

De bodemsaneringsdeskundige verklaart dat hij voor het uitvoeren van deze opdracht niet in onverenigbaarheid verkeert of dat hij bij een situatie van onverenigbaarheid beheersmaatregelen heeft genomen.

Dit document is het pdf-bestand met de relevante bijlagen en figuren zoals omschreven in de recente standaardprocedure (september 2020), dat als pdf-bijlage van het xml-bestand aan de OVAM wordt overgemaakt.

De volgende bijlagen zijn van toepassing voor TTR, met verwijzing naar de relevante sectie in het rapport:

Tabel 1.1 Overzicht relevante secties TTR

Onderwerp	Verwijzing
Afwijking op de standaardprocedure	Hoofdstuk 0
Wijzigingen ten opzichte van het conform verklaard BSP	Hoofdstuk 2.5
Interventies van de OVAM	Geen interventies vereist
Vraag tot éénmalige verlenging van de termijn van 180 dagen	N.v.t.
Emissienormen en hinder	Hoofdstuk 4
Verslag met betrekking tot Achilles	N.v.t.
Keuringsattest van de installatie	N.v.t.
Verloop van de bodemsaneringswerken	N.v.t.
Financiële zekerheid	N.v.t.
Informatie over het veldwerk & analyses	Hoofdstuk 3
Gegevens met betrekking tot het onttrekkings- of injectiesysteem	Hoofdstuk 3
Onttrekkingsdebieten	Hoofdstuk 3
Gegevens met betrekking tot de ontgraving	N.v.t.
Gegevens met betrekking tot de monitoring	Hoofdstuk 4
Gegevens met betrekking tot de aangebrachte isolatie	N.v.t.
Gegevens met betrekking tot onsite grondreiniging	N.v.t.

2. PROJECTINFORMATIE

2.1 Administratieve gegevens

De administratieve gegevens zijn opgenomen in Tabel 2.1.

Tabel 2.1 Administratieve gegevens

Dossiernummer OVAM	732
Karakteristieke naam OVAM	Eerste gefaseerd bodemsaneringsproject – 9 ^{de} tussentijds verslag bodemsaneringswerken: Periode augustus 2017 – juli 2020
	3M Belgium bvba Canadastraat 11 te 2070 Zwijndrecht
Naam opdrachtgever:	3M Belgium bvba
Contactpersoon opdrachtgever:	Emma Tavernier
Adres:	Hermeslaan 7, 1831 Machelen
Telefoon:	[REDACTED]
E-mail:	[REDACTED]
Naam eigenaar bronperceel:	3M Belgium bvba
Contactpersoon eigenaar:	Emma Tavernier
Adres:	Canadastraat 11
Gemeente:	2070 Zwijndrecht
Kadastrale gegevens:	A467E
Bestemmingstype:	V (industrie)
Gelegen in waterbeschermingszone:	Neen
Kwetsbaarheid grondwater:	Zeer kwetsbaar (Ca1)
X-coördinaat centraal punt:	147.599 m
Y-coördinaat centraal punt:	213.626 m

2.2 Omgevingskenmerken en beschrijving van het terrein

De topografische ligging van de onderzoekslocatie is weergegeven op Kaart 1 en Figuur 2.1. De 3M site, alsook de terreinen gelegen aan de noordelijke en westelijke zijde ervan, hebben als bestemming industriegebied. Ten zuiden van de Expresweg is een agrarisch waardevol gebied aanwezig. Ten oosten is het natuurgebied Blokkersdijk gelegen.

Figuur 2.1 geeft een overzicht van de site en omgeving weer. Voor meer details in verband met de omgevingskenmerken wordt verwezen naar het eerste tussentijds verslag (ERM, Ref. 0102548, periode juli 2009 – juni 2010). Op deze figuur zijn eveneens enkele belangrijke zones aangeduid in het kader van de lopende saneringswerken.

Figuur 2.1 Situering van de 3M site



Het bedrijfsterrein van 3M beslaat verschillende percelen, waarvan de bedrijfsactiviteiten zich beperken tot perceel 467E met een oppervlakte van circa 32 ha. De bedrijfsactiviteiten omvatten de productie van fijnchemicaliën, waaronder gefluoreerde verbindingen en niet-gefluoreerde verbindingen, en het opwekken van fluorelastomeren.

De fabriek is gebouwd in 1970 en de productie is opgestart in 1971. In de daaropvolgende jaren is de productie (met de nodige vergunningen) verder uitgebreid. Voor meer details in verband met de historiek wordt verwezen naar het conform verklaarde bodemsaneringsproject (Eerste gefaseerd BSP, Arcadis, Ref. 11/003460, oktober 2008) en andere voorgaande onderzoeken.

2.3 Verontreinigingstoestand, saneringsconcept en belangrijkste acties voorafgaand aan voorliggend TTR

Het 1^e gefaseerd BSP heeft betrekking op de historische grondwaterverontreiniging met FC's ter hoogte van de bronzones en vluchtige aromaten, alsook op een grondhoop met licht verhoogde kwik en FC-concentraties.

Onderstaande saneringsaanpak is conform het BSP van 2008, aangevuld met de aanbevelingen opgenomen in voorgaande tussentijdse verslagen.

2.3.1 Grondhoop verontreinigd met kwik en FC-verbindingen

De afdeklaag op de grondhoop met verhoogde kwik- en FC-verbindingen is gedurende de eerste vijf jaar met een jaarlijkse frequentie gecontroleerd met het oog op erosie en de vaststelling van eventueel noodzakelijke herstelwerkzaamheden.

In 2012 is een nieuwe afdekking geplaatst waardoor het risico op uitlozing door infiltrerend regenwater geminimaliseerd is. In het 4^{de} TTR is, na overleg met OVAM (dd. 22 oktober 2013), de controlefrequentie verlaagd naar vijfjaarlijks. Op vraag van OVAM d.d. 13 april 2016 is nog een

tussentijds controle van het afdek uitgevoerd op 26 april 2016. De afdekking was op dat moment in goede staat.

2.3.2 *Vluchtige aromatenverontreiniging ter hoogte van de ondergrondse tanken*

In het BSP van 2008 was voorzien om de grondwaterverontreiniging met vluchtige aromaten ter hoogte van de ondergrondse tanken te saneren door middel van gestimuleerde biologische afbraak. In het 1ste TTR is beslist dat een actieve saneringsaanpak voor de BTEX-verontreiniging in het grondwater niet noodzakelijk was aangezien de concentraties in het grondwater sterk waren afgenoem en natuurlijke afbraak plaatsgevonden heeft. Bijgevolg werd een monitoring voorgesteld om de daling van de BTEX-grondwaterconcentraties te bevestigen. Uit de monitoring bleek dat de concentraties gedaald waren tot onder de terugsaneerwaarde; de saneringsdoelstelling werd behaald. In oktober 2014 is een eerste gefaseerd eindevaluatieonderzoek opgemaakt voor de (voormalige) verontreiniging met BTEX in het grondwater (ref. R001-0243999-v2). Dit is in juli 2015 door de OVAM conform verklaard (ref. BB-O-DS-20150289277).

2.3.3 *FC-verontreiniging*

2.3.3.1 *Algemene saneringsdoelstelling en -aanpak*

De algemene doelstelling van de sanering bestaat erin de verontreiniging met FC's in de bronzones te beheersen en zover technisch/financieel mogelijk te reduceren, en hierbij de verspreiding van de verontreiniging buiten de terreingrenzen te verminderen.

De verontreiniging op de site zal met volgende maatregelen beheerd worden:

- Grondwateronttrekking ter hoogte van de productiezone, waterzuiveringsinstallatie en de voormalige slibbekkens;
- Beheer van alle gronden op de site; en
- Plaatsing van actief koolfilters op de regenwaterriolering ter verminderen van de FC-vuilvracht naar de Schelde.

Het grondwater en oppervlaktewater buiten de terreingrenzen wordt eveneens gemonitord.

2.3.3.2 *Gebouw 16 en zone WWTP - 1^e aquifer*

De sanering is erop gericht om de verspreiding van verontreinigd grondwater tegen te gaan vanuit de gedefinieerde bronzones, en meer bepaald de concentraties hoger dan 10.000 µg/L voor de som van PFOS, PFOA en PFHS.

Na bijkomend onderzoek en verdere afperking van de 10.000 µg/l contour, was in het BSP een gefaseerde opbouw van het grondwaterextractiesysteem voorzien. Het ontrokken grondwater wordt over de afvalwaterzuiveringsinstallatie van 3M geleid.

De voorwaarden opgenomen in het BSP waarbij het extractiesysteem verder diende uitgebreid te worden, zijn:

- Gebouw 16:
 - De vuilvrachtverwijdering per tijdseenheid blijft behouden of neemt toe; en
 - Het onttrekkingssdebit bedraagt 30 L/u of meer.
- WWTP
 - Een uitbreiding van de 10.000 µg/L contourlijn voor de som van PFOA, PFHS en PFOS; en

- Het uitblijven van een reductie van de massatransfer van FC-verbindingen van de eerste naar de tweede aquifer.

De fasering en het design van de grondwateronttrekking werd gewijzigd ten opzichte van het oorspronkelijke bodemsaneringsproject; de details hierrond zijn reeds in eerdere TTR beschreven.

De grondwaterextractie bestaat (tot 2017) uit 3 extractieputten (PP01-PP04 en PP05) in de zone gebouw 16 en 6 extractieputten in de zone WWTP (PP02-06-07-08-09-10). Nadien zijn bepaalde extractieputten herplaatst en bijgeplaatst, zoals verder in voorliggend verslag beschreven zal worden (zie paragraaf 3.2).

De efficiëntie van de onttrekking wordt nagegaan en opgevolgd middels een periodieke monitoring van het grondwater.

Het opgepompte grondwater en het effluent van de zuiveringsinstallatie wordt eveneens periodiek bemonsterd voor analyse op FC-verbindingen.

2.3.3.3 Tweede aquifer

De actieve sanering van de 1^e aquifer ter hoogte van gebouw 16 en de zone WWTP is er ook op gericht om de verspreiding van met FC verontreinigd grondwater naar de 2^e aquifer te minimaliseren.

Voor de 2^e aquifer dient een reductie van de massatransfer (flux) van FC-verbindingen vanuit de eerste aquifer gerealiseerd te worden. Meer concreet is dit ingevuld als volgt:

- De concentraties aan FC verbindingen ter hoogte van de terreingrenzen mogen de 10% oplosbaarheid niet overschrijden, met PFOA als triggerfactor ⁽²⁾; en
- De concentraties aan individuele FC-verbindingen in de bronzone in de 2^e aquifer mogen maximaal met het vijfvoud stijgen ten opzichte van de concentraties bij opstart van de sanering.

Het grondwater van de 2^e aquifer wordt periodiek gemonitord om de evolutie in concentraties op te volgen. Jaarlijks wordt de monitoringsfrequentie opnieuw geëvalueerd; dit wordt telkens in de tussentijdse rapportages besproken. In 2016 is een reeks aan peilbuizen met een filterstelling in de 2^e aquifer aan het monitoringsprogramma toegevoegd.

2.3.3.4 Regenwaterriolering

In het BSP van 2008 is voorzien om het water uit de regenwaterriolering van de site dat in de Schelde geloosd wordt te behandelen met een actief koolfiltersysteem. Er dient gestreefd te worden naar een effluentconcentratie van 30 µg/L voor PFOS. Vanuit risicogebaseerd oogpunt was in het BSP van 2008 gesteld dat het niet noodzakelijk was de vuilvracht, die dagelijks via de regenwaterriolering geloosd wordt, te reduceren omdat de dagelijkse PFOS-vuilvracht naar de Schelde de ecotoxicologisch veilige waarde niet overschrijdt. De reductie van de vuilvracht in het regenwater kadert binnen de beheersing van de verontreiniging op het terrein van 3M.

In 2012 is de collector put gereinigd door het aanwezig slib te verwijderen. Dit heeft geresulteerd in een positief effect op de vuilvracht die via de regenwaterriolering in de Schelde terecht komt.

Om de vuilvracht, die het 3M terrein verlaat als gevolg van het infiltreren van potentieel verontreinigd grondwater in het regenwaterriolatingsstelsel, op te volgen, worden het debiet en de FC-concentraties van het regenwater op regelmatige tijdstippen gemonitord. Omdat de concentraties gemeten in de regenwaterriolering zeer gevoelig zijn aan fluctuaties als gevolg van verdunning door neerslag is, in overleg met OVAM, besloten om vanaf 2014 de driemaandelijkse monitoring van de

⁽²⁾ In het BSP wordt vermeld dat in de 2^{de} aquifer PFOA concentraties gemeten zijn hoger dan 10% van de oplosbaarheid (900 µg/l), waardoor de opname van de 2^{de} aquifer in het BSP noodzakelijk was. Voor PFOS, PFHS en PFOSA waren geen concentraties gemeten hoger dan 10% van de oplosbaarheid. De oplosbaarheid van PFOA ligt echter rond $9,5 \cdot 10^3$ mg/l; 10% oplosbaarheid is 950.000 µg/l.

regenwaterriolering (RW3, RW4, RW12 en RW13) te schrappen en te vervangen door een periodieke monitoring van de collector put.

3M heeft een actieplan uitgewerkt, dat momenteel in uitvoering is, om de effluentconcentraties van het regenwater, maar ook van de WWTP te reduceren. Meer details hierond zijn opgenomen onder paragraaf 4.8.2.

2.3.3.5 Natuurgebied Blokkersdijk

Om ecotoxicologische risico's te vermijden, dient een significante stijging van de concentraties aan PFOS in het oppervlaktewater van de Blokkersdijkvijver vermeden te worden.

Ter hoogte van het natuurgebied Blokkersdijk is geen actieve sanering voorzien. De concentraties in het oppervlaktewater van de Blokkersdijkvijver en de 3M vijver, alsook het grondwater stroomopwaarts van de vijvers worden op regelmatige tijdstippen opgevolgd. De frequentie van bemonstering wordt jaarlijks geëvalueerd.

Door middel van een statistische analyse van de PFOS-concentraties in het water van de Blokkersdijkvijver dient aangetoond te worden dat de concentraties statistisch niet significant stijgen. Indien een significant stijgende trend wordt waargenomen, zullen in eerste instantie meer frequent stalen genomen worden. Indien deze trend hierdoor bevestigd wordt, dient overgegaan te worden tot de implementatie van een actieve sanering. Als back-up variant is in het BSP een hydrogeologische barrière voorzien ter hoogte van het 3M pad (verbindingssweg tussen het 3M terrein en de Schelde). Deze variant is echter enkel zinvol indien de stijging van de concentraties aan PFOS in de Blokkersdijkvijver het gevolg is van de instroom van verontreinigd grondwater.

Op de grondwaterconcentraties stroomopwaarts van Blokkersdijk wordt eveneens een statistische analyse uitgevoerd. Indien een statistische stijging wordt aangetoond van de concentraties in het grondwater, dient de monitoringsfrequentie van de 3M vijver en de Blokkersdijkvijver opgedreven te worden. Een statistisch significante stijging in het grondwater is niet noodzakelijk een aanleiding om tot een actieve saneringsvariant over te gaan. Hiervoor dient een statistisch significante stijging van de PFOS-concentratie in de Blokkersdijkvijver aangetoond te zijn.

Sinds de start van de monitoring zijn er verschillende laboratoria gebruikt voor de analyse van de waterstalen. Mogelijk heeft het gebruik van verschillende labo's een impact gehad op het resultaat van de PFOS-trend analyse. Uit een evaluatie uitgevoerd in 2015 (zie TTV6), waarbij de resultaten van de statistische evaluatie met de volledige dataset (verschillende labo's) zijn vergeleken met de resultaten van de statistische evaluatie enkel gebruik makend van de dataset van het 3M Environmental laboratorium in de Verenigde Staten, blijkt dat de wisseling van labo's invloed heeft gehad op de PFOS-trend analyse. Daarom is in overleg met en akkoord van OVAM enkel nog de data van het 3M Environmental laboratorium in de Verenigde Staten gebruikt, met controle analyses door SGS nv.

2.3.3.6 Zuidelijke terreingrens, Palingbeek en Tophatgracht

De aanpak van de grondwaterverontreiniging aan de zuidelijke perceelgrens, inclusief de Palingbeek en de Tophatgracht, zal in het kader van een 2^e gefaseerd BSP uitgewerkt worden. Dit 2^e gefaseerd BSP zal immers rekening moeten houden met de topografische en hydrogeologische wijzigingen als gevolg van de Oosterweelwerken. In afwachting van het 2^e gefaseerd BSP wordt de grondwaterkwaliteit ter hoogte van de zuidelijke perceelsgrens opgevolgd, alsook het oppervlaktewater van de Palingbeek en Tophat gracht.

2.3.3.7 Gebied ten zuiden van de Expresweg (Z-peilbuizen)

Om de evolutie van de FC-concentraties in het landbouwgebied ten zuiden van de Expresweg na te gaan, zijn enkele peilbuizen periodiek bemonsterd geweest voor analyse op FC. Uit de monitoring bleek dat de concentraties fluctueren maar globaal gezien stabiel bleven en vergelijkbaar waren met de concentraties gemeten voor de start van de sanering. De op dat moment geldende

saneringsdoelstelling was behaald. Een tweede gefaseerd eindevaluatieonderzoek met referentie R005-0243999 is op 1 juli 2015 door de OVAM conform verklaard (ref. BB-O-DS-20150289432). Het momenteel lopende beschrijvend bodemonderzoek zal ook voor deze zone een uitspraak doen over mogelijke risico's en saneringsnoodzaak, rekening houdende met het huidige regelgevend kader.

2.3.3.8 Vuilvracht richting de Schelde

De geraamde vuilvracht van PFOS naar de Schelde, indien de hogervermelde saneringsaanpak zoals bepaald in het BSP van 2008 wordt gevolgd, is samengevat in onderstaande tabel.

Tabel 2.2 Geraamde PFOS vuilvracht naar de Schelde zoals opgenomen in het 1^e gefaseerd BSP tijdens en na de sanering

Vuilvracht naar de Schelde	Debit (m ³ /dag)	Geraamde PFOS-concentratie (µg/L)	Geraamde vuilvracht (g/dag)
Effluent van het bedrijfsafvalwater	700	30 (worst case)	21
Effluent van de grondwaterextractie	33	30 (worst case)	1,0
Effluent Regenwaterriolering	325	30 (streefwaarde)	9,8
Palingbeek	2.865	43,7	125
Totaal			157

Hierbij is in het BSP van 2008 uitgegaan van een PFOS-concentratie gelijk aan de lozingsnorm voor het effluent van de waterzuiveringsinstallatie en een concentratie van 30 µg/L voor de regenwaterriolering. Deze laatste wordt in het BSP van 2008 beschouwd als een streefwaarde.

De evaluatie van de Best Beschikbare Techniek voor de waterzuivering is opgesteld door EPAS (referentie PR04.042.06). In deze studie is de veilige PFOS-vuilvracht voor de Schelde geraamd op 370 g/dag.

Tijdens de sanering wordt ieder jaar de totale vuilvracht (PFOS) naar de Schelde berekend op basis van de gegevens verzameld tijdens de monitoringscampagne.

2.3.4 Betrokken partijen

De installatiewerken en het onderhoud van de installatie zijn uitgevoerd door DEC nv tot juni 2019. Vanaf juli 2019 is Envisan nv (Envisan) aangesteld als saneringsaannemer op de site van 3M. Envisan is lid van de OVB (Ondernemers Vereniging Bodemsaneerders vzw) en is gecertificeerd volgens het Achilles Zorgsysteem.

Als erkend bodemsaneringsdeskundige type 2 staat ERM in voor de nodige monstername, analyse en rapportage.

De coördinaten van de bij de sanering betrokken partijen zijn in Tabel 2.3 weergegeven.

Tabel 2.3 Betrokken partijen

Bouwheer	
Volledige naam	3M Belgium bvba
Adres	Hermeslaan 7, 1831 Machelen
Telefoon	[REDACTED]
Contactpersoon	Emma Tavernier
Telefoon contactpersoon	[REDACTED]
E-mail	[REDACTED]
Erkende bodemsaneringsdeskundige (EBSD, type 2)	
Volledige naam	ERM nv
Adres	Posthoflei 5 bus 6, 2600 Antwerpen (Berchem)
Telefoon	[REDACTED]
Contactpersoon	Lieselotte Sorgeloos
Telefoon contactpersoon	[REDACTED]
E-mail	[REDACTED]
Veiligheidscoördinator (ontwerp en realisatie)	
Volledige naam	ERM NV
Adres	Posthoflei 5 bus 6, 2600 Antwerpen (Berchem)
Telefoon	[REDACTED]
Contactpersoon	Erik Boeckx
E-mail	[REDACTED]
Hoofdaannemer	
Volledige naam	Envisan NV
Adres	Tragel 60, 9308 Hofstade (Aalst)
Achilles	Lid OVB – Achilles Zorgsysteem
Telefoon	[REDACTED]
Contactpersoon	Kris Dendoncker
GSM	[REDACTED]

2.4 Afwijkingen op de standaardprocedure

Voor de opvolging van de lopende saneringswerken op de site te Zwijndrecht zijn er geen afwijkingen op de standaardprocedure van toepassing.

2.5 Wijzigingen ten opzichte van het conform verklaarde BSP

De wijzigingen relevant voor de beschouwde periode van dit TTV worden hieronder overzichtelijk weergegeven. Meer details kunnen teruggevonden worden in navolgende secties.

2.5.1 Kleine wijziging – Verplaatsing PP01 en PP09

Zoals met OVAM besproken tijdens een overleg op 28 september 2018 en via email dd. 9 maart 2018 gecommuniceerd, worden pompputten PP01 en PP09 in 2018-2019 verplaatst omwille van

toekomstige werken nabij de pompputten. De communicatie met OVAM hierond is in Bijlage 1 opgenomen.

Op 16 oktober 2018 is pompput PP01 ter hoogte van gebouw 16 (zie locatie Kaart 5) buiten gebruik gesteld. Deze pompput moest verplaatst worden omwille van de constructie van het nieuwe gebouw 036 (project CS17). Op 4 december 2018 is pompput PP11 ter vervanging geïnstalleerd door Bouter/Geotron door middel van een pulsboor.

PP09 is op 4 juli 2019 buiten gebruik gesteld door Envisan. De pompput moet verplaatst worden omwille van het plaatsen van een berm in het kader van de Oosterweel werken. Om de captatie van de verontreiniging zo efficiënt mogelijk te houden, is besloten om PP09 te vervangen door twee nieuwe pompputten: PP12 en PP13. De effectieve installatie van de pompputten was ingepland in februari 2020, maar is omwille van COVID-19 eind augustus 2020 uitgevoerd.

Het verplaatsen van de twee pompputten wordt beschouwd als een kleine wijziging.

2.5.2 Kleine wijziging – Duplicaatstalen SGS

Tijdens het overleg van 28 september 2018 is aan OVAM voorgesteld om de duplicaat-analyses, die uitgevoerd worden door het door OVAM erkend laboratorium SGS en die dienen als verificatie van de analyses uitgevoerd door het 3M Environmental laboratorium in de VS, af te bouwen. Voordien werden alle grondwaterstalen op FC's in het 3M Environmental laboratorium geanalyseerd. Aangezien dit geen door OVAM erkend labo is, is in oktober 2013 met OVAM afgesproken dat van elke meetlocatie minstens één grondwaterstaal jaarlijks in duplicaat geanalyseerd wordt door SGS. De volgende aanpassing van het analyseschema werd voorgesteld voor de monitoringsronden vanaf januari 2019:

- Alle waterstalen die in het kader van de saneringsmonitoring worden genomen, zullen nog steeds geanalyseerd worden in het 3M Environmental laboratorium in de VS;
- Alle stalen die nodig zijn voor de PNEC-evaluatie, meer bepaald de waterstalen van het effluent van de bedrijfswaterzuivering, het regenwater van de collector put en het oppervlaktewater ter hoogte van het bemalingsstation, zullen viermaal per jaar geverifieerd worden door duplicaatanalyses die door SGS uitgevoerd worden;
- De oppervlaktewaterstalen van de Blokkersdijkvijver en 3M vijver, en de grondwaterstalen van de monitoringspeilbuizen ter hoogte van het 3M pad (aanpalend aan het natuurreervaat Blokkersdijk) zullen éénmaal per jaar geverifieerd worden door duplicaatanalyses door SGS;
- De grondwaterstalen van de monitoringspeilbuizen ter hoogte van de twee bronzones (gebouw 16 en zone bedrijfswaterzuivering), zowel van de eerste als de tweede aquifer, zullen éénmaal in de drie jaar geverifieerd worden door duplicaatanalyses die door SGS uitgevoerd worden; en
- De grondwaterstalen van de extractieputten zullen éénmaal per jaar geverifieerd worden door SGS door duplicaatanalyses.

Verder is voorzien dat, indien er tijdens een monitoringsronde afwijkende resultaten worden gemeten, geëvalueerd zal worden of een heranalyse dan wel een bijkomende duplicaat staalname tijdens de volgende monitoring noodzakelijk is. De communicatie met OVAM en memo (Ref. M005-0451640-V1) hieromtrent zijn in Bijlage 1 terug te vinden.

2.5.3 Kleine wijziging – Verandering van laboratorium

Er wordt voorgesteld om vanaf juli 2020 voor het uitvoeren van de grond- en oppervlaktewater analyses volledig over te stappen naar het door OVAM erkende laboratorium SGS nv. Echter, aangezien er voor de statistische analyse (paragraaf 4.6.3) sinds 2014 gebruik wordt gemaakt van de resultaten van het 3M Environmental laboratorium in de VS, wordt er voorgesteld om voor deze 10 locatiepunten (L4, L21, L22, L31, P114bis, P115, P116, Blokkersdijk noord, Blokkersdijk standaard,

3M vijver) de analyses door het 3M Environmental laboratorium in de VS te behouden. De stalen zullen jaarlijks wel nog geverifieerd worden door dupliaatanalyses van SGS.

De wekelijkse analyses op het effluent van de WWTP en de collector put door het intern laboratorium van 3M Zwijndrecht blijft wel behouden.

Het veranderen van laboratorium voor het uitvoeren van de periodieke analyses wordt beschouwd als een kleine wijziging.

2.5.4 Kleine wijziging – Monitoringsfrequentie peilbuizen

De monitoringsfrequentie wordt jaarlijks herzien en aangepast op basis van de resultaten. Eventuele aanpassingen die in de periode augustus 2017 – juli 2020 zijn gebeurd, zijn verwerkt in hoofdstuk 4.

Er wordt voorgesteld om vanaf 2021 de volgende wijzigingen in het monitoringsprogramma door te voeren:

- Verhoging van monitoringsfrequentie van jaarlijkse naar halfjaarlijks voor de volgende peilbuizen: K3 en P382. De monitoringsfrequentie wordt verhoogd voor K3 om de 10.000 µg/L contour beter te kunnen opvolgen. Voor P382 wordt het verhoogd om de effecten van de Oosterweel werken en de onttrekking door de nieuwe pompput PP13 op te volgen;
- Verlaging van de monitoringsfrequentie omwille van continue lage en stabiele concentraties voor de volgende peilbuizen:
 - Driemaandelijkse naar halfjaarlijks: P21B, P321 en L21, L22, L31, L4, P114bis en P116; en
 - Halfjaarlijkse naar jaarlijks: P118C, P264, P340, P341, P379, D17, D18, D14, P121, P119A en P119B.
- Door de verandering in het gebruik van laboratoria, zullen de stalen van het effluent van de WWTP en de collector put maandelijks door het erkende SGS laboratorium geanalyseerd worden in plaats van driemaandelijkse door het 3M Environmental laboratorium in de VS (met jaarlijkse verificatie door SGS); en
- Stopzetting van monitoring van peilbuizen P381 en P265B omwille van stabiele en voldoende lage concentraties. P382 ligt vlak naast peilbuis P381 en peilbuis P265B naast P118C. Filterstellingen zijn gelijkaardig en P382 en P118C zullen wel in het monitoringsprogramma blijven.

De volgende peilbuizen zijn gedurende de periode augustus 2017 – juli 2020 buiten gebruik gesteld of beschadigd geweest: D09, L22, P262, P56, P300, P343, D16, P28, B3-bis, B7, P378, PA109A, PA111A en PA112.

- Peilbuis P262 werd vervangen in juli 2019 en peilbuizen L22 en D09 zullen vervangen worden in augustus 2020;
- Er wordt aangeraden om peilbuis P28, P56, P300, P343 en D16 zo snel mogelijk te vervangen na afronding van de constructiewerken van het gebouw 036; en
- Peilbuizen B3-bis, B7, P378, PA109A, PA111A en PA112 zijn peilbuizen ter hoogte van de zuidelijke perceelsgrens die in het kader van de Oosterweelwerken buiten gebruik gesteld zijn. In deze zone zal na het afronden van de Oosterweelwerken kritisch beoordeeld worden waar er nieuwe peilbuizen geplaatst moeten worden.

Het volledig aangepast monitoringsprogramma voor 2021 is eveneens opgenomen in Tabel 5.1.

3. OPVOLGING VAN HET GRONDWATERONTTREKKINGSSYSTEEM

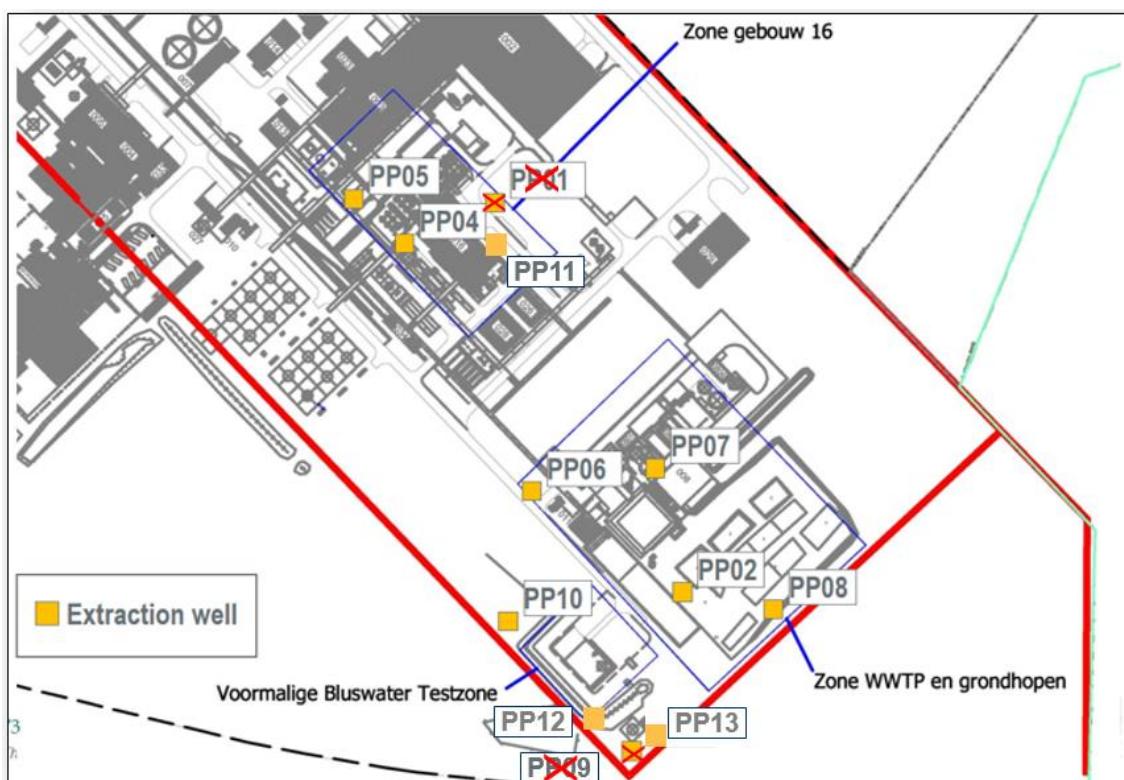
3.1 Algemeen en overzicht uitgevoerde werken

Via het P&T-systeem worden saneringswerkzaamheden uitgevoerd om de migratie van FC verontreinigd grondwater uit de twee bronzones (gebouw 16 en WWTP) te beperken.

In totaal zijn er negen pompputten in werking. Er zijn drie pompputten in de zone gebouw 16 (PP01/PP11-PP04-05) en zes pompputten in de zone WWTP (PP02-06-07-08-09-10).

De locaties van de respectievelijke pompputten zijn aangeduid op Kaart 5 en in Figuur 3.1.

Figuur 3.1 Locatie pompputten P&T systeem



De hieronder besproken resultaten hebben betrekking op de periode van 1 augustus 2017 tot 31 juli 2020. Tot juni 2019 was DEC nv de saneringsaannemer op de site van 3M. Vanaf juli 2019 heeft Envisan nv deze rol overgenomen.

Volgende activiteiten zijn uitgevoerd:

- Exploitatie en onderhoud van het pump & treat (P&T) systeem dat in de twee bronzones (gebouw 16 en WWTP) is geïnstalleerd:
 - Elk kwartaal is het grondwater in de onttrekkingsputten bemonsterd; en
 - Een chemische reiniging van de P&T installatie is uitgevoerd in september 2017, maart 2018, september 2018, juli 2019, januari 2020 en juli 2020. In december 2017, december 2018, oktober 2019, april 2020 en november 2020 is een lichtere reiniging uitgevoerd.
- Ontmanteling van de extractieputten PP01 (oktober 2018) en PP09 (juli 2019) in verband met bouwwerkzaamheden. PP11 is in de plaats van PP01 geïnstalleerd (december 2018). PP09 is vervangen door de nieuwe pompputten PP12 en PP13 (augustus 2020).

3.2 Verplaatsing PP01 en PP09

Op 16 oktober 2018 en 4 juli 2019 zijn, respectievelijk, pomputten PP01 en PP09 met bentoniet buiten gebruik gesteld. Pompput PP01 is buiten gebruik gesteld omwille van de constructie van gebouw 036 (project CS17) en is vervolgens op 4 december 2018 vervangen geweest door pompput PP11. In overleg met 3M is de meest praktisch haalbare locatie uitgekozen om PP11 te plaatsen. De werken zijn uitgevoerd door Bouting/Geotron met behulp van een pulsboor.

Pompput PP09 is op 4 juli 2019 buiten gebruik gesteld omwille van het plaatsen van een berm voor de Oosterweelwerken. Om een maximale captatie te voorzien is stroomafwaarts van de verontreiniging besloten om PP09 te vervangen door twee nieuwe pomppetten: PP12 en PP13. Om de meest geschikte locatie en filterdiepte te bepalen van de nieuwe pomppetten zijn in oktober 2019 onder supervisie van ERM zes geoprobe boringen uitgevoerd door de boorfirma BP². Op de grondstalen zijn vervolgens korrelgrootte analyses uitgevoerd om de meest geschikte diepte voor de filters te bepalen. De effectieve installatie van de pomppetten was ingepland in februari 2020, maar is omwille van COVID-19 restricties pas uitgevoerd eind augustus 2020 uitgevoerd. In februari 2020 zijn echter wel al de volgende werken uitgevoerd: het graven van sleuven en het plaatsen van tijdelijke toezichtspotten. Op basis van de geoprobe boringen en korrelgrootte analyses zullen de pomppetten met de volgende filterstellingen geïnstalleerd worden: PP12 (3-4,7 m-mv) en PP13 (3,5-5,9 m-mv).

De verschillende milieudagboeken van deze werken (installatie PP11, buiten gebruik stelling pomppetten, geoprobe boringen) en boorprofielen zijn te vinden in Bijlage 2.

3.3 Onderhoud onttrekkingssysteem

ERM inspecteert regelmatig het P&T systeem om de werking van het systeem te controleren. Aan de hand van milieudagboeken wordt vervolgens de saneringsaannemer en 3M op de hoogte gehouden van de status van de installatie.

In de beschouwde periode (augustus 2017- juli 2020) werd het onttrekkingssysteem periodiek gereinigd om het rendement van de onttrekkingssputten zo hoog mogelijk te houden. Deze reinigingen werden van september 2017 tot en met december 2018 uitgevoerd door DEC nv, en vanaf juli 2019 tot en met juli 2020 door Envisan nv, steeds onder de supervisie van ERM.

De voornaamste reden voor een daling in rendement zonder aantoonbare technische reden is de verstopping van het filterpakket met ijzerslib. Om deze verstopping tegen te gaan wordt om de 6 maanden een uitgebreide chemische reiniging uitgevoerd. Deze chemische reiniging wordt voorafgegaan door een reiniging met een vacuümtruck. Na de chemische reiniging worden de putten opnieuw gespoeld en worden de pompen teruggehangen. Een dergelijke chemische reiniging vond in desbetreffende periode plaats in september 2017, maart 2018, september 2018, juli 2019, januari 2020 en juli 2020. Drie maanden na elke uitgebreide chemische reiniging wordt ook steeds een beperkte reiniging van het onttrekkingssysteem uitgevoerd. Hierbij worden de putten opnieuw leeg getrokken met een vacuümtruck en gespoeld. Dergelijke beperkte reinigingen vonden plaats in december 2017, december 2018, oktober 2019 en april 2020. Bij de overgang van subcontractor hebben zowel de uitgebreide chemische reiniging van maart 2019 als de beperkte reiniging van juni 2019 niet plaatsgevonden, deze zijn respectievelijk in juli en oktober 2019 heropgestart waarna de halfjaarlijkse frequentie opnieuw is aangehouden.

De schematische weergave van de inspectiekamer van een pompput is te vinden in Bijlage 3. Bijlage 4 bevat de rapporten en dagboeken van de uitgevoerde reinigingen door DEC en Envisan.

3.4 Bespreking resultaten

3.4.1 Uptime en ontrokken debieten

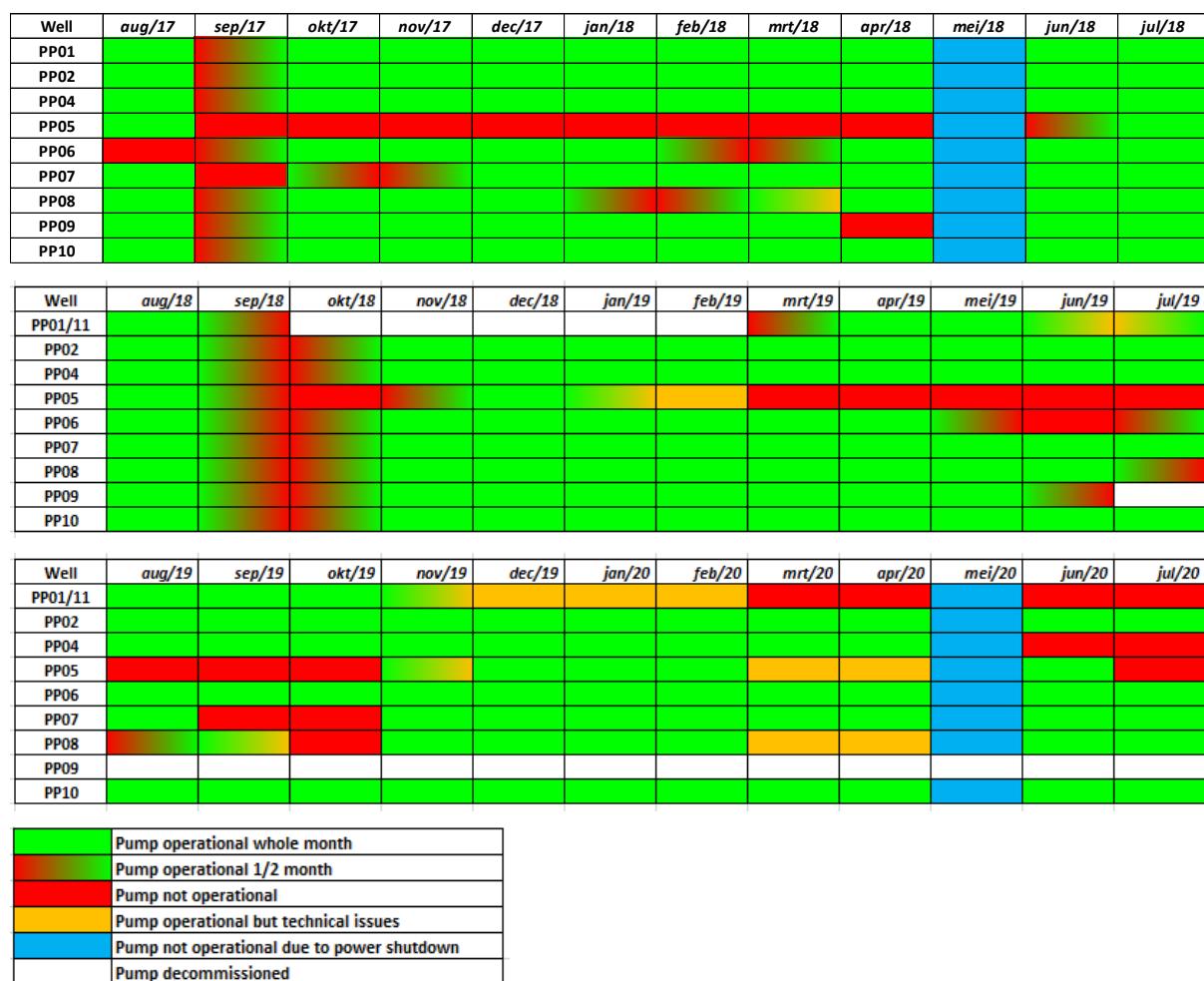
De jaarlijkse gemiddelde uptime van het P&T systeem is weergegeven in onderstaande tabel. Sinds 2017 is de uptime van het systeem relatief constant.

Tabel 3.1 Jaarlijkse gemiddelde uptime van het P&T systeem

Periode	Gemiddelde uptime (%)
2019-2020	78
2018-2019	79
2017-2018	76
2016-2017	81
2015-2016	53
2014-2015	73
2013-2014	71

Zoals uit onderstaande Figuur 3.2 en Tabel 3.2 blijkt, is er een groot verschil tussen de werking van de verschillende pompputten. Zo heeft gedurende de periode augustus 2017 tot juli 2020, pompput PP05 een minimale uptime van 29% gehaald, terwijl pompputten PP02, PP04, PP06, PP07 en PP10 een uptime hebben gerealiseerd tot 92%.

Figuur 3.2 P&T systeem – Maandelijks overzicht van uptime



Tabel 3.2 Overview uptime per pompput

Pompputten	Uptime 2017-2018 (%)	Uptime 2018-2019 (%)	Uptime 2019-2020 (%)
PP01/PP11	88	86	58*
PP02	88	92	92
PP04	88	92	75
PP05	29**	46***	58***
PP06	71	75	92
PP07	79	92	75
PP08	79	88	81
PP09	79	86	/
PP10	88	92	92

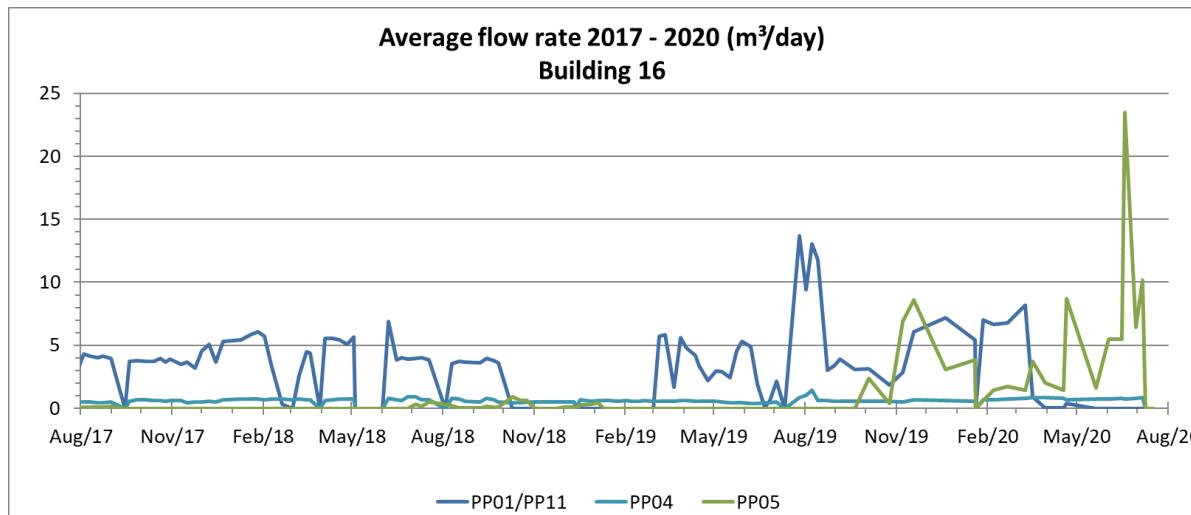
* PP11 heeft van maart '20 tot juli '20 afgestaan omdat van de lange wachttijd voor de levering van een nieuwe pomp.

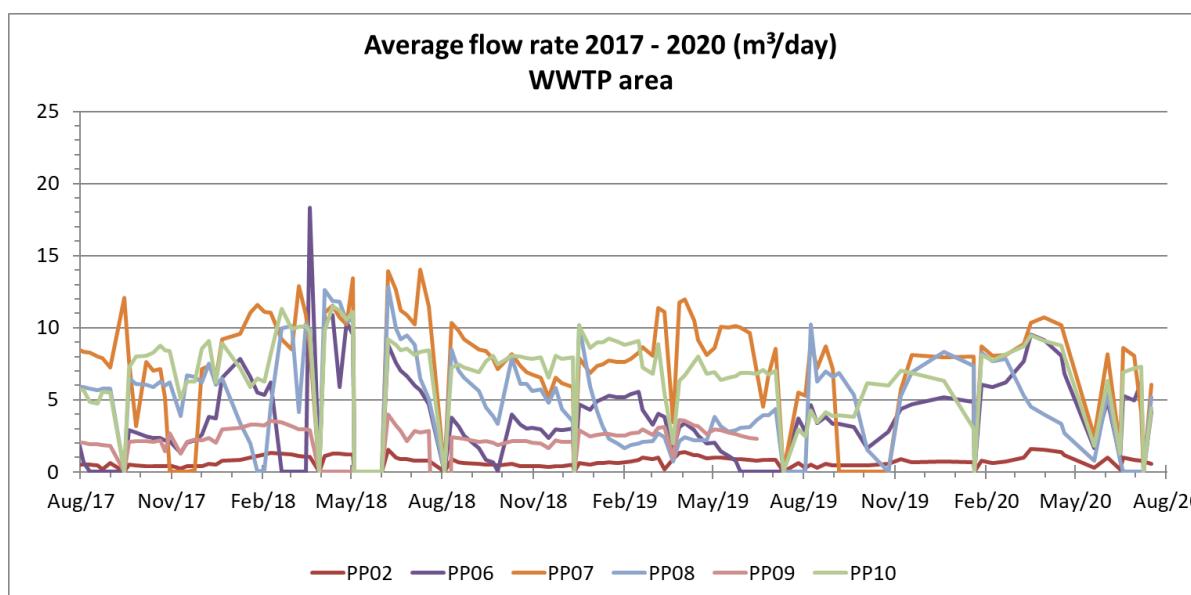
** PP05 heeft van september '17 tot juli '18 niet gewerkt wegens aantasting van de pomp door zwarte depositie.

*** PP05 heeft van maart '19 tot oktober '19 afgestaan wegens de werken die in de omgeving hebben plaatsgevonden (plaatsen van een nieuwe bovengrondse tank).

3M bezorgt ERM en Envisan wekelijkse PLC-data om de werking van het systeem op te volgen. De onderstaande grafieken stellen de wekelijks gemiddelde debieten voor per pompput per bronzone voor de periode van augustus 2017 tot juli 2020. Algemeen liggen de debieten in pompputten PP02, PP04 en PP05 vrij laag, hoofdzakelijk doordat de toestroming in de pompputten erg gering is. Bij PP05 is een harde afzetting geconstateerd in de pomp en debietmeter dat voor herhaalde schade aan respectievelijk de pomp en de registratie in de debietsmeter zorgt.

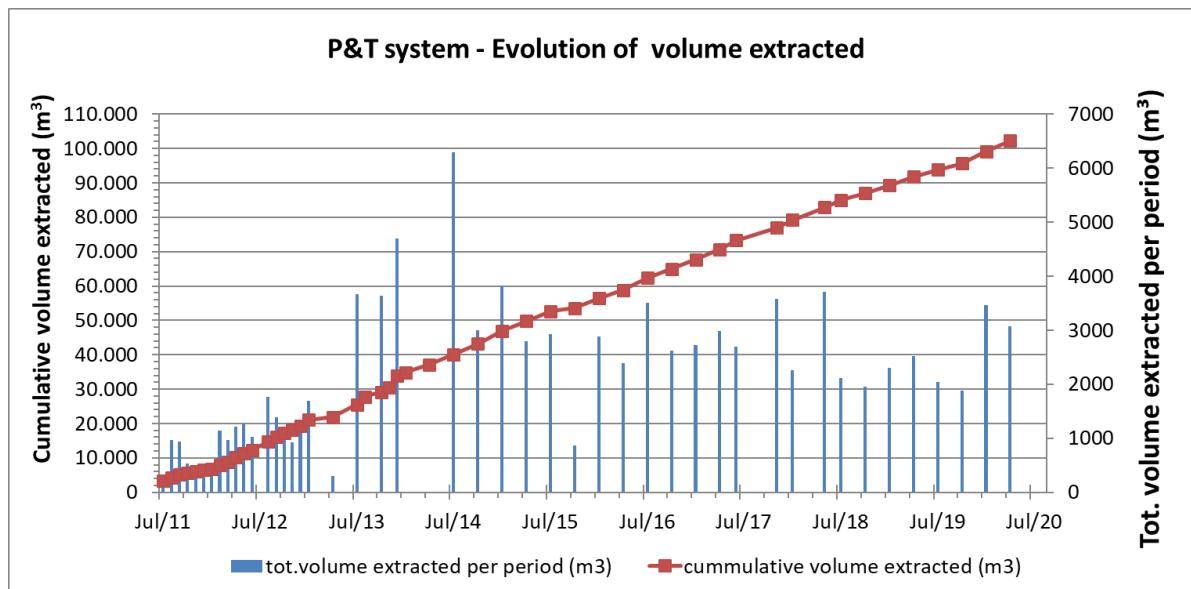
Figuur 3.3 Gemiddeld debiet per onttrekkingsput per bronzone (augustus 2017 tot juli 2020)





Onderstaande grafiek geeft het cumulatief onttrokken volume door het P&T systeem weer sinds de start van de installatie. Tussen augustus 2017 en juli 2020 is ongeveer 31.100 m³ verontreinigd grondwater onttrokken. In totaal is sinds juli 2011 circa 108.200 m³ verontreinigd grondwater onttrokken.

Figuur 3.4 Cumulatief onttrokken volume van het P&T systeem



3.4.2 Monitoring grondwaterstanden

Om de seizoenale fluctuaties van het grondwaterpeil en de invloed van de pompputten beter te kunnen opvolgen, wordt er sinds 2015 elk kwartaal een dipronde uitgevoerd samen met de bemonsteringscampagnes voorzien in januari, april, juli en oktober.

In oktober 2017, april 2018, april 2019 en april 2020 is een uitgebreide dipronde uitgevoerd waarbij de grondwaterstand in een grote hoeveelheid peilbuizen op en in de onmiddellijke nabijheid van de site

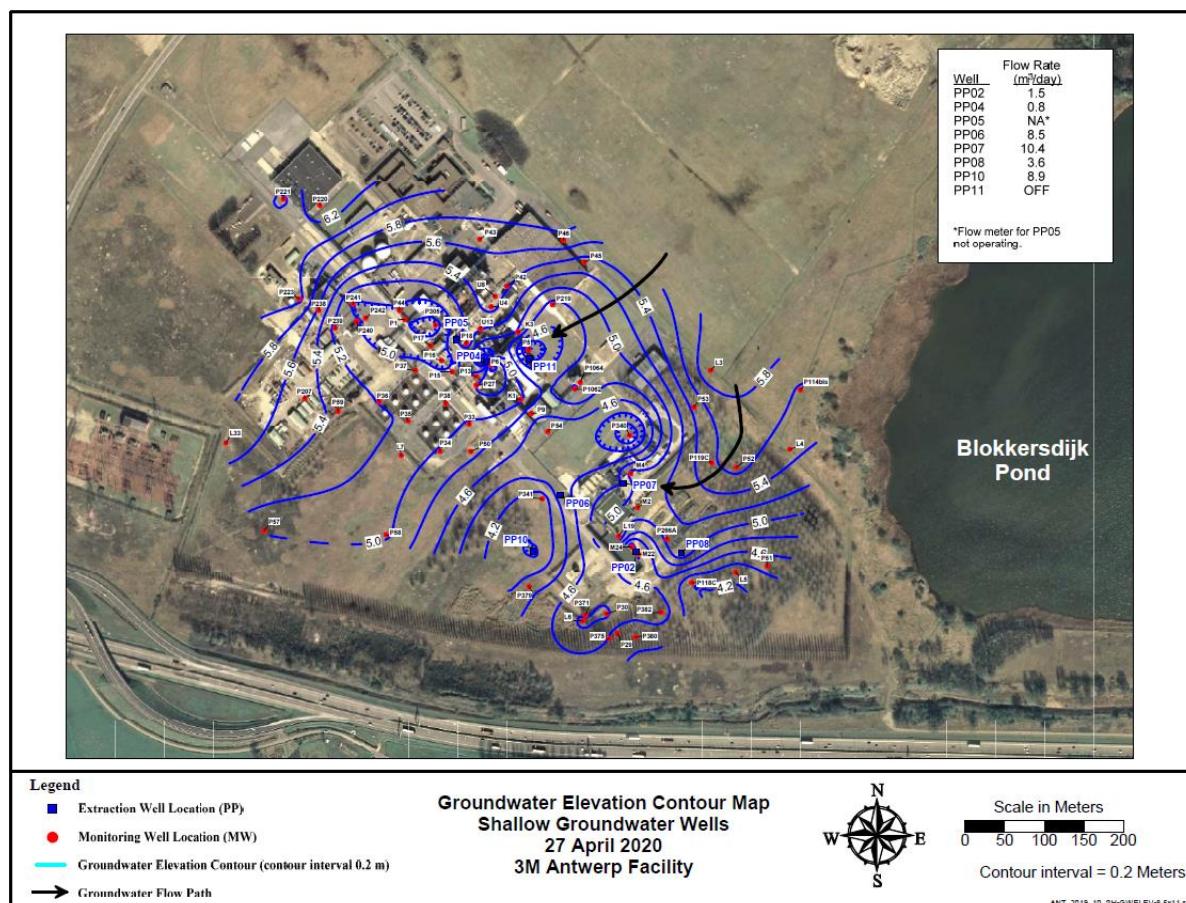
is opgemeten. Gedurende deze uitgebreide diprondes worden zowel de grondwaterstanden in de 1^e als in de 2^e aquifer opgemeten. Tijdens de overige diprondes (januari 2018, juli 2018, januari 2019, juli 2019, oktober 2019, januari 2020 en augustus 2020) is een beperkte dipronde uitgevoerd waarbij enkel de grondwaterstand in peilbuizen in de 1^e freatiche aquifer zijn opgemeten.

De isohypsenkaarten die opgemaakt zijn op basis van de meetresultaten van de meest recente uitgebreide dipronde (april 2020) zijn weergegeven in Figuur 3.5.

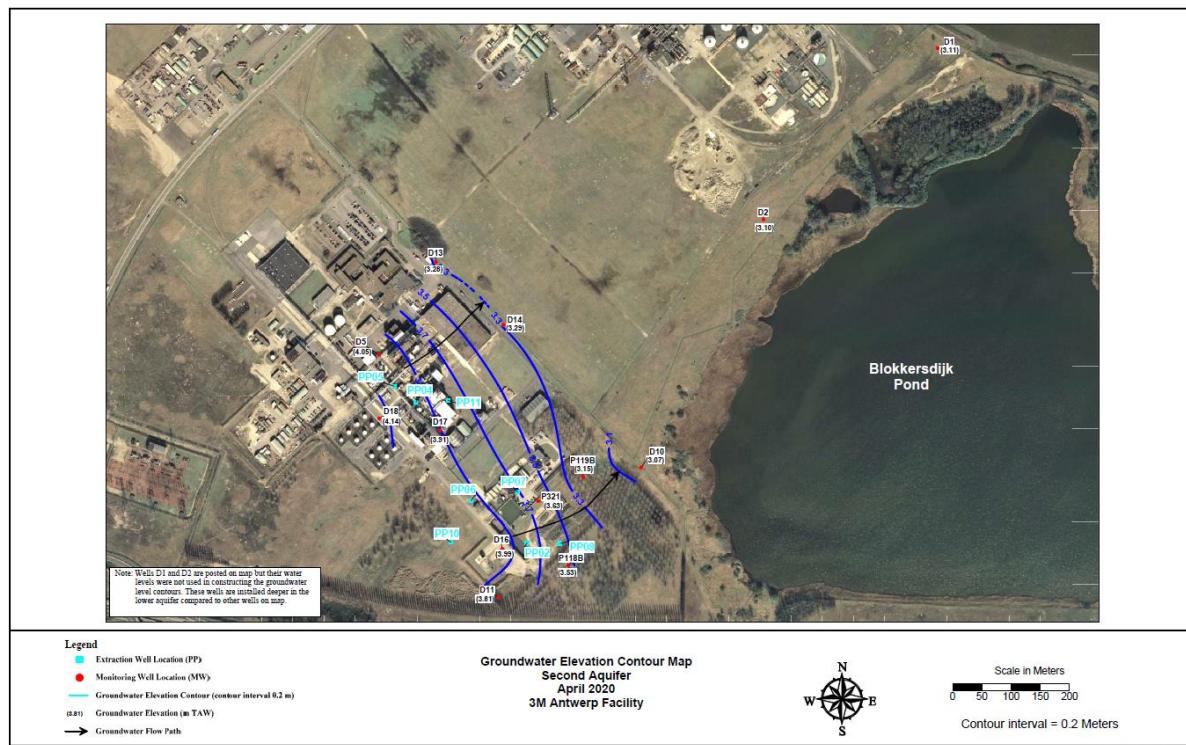
In de 1^e aquifer is een duidelijke invloedszone van de pompputten op te merken, in de vorm van lokale dalingen in grondniveau rond de pompputten. Deze komen min of meer overeen met de voorspelde toestand van het voor de sanering opgestelde grondwatermodel (zie TTV4). De grondwaterstanden zullen volgende periode verder worden opgevolgd met een ongewijzigde frequentie van viermaal per jaar. In de 2^e aquifer is de invloed van de pompputten niet zichtbaar.

Figuur 3.5 Isohypsenkaart 1^e en 2^e aquifer (april 2020)

Isohypsenkaart 1^e freatiche aquifer april 2020



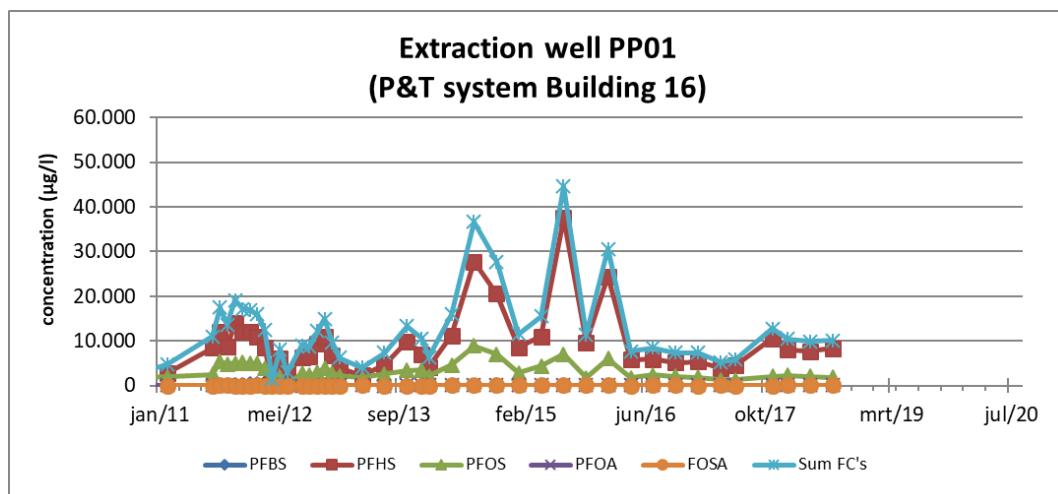
Isohypsenkaart 2^e aquifer april 2020

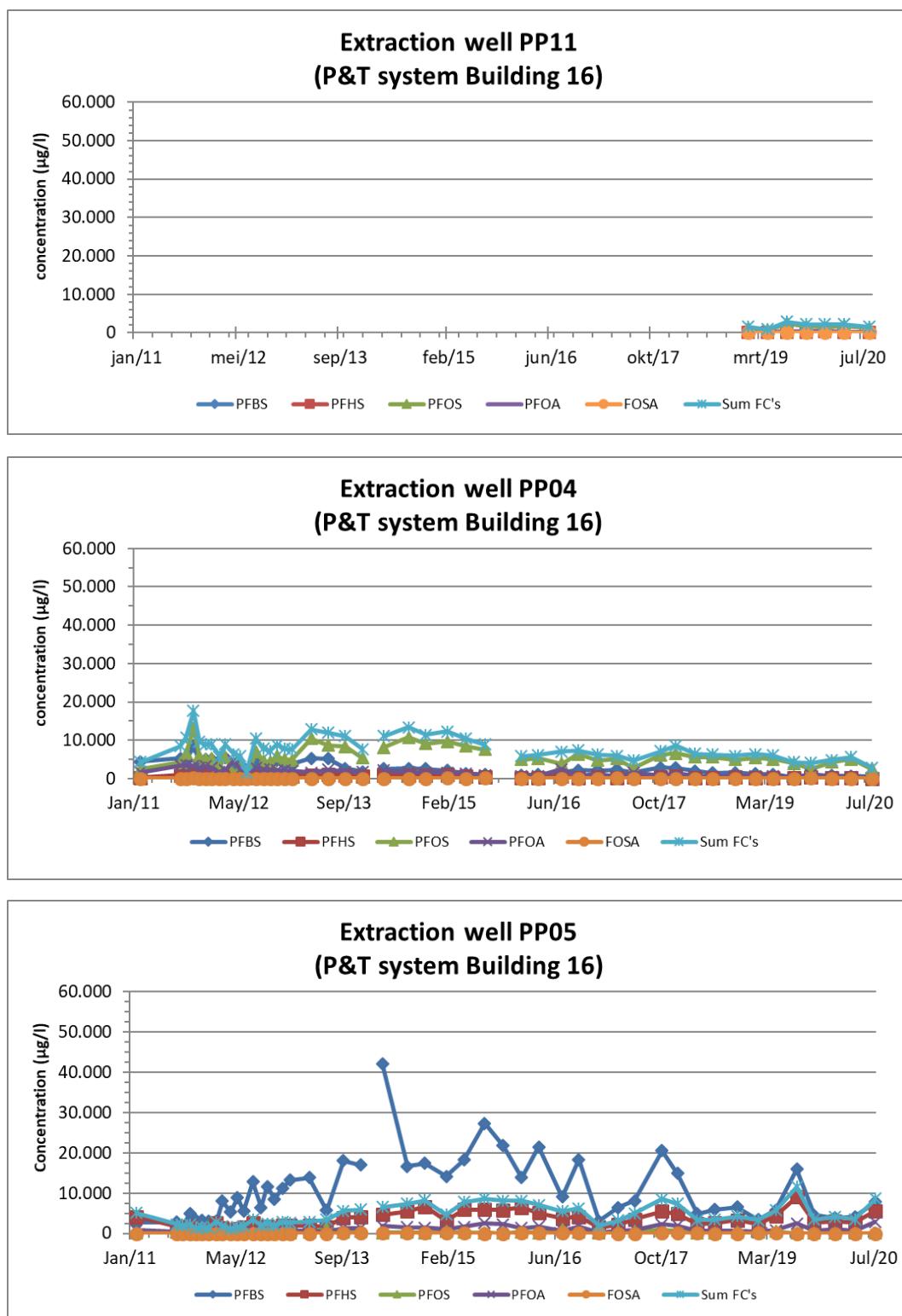


3.4.3 Concentraties in het ontrokken grondwater

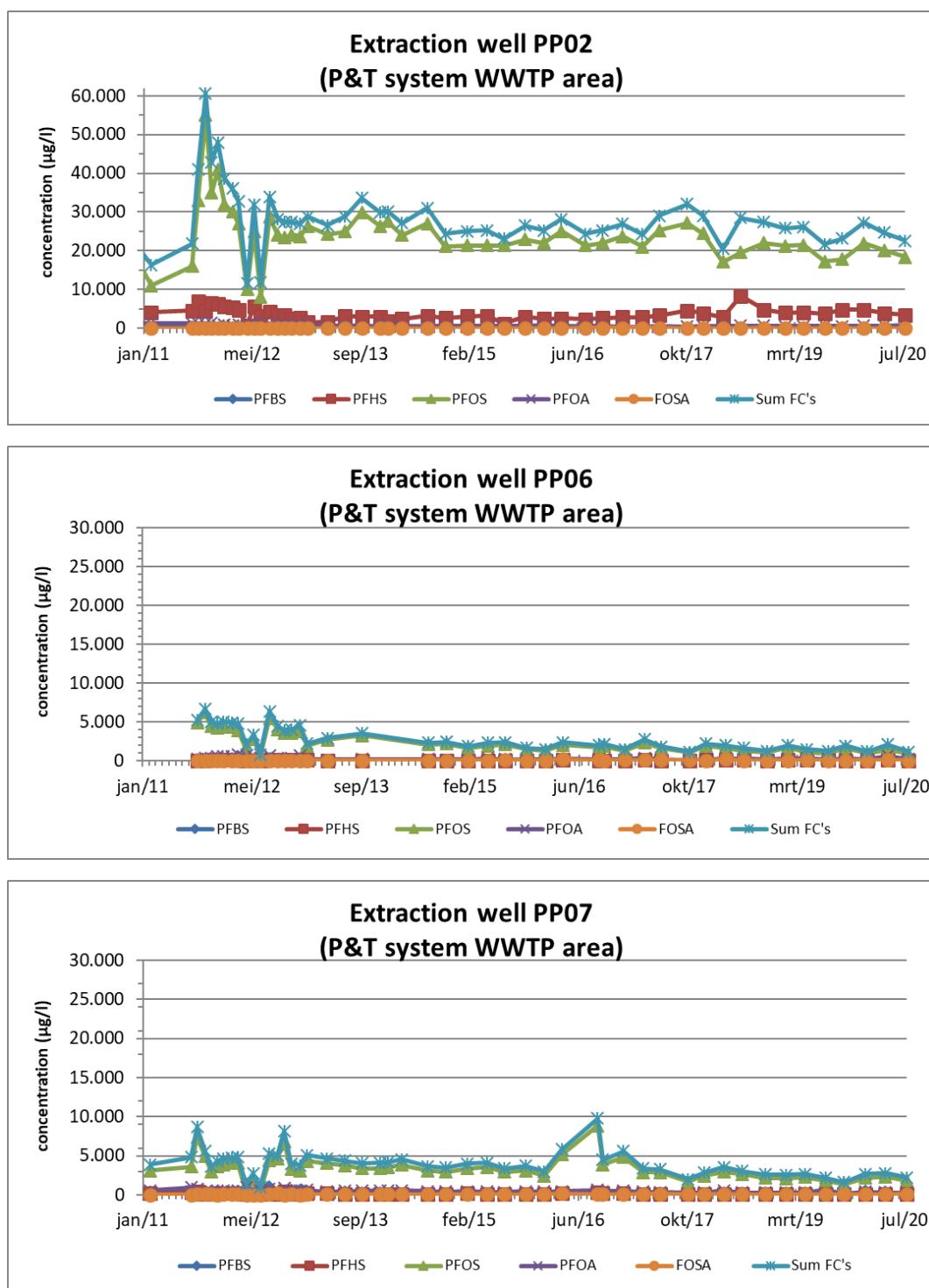
Grondwaterstalen van alle pompputten worden genomen om de FC-concentratie trends op te volgen. In onderstaande figuren zijn de FC-concentraties per pompput en per bronzone (gebouw 16 en WWTP) weergegeven. De “som FC’s” in de grafiek geven de som van de parameters PFOS, PFOA en PFHS weer.

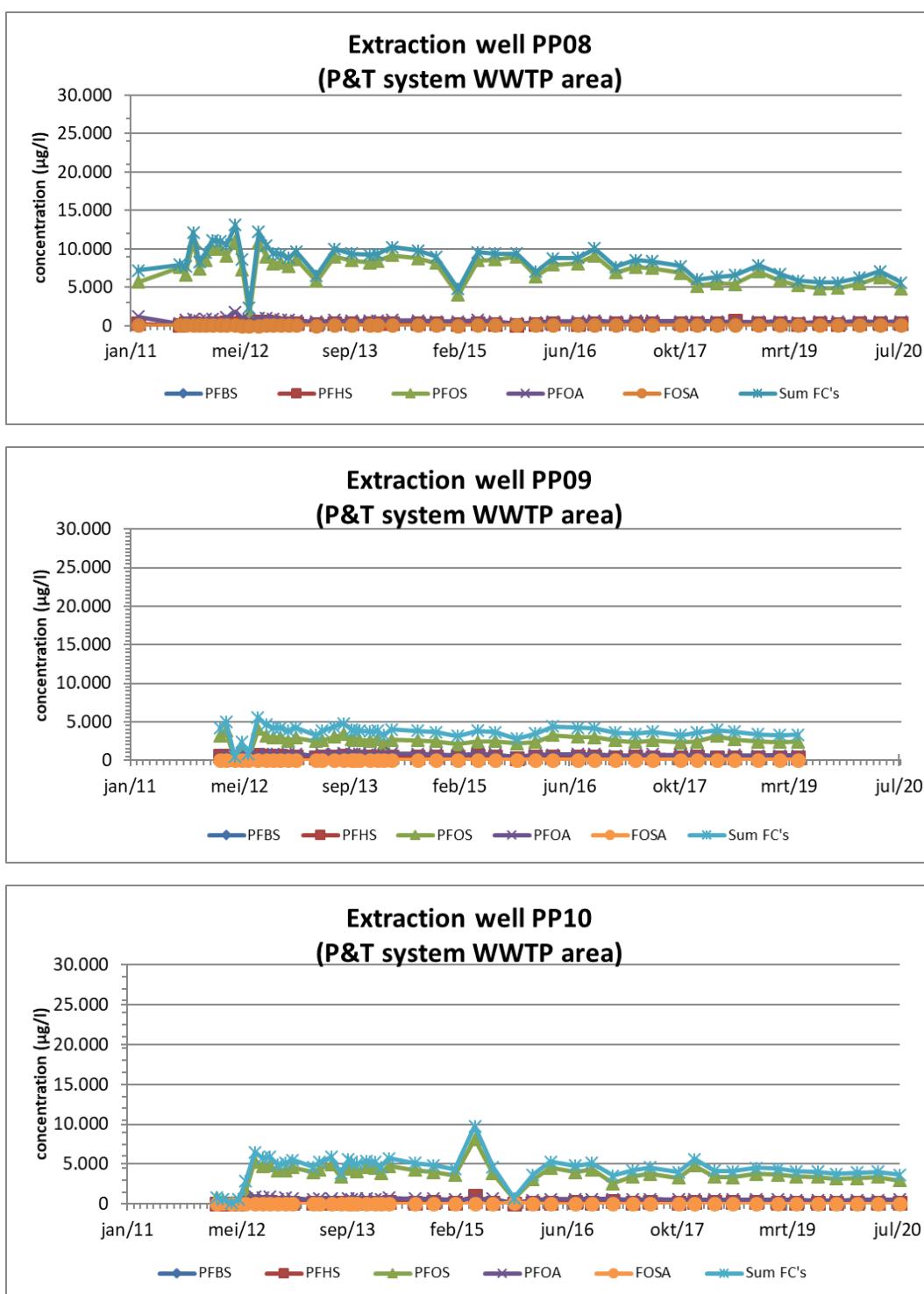
Figuur 3.6 Evolutie FC-concentraties t.h.v. gebouw 16 (pompput PP01/PP11, PP04 en PP05)



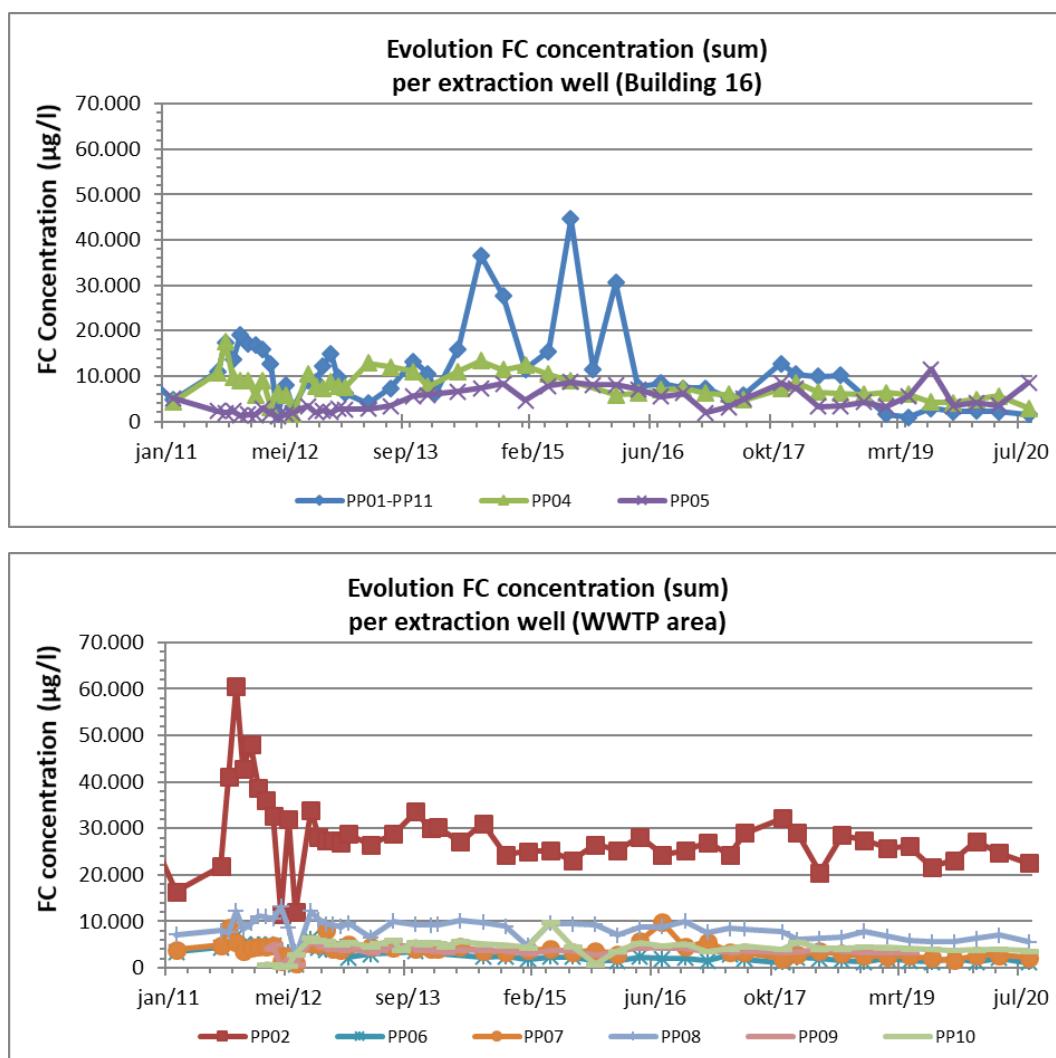


Figuur 3.7 Evolutie FC-concentraties t.h.v. WWTP zone (pompput PP02, PP06, PP07, PP08, PP09 en PP10)





Figuur 3.8 Overzicht evolutie som FC-concentraties (PFOS, PFOA en PFHS) t.h.v. gebouw 16 en WWTP zone



Uit bovenstaande grafieken kunnen volgende zaken afgeleid worden:

- Er is een verschil in samenstelling van het met FC's verontreinigde grondwater in de zone rond gebouw 16 (PP01, PP04 en PP05) en van het grondwater in de zone WWTP (overige pompputten):
 - In PP01 is PFHS de component met de hoogste concentraties;
 - In pompput PP05 wordt naast de "standaard" FC's (PFOS, PFOA, PFHS en PFOSA) ook een verhoogde concentratie PFBS (perfluorbutaansulfonzuur) gemeten; en
 - In de overige pompputten wordt voornamelijk PFOS gemeten.
- De hoogste concentraties aan som FC's (PFOS, PFOA en PFHS) worden gemeten in PP02.
- De opgepompte concentraties aan som FC's fluctueren (hoofdzakelijk in PP02 en PP05), maar in de meeste pompputten zijn de concentratietrends eerder stabiel of zwak dalend. Deze variatie in concentratie wordt mogelijk deels veroorzaakt door seizoenale variatie in de grondwaterstand.
 - Voor PP06 ligt de gemeten concentratie aan som FC's rond 2.000 $\mu\text{g}/\text{l}$.

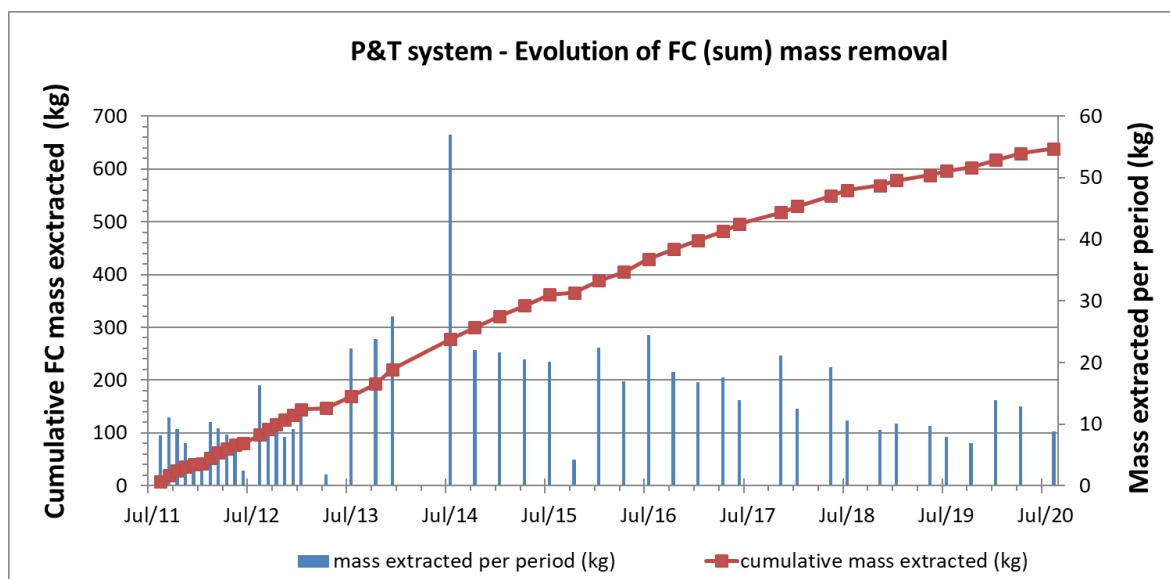
- De gemeten concentraties aan som FC's (PFOS, PFOA en PFHS) in PP04, PP08, PP09 en PP10 liggen doorgaans rond 5.000 µg/L.
- In pompput PP02 ligt de som FC-concentratie rond 25.000 µg/L.
- De gemeten PFHS en PFBS-concentraties fluctueren in PP05, met een concentratie aan som FC's rond 8.000 µg/L.
- De concentratie aan som FC's nam tussen juli 2017 en juli 2018 toe in PP01 tot ongeveer 10.000 µg/L. PP11 wordt sinds 2019 ter vervanging van PP01 bemonsterd. Hier worden stabiele concentraties aan som FC's van ongeveer 1.500 µg/L gemeten. De evolutie van de concentraties in PP11 zal verder opgevolgd worden om na te gaan wat de invloed van de onttrekking is op de opgepompte concentraties.

Over het algemeen zijn de concentraties in de pompputtonen stabiel.

3.4.4 Vuilvrachtverwijdering

Figuur 3.9 geeft de cumulatieve hoeveelheid FC (PFOS, PFOA en PFHS) vuilvracht weer die is verwijderd via het saneringssysteem. In de periode is augustus 2017 – juli 2020 is ongeveer 143 kg FC's verwijderd. In totaal is sinds juli 2011 circa 656 kg FC verwijderd.

Figuur 3.9 Cumulatieve vuilvrachtverwijdering van het totaal saneringssysteem



3.4.5 Stabiliteit

Uit de evaluatie door de onafhankelijk stabiliteitsexpert Grontmij van de zettingsmetingen uitgevoerd in de periode 2009 tot en met 2013 (zie TTV5) blijkt dat het toetsingscriterium (maximale zetting: 3 mm) nooit is bereikt. De hoogst gemeten zetting bedraagt 2,9 mm (gemeten dd.15/02/2011). De laatste zettingsmeting (dd. 2/07/13) toont dat de zettingen in bijna elk meetpunt verwaarloosbaar klein zijn, enkel twee punten (9 en 16) vertonen nog zettingen groter dan 1 mm.

De stabiliteitsdeskundige Grontmij adviseert dan ook om de zettingsmetingen nog eenmaal te herhalen en op basis van deze meting te beslissen of de opvolging van de zettingen al dan niet kunnen worden stopgezet (zie nota opgenomen in TTV5). Belangrijke randvoorwaarde voor stopzetting is dat de opgepompte debieten in de toekomst, nooit de tot op heden opgepompte debieten mogen overschrijden. Deze eindmeting is nog niet uitgevoerd.

ERM adviseert een nieuwe stabiliteitsmeting uit te voeren, gezien de grote tijdspanne tussen de laatste meting en nu; na het installeren van de nieuwe onttrekkingsputten ter vervanging van PP09, in een periode dat alle onttrekkingsputten goed werken.

3.5 Conclusie en aanbevelingen

Na het uitvoeren van de pomptest en de verdere optimalisatie van het systeem, zal de frequentie van de periodieke reiniging van de installatie herbekeken worden. De periodieke opvolging van de grondwaterstanden, concentraties en vuilvrachtverwijdering dient verder gezet te worden.

Er wordt aanbevolen een stabiliteitsmeting uit te voeren van zodra de nieuwe extractieputten (en reeds aanwezige putten) in werking zijn.

4. MONITORING GROND- EN OPPERVLAKTEWATER

4.1 Algemeen en overzicht uitgevoerde werken

In de periode van 1 augustus 2017 tot 31 juli 2020 zijn volgende activiteiten uitgevoerd:

- Periodieke controle van de waterkwaliteit op de volgende locaties:
 - Grondwater in peilbuizen (driemaandelijks, halfjaarlijks of jaarlijks):
 - Bronzones: gebouw 16 en WWTP;
 - Tweede aquifer;
 - Zuidelijke perceelsgrens;
 - 3M pad langs het Blokkersdijk natuurgebied; en
 - Twee peilbuizen (P18 en P28) in het gebied van de tankparken (bij gebouw 03) voor minerale olieanalyses.
 - Oppervlaktewater (driemaandelijks en maandelijks):
 - Blokkersdijkvijver en 3M vijver; en
 - Palingbeek en Tophatgracht.
 - Regenwater van de collector put en effluent van de WWTP (wekelijks en driemaandelijks).
- Berekening van de PFOS-vuilvracht die via de collector put, het effluent van de WWTP en de Palingbeek en Tophatgracht in de Schelde terechtkomt;
- Evaluatie van de analyseresultaten gerapporteerd door het 3M Environmental Laboratorium in de VS versus de analyseresultaten van de dupliecatstalen gerapporteerd door het SGS laboratorium;
- Jaarlijkse statistische evaluatie van de PFOS-concentratie trend in het grondwater en oppervlaktewater van Blokkersdijk en de 3M vijver; en
- Visuele check van de kwik grondhopen.

In de figuren en grafieken opgenomen in dit verslag zijn, omwille van consistentie, tot juli 2020 enkel de analyseresultaten van het 3M Environmental Laboratorium gebruikt. Vanaf juli 2020 is de opgenomen data afkomstig van het SGS laboratorium (zie kleine wijziging in paragraaf 2.5.3).

4.2 Eerste aquifer – Gebouw 16 en WWTP

4.2.1 Uitgevoerd veldwerk

De peilbuizen bemonsterd tijdens de periodieke grondwaterstaalname in de eerste aquifer ter hoogte van de twee bronzones (gebouw 16 en WWTP) zijn opgeliist in onderstaande tabel.

De locaties van deze peilbuizen zijn aangeduid op Kaart 4A en Kaart 4B

Tabel 4.1 Overzicht bemonsterde peilbuizen eerste aquifer (2017-2020)

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum												Analyse	
		Okt 2017	Jan 2018	Apr 2018	Jul 2018	Okt 2018	Jan 2019	Apr 2019	Jul 2019	Okt 2019	Jan 2020	Apr 2020	Jul 2020		
Zone Building 16															
K3	3,6-5,6				X				X					X	FC inclusief PFBS
P21B	3,5-5,5	X	X	X	X	X	X	X	X		X			X	FC inclusief PFBS
P27	0,9-2,9		X		X		X		X		X			X	FC inclusief PFBS
P304	4,0-6,0		X		X		X		X		X			X	FC inclusief PFBS
P305	3,5-5,5		X		X		X		X		X			X	FC inclusief PFBS
P42	3,0-5,0		X		X		X		X		X			X	FC inclusief PFBS
P56	1,2-3,2				X		Niet**								FC inclusief PFBS
Zone WWTP															
P118C	2.5-4.5						X		X		X			X	FC
P119C	2.5-4.5						X		X		X			X	FC
L19	2,8-4,8		X		X		X		X		X			X	FC
M4	1,0-3,0		X		X		X		X		X			X	FC
P262	6,2-7,2		X		X		Niet***								FC
P262bis****	6.5-7.5						-		X		X			X	FC
P263	7,4-8,4	X	X		X		X		X		X			X	FC
P264	5,0-5,5		X		X		X		X		X			X	FC
P265B	2,8-3,8		X		Niet*		X		X		X			X	FC
P265C	4,5-5,5		Niet*												FC
P340	4,0-6,0		X		X		X		X		X			X	FC

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum												Analyse
		Okt 2017	Jan 2018	Apr 2018	Jul 2018	Okt 2018	Jan 2019	Apr 2019	Jul 2019	Okt 2019	Jan 2020	Apr 2020	Jul 2020	
P341	3,6-5,6				X		X		X		X		X	FC
P343	4,8-6,8		X		X		X		Niet***					FC
P371	1,5-3,5		X		X		X		X		X		X	FC
P374	5,0-6,0		X		X		X		X		X		X	FC
P379	3,5-5,5				X		X		X		X		X	FC
P380	3,5-5,5				X		X		X		X		X	FC
P381	5,0-6,0				X				X				X	FC
P382	2,5-3,5				X				X				X	FC

FC: PFOS, PFOA, PFHS, FOSA

*Peilbuis P265B was op het moment van staalname niet bereikbaar voor monitoring, en P265C werd permanent beschadigd en is buiten gebruik.

** Peilbuis P56 is buiten gebruik gesteld in oktober 2018 door constructiewerken aan gebouw 036.

*** Peilbuis P262 en P343 waren beschadigd en konden niet bemonsterd worden.

**** Peilbuis P26bis is in juli 2019 ter vervanging van P262 geïnstalleerd.

- Staalnames zijn uitgevoerd conform de CMA-procedures (Compendium voor monsterneming en analyse, meest recente versie)

- Temperatuur, zuurtegraad (pH), elektrische conductiviteit en grondwaterniveaus zijn gemeten.

4.2.2 Bespreking resultaten

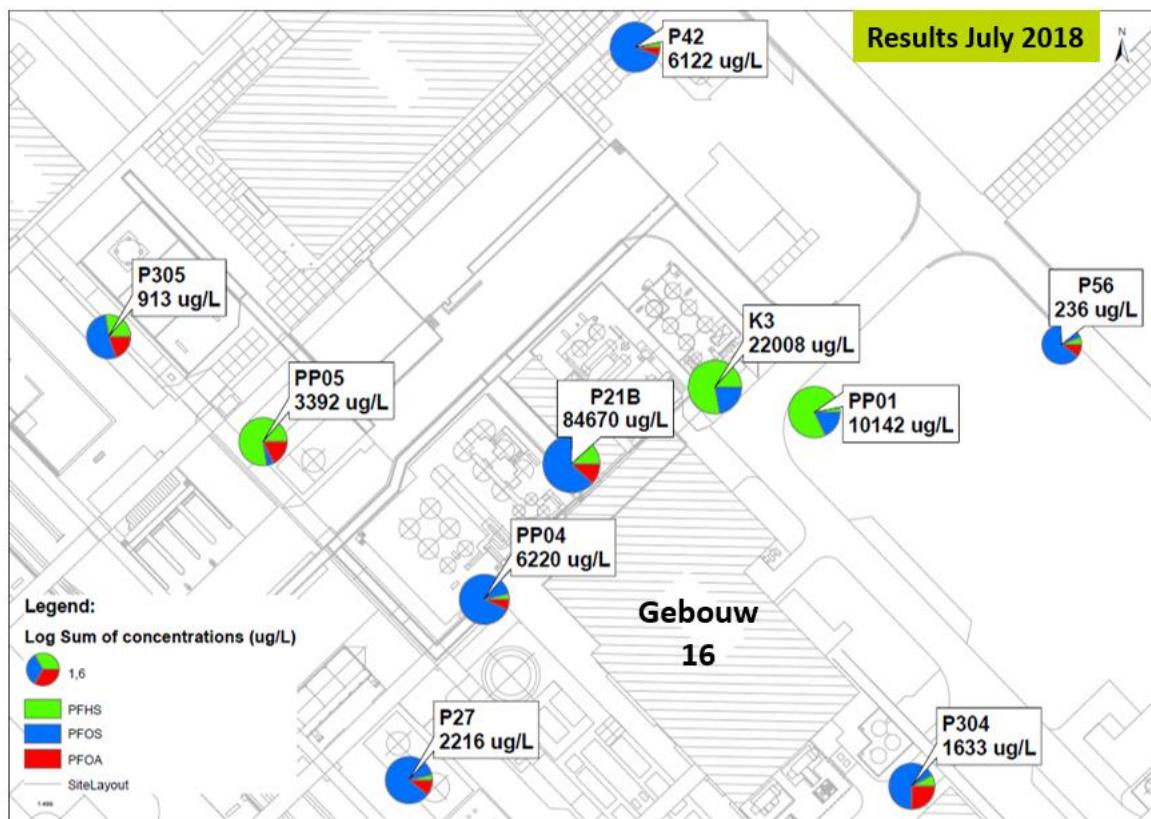
De analyseverslagen zijn toegevoegd in Bijlage 5. Een tabel met de analyseresultaten en veldmetingen is opgenomen in Bijlage 6.

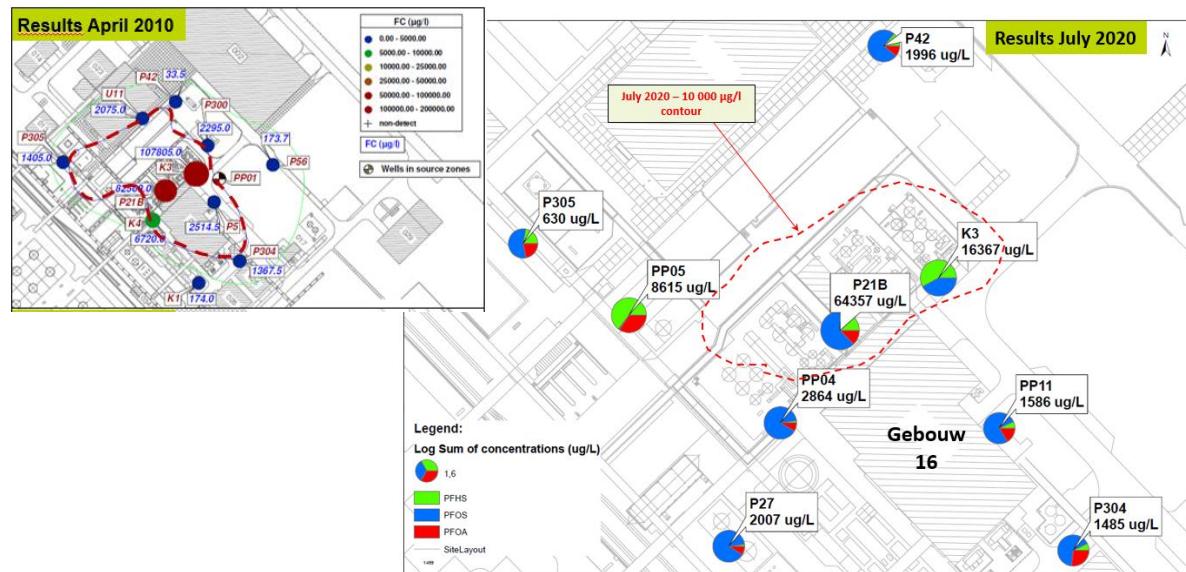
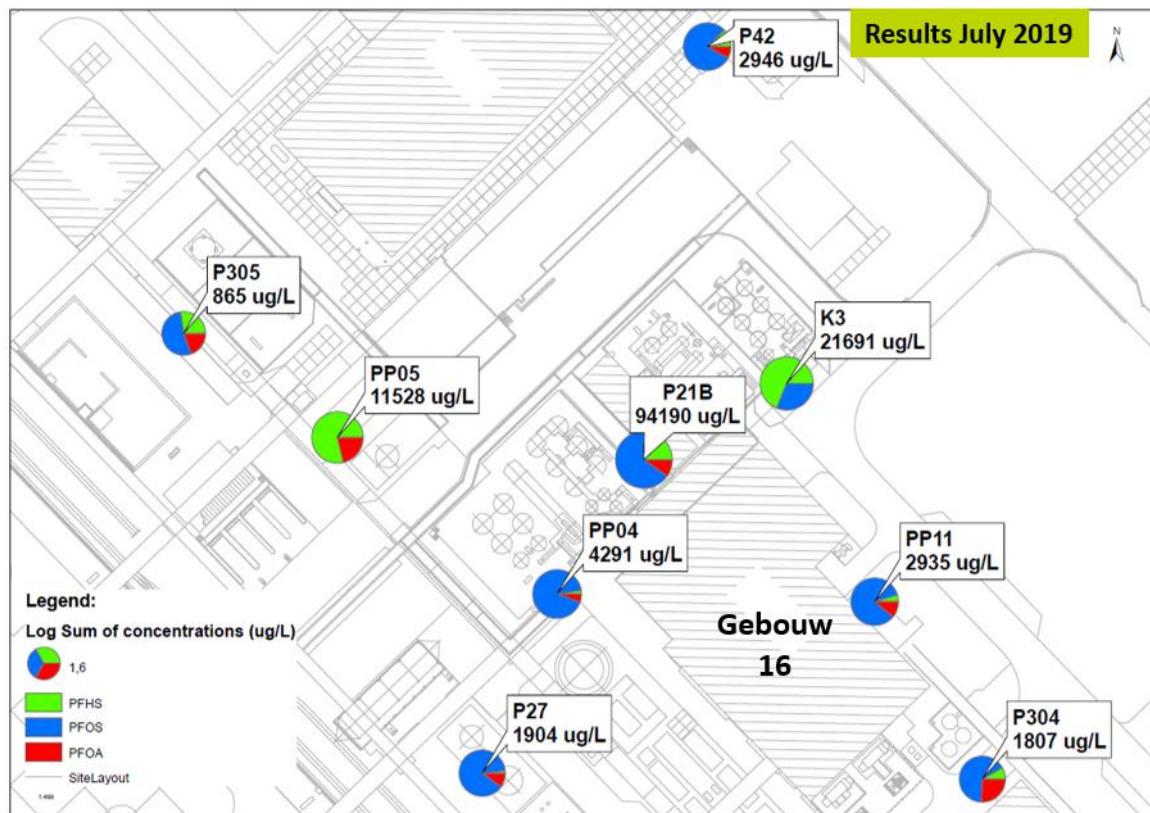
De concentratie trends voor de componenten PFOS, PFOA, PFHS, PFOSA en PFBS ter hoogte van de verschillende peilbuizen in de 1^e aquifer rond de bronzones gebouw 16 en WWTP zijn opgenomen in Bijlage 7.

4.2.2.1 Gebouw 16

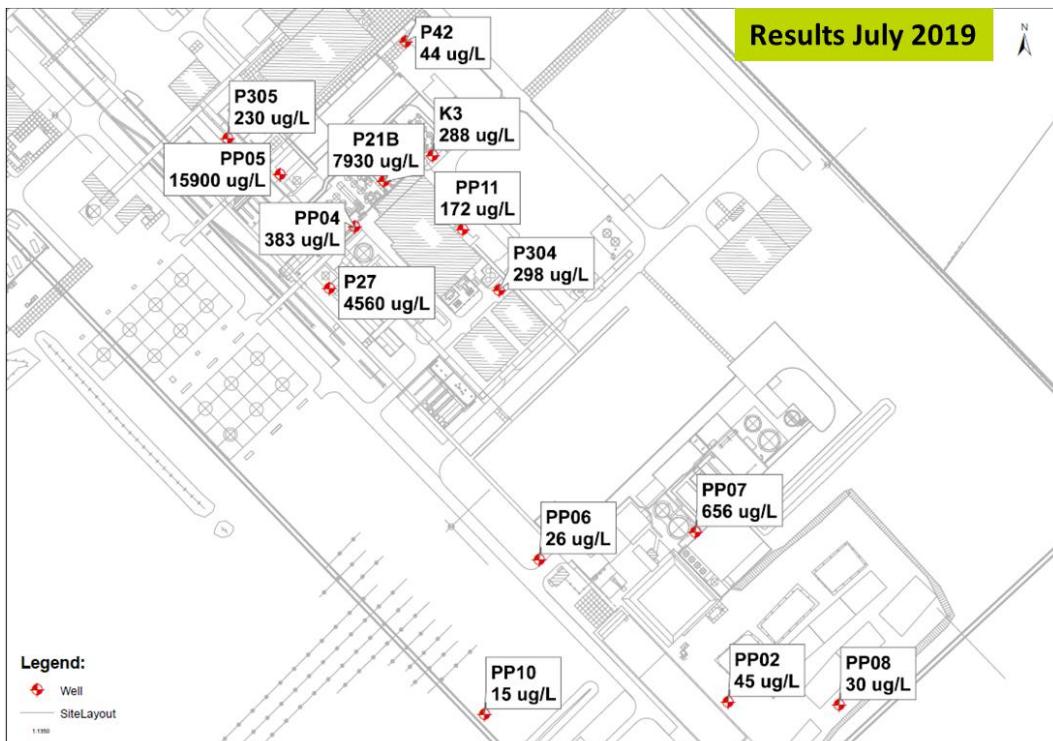
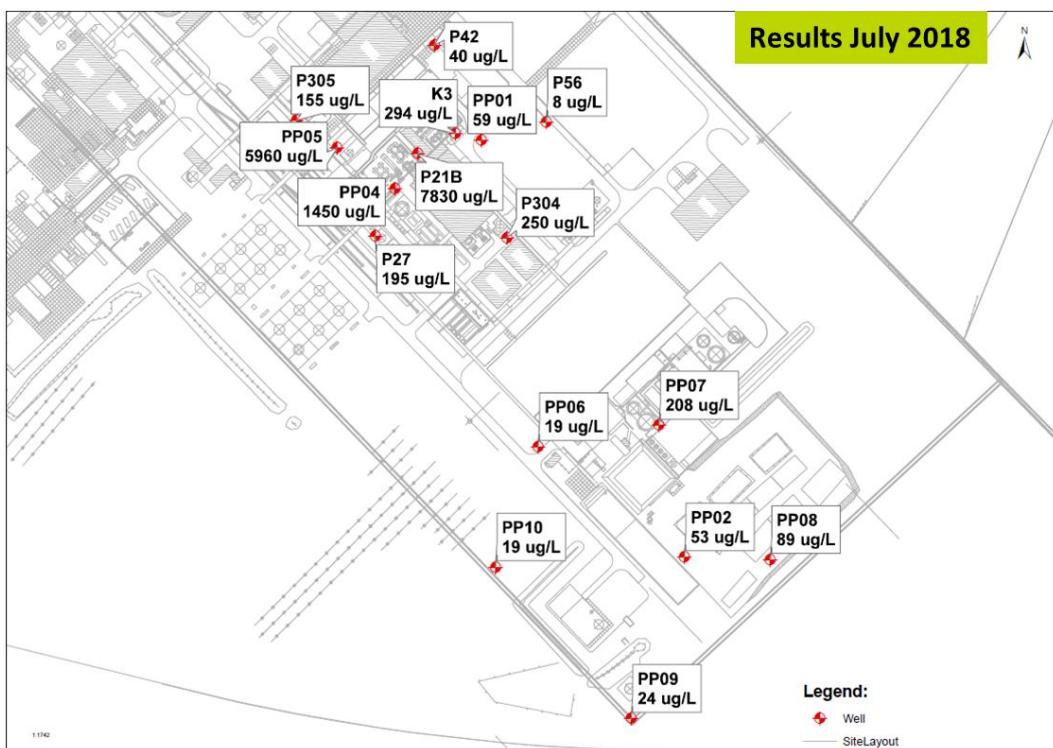
De grondwateranalyseresultaten voor de som van de FC-concentraties (PFOS, PFHS en PFOA) en PFBS van juli 2018, 2019 en 2020 ter hoogte van gebouw 16 zijn in de volgende figuren voorgesteld. De rode lijn is de contour van 10.000 µg/L voor de som van de FC-concentraties van de meest recente situatie tijdens deze rapportage periode.

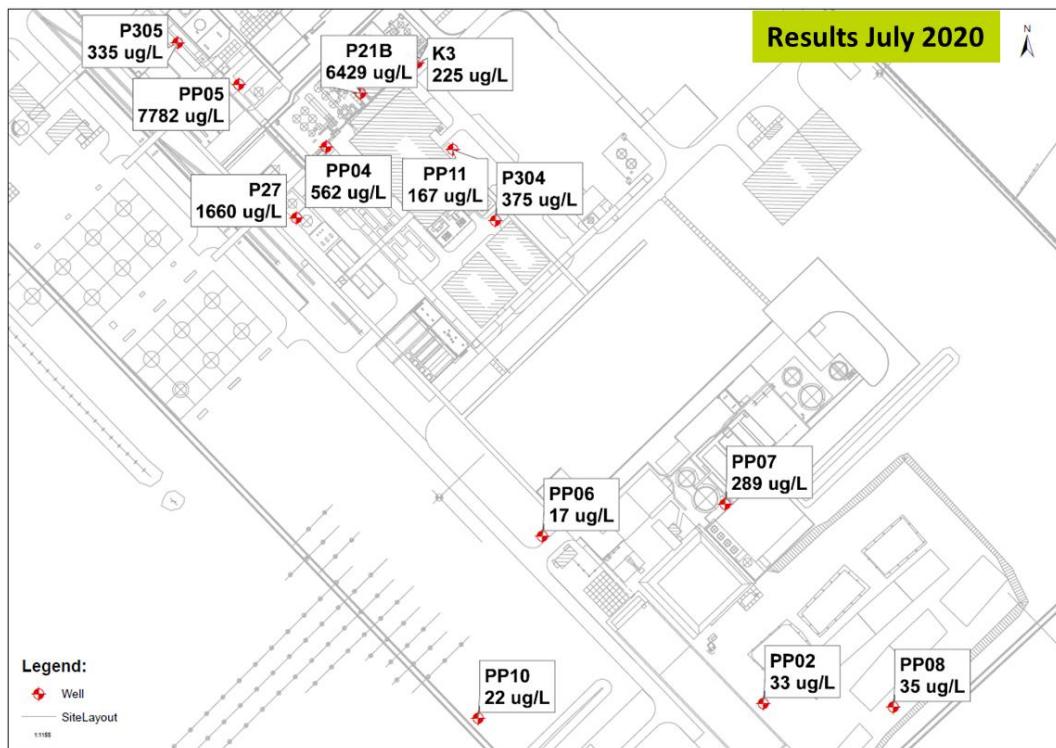
Figuur 4.1 Schematische voorstelling FC-concentraties in grondwater t.h.v. gebouw 16 (augustus 2017 tot juli 2020)





Figuur 4.2 Schematische voorstelling PFBS-concentraties in grondwater t.h.v. gebouw 16 (augustus 2017 tot juli 2020)

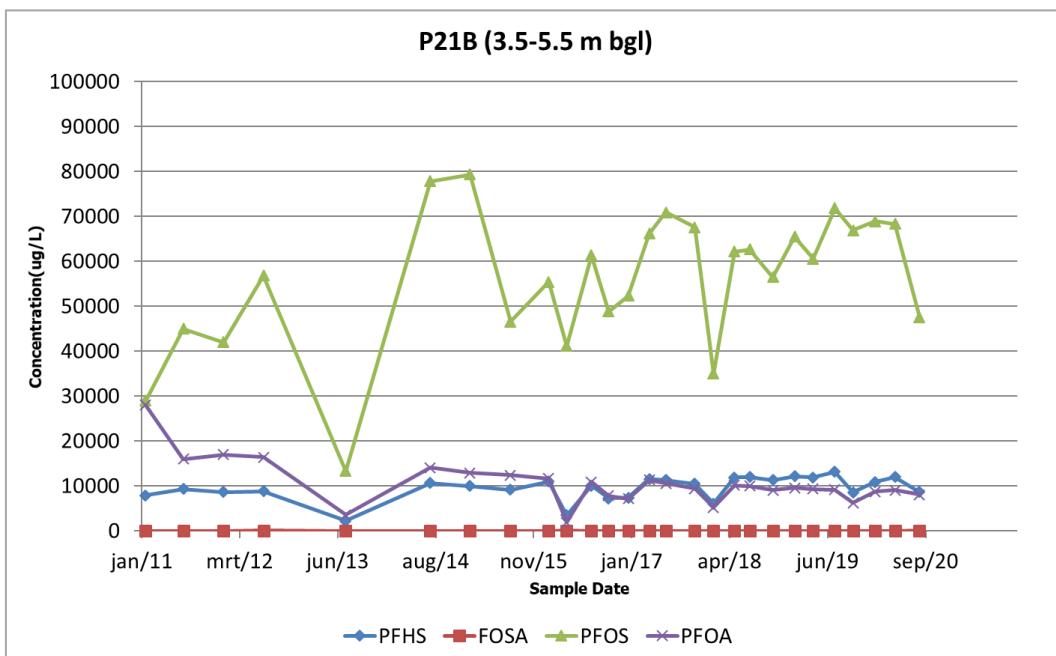




Enkel van de meetpunten waar belangrijke waarnemingen/trends zijn aangetoond, zijn hieronder de grafieken weergegeven.

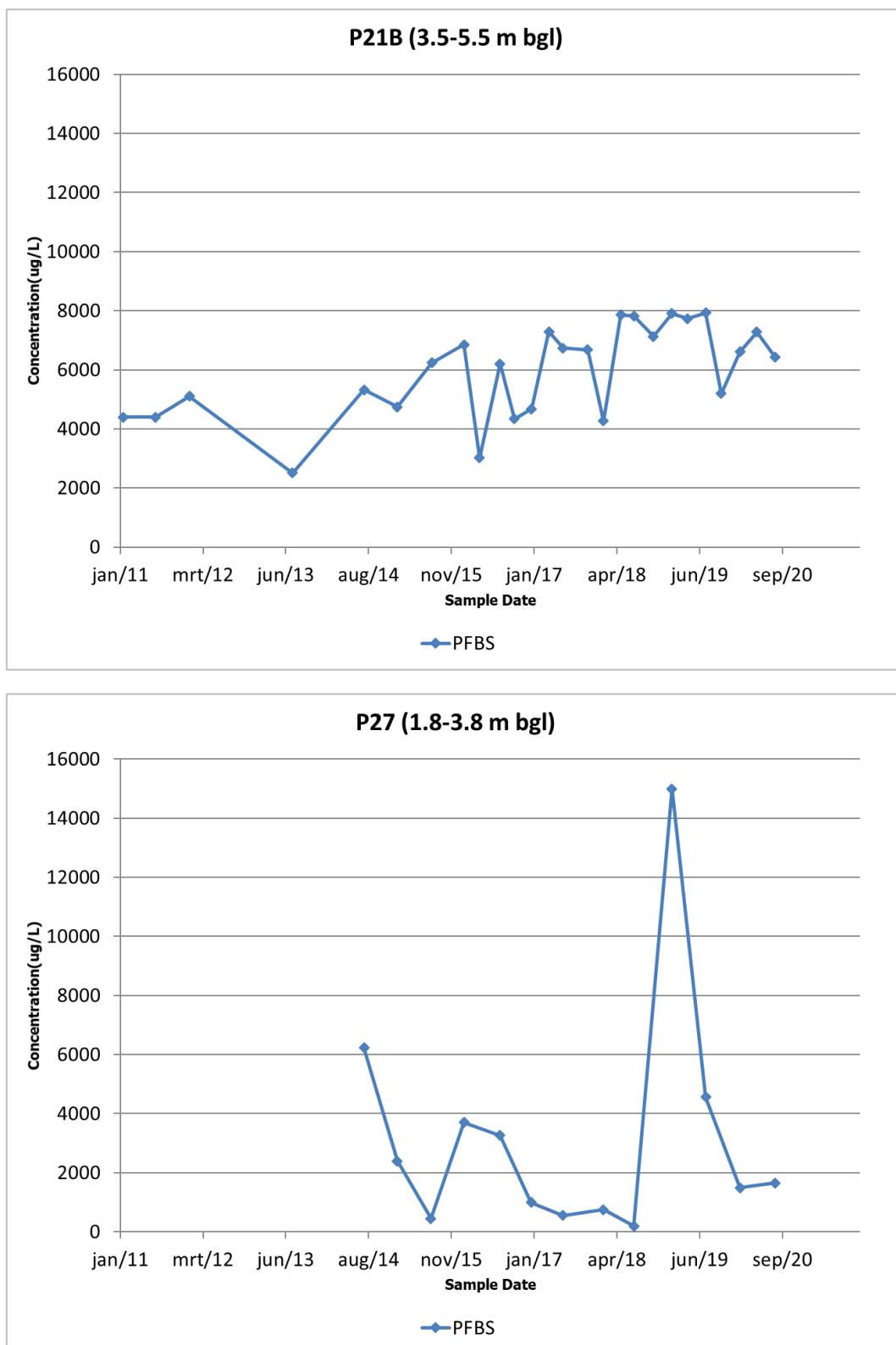
De evolutie van de grondwaterconcentraties aan FC's ter hoogte van peilbuis P21B is voorgesteld in onderstaande grafiek.

Figuur 4.3 Evolutie FC-concentraties t.h.v. gebouw 16



De evolutie van de PFBS-concentratie in het grondwater ter hoogte van peilbuizen P21B en P27 is voorgesteld in onderstaande grafieken.

Figuur 4.4 Evolutie PFBS concentraties t.h.v. gebouw 16



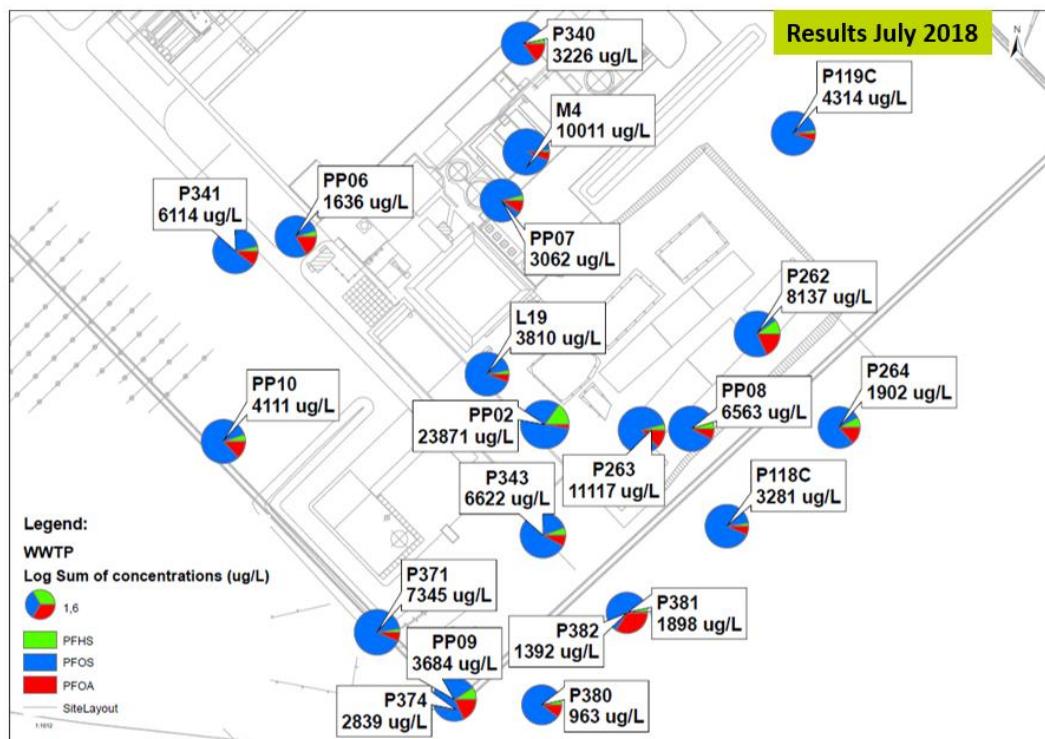
Belangrijkste vaststellingen:

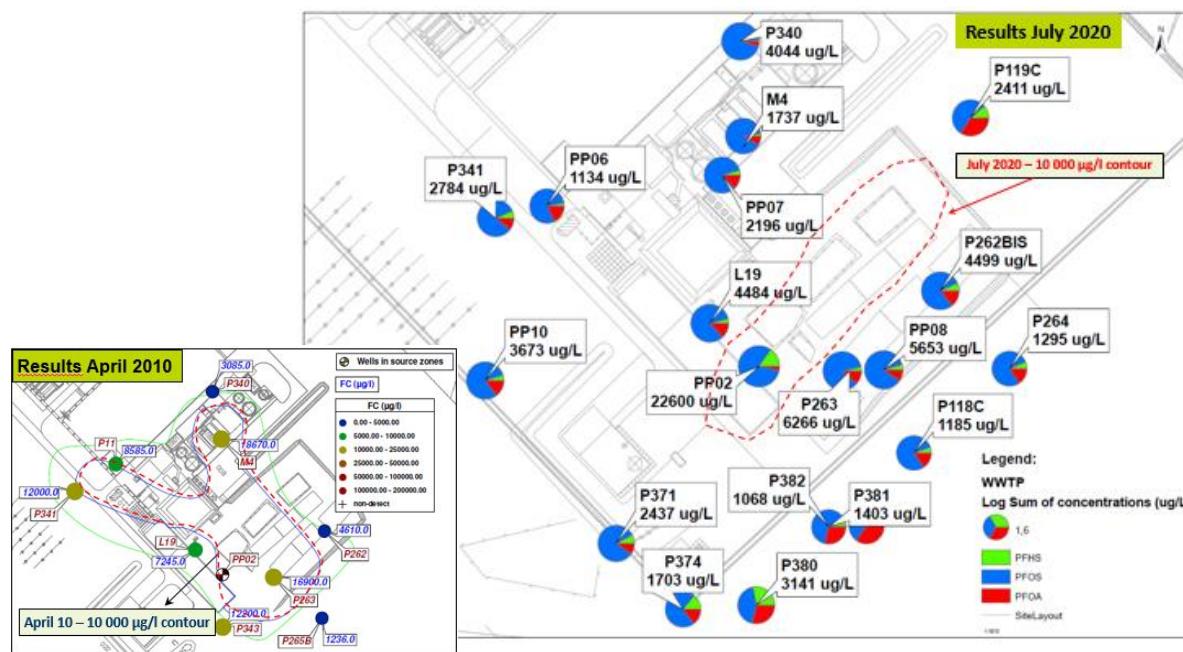
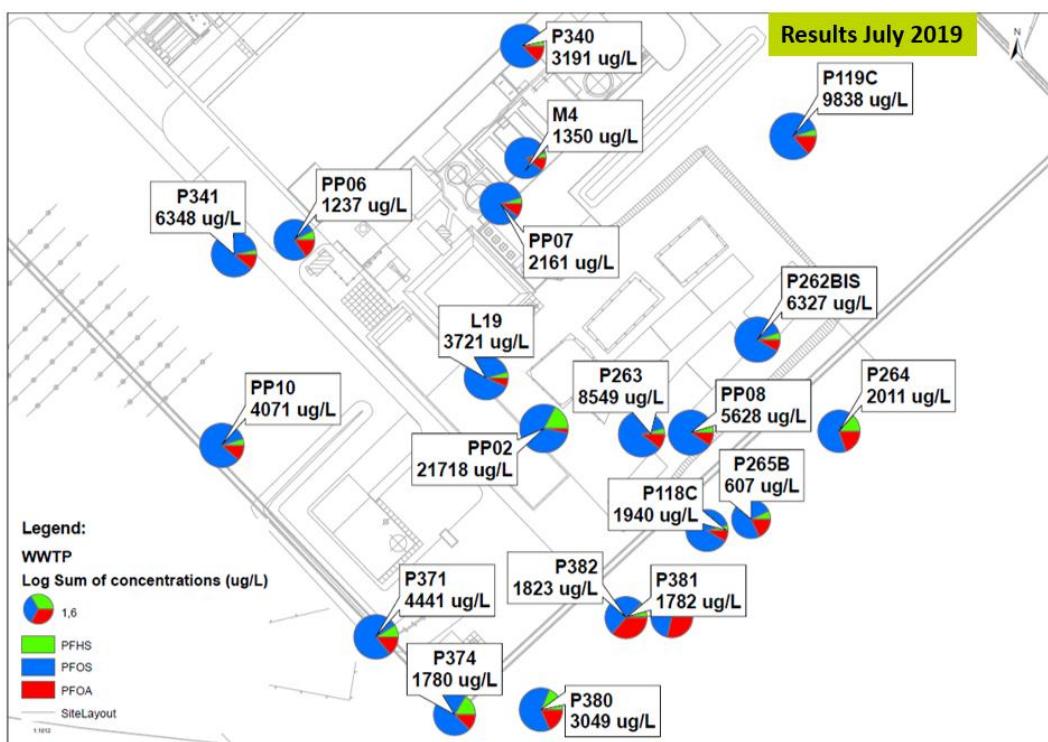
- De som van FC-concentraties ($10.000 \mu\text{g/L}$ contour) neemt in omvang af sinds de start van de sanering;
- De meest voorkomende component ter hoogte van gebouw 16 is PFOS, met uitzondering van peilbuis K3 (en pompput PP05 - zie paragraaf 0) waar PFHS de meest voorkomende component is;
- In het algemeen kan gesteld worden dat de FC-concentraties in het grondwater ter hoogte van gebouw 16 dalen of stabiel zijn in de tijd, maar de concentraties fluctueren. De concentratie aan PFOS ter hoogte van peilbuis P21B lijkt over het algemeen een sterk fluctuerende maar licht stijgende trend te vertonen. Deze peilbuis is centraal gelegen in de $10.000 \mu\text{g/L}$ contour ter hoogte van gebouw 16. Mogelijk is dit een gevolg van de lopende onttrekking, verdere monitoring is noodzakelijk om aan te tonen of deze trend significant is;
- De component PFBS (vervagt PFOS sinds 2002 in de productie) is eveneens in relatief hoge concentraties aanwezig in de omgeving van gebouw 16; en
- In peilbuis P21B is een fluctuerende maar licht stijgende trend in PFBS aangetoond, gelijkaardig aan de trend in PFOS-concentratie. Begin 2019 is een sterk verhoogde PFBS-concentratie gemeten in peilbuis P27. Actueel is de concentratie in P27 terug gedaald naar het niveau voor januari 2019. De oorzaak voor deze temporele concentratietoename is niet duidelijk, mogelijk gerelateerd aan de actieve onttrekking en wordt verder opgevolgd.

4.2.2.2 Zone WWTP

De grondwateranalyseresultaten voor de som van de FC-concentraties (PFOS, PFHS en PFOA) van juli 2018, 2019 en 2020 ter hoogte van de WWTP zijn op de volgende figuur voorgesteld. De rode lijn is de contour van $10.000 \mu\text{g/L}$ voor de som van de FC-concentraties van de meest recente situatie.

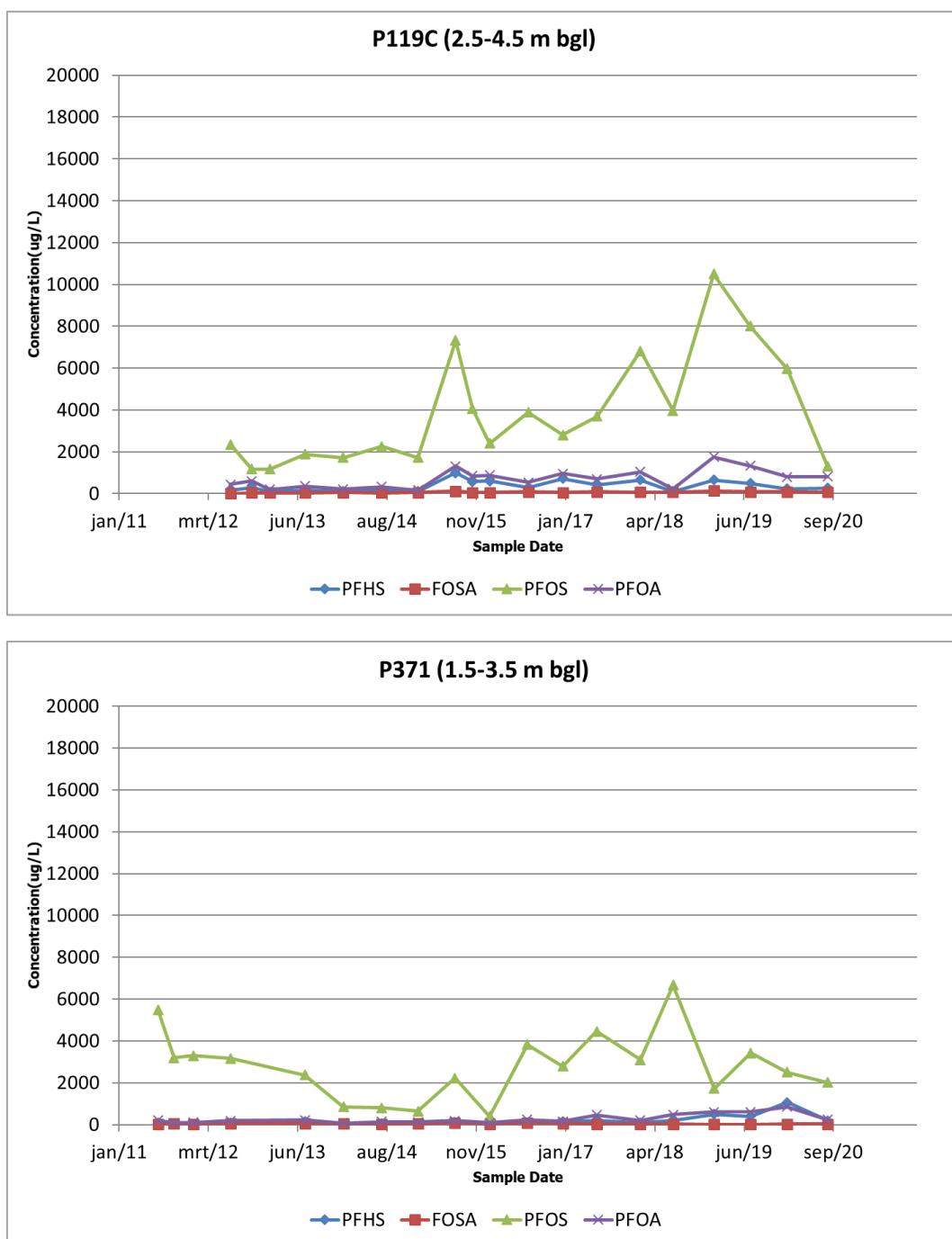
Figuur 4.5 Schematische voorstelling FC-concentraties in grondwater t.h.v. WWTP (augustus 2017 tot juli 2020)

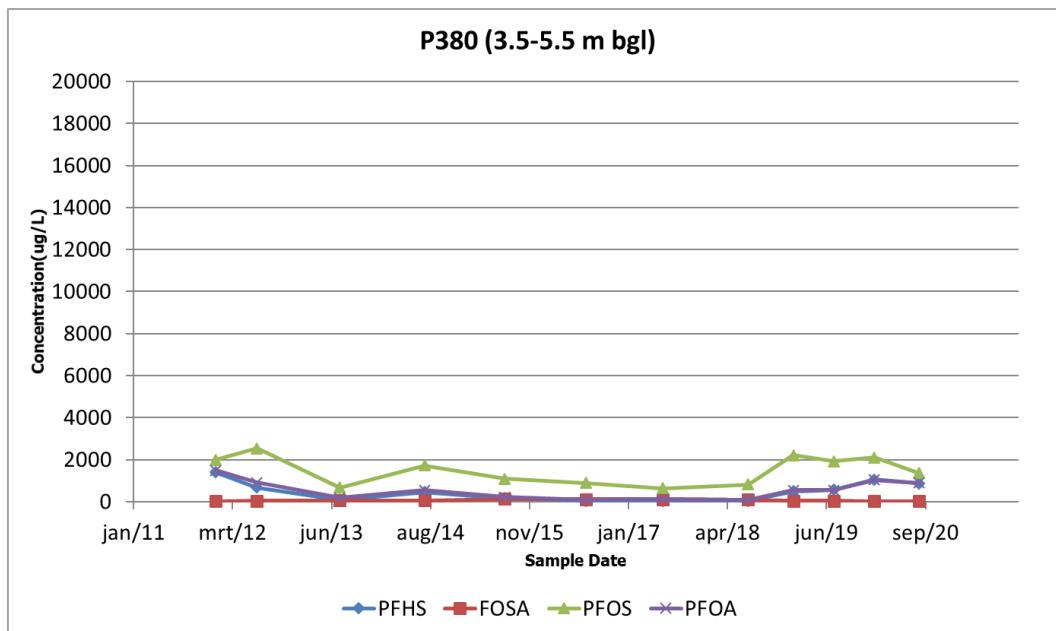




Enkel van de meetpunten waar belangrijke waarnemingen/trends zijn aangetoond, zijn hieronder de grafieken weergegeven.

Figuur 4.6 Evolutie FC-concentraties t.h.v. zone WWTP





Belangrijkste vaststellingen:

- De som van FC-concentraties (10.000 µg/L contour) is in omvang kleiner ten opzichte van de start van de sanering;
- De meest voorkomende component ter hoogte van de WWTP is PFOS;
- In het algemeen kan gesteld worden dat de concentraties aan FC verbindingen in het grondwater ter hoogte van de WWTP dalen of stabiel zijn in de tijd, maar de concentraties fluctueren. Ter hoogte van peilbuis P380 zijn licht hogere concentraties gemeten tijdens deze rapportageperiode ten opzichte van de vorige monitoringsperiode; de laatste meting toont echter terug een lagere concentratie aan; en
- De lichte concentratiestijgingen voor PFOS in het grondwater ter hoogte van peilbuizen P119C en P371 die in het TTV8 besproken werden, zijn niet bevestigd.

4.2.3 Conclusies en aanbevelingen

- De algemene stabiele/dalende trend in FC-concentraties lijkt erop te duiden dat het onttrekkingssysteem de 10.000 µg/l contouren in het grondwater in de twee bronzones (gebouw 16 en WWTP) kan beheersen; en
- ERM raadt aan om peilbuizen P56 en P343 te vervangen zodra dit mogelijk is, omdat de peilbuizen op strategische plaatsen liggen om na te gaan dat er geen migratie van de grondwaterverontreiniging plaatsvindt.

4.3 Tweede aquifer – Gebouw 16 en WWTP

4.3.1 Uitgevoerd veldwerk

De peilbuizen bemonsterd tijdens de periodieke grondwaterstaalname in de 2^e aquifer ter hoogte van de twee bronzones (gebouw 16 en WWTP) zijn opgeliist in onderstaande tabel.

De locaties van deze peilbuizen zijn aangeduid op Kaart 4C.

Tabel 4.2 Overzicht bemonsterde peilbuizen 2^e aquifer (2017-2020)

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum												Analyse
		Okt 2017	Jan 2018	Apr 2018	Jul 2018	Okt 2018	Jan 2019	Apr 2019	Jul 2019	Okt 2019	Jan 2020	Apr 2020	Jul 2020	
Zone Building 16														
D9	13,0-15,0	X	X		X		X		Niet**		Niet**		Niet**	FC
D14	15,5-16,5		X		X		X		X		X		X	FC
D17	15-16		X		X		X		X		X		X	FC
D18	14,0-15,0		X		X		X		X		X		X	FC
D5	15,0-16,0		X		X		X		X		X		X	FC
ND7	15,0-16,0		X		X		X		X		X		X	FC
Zone WWTP														
D10	15,0-16,0		X		X		X		X		X		X	FC
D11	14,9-15,9		X		X		X		X		X		X	FC
BD24-3	17,0-18,0	X	X		X		X		X		X		X	FC
BD24-4	22,0-24,0		X		X		X		X		X		X	FC
P121	23,5-24,5	X	X	X	X		X	X	X		X	X	X	FC
P321	15,5-15,6	X	X	X	X	X	X	X	X	X	X	X	X	FC
D16	14,0-15,0		Niet*	X	X		X	X	X	X	X		X	FC
D2	24,3-25,3				X				X				X	FC
P118A	22,5-23,5		X		X		X		X		X		X	FC
P118B	13,0-14,0		X		X		X		X		X		X	FC
P119A	23,5-24,5		X		X		X		X		X		X	FC
P119B	13,0-14,0		X		X		X		X		X		X	FC

FC: PFOS, PFOA, PFHS, FOSA

*D16 kon niet bemonsterd worden in januari (niet bereikbaar), en werd ter vervanging in april bemonsterd.

** Peilbuis D9 werd beschadigd in juli 2019 tijdens de doorgaande constructiewerken rondom gebouw 036 en kon daardoor niet meer bemonsterd worden.

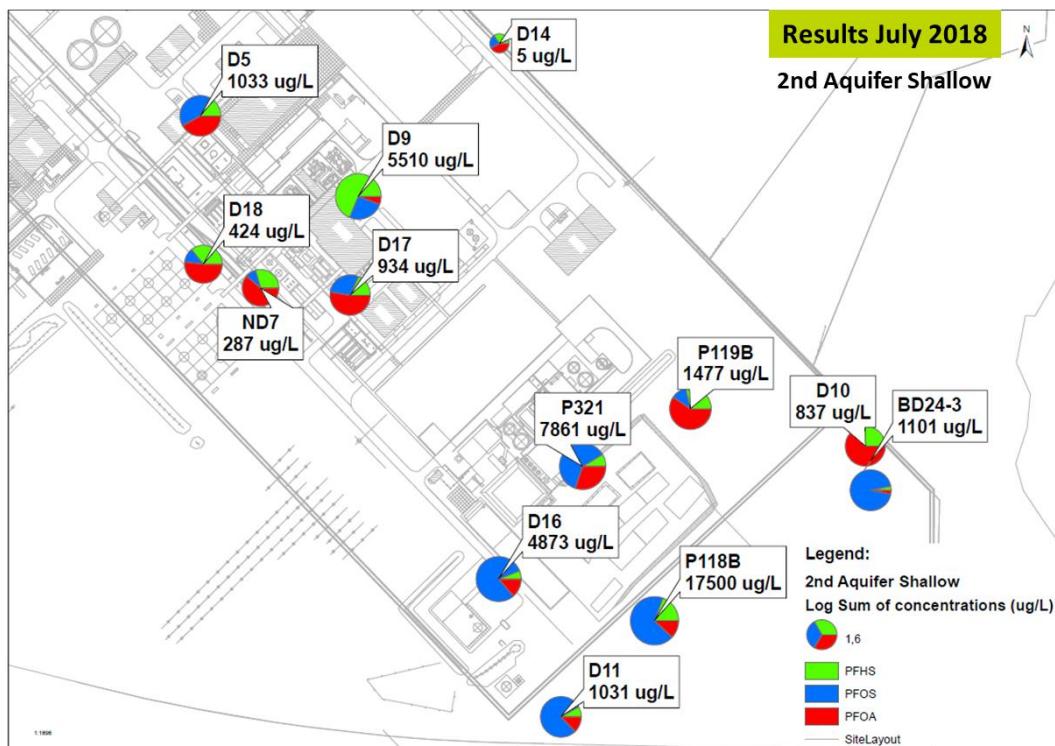
4.3.2 Bespreking resultaten

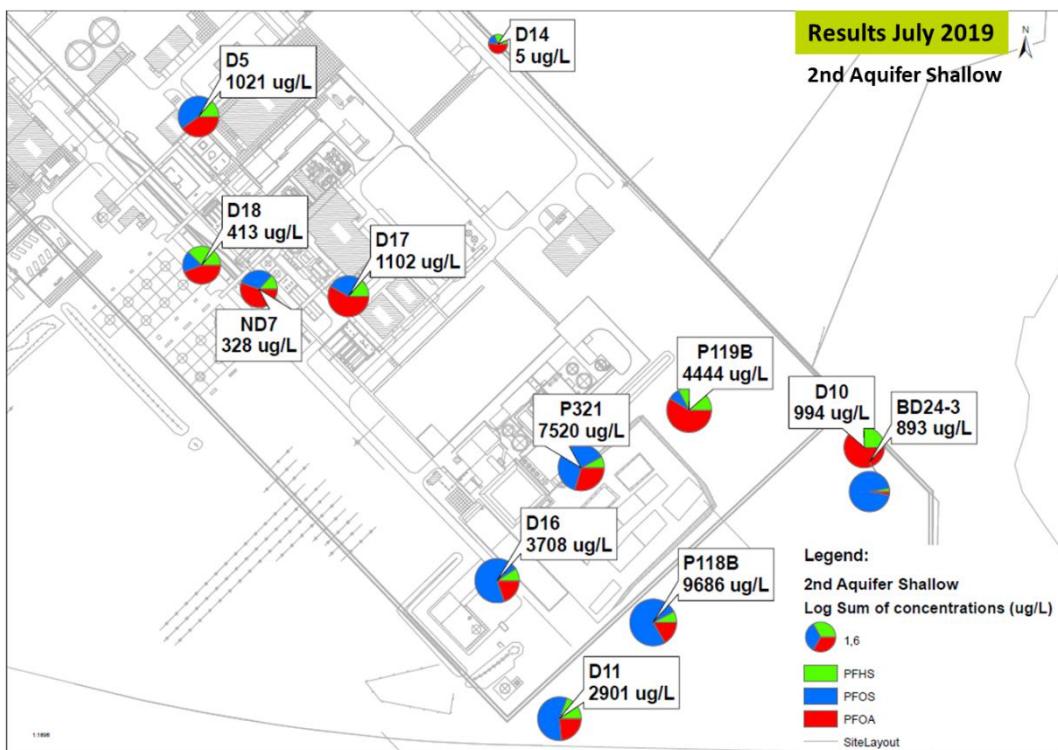
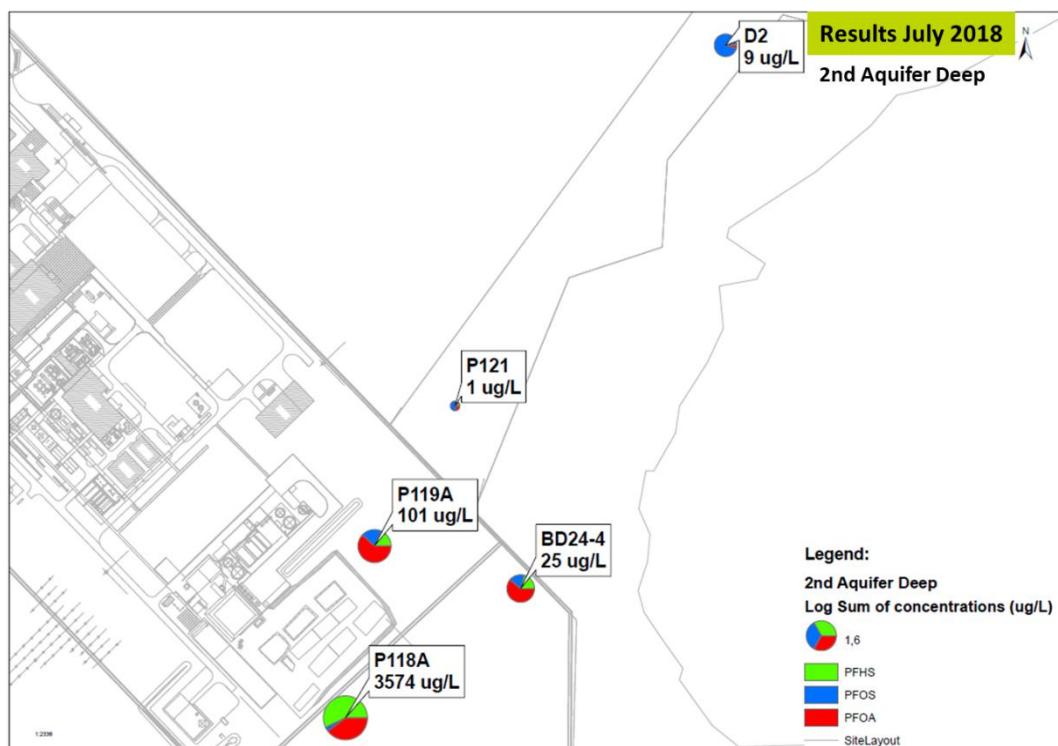
De analyseverslagen zijn toegevoegd in Bijlage 5. Een tabel met de analyseresultaten en veldmetingen is opgenomen in Bijlage 6.

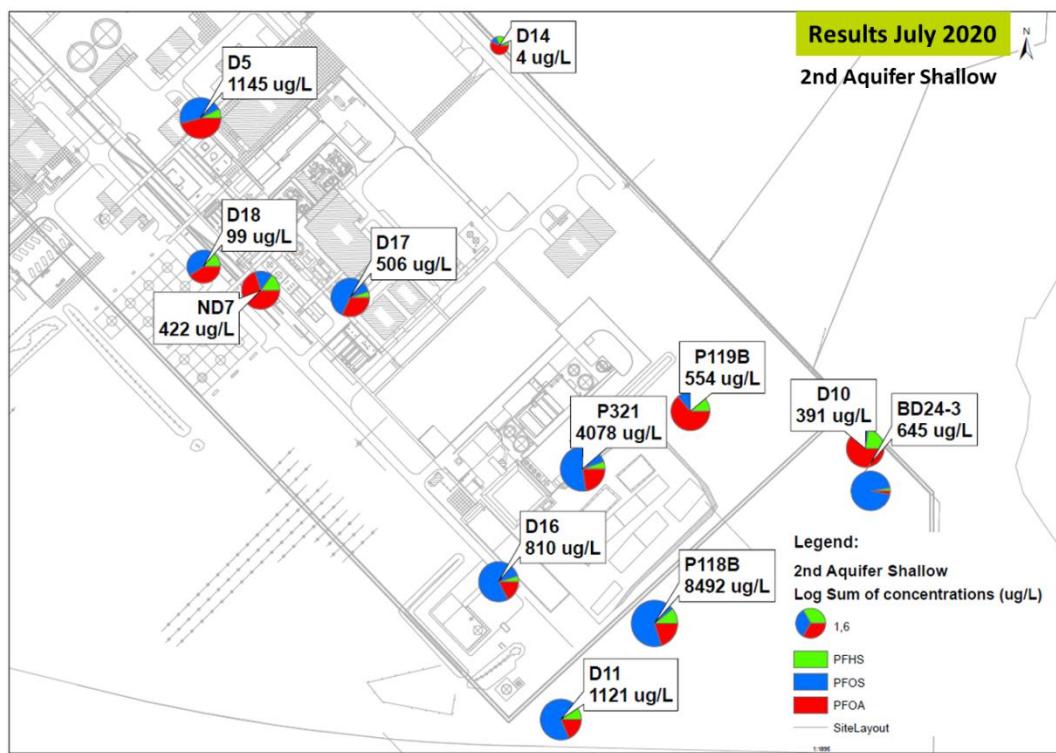
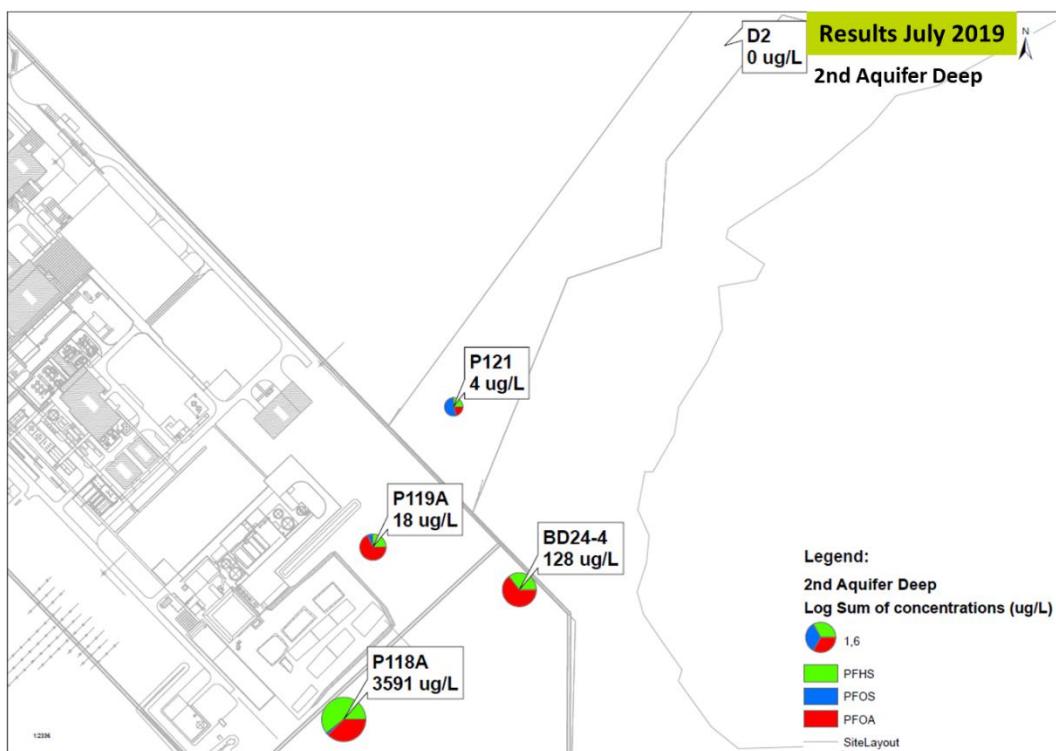
De concentratie trends voor de componenten PFOS, PFOA, PFHS, PFOSA ter hoogte van de verschillende peilbuizen in de 2^e aquifer rond de bronzones gebouw 16 en WWTP zijn opgenomen in Bijlage 7.

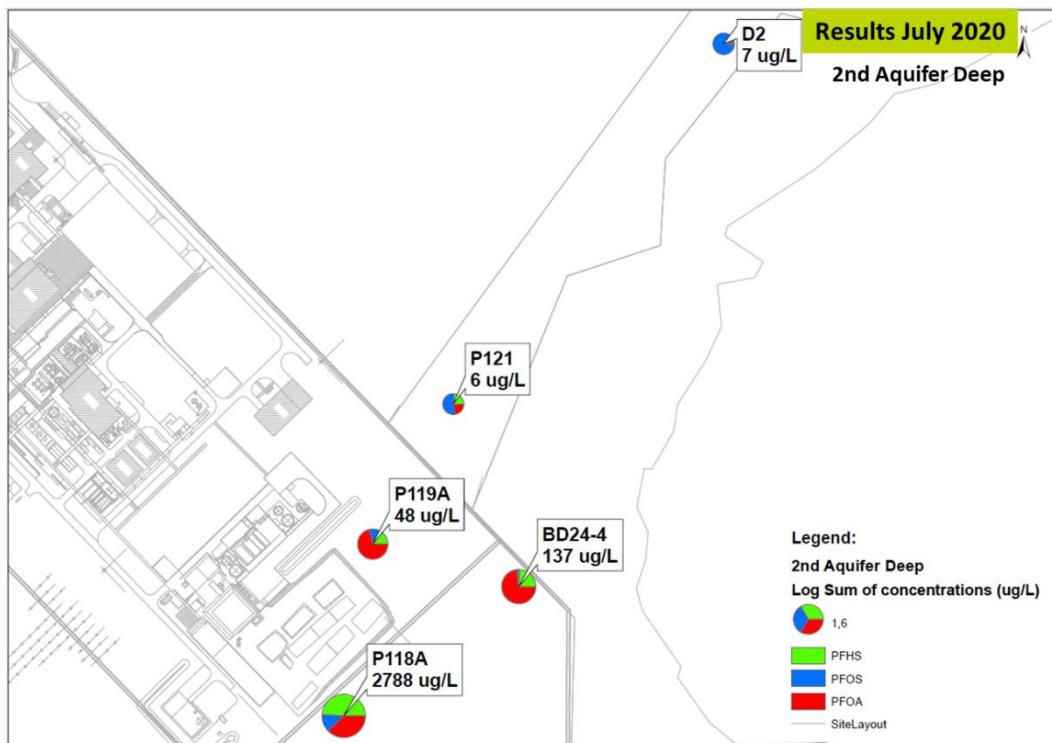
De grondwateranalyseresultaten voor de som van de FC-concentraties (PFOS, PFHS en PFOA) van juli 2018, 2019 en 2020 in de 2^e aquifer zijn in de volgende figuren voorgesteld. Er is telkens een onderscheid gemaakt tussen de middeldiepe (max. diepte 16 m-mv) en de diepe peilbuizen (max. diepte 24 m-mv).

Figuur 4.7 Schematische voorstelling FC-concentraties in grondwater 2^e aquifer (augustus 2017 tot juli 2020)





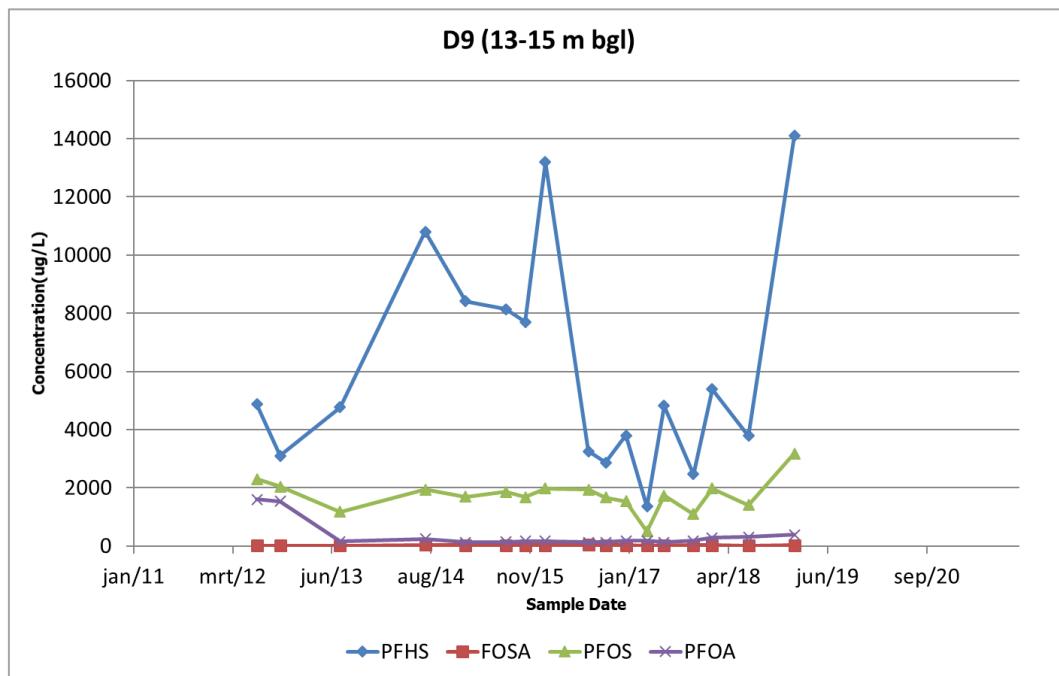




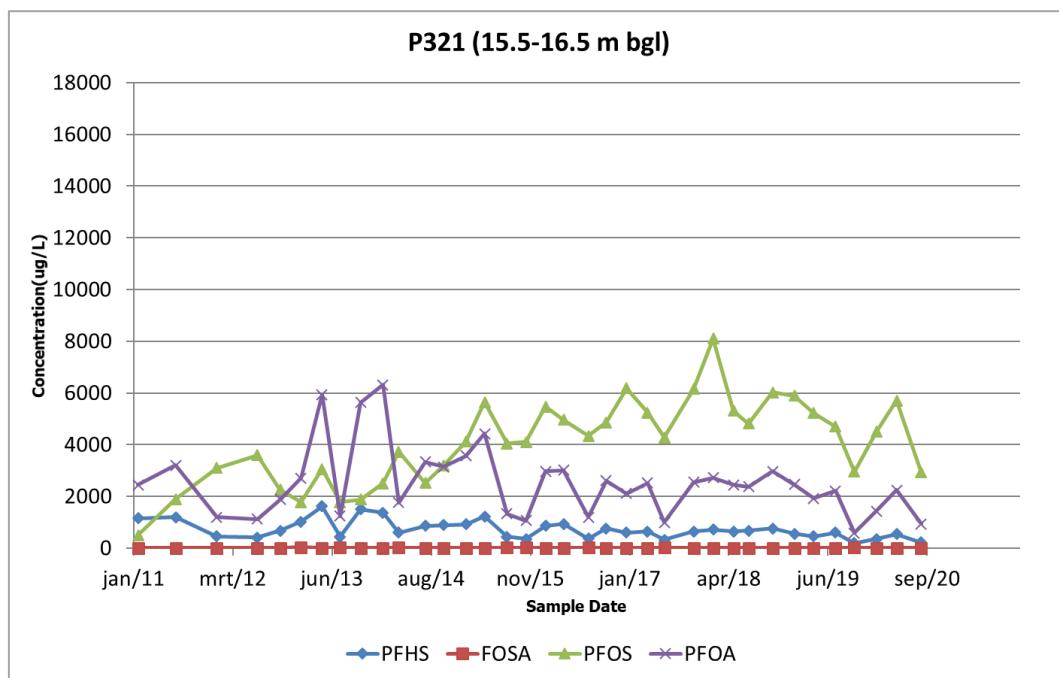
Enkel van de meetpunten waar belangrijke waarnemingen/trends zijn aangetoond, zijn hieronder de grafieken weergegeven.

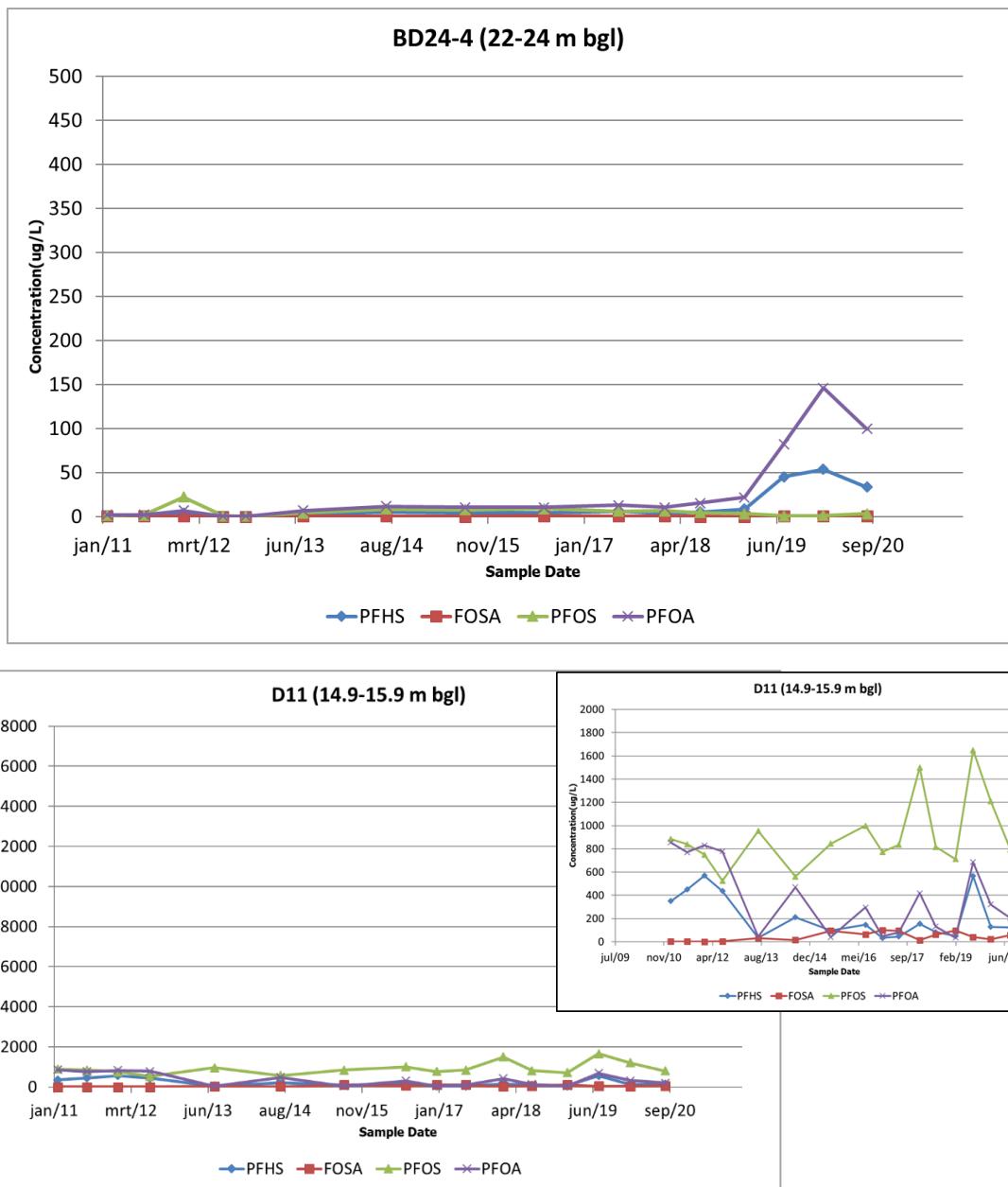
Figuur 4.8 Evolutie FC-concentraties in 2^e aquifer t.h.v. Gebouw 16 en zone WWTP

Bronzone Gebouw 16



Bronzone WWTP





Belangrijkste vaststellingen:

Gebouw 16:

- De meest voorkomende component in de 2^e aquifer is hoofdzakelijk PFOA, met ter hoogte van sommige peilbuizen PFOS;
- In het algemeen kan gesteld worden dat de concentraties aan FC verbindingen in het grondwater ter hoogte van gebouw 16 dalen of stabiel zijn in de tijd, maar de concentraties fluctueren; en
- In peilbuis D9 is al meermaals een verhoogde concentratie aan PFHS in het grondwater gemeten. De concentratie gemeten in januari 2019 (14.100 µg/l) ligt nog steeds lager dan vijfmaal de concentratie die gemeten is bij de start van de monitoring³ in deze peilbuis (5*4.876 µg/l in juli 2012). Deze verhoogde concentratie kon niet bevestigd worden omdat in

³ Te verwachten eindresultaat conform BSP 2008 voor 2e aquifer ter hoogte van de kernzones

juli 2019 de peilbuis beschadigd is geweest door de constructiewerken in de omgeving. De peilbuis zal eind augustus 2020 worden herplaatst.

■ WWTP:

- De meest voorkomende component in 2^e aquifer is hoofdzakelijk PFOS, hoewel dit op sommige locaties PFOA is;
- In het algemeen kan gesteld worden dat de concentraties aan FC verbindingen in het grondwater ter hoogte van de WWTP dalen of stabiel zijn in de tijd, maar de concentraties fluctueren;
- In peilbuis P118B is een dalende concentratie (som FC) aangetoond van 17500 µg/L naar 8492 µg/L;
- De concentratietrend ter hoogte van P321 was in het vorige tussentijdse rapport (TTR8) als stijgend beschouwd, met een maximale concentratie aan PFOS van 8.100 µg/L in februari 2018. Sinds 2019 is deze stijgende trend niet meer bevestigd. De PFOS-concentraties zijn lager dan vijfmaal de beginconcentratie bij opstart (5*1.900 µg/l in juli 2011); en
- Een licht stijgende trend is aangetoond in de grenspeilbuis BD24-4, nabij het Blokkersdijk natuurreervaat, voor de componenten PFOA en PFHS, en in peilbuis D11 aan de zuidelijke perceelsgrens voor componenten PFOS, PFOA en PFHS. De concentraties zijn lager dan 10% van de oplosbaarheid van deze parameters⁴.

4.3.3 Conclusies en aanbevelingen

- De gemeten concentraties in het grondwater van de 2^e aquifer ter hoogte van de bronzones zijn lager dan het vijfvoud van de concentraties bij de opstart van de sanering (juli 2011). Ter hoogte van de terreingrenzen zijn de concentraties aan FC-verbindingen lager dan 10% van de oplosbaarheid. Hieruit volgt dat wordt voldaan aan de saneringsdoelstellingen voor de tweede aquifer volgens het BSP van 2008;
- De monitoring in de 2^e aquifer dient verder gezet te worden; en
- Ter vervanging van peilbuis D9 dient een nieuwe peilbuis geïnstalleerd te worden om de PFHS-concentratie in de 2^e aquifer rondom gebouw 16 verder op te volgen. Hiervoor zal in augustus 2020 peilbuis D9bis op ongeveer 25 meter afstand van D9, nabij pompput PP11, geïnstalleerd worden. De locatie van D9 is niet langer beschikbaar door het plaatsen van een nieuw pijpenrek.

4.4 Zuidelijke perceelsgrens

4.4.1 Uitgevoerd veldwerk

De peilbuizen bemonsterd tijdens de periodieke grondwaterstaalname langs de zuidelijke perceelsgrens zijn opgeliist in onderstaande tabel.

De locaties van deze peilbuizen zijn aangeduid op Kaart 4D.

⁴ Te verwachten eindresultaat conform BSP 2008 voor 2e aquifer ter hoogte van de terreingrenzen

Tabel 4.3 Overzicht bemonsterde peilbuizen zuidelijke terreingrens (2017-2020)

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum						Analyse
		Jan 2018	Jul 2018	Jan 2019	Jul 2019	Jan 2020	Jul 2020	
B3-bis	2,5-3,5	X	Niet*	Niet**	Niet**	Niet**	Niet**	FC
B7	1,8-3,8	X	Niet*	Niet**	Niet**	Niet**	Niet**	FC
P372	4,8-5,8	X	X	X	X	X	X	FC
P378	3,5-5,5	X	X	Niet**	Niet**	Niet**	Niet**	FC
PA109A	5,0-6,0	X	X	Niet**	Niet**	Niet**	Niet**	FC
PA111A	4,0-6,0	X	X	Niet**	Niet**	Niet**	Niet**	FC
PA112	3,5-5,5	X	X	Niet**	Niet**	Niet**	Niet**	FC

FC: PFOS, PFOA, PFHS, FOSA

*Peilbuizen B3-bis en B7 waren niet aanwezig tijdens de monitoringsronde van juli 2018 omwille van permanent verlies door de start van de Oosterweel werken.

**Peilbuizen niet langer beschikbaar sinds januari 2019 door de start van de Oosterweel werken.

4.4.2 Bespreking resultaten

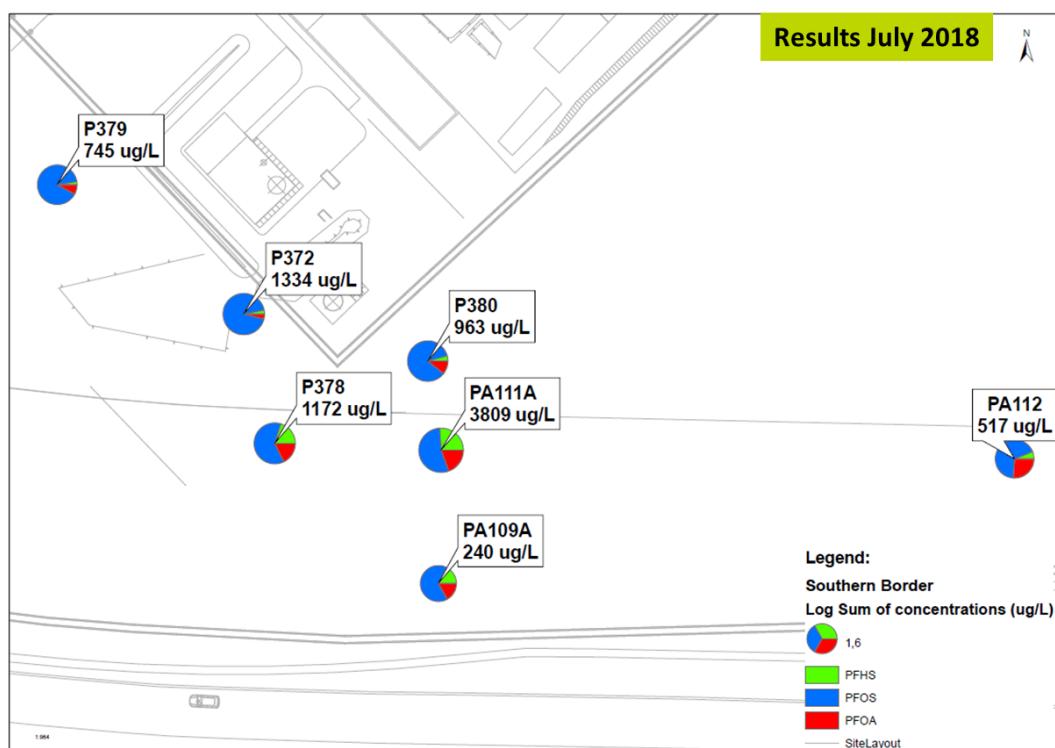
De analyseverslagen zijn toegevoegd in Bijlage 5. Een tabel met de analyseresultaten en veldmetingen is opgenomen in Bijlage 6.

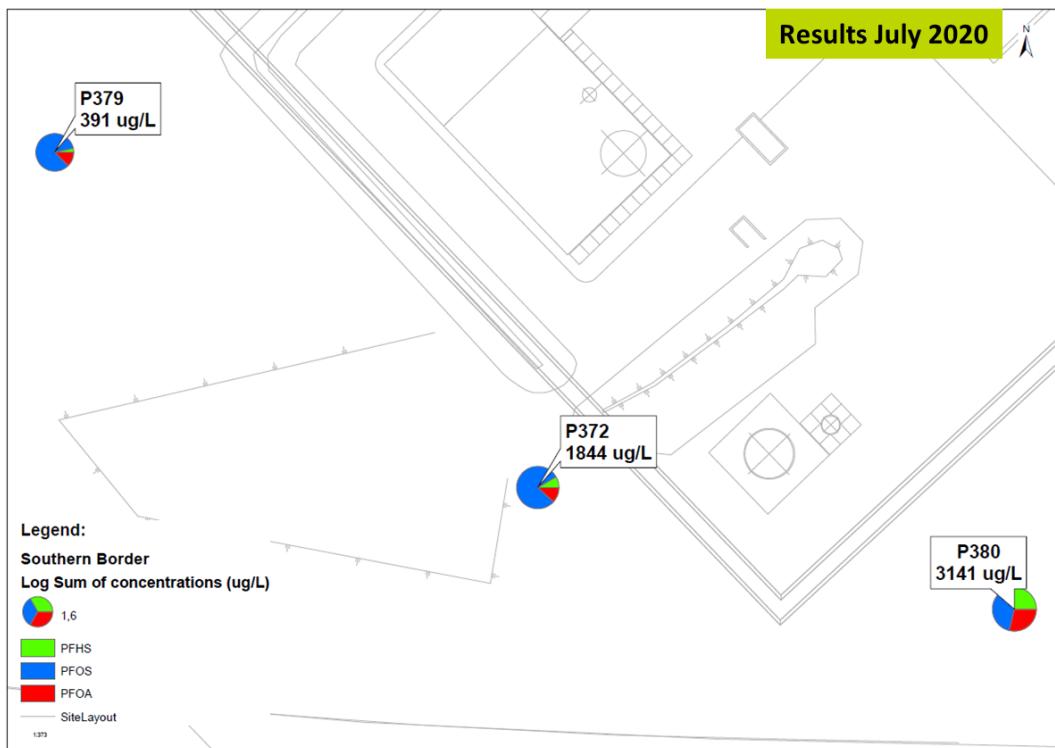
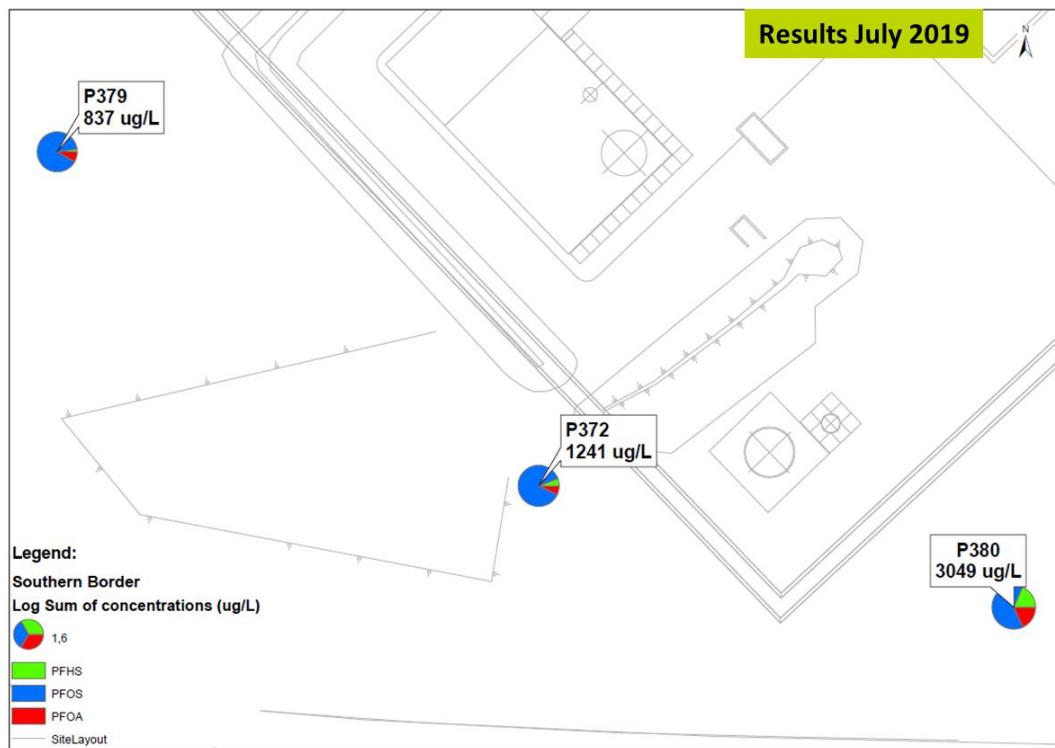
De concentratie trends voor de componenten PFOS, PFOA, PFHS, PFOSA van de verschillende peilbuizen ter hoogte van de zuidelijke perceelsgrens zijn opgenomen in Bijlage 7.

De grondwateranalyseresultaten voor de som van de FC-concentraties (PFOS, PFHS en PFOA) van juli 2018, 2019 en 2020 aan de zuidelijke perceelsgrens zijn in de volgende figuren voorgesteld.

Peilbuizen P379 en P380 maken eveneens deel uit van de groep van peilbuizen die wordt opgevolgd in de WWTP zone van de 1^e aquifer.

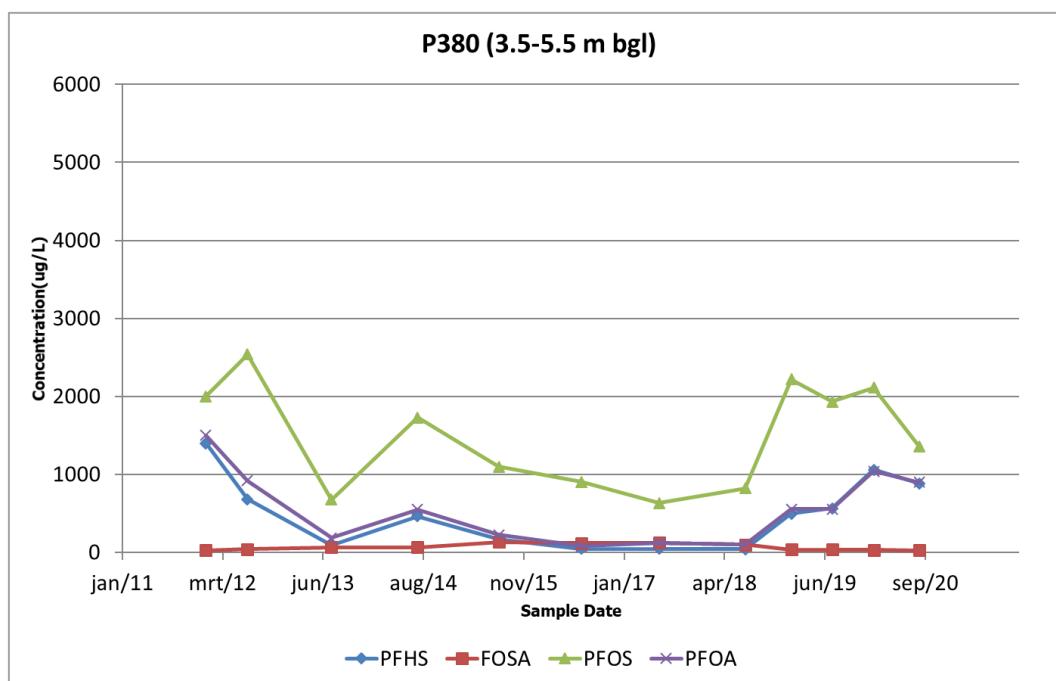
Figuur 4.9 Schematische voorstelling FC-concentraties in grondwater ter hoogte van zuidelijke perceelsgrens (augustus 2017 tot juli 2020)





Enkel van de meetpunten waar belangrijke waarnemingen/trends zijn aangetoond, zijn hieronder de grafieken weergegeven.

Figuur 4.10 Evolutie FC-concentraties t.h.v. zuidelijke perceelsgrens



Belangrijkste vaststellingen:

- De meest voorkomende component ter hoogte van de zuidelijke perceelsgrens is PFOS;
- In het algemeen kan gesteld worden dat de concentraties aan FC verbindingen in het grondwater ter hoogte van de zuidelijke perceelsgrens dalen of stabiel zijn in de tijd, maar de concentraties fluctueren; en
- Ter hoogte van peilbuis P380 zijn licht hogere concentraties gemeten tijdens deze rapportageperiode ten opzichte van de vorige monitoringsperiode; de laatste meting toont echter terug een lagere concentratie aan.

4.4.3 Conclusies en aanbevelingen

- Algemeen kan gesteld worden dat de gemeten concentraties FC-verbindingen in grondwater ter hoogte van de zuidelijke perceelsgrens in dezelfde lijn liggen als de concentraties gemeten tijdens voorgaande bemonsteringen; en
- De grondwaterconcentraties ter hoogte van de zuidelijke perceelsgrens dienen verder opgevolgd te worden. Van zodra de werken in het kader van de Oosterweelverbinding het toelaten, dient opnieuw een uitgebreider netwerk aan peilbuizen in deze zone gemonitord te worden en dienen verdere acties genomen te worden waar nodig.

4.5 Tankenpark – Minerale olie (P18 en P28)

4.5.1 Uitgevoerd veldwerk

Op vraag van OVAM in 2016 zijn twee peilbuizen (P18 en P28) mee opgenomen in de jaarlijkse monitoring voor analyse op minerale olie. Deze peilbuizen bevinden zich nabij de ondergrondse tanks ter hoogte van gebouw 3. De locaties van deze peilbuizen zijn aangeduid op Kaart 4A.

De peilbuizen bemonsterd tijdens de periodieke grondwaterstaalname zijn opgeliist in onderstaande tabel.

Tabel 4.4 Overzicht bemonsterde peilbuizen Tankenpark (2017-2020)

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum			Analyse
		Jul 2018	Jul 2019	Aug 2020	
P18	1,4-3,4	X	X	X	Minerale olie
P28	1,1-3,1	X	Niet*	Niet*	Minerale olie

* Peilbuis P28 kon niet bemonsterd worden omdat van schade opgelopen tijdens werken rond pompput PP05. Op 4 maart 2019 werd deze peilbuis gevuld met bentoniet door ERM.

4.5.2 Bespreking resultaten

De analyseverslagen zijn toegevoegd in Bijlage 5. Een tabel met de analyseresultaten en veldmetingen is opgenomen in Bijlage 6.

De concentratie trends voor minerale olie voor beide peilbuizen zijn opgenomen in Bijlage 7.

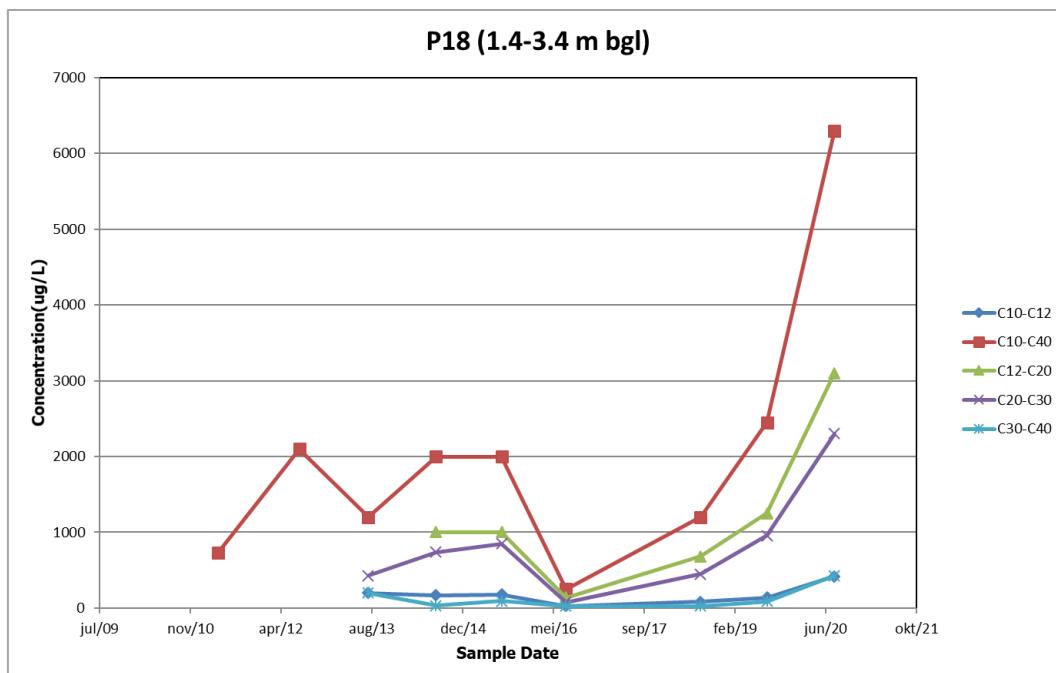
4.5.2.1 Veldparameters

De pH en geleidbaarheid van het grondwater ter hoogte van peilbuis P28 zijn niet tijdens de bemonstering gemeten omdat er, omwille van sterk verhoogde pH (circa 14), een reële kans bestaat op beschadiging van de veldmeetapparatuur.

4.5.2.2 Minerale olie

Onderstaande grafiek geeft de concentratietrend in peilbuis P18 weer. De laatste jaren (2018-2020) is er een stijgende trend in totale minerale olie concentratie (C₁₀-C₄₀) aangetoond, met een hoogst gemeten concentratie van 6.300 µg/L in augustus 2020. Deze concentratie ligt meer dan 10 keer boven de bodemsaneringsnorm (BSN) van 500 µg/l. P28 is buiten gebruik en is sinds juli 2018 niet meer bemonsterd.

Figuur 4.11 Evolutie minerale olie concentraties t.h.v. tankenpark



4.5.3 Conclusies en aanbevelingen

- De concentratie aan minerale olie in P18 stijgt sinds 2018;
- Peilbuis P28 dient vervangen te worden om de monitoring opnieuw optimaal uit te kunnen voeren; en
- Momenteel kan geen verklaring worden gegeven voor de gemeten verhoogde waarden aan minerale olie in het grondwater tijdens de voorgaande monitoringsronden.

Bijkomend onderzoek naar de oorzaak, de omvang en de ernst van deze verhoogde concentraties aan minerale olie dient uitgevoerd te worden, onder de vorm van een beschrijvend bodemonderzoek.

4.6 Blokkersdijk naturreervaat

De kwaliteit van het grond- en oppervlaktewater in het natuurgebied Blokkersdijk wordt gemonitord via de periodieke bemonstering van zeven peilbuizen en via een periodieke staalname van het oppervlaktewater van de 3M- en Blokkersdijkvijvers. Door middel van een statistische analyse van de PFOS-concentraties wordt nagegaan of er een concentratietrend aanwezig is.

De resultaten van de grond- en oppervlaktewatermonitoring en de herevaluatie van de statistische analyse van de PFOS-concentraties zijn hierna beschreven.

4.6.1 Uitgevoerd veldwerk

De peilbuizen langs het 3M pad ten westen van het natuurgebied Blokkersdijk en het oppervlaktewater van de 3M vijver en de Blokkersdijkvijver zijn driemaandelijks bemonsterd om de evolutie in FC-concentraties in deze zones na te gaan.

In onderstaande tabellen is een overzicht gegeven van de grondwater en oppervlaktewater monitoring. De locaties van de peilbuizen en de monsternamelpunten voor het oppervlaktewater zijn aangeduid op Kaart 4E.

Tabel 4.5 Overzicht staalnamelocaties natuurgebied Blokkersdijk (2017-2020)

Meetpunt	Filterdiepte (m-mv)	Bemonsteringsdatum												Analyse
		Okt 2017	Jan 2018	Apr 2018	Jul 2018	Okt 2018	Jan 2019	Apr 2019	Jul 2019	Okt 2019	Jan 2020	Apr 2020	Jul 2020	
Peilbuizen														
L21	3.0-5.0	X	X	X	X	X	X	X	X	X	X	X	X	FC
L22	2.0-4.0	X	X	X	X	X	X	X	X	X	X	Niet**	Niet**	FC
L30	1.0-3.0												X	X
L31	1.2-3.2	X	X	X	X	X	X	X	X	X	X	X	X	FC
L4	2.0-4.0	X	X	X	X	X	X	X	X	X	X	X	X	FC
P114bis	3.6-4.6	X	X	X	X	X	X	X	X	X	X	X	X	FC
P115	3.3-4.3	X	X	X	X	X	X	X	X	X	X	X	X	FC
P116	2.5-3.5	X	X	X	X	X	X	X	X	X	X	X	X	FC
Vijvers - Oppervlaktewater														
3M Pond	-	X	X	X	X	X	X	X	X	X	X	X	X	FC
Blokkersdijk standaard	-	X	X	X	X	X	X	X	X	X	X	X	X	FC
Blokkersdijk Noord	-	X	X	Niet*	X	X	X	X	X	X	X	X	X	FC

FC: PFOS, PFOA, PFHS, FOSA

*Blokkersdijk Noord werd niet bemonsterd in april 2018.

** Peilbuis L22 ging verloren in april 2020, peilbuis L30 werd in de plaats bemonsterd.

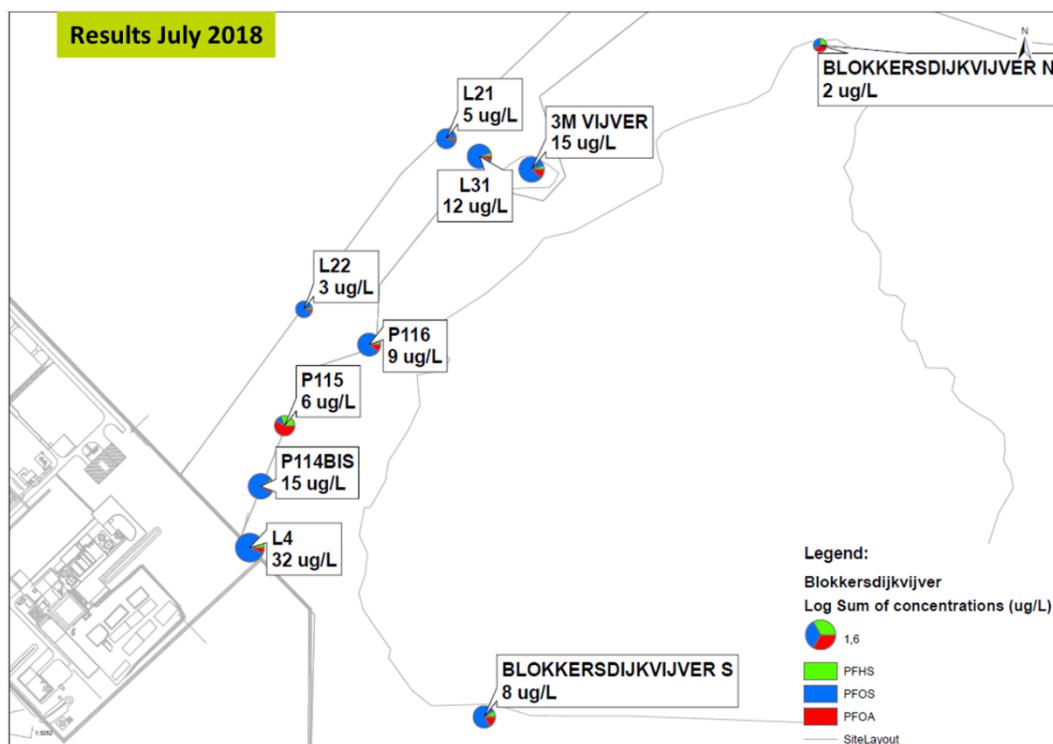
4.6.2 Bespreking resultaten

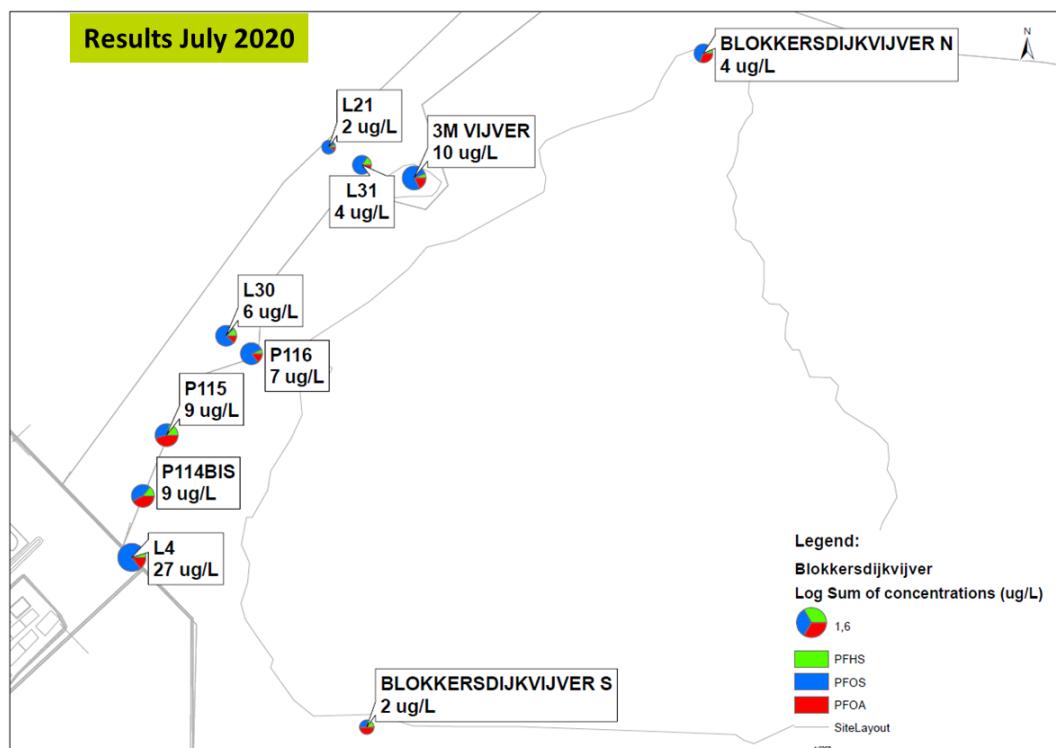
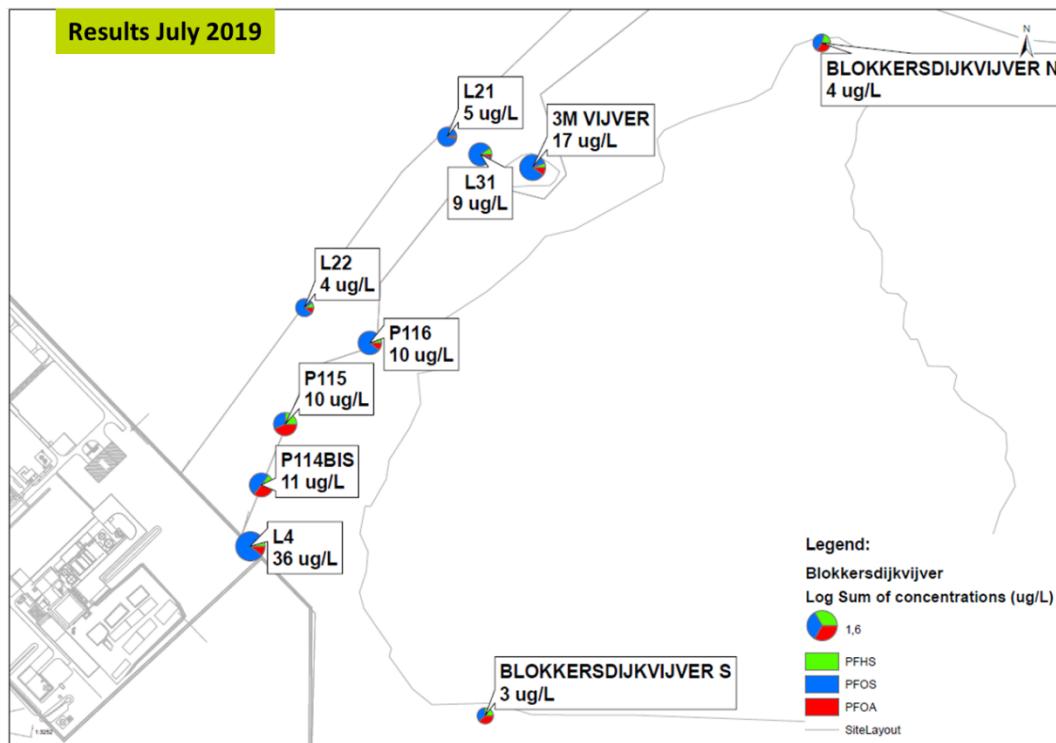
De analyseverslagen zijn toegevoegd in Bijlage 5. Een tabel met de analyseresultaten en veldmetingen is opgenomen in Bijlage 6.

De concentratie trends voor de componenten PFOS, PFOA, PFHS, PFOSA van de verschillende peilbuizen/oppervlaktewaterstalen ter hoogte van Blokkersdijk zijn opgenomen in Bijlage 7.

De grondwateranalyseresultaten voor de som van de FC-concentraties (PFOS, PFHS en PFOA) van juli 2018, 2019 en 2020 langs het 3M pad ten westen van het natuurgebied Blokkersdijk en het oppervlaktewater van de 3M vijver en de Blokkersdijkvijver zijn in de volgende figuren voorgesteld.

Figuur 4.12 Schematische voorstelling FC-concentraties in grond- en oppervlaktewater ter hoogte van het 3M pad en het natuurgebied Blokkersdijk (augustus 2017 tot juli 2020)





Belangrijkste vaststellingen:

- De meest voorkomende component in deze zone is PFOS;
- Algemeen zijn de gemeten concentraties in deze zone in het grondwater en het oppervlaktewater laag; en

- In zowel de 3M vijver als de Blokkersdijkvijver is duidelijk een fluctuatie in de PFOS-concentratie te zien. Voornamelijk voor de 3M vijver is deze fluctuatie seizoenaal, waar er hogere concentraties gemeten worden in de zomer omwille van lagere waterstanden.

4.6.3 Statistische evaluatie PFOS-concentraties

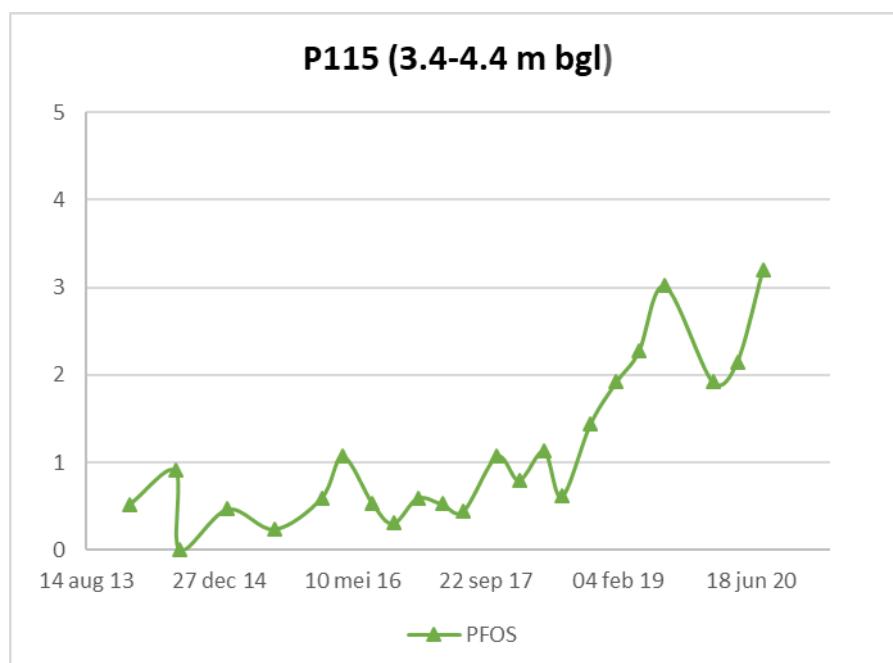
Na de goedkeuring van het bodemsaneringsproject (Eerste gefaseerd BSP, Arcadis, Ref. 11/003460, oktober 2008) in 2009 zijn er periodiek waterstalen genomen van een aantal monitoringspeilbuizen en de twee vijvers in het natuurreervaat Blokkersdijk. De analyseresultaten van deze waterstalen worden, conform het bodemsaneringsproject, gebruikt voor de statistische evaluatie van de PFOS-concentratie, om na te gaan of er een trend is. Voor de evaluatie worden twee statistische tests gebruikt: de Mann-Kendall test en de lineaire regressie test. Enkel indien beide statische tests een stijgende trend als resultaat geven, wordt gesproken van een significante stijging.

Sinds de start van de monitoring zijn er verschillende laboratoria gebruikt voor de analyse van de waterstalen. De wisseling van labo's is een gevolg van vastgestelde kwaliteitsproblemen en is gebeurd in overleg met OVAM.

Vanaf 2014 zijn alle analyses uitgevoerd door het 3M Environmental laboratorium in de Verenigde Staten met controle analyses door SGS nv. Op basis van de vergelijking met de controle analyses en de grondige kwaliteitscontrole van het 3M lab wordt aangenomen dat de dataset van het 3M lab de meest representatieve is.

Uit de evaluatie (zie Bijlage 8) blijkt dat de PFOS-concentraties ter hoogte van peilbuizen L22, L31, L4, P114bis en P116 een significant dalende trend vertonen voor beide tests. Peilbuis P115 vertoont wel een significant stijgende trend voor PFOS volgens voor beide tests. De absolute PFOS concentratie is echter algemeen in lijn of lager dan omliggende peilbuizen. Gelet op de dalende trend in de overige peilbuizen en de absolute concentraties ter hoogte van peilbuis P115 wordt gesteld dat het momenteel niet noodzakelijk is om eventuele bijkomende acties te nemen. Wel wordt voorgesteld om dezelfde frequentie van bemonstering aan te houden voor peilbuis P115 (driemaandelijks). De evolutie van de PFOS-concentraties ter hoogte van peilbuis P115 zijn weergegeven in onderstaande grafiek. De gemeten concentraties in de peilbuis L21 en de oppervlaktewaterstalen in de Blokkersdijkvijver en 3M vijver geven geen statistisch significante trend weer.

Figuur 4.13 Evolutie PFOS-concentratie t.h.v. P115



Figuur 4.14 Blokkersdijk



4.6.4 Conclusies en aanbevelingen

- Er is geen statistisch stijgende trend voor de gemeten PFOS concentraties ter hoogte van de Blokkersdijk vijver aangetoond;
- De PFOS-concentratie trend in de Blokkersdijk peilbuizen, de 3M vijver en de Blokkersdijkvijver dient verder opgevolgd te worden via monitoring en een statistische evaluatie. Hierbij wordt aangeraden om de data van het 3M US lab te blijven gebruiken; en
- Op basis van de statistisch stijgende trend voor PFOS die is aangetoond in peilbuis P115, wordt aangeraden om deze peilbuis driemaandelijks te blijven monitoren. Gelet op de dalende trend in de overige peilbuizen en de absolute concentraties ter hoogte van peilbuis P115 wordt gesteld dat er momenteel geen noodzaak is voor bijkomende saneringsacties in de Blokkersdijkzone, of om de monitoringsfrequentie voor het oppervlaktewater te verhogen (reeds 4-maal per jaar).
- Uit de resultaten van het geohydrologisch onderzoek uitgevoerd in 2014 (zie TTR6) blijkt dat de oude chemische riolering die ter hoogte van het 3M pad loopt mogelijk een secundaire verontreinigingsbron is. 3M zal van start gaan met het uitwerken van een plan van aanpak om deze oude riolering te verwijderen.

4.7 Palingbeek en Tophatgracht

4.7.1 Uitgevoerd veldwerk

Om de PFOS-vuilvracht op te volgen dat de 3M site verlaat richting de Schelde wordt het oppervlaktewater van de Palingbeek en de Tophatgracht en het oppervlaktewater ter hoogte van het bemalingsstation driemaandelijks bemonsterd. De PFOS-concentraties worden gebruikt bij de berekening van de totale vuilvracht die de Schelde bereikt (zie paragraaf 4.9 voor de berekening). Sinds november 2017 worden de grachten eveneens maandelijk door ERM bemonsterd en door het intern labo van 3M Zwijndrecht geanalyseerd, gezien de nabijheid van de Oosterweel werken.

De staalnamelocaties zijn aangeduid op Kaart 4F.

Tabel 4.6 Overzicht staalnamelocaties Palingbeek en Tophatgracht

Meetpunt	Filterdiepte (m-mv)	Bemonsteringsdatum												Analyse
		Okt 2017	Jan 2018	Apr 2018	Jul 2018	Okt 2018	Jan 2019	Apr 2019	Jul 2019	Okt 2019	Jan 2020	Apr 2020	Jul 2020	
Palingbeek														
12	-	X	X	X	X	X	X	X	X	X	X	X	X	FC
13	-	X	X	X	X	X	X	X	X	X	X	X	X	FC
Tophatgracht														
5	-	X	X	X	X	X	X	X	X	X	X	X	X	FC
Pumping station	-	X	X	X	X	X	X	X	X	X	X	X	X	FC

FC: PFOS, PFOA, PFHS, FOSA

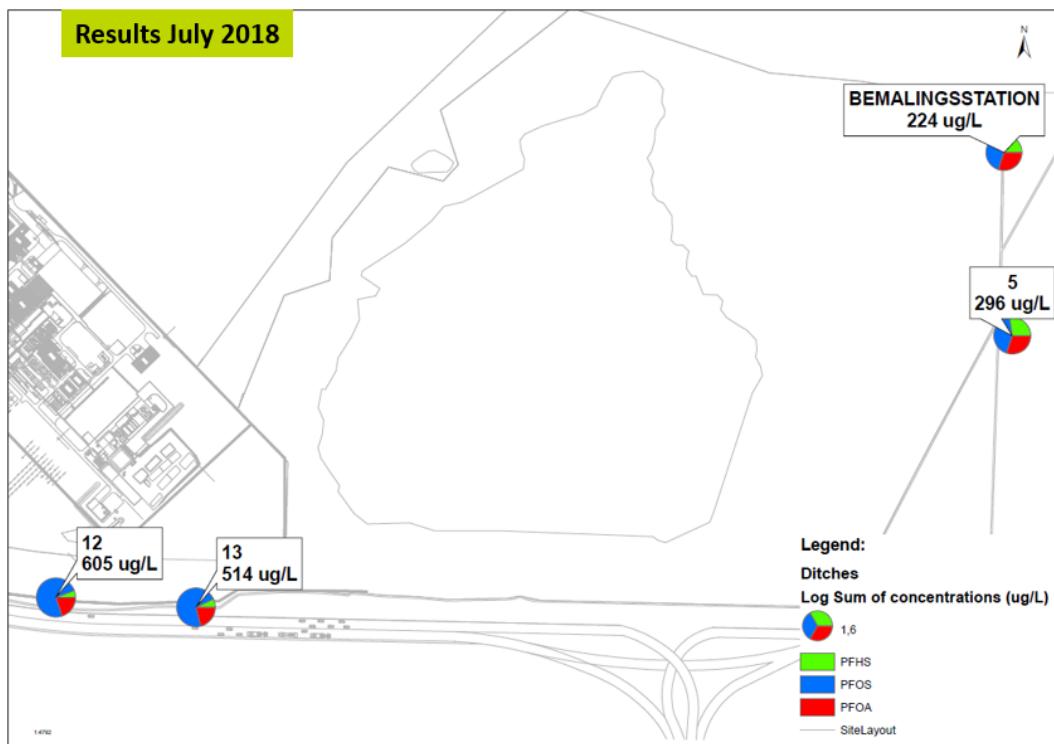
4.7.2 Bespreking resultaten

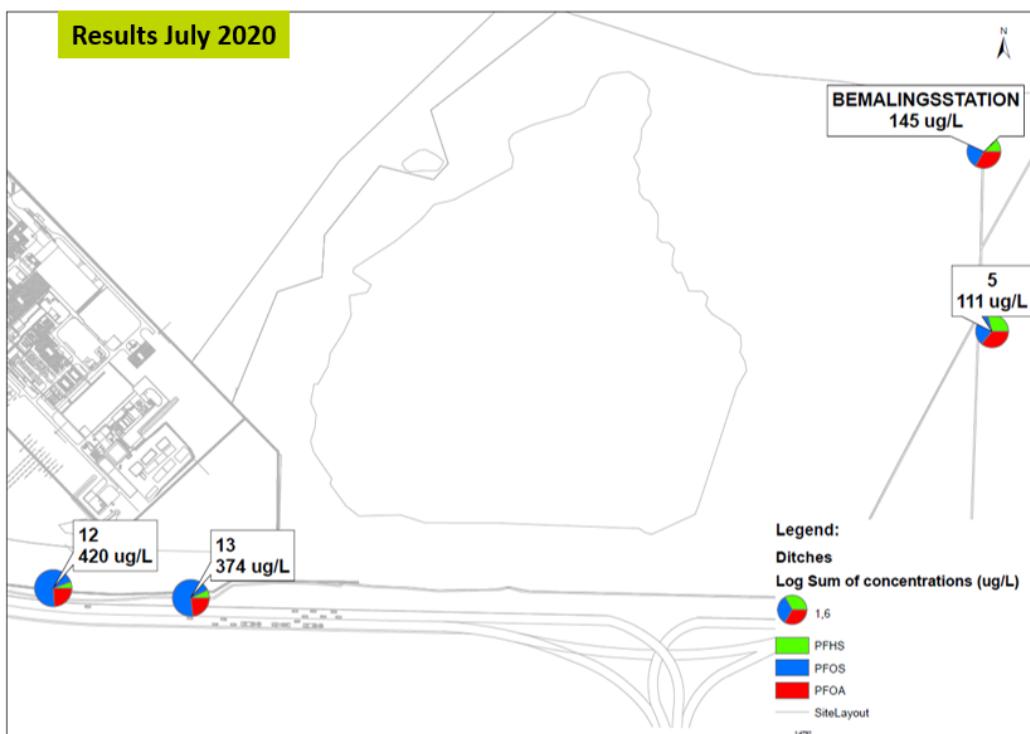
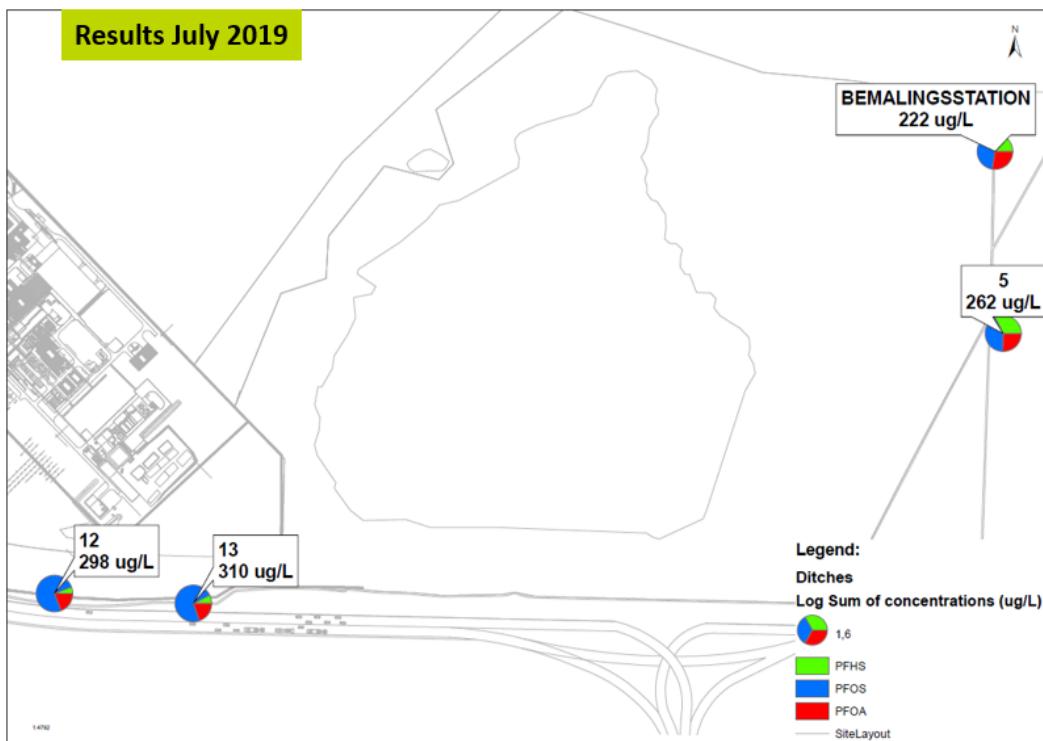
De analyseverslagen zijn toegevoegd in Bijlage 5. Een tabel met de analyseresultaten en veldmetingen is opgenomen in Bijlage 6.

De concentratie trends voor de componenten PFOS, PFOA, PFHS, PFOSA ter hoogte van de Palingbeek en Tophatgracht zijn opgenomen in Bijlage 7.

De analyseresultaten voor de som van de FC-concentraties (PFOS, PFHS en PFOA) van juli 2018, 2019 en 2020 aan de Palingbeek en Tophatgracht zijn in de volgende figuren voorgesteld.

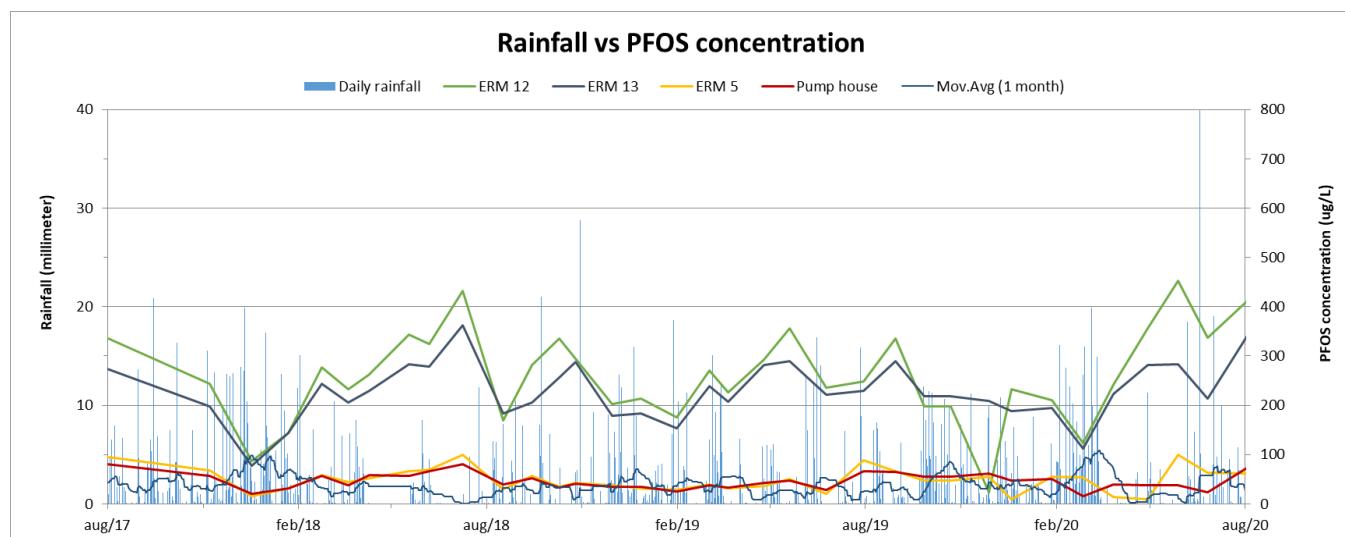
Figuur 4.15 Schematische voorstelling FC-concentraties in oppervlaktewater van de Palingbeek en de Tophatgracht (augustus 2017 tot juli 2020)





Onderstaande grafiek geeft de gemeten PFOS-concentraties in de Palingbeek en Tophatgracht weer ten opzichte van de dagelijkse neerslag voor de periode 2017-2020.

Figuur 4.16 PFOS concentratie in de grachten t.o.v. dagelijkse neerslag



ERM12 en ERM13 thv Palingbeek; ERM5 en Pump house thv Tophatgracht.

Belangrijkste vaststellingen:

- Palingbeek:
 - De FC-concentraties ter hoogte van de Palingbeek vertonen globaal gezien een stabiel verloop sinds 2012.
- Tophatgracht:
 - De FC-concentraties ter hoogte van de Tophat gracht liggen algemeen lager dan in de Palingbeek; en
 - De FC-concentraties ter hoogte van de Tophatgracht vertonen globaal gezien een stabiel verloop sinds 2012.
- In beide grachten fluctueren de concentraties sterk. Deze fluctuaties blijken gerelateerd aan de neerslaghoeveelheid; de concentraties in het oppervlaktewater worden meer verduld bij meer neerslag.

4.7.3 Conclusies en aanbevelingen

- De FC-concentraties in de grachten zijn het hoogste nabij het 3M terrein (Palingbeek: locaties 12 en 13) en nemen af naarmate de afstand tot het 3M terrein groter wordt (Tophatgracht). Dit is vermoedelijk het gevolg van verdunning door het instromen van minder of niet-verontreinigd grond- of oppervlaktewater. De concentraties in het oppervlaktewater van de Tophatgracht (locatie 5) en het bemalingsstation zijn vergelijkbaar en fluctueren sterk. Deze fluctuatie verloopt samen met neerslaghoeveelheid; en
- De driemaandelijkse bemonsteringsfrequentie met analyses door het SGS labo dienen behouden te blijven; de concentratiefluctuaties en hun impact op de vuilvracht dienen verder opgevolgd te worden. Ook wordt er aanbevolen de bijkomende maandelijkse monitoring met aanvullende analyses te behouden zolang de Oosterweelwerken bezig zijn.

4.8 Regenwatercollector put en bedrijfswaterzuivering

Om de PFOS-vuilvracht die het 3M terrein verlaat als gevolg van de lozing vanuit de waterzuiveringsinstallatie (WWTP) en de infiltratie van mogelijk verontreinigd grondwater in het regenwaterrioolsysteem te monitoren, worden er stalen genomen van het effluent van de WWTP en

de collector put. De collector put is de verzamelpunt waarin al het water van de regenwaterriolering wordt verzameld voor het, samen met het gezuiverde bedrijfsafvalwater, in de Schelde geloosd wordt.

In april 2019 heeft OVAM 3M gevraagd een actieplan op te maken om de concentraties aan PFOS in het regenwater te reduceren. Dit actieplan is in mei 2019 aan de OVAM overgemaakt. Infiltrerend grondwater verontreinigd met FC's zorgt er immers voor dat via deze weg een relevante hoeveelheid verontreinigd grondwater in het regenwater rioolsysteem infiltrert, op te sporen en vervolgens aan te pakken.

Eind 2019 is de collector put door 3M gereinigd. In januari 2020 is een actief koolfilter systeem geïnstalleerd om het water uit de regenwatercollector put te behandelen. Nadien zijn verscheidene aanpassingen gedaan (zoals het installeren van een zandfilter en het verminderen van de hoeveelheid regenwater die bij hevige regenval via een bypass geloosd wordt) om de efficiëntie van het systeem te verbeteren. De bijkomende behandelingsstap vermindert de vuilvracht vanuit het regenwater rioleringssysteem naar de Schelde. In ditzelfde kader zal in augustus 2020 eveneens een camera inspectie van de regenwaterriolering uitgevoerd worden om zo lekken, waarschijnlijk verontreinigd grondwater in het regenwater rioolsysteem infiltrert, op te sporen en vervolgens aan te pakken.

Bovendien heeft 3M verschillende projecten lopende om de FC concentraties in het effluent van de waterzuivering verder te verlagen, zodat de activiteiten verder gezet kunnen worden binnen de geldende omgevingsvergunning⁵. In het volgende tussentijdse rapport zal hier dieper op ingegaan worden.

4.8.1 Uitgevoerd veldwerk

Stalen van het effluent van de WWTP en van de collector put worden wekelijks door 3M genomen en geanalyseerd door het labo van 3M Zwijndrecht. De wekelijkse monitoring door 3M vindt plaats in het kader van de geldende milieuvergunning; deze data worden door ERM gebruikt voor de opvolging van de geloosde concentraties in het kader van de lopende sanering.

Daarnaast worden elk kwartaal waterstalen genomen door ERM op deze locaties, die geanalyseerd worden op FC-verbindingen door het laboratorium van 3M Environmental en/of SGS.

De locatie van de collector put en de bedrijfswaterzuivering (WWTP) is aangeduid op Kaart 4G.

⁵ 3M heeft een nieuwe omgevingsvergunning vanaf september 2020 (OMGP-2020-0032 ; dd 17 september 2020). Hierin zullen nieuwe lozingsnormen voor o.a. PFOS (1 µg/l) opgenomen worden.

Tabel 4.7 Overzicht staalnamelocaties ERM effluent WWTP en collector put

Meetpunt	Filterdiepte (m-mv)	Bemonsteringsdatum													Analyse
		Okt 2017	Jan 2018	Apr 2018	Jul 2018	Okt 2018	Jan 2019	Apr 2019	Jul 2019	Okt 2019	Jan 2020	Apr 2020	Jul 2020	Wekelijks	
Effluent WWTP	-	X	X	X	X	X	X	X	X	X	X	X	X	X	FC
Regenwatercollect or put	-	X	X	X	X	X	X	X	X	X	X	X	X	X	FC

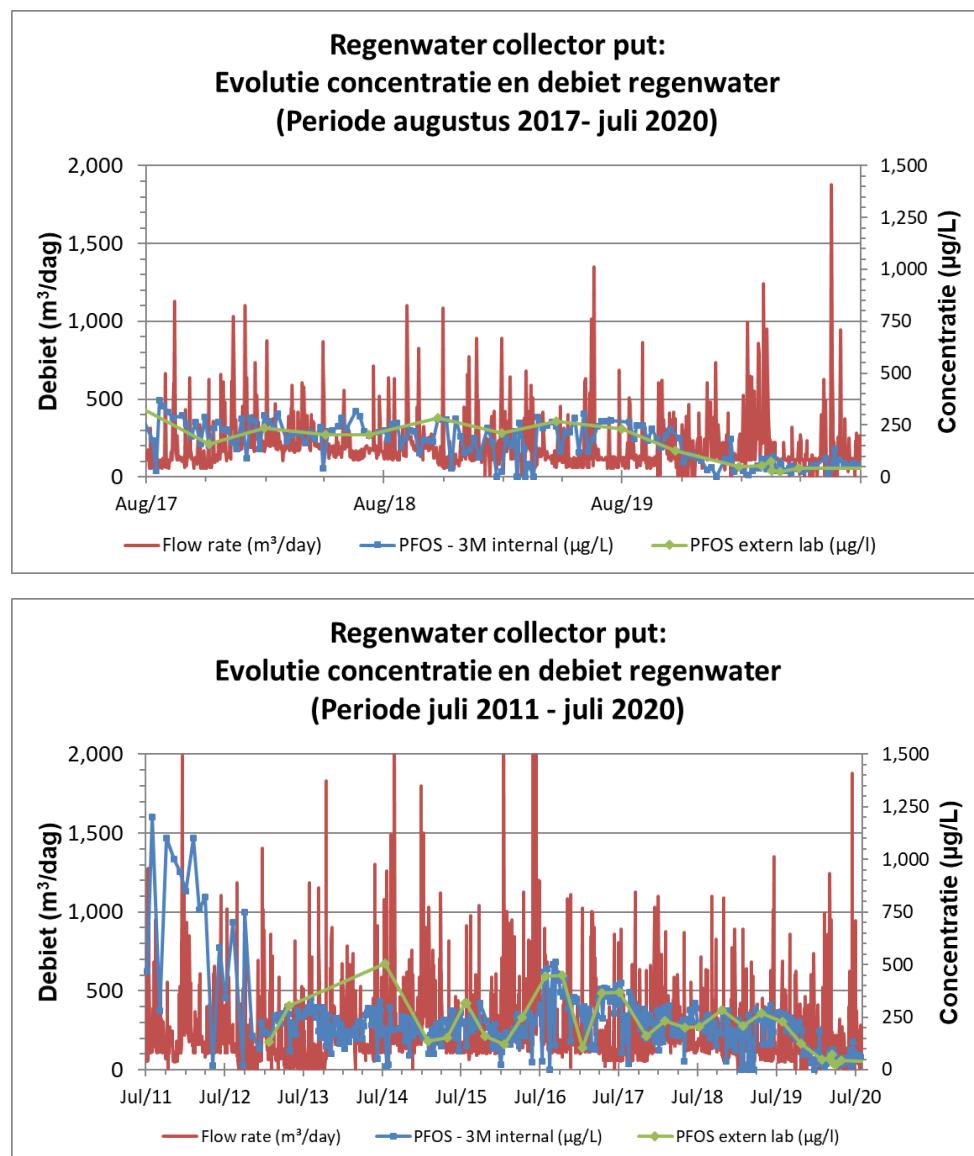
FC: PFOS, PFOA, PFHS, FOSA

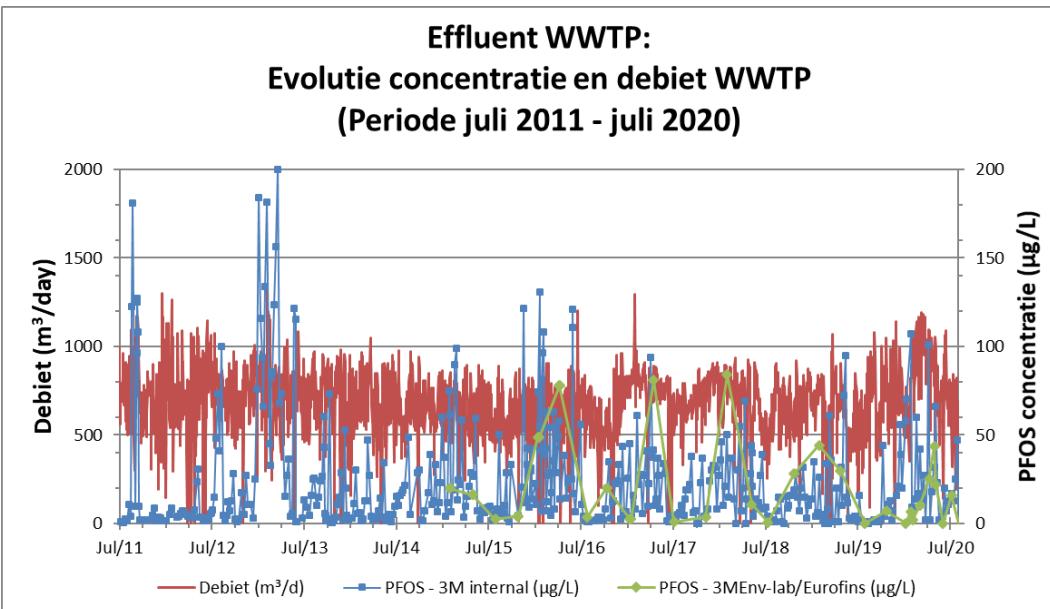
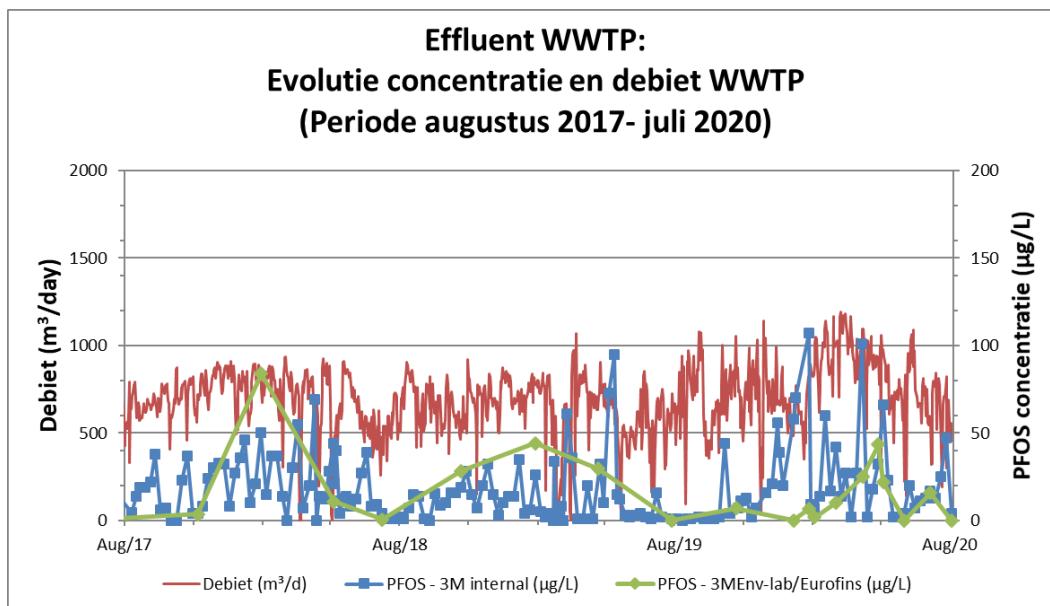
4.8.2 Bespreking resultaten

De analyseverslagen zijn toegevoegd in Bijlage 5. Een tabel met de analyseresultaten en veldmetingen is opgenomen in Bijlage 6.

In onderstaande grafieken is de evolutie van concentratie aan PFOS ter hoogte van de regenwatercollector put en in het effluent van de WWTP weergegeven, in combinatie met het gemeten debiet. Onderstaande grafieken en gemiddelden zijn gebaseerd op de wekelijkse analyseresultaten van het intern labo van 3M in Zwijndrecht. De wekelijkse monitoring door 3M vindt plaats in het kader van de geldende milieuvergunning; deze data worden door ERM gebruikt voor de opvolging van de geloosde concentraties in het kader van de lopende sanering. Merk op dat de gemeten concentraties ter hoogte van de regenwater collectorput concentraties betreffen die in de collector put gemeten zijn, en waar mogelijk nog een verdunningseffect optreedt bij regenval op het moment dat het water verpompt wordt naar de Schelde. Ter vergelijking zijn de driemaandelijkse concentraties gemeten door het 3M US lab/SGS ook weergegeven in de grafieken.

Figuur 4.17 Evolutie PFOS concentratie in collector put en effluent WWTP





Belangrijkste vaststellingen:

- De PFOS-concentraties in de regenwatercollector put zijn sinds februari 2020, na installatie van het actief koolfilter systeem, aanzienlijk gedaald:
 - In de periode juli 2017-januari 2020 is een gemiddelde PFOS concentratie van 190 µg/l in de collector put gemeten; en
 - In de periode februari 2020-juli 2020 is een gemiddelde PFOS concentratie van 65 µg/l in de collector put gemeten.

De gemeten concentraties (intern 3M lab) zijn geregeld nog hoger dan de ‘streefwaarde’ van 30 µg/l zoals opgenomen in het BSP van 2008; er dient evenwel nog een meetonzekerheid van 50% in rekening gebracht te worden bij de afvoetsing met de vooropgestelde ‘streefwaarde’.

Merk op dat vanzodra er een relatief hoge concentratie in het effluent gemeten werd, een bijkomende controle-analyse gedaan werd door een extern labo op aangeven van 3M.

In de periode volgend op deze rapportage periode heeft/zal 3M bijkomende maatregelen treffen om deze concentraties verder te reduceren.

- De PFOS-concentraties in het effluent van de WWTP fluctueren sterk, en vertonen noch een dalende noch een stijgende trend:
 - De gemiddelde PFOS-concentratie in het effluent van de WWTP gedurende deze rapportageperiode bedraagt 19 µg/L (obv analyseresultaten van het intern 3M labo).

Ook hier werkt 3M momenteel verscheidene projecten uit om in lijn met de geldende vergunningen de activiteiten te kunnen verder zetten.

4.8.3 Conclusies en aanbevelingen

Aanbevolen wordt om de dagelijkse opvolging van het debiet en de wekelijkse opvolging van de concentraties in het effluent van de bedrijfswaterzuivering en in de collector put/effluent van de collector put door 3M te behouden, alsook de driemaandelijkse opvolging door ERM/SGS.

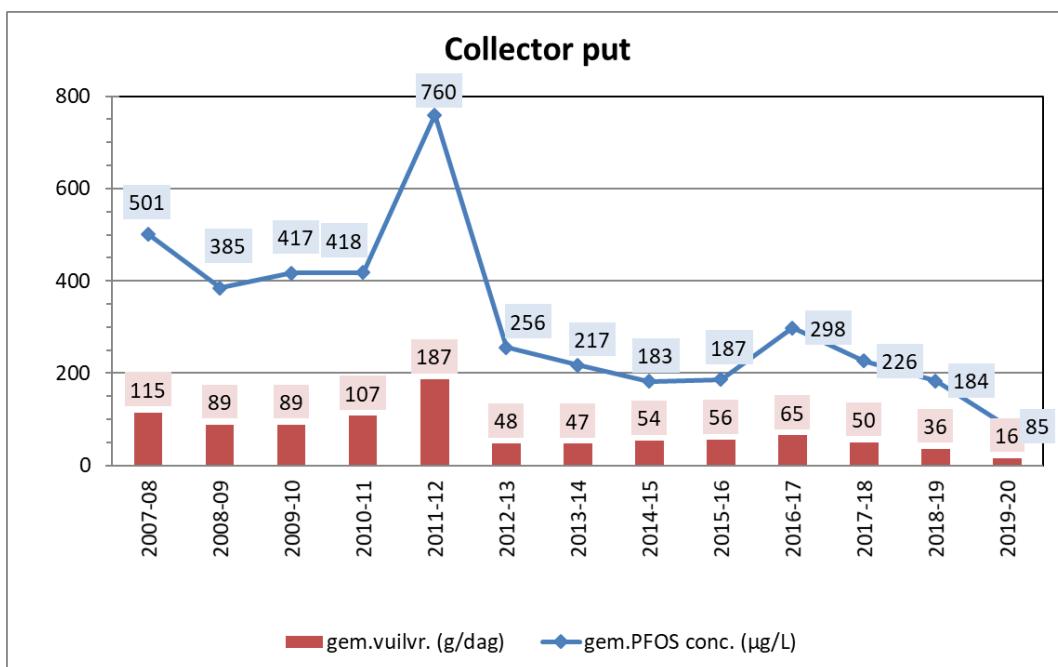
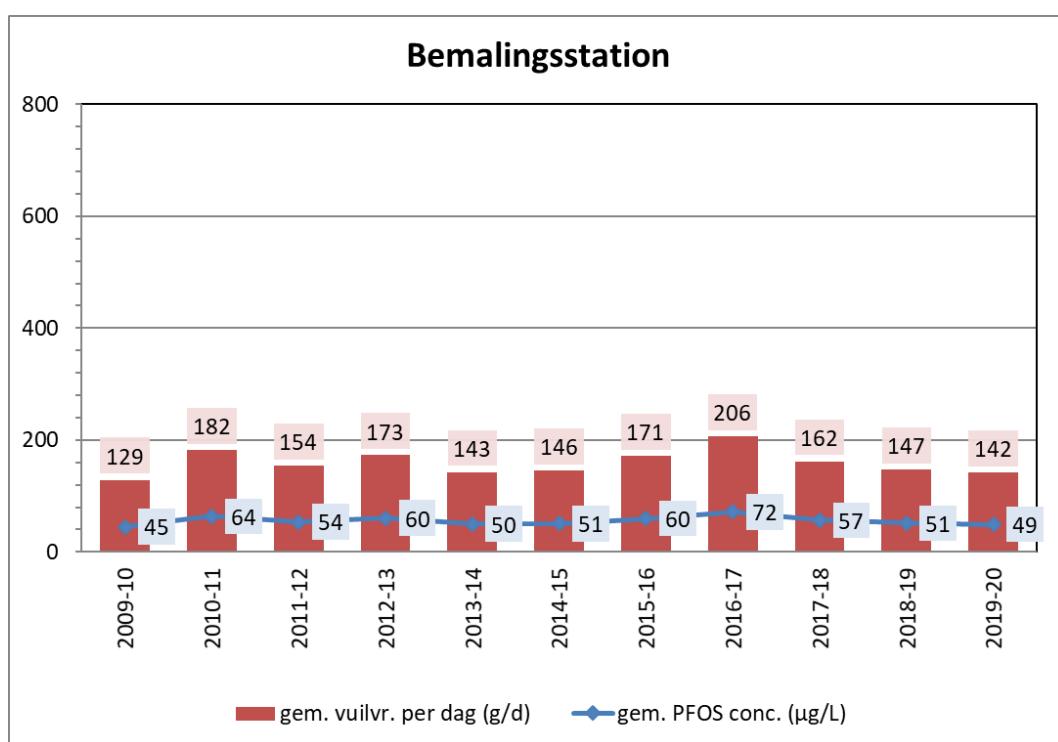
Een verdere optimalisatie van de opstelling van het actief koolfilter systeem bij de collector put is momenteel in uitvoering door 3M en dient de concentraties verder te verlagen. 3M heeft eveneens een aantal projecten lopende om de FC concentraties in het effluent van de WWTP verder te verlagen.

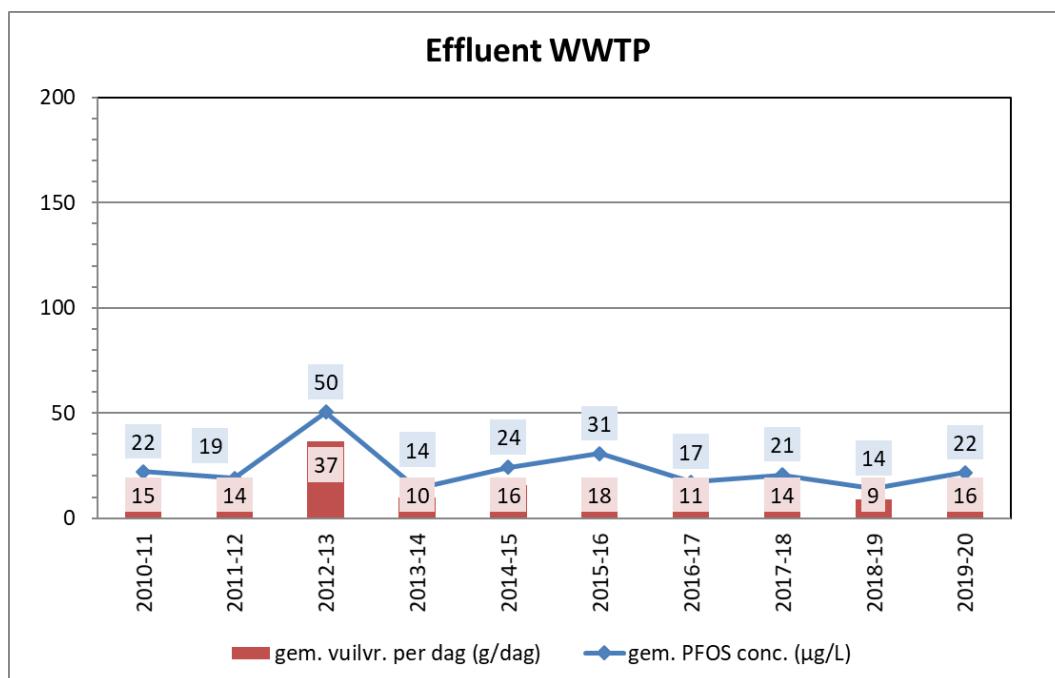
4.9 PFOS-vuilvracht richting Schelde

De gemiddelde dagelijkse PFOS-vuilvracht die het 3M terrein verlaat naar de Schelde wordt berekend aan de hand van de concentraties gemeten over een periode van één jaar ter hoogte van het bemalingsstation in de Tophatgracht (driemaandelijkse monitoring, 3M Environmental lab in de Verenigde Staten), in de regenwatercollector put (wekelijkse monitoring, 3M QC lab) en het effluent van de WWTP (wekelijkse monitoring, 3M QC lab). De wekelijkse monitoring door 3M vindt plaats in het kader van de geldende milieuvergunning; deze data worden door ERM gebruikt voor de vuilvrachtberekening in het kader van de lopende sanering.

In onderstaande grafieken worden de gemiddelde PFOS-concentratie (resultaten intern 3M labo) en vuilvracht uitgaande van de verschillende locaties weergegeven.

Figuur 4.18 Gemiddelde PFOS concentratie en vuilvrachtberekening per locatie





De gemiddelde vuilvracht vanuit de collector put en het bemalingsstation vertonen een dalende trend ten opzichte van vorige monitoringsperiodes. Er kan opgemerkt worden dat er vanuit de collector put in 2011-2012 een sterk verhoogde vuilvracht was gemeten. Dit was waarschijnlijk te wijten aan het slijb dat in de collectorput aanwezig was. Intussen is dit slijb verwijderd, en is de vuilvracht terug gedaald. In 2020 is eveneens een actief koolfilter systeem geplaatst voor behandeling van het water uit de collector put, wat bijgedragen heeft tot een verdere daling in concentraties en vuilvracht. Een grotere positieve impact van het actief koolfilter systeem dat geplaatst is, zal de komende jaren duidelijker worden.

De gemiddelde dagelijkse PFOS-vuilvracht naar de Schelde wordt vergeleken met de PNEC-toetsingwaarde zoals gedefinieerd in het BSP van 2008, 370 g/dag. De gemiddelde PFOS-vuilvracht van het 3M terrein naar de Schelde is in het eerste gefaseerd bodemsaneringsproject geraamd op 290 g/dag. De veilige vuilvracht voor PFOS (PNEC-waarde) is toen vastgelegd op 370 g/dag en wordt gebruikt als toetsingwaarde om de gemiddelde dagelijks PFOS-vuilvracht naar de Schelde vanuit de aanwezige bodem- en grondwaterverontreiniging te evalueren. Er dient opgemerkt te worden dat deze PNEC-waarde geherevalueerd zal worden, rekening houdend met de nieuwe inzichten rond FC's.

Op basis van gegevens bekomen uit de monitoringscampagne van 1 augustus 2017 tot en met 31 juli 2020 is de gemiddelde vuilvracht in onderstaande tabel opgenomen voor de drie laatste monitoringsperiodes (zie Tabel 4.8).

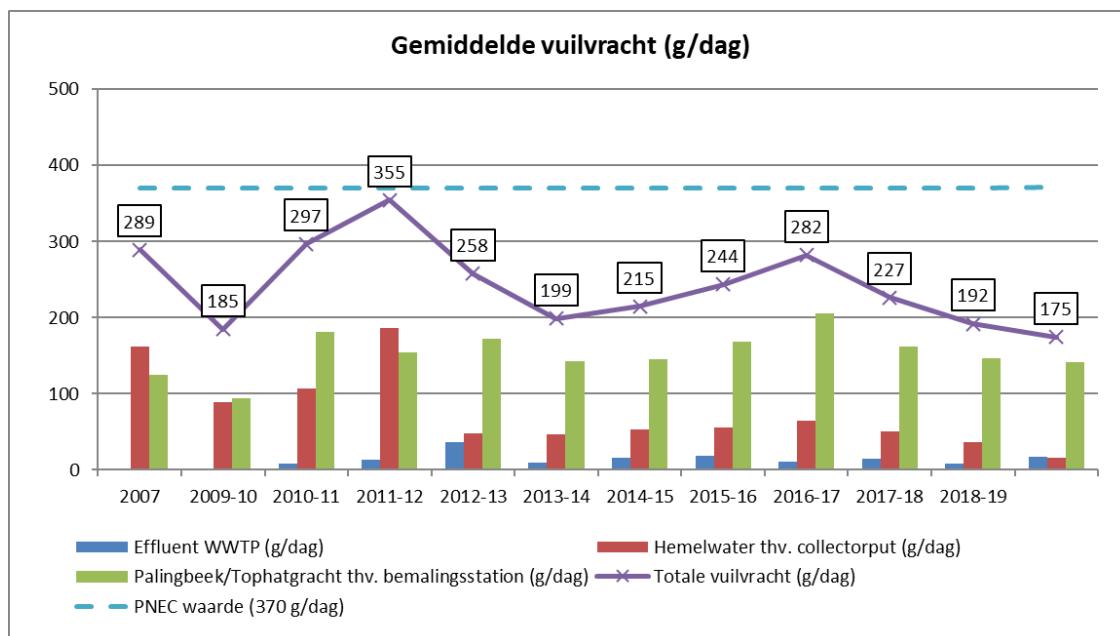
Tabel 4.8 PFOS vuilvracht richting de Schelde (augustus 2017 t.e.m. juli 2020)

Influx naar de Schelde	Gemiddeld debiet (m³/dag)	Gemiddelde PFOS concentratie (µg/L)	Gemiddelde vuilvracht (g/dag)
augustus 2017 – juli 2018			
Effluent van het bedrijfsafvalwater (incl. opgepompt grondwater)	685	21	14
Regenwaterriolering	223	226	50
Palingbeek/Tophatgracht*	2865	57	162
Totale vuilvracht			227
augustus 2018 – juli 2019			
Effluent van het bedrijfsafvalwater (incl. opgepompt grondwater)	636	14	9
Regenwaterriolering	197	184	36
Palingbeek/Tophatgracht*	2865	51	147
Totale vuilvracht			192
augustus 2019 – juli 2020			
Effluent van het bedrijfsafvalwater (incl. opgepompt grondwater)	760	22	17
Regenwaterriolering	189	102	16
Palingbeek/Tophatgracht*	2865	50	142
Totale vuilvracht			175

* Berekend op basis van de gemiddelde concentraties ter hoogte van het bemalingsstation

Een overzicht van de berekende gemiddelde vuilvracht naar de Schelde is eveneens samengevat in onderstaande grafiek.

Figuur 4.19 Evolutie gemiddelde PFOS vuilvracht naar de Schelde



De gemiddelde vuilvracht neemt sinds 2017 gestaag af, in tegenstelling tot in het vorige tussentijdse verslag waar een periode van toename werd gerapporteerd. Deze afname in vuilvracht is hoofdzakelijk het gevolg van een daling in vuilvracht vanuit de regenwatercollector put en de grachten.

4.10 Grondhoop verontreinigd met kwik en FC-verbindingen

Op het terrein is een hoop grond verontreinigd met kwik en FC-verbindingen opgeslagen. Het depot situeert zich in het oosten van het bedrijfsterrein, op een braakliggende zone. Op Kaart 3 is de locatie van deze hoop aangeduid.

Na overleg met OVAM (dd. 22 oktober 2013), is besloten de monitoringsfrequentie te verlagen van jaarlijks naar vijfjaarlijks. Op vraag van OVAM d.d. 13 april 2016 is nog een tussentijds controle van het afdek uitgevoerd op 26 april 2016. Aangezien de afdekking op dat moment in goede staat was, werd de volgende controle in 2018 gepland. In de periode augustus 2017 tot juli 2020 werd de afdekking gecontroleerd in augustus 2018. Tijdens deze controle is er geen schade geobserveerd. Het milieudagboek van deze controle is opgenomen in Bijlage 9.

De afdekking bevindt zich nog in een goede staat. ERM stelt voor om de volgende controleronde uit te voeren in 2023.

4.11 Data verificatie

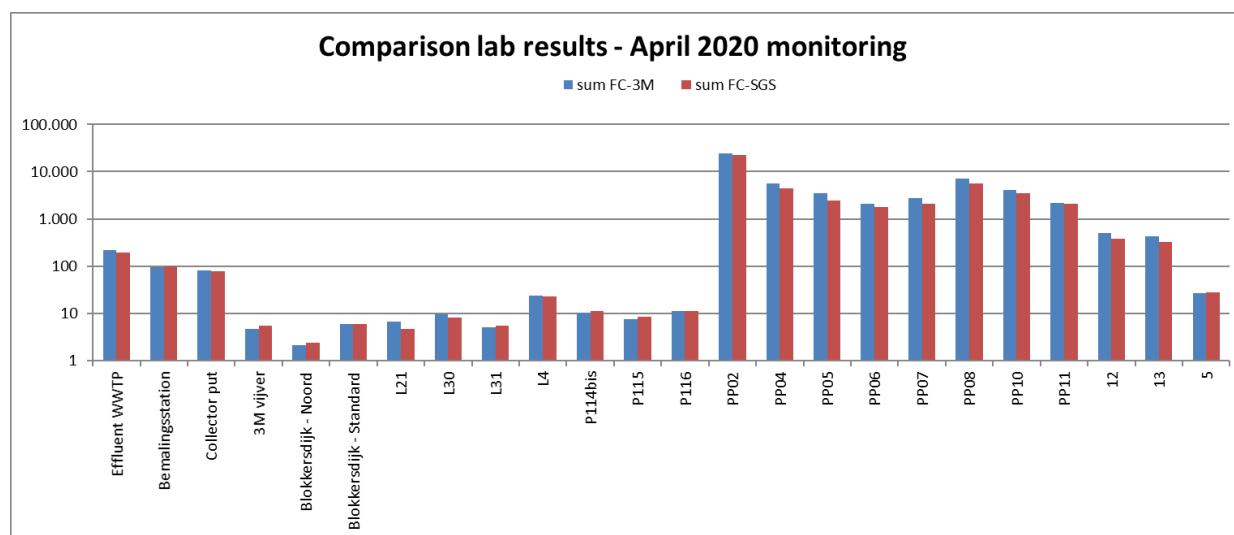
Zoals overeengekomen met OVAM (vergadering dd. 22 oktober 2013) zijn alle waterstalen die tot en met 2018 zijn genomen, door het 3M Environmental Laboratorium in de Verenigde Staten geanalyseerd, waarbij de duplicaten minstens één keer door SGS nv zijn geanalyseerd op precisie en om te voldoen aan de lokale wetgeving. SGS is een OVAM-gecertificeerd labo. De bemonsterings- en analysecontrole zijn gespreid over de vier controleperiodes.

Volgens het akkoord dat in februari 2019 met de OVAM werd bereikt (brief dd. 19 februari 2019), is de volgende dupliecatbemonstering vanaf 2019 tot juli 2020 toegepast. Alle watermonsters zijn in deze periode geanalyseerd door het 3M Environmental Laboratorium in de VS. Ter controle zijn verschillende dupliecatmonsters ook geanalyseerd door SGS nv. Een overzicht van de uitgevoerde analyses van dupliecatmonsters door SGS is hieronder gegeven:

- Monsters nodig voor de PNEC-evaluatie (effluent WWTP, verzamelput, pompstation): 3M Dupliecatmonsters vier keer per jaar: SGS;
- Oppervlaktewatermonsters Blokkersdijk en 3M vijver en grondwatermonsters 3M pad: 3M Dupliecatmonster één keer per jaar (april): SGS;
- Grondwatermonsters peilbuizen brongebieden (eerste en tweede aquifer): 3M Dupliecatmonster één keer per drie jaar (juli 2020): SGS; en
- Grondwatermonsters pompputten: 3M Dupliecatmonster één keer per jaar: SGS.

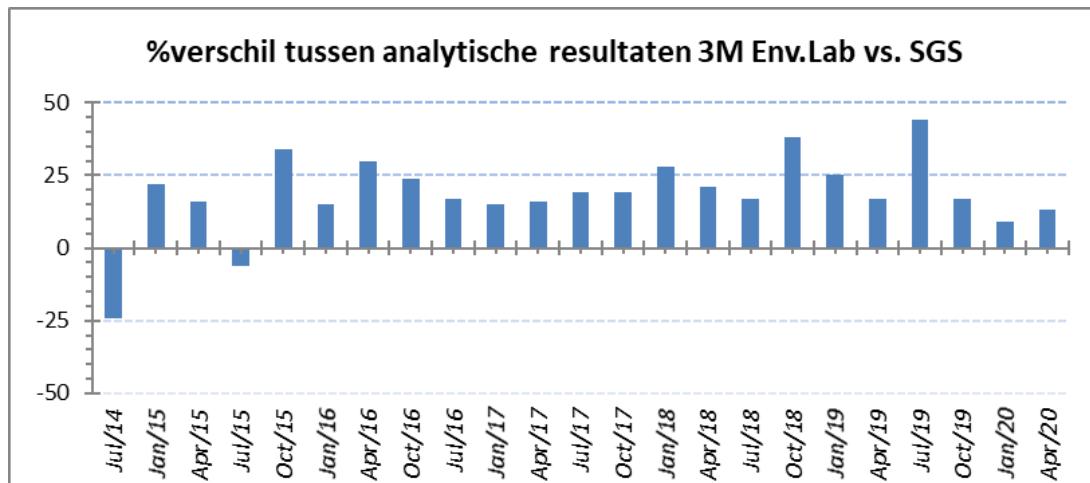
Uit een evaluatie van de datasets voor desbetreffende periode (SGS vs. 3M labo) volgt dat de resultaten van beide labo's in grote lijnen vergelijkbaar zijn.

Figuur 4.20 Vergelijking som FC resultaten SGS vs. 3M Environmental Laboratorium, april 2020



De volgende grafiek geeft ook de historische data vanaf 2014 weer. Hier is te zien hoe de gemiddelde FC-concentraties over het algemeen hoger liggen bij het 3M lab in vergelijking met SGS.

Figuur 4.21 Percentageverschil som FC resultaten SGS vs. 3M Environmental laboratorium 2014-2020



Het verschil tussen de datasets wordt waarschijnlijk veroorzaakt door een verschil in de voorbereiding van de waterstalen en de analytische methode (de gebruikte apparatuur en de kalibratie ervan) die elk labo gebruikt.

Echter, sinds juli 2020 worden op vraag van 3M alle analyses uitgevoerd door het door OVAM erkende laboratorium SGS, met uitzondering van de oppervlakewatermonsters van de Blokkersdijk- en 3M vijver, en de grondwatermonsters van het 3M pad (in totaal 10 stalen). Deze stalen zullen door het 3M Environmental Laboratorium in de Verenigde Staten geanalyseerd blijven worden zodat de dataset voor de statistische analyse consequent van hetzelfde laboratorium blijft komen (zie ook kleine wijziging in paragraaf 2.5.3).

5. VOORZIENE WERKEN IN VOLGENDE JAAR VAN DE SANERING

5.1 Opvolging grondwaterextractiesysteem

De controle van het P&T systeem zal maandelijks worden uitgevoerd door ERM.

Om een inschatting te kunnen maken van de ontrokken vuilvracht via de waterfase wordt voorgesteld om de driemaandelijkse monitoring van het opgepompte water te behouden voor alle pompputten.

ERM raadt aan het onttrekkingssysteem op regelmatige basis te onderhouden.

5.1.1 Plaatsing PP12 en PP13

Zoals vermeld in paragraaf 3.2, is pompput PP09 in juli 2019 buiten gebruik gesteld in het kader van de Oosterweel werken. Een deel van de voorbereidingswerken zoals graven sleuven en het plaatsen van tijdelijke toezichtputten heeft in het voorjaar van 2020 plaatsgevonden. De effectieve installatie van de pompputten is omwille van COVID-19-maatregelen uitgesteld tot eind augustus 2020. De twee nieuwe pompputten zullen met de volgende filterstelling geïnstalleerd worden: PP12 (3-4,7 m-mv) en PP13 (3,5-5,9 m-mv).

5.1.2 P&T systeem – Optimalisatie en pomptest

De huidige saneringsinstallatie heeft, mede gezien de aard van de verontreiniging, vrij intensief onderhoud nodig (zie paragraaf 3.3). Om het systeem onderhoudsvriendelijker te maken, wordt onderzocht om de dompelpompen te vervangen door een kleiner aantal gecentraliseerde slangenzuigen waarop de verschillende putten via een manifold zouden aangesloten worden. Hierdoor zullen de pompen niet meer in rechtstreeks contact zijn met de verontreiniging, en zouden er eveneens minder pompen nodig zijn om het systeem te opereren, waardoor onderhoud minder intensief zou moeten zijn. In 2021 zullen de nodige pomptesten uitgevoerd worden om de technisch meest geschikte opbouw van de installatie te bepalen.

5.2 Monitoring grond, grondwater en oppervlaktewater

In onderstaande tabel is een overzicht gegeven van het voorgestelde monitoringsprogramma voor 2021. Per zone is weergegeven welke staalnamelocaties zullen bemonsterd en/of opgevolgd worden in het kader van het bodemsaneringsproject, alsook de frequentie die gevolgd zal worden.

Elk kwartaal zal een monitoringsronde uitgevoerd worden waarbij de grondwaterstand in een geselecteerd aantal peilbuizen gemeten zal worden om zo de grondwaterstromingsrichting ter hoogte van de saneringsinstallatie op te volgen.

Voor de monitoringscampagne ingepland in januari 2021 zal het monitoringsprogramma van 2020 nog gevuld worden. Vanaf de monitoringscampagne van april 2021 zal het monitoringsprogramma van 2021 in werking treden. Het monitoringsprogramma zal jaarlijks opnieuw geëvalueerd worden.

Peilbuizen die omwille van werken op of nabij de site niet meer in gebruik zijn, maar die wel belangrijk zijn voor de opvolging van de verontreinigingssituatie (zowel bronzones als zuidelijke perceelsgrens), zullen herplaatst worden van zodra de activiteiten dit toelaten.

Tabel 5.1 Monitoringsprogramma 2021

Locatie	Aantal peilbuizen	Frequentie	Peilbus ID	Verantwoording	Opmerkingen
P&T systeem	10	Driemaandelijks	PP02, PP04, PP05, PP06, PP07, PP08, PP10, PP11, PP12 en PP13	-	Minstens maandelijkse inspecties van de pompputten en installatie. PP01 werd in oktober 2018 buiten gebruik gesteld en vervangen door PP11. PP09 werd in juli 2019 buiten gebruik gesteld. Ter vervanging zullen twee nieuwe pompputten (PP12 en PP13) geïnstalleerd worden in de zomer van 2020.
Stabiliteit	-	Eenmalig (te annuleren indien expert stabiliteit kan bevestigen)		-	Uit te voeren na de afronding van de constructiewerken Oosterweel en wanneer PP13 en PP12 in gebruik zijn.
Grondhoop	-	Vijfjaarlijks	Visuele inspectie van de grondhoop	-	Vanaf 2013, één controle om de vijf jaar sinds de vervanging van de afdekking in 2012 en de controle in 2013, 2016 en 2018; Volgende controle: 2023.
FC Bronzones					
Gebouw 16	1	Halfjaarlijks	K3	De monitoringsfrequentie van K3 wordt verhoogd van jaarlijks naar halfjaarlijks om de contour van de kernzone (>10.000 µg/L) beter op te volgen.	-
	4	Halfjaarlijks	P27, P42, P304 en P305	-	P56 en P300 zijn in oktober 2018 buiten gebruik gesteld in verband met de bouwwerkzaamheden aan gebouw 036. Om de contour volledig op te volgen dienen P56 en P300 vervangen te worden.

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
	1	Halfjaarlijks	P21B	Peilbuis P21B bevindt zich in kern van bronzone waardoor de concentraties hoog blijven. Monitoringsfrequentie wordt verlaagd van driemaandelijks naar halfjaarlijks.	
WWTP	5	Jaarlijks	P118C, P264, P340, P341 en P379	Omwille van stabiele concentratie verlopen ter hoogte van P340, P379, P118C en P341 worden deze peilbuizen in 2021 jaarlijks in plaats van halfjaarlijks bemonsterd.	P381 en P265B zullen niet meer bemonsterd worden omdat de concentraties continu en voldoende laag waren. P382 ligt vlak naast peilbuis P381 en peilbuis P265B naast P118C. Filterstellingen zijn gelijkaardig en P382 en P118C zullen wel in monitoringsprogramma blijven.
	8	Halfjaarlijks	L19, P262, P263, M4 ,P371, P374, P380 en P119C	-	P343 is in april 2019 beschadigd geweest en kan niet meer gebruikt worden. Moet herplaatst worden zodra werken in deze omgeving zijn afgerond.
	1	Halfjaarlijks	P382	De monitoringsfrequentie wordt verhoogd van jaarlijks naar halfjaarlijks om de effecten van de Oosterweel werken en PP13 op te volgen.	-
Tweede aquifer	1	Jaarlijks	D2	-	-
	10	Halfjaarlijks	BD24-3, BD24-4, D09bis, D10, D11, D16, D5, ND7, P118A en P118B	-	D16 is in augustus 2020 beschadigd geweest, en moet vervangen worden in 2021.

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
	6	Jaarlijks	D17, D18, D14, P121, P119A en P119B	Monitoringsfrequentie wordt verlaagd van halfjaarlijks naar jaarlijks omdat er continue lage en stabiele concentraties zijn waargenomen.	-
	1	Halfjaarlijks	P321	Monitoringsfrequentie aangepast van driemaandelijks naar halfjaarlijks omwille van de stabiliserende concentratietrend.	-
Zuidelijke perceelsgrens	1	Halfjaarlijks	P372		Peilbuizen B3-bis, B7, P378, PA109A, PA111A, PA112 zijn in januari 2019 buiten gebruik gesteld in het kader van de Oosterweel werken.

Ondergrondse tanken

TPH	1	Jaarlijks	P18	TPH-monitoring op verzoek van de OVAM	Brononderzoek lopende. P28 is beschadigd en moet vervangen worden in 2021.
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Blokkersdijk Natuurgebied

Peilbuizen	7	Halfjaarlijks	L21, L22, L31, L4, P114bis, P116	De monitoringsfrequentie wordt verlaagd van driemaandelijks naar halfjaarlijks omwille van algemeen constante concentratie verlopen en voldoende beschikbare data om de trend op te volgen.	De stalen worden geanalyseerd door het 3M Environmental Laboratorium in de Verenigde Staten om de statistische evaluatie hier op uit te kunnen voeren.
Oppervlaktewater	3	Driemaandelijks	P115, Blokkersdijk standaard, 3M pond, Blokkersdijk Noord	-	-

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
Palingbeek en Tophatgracht	4	Maandelijks	12, 13, 5, Bemalingsstation	-	-
Regenwater	1	Maandelijks	Collector put	Driemaandelijkse analyse zullen maandelijks uitgevoerd worden door het SGS lab.	Wekelijkse monsters geanalyseerd door het 3M intern laboratorium.
Effluent WWTP	1	Maandelijkse	Effluent WWTP	Driemaandelijkse analyse zullen maandelijks uitgevoerd worden door het SGS lab.	Wekelijkse monsters geanalyseerd door het 3M intern laboratorium.

Legende	
Verhoogde monitoringsfrequentie	
Verlaagde monitoringsfrequentie	
Geen wijzigingen	

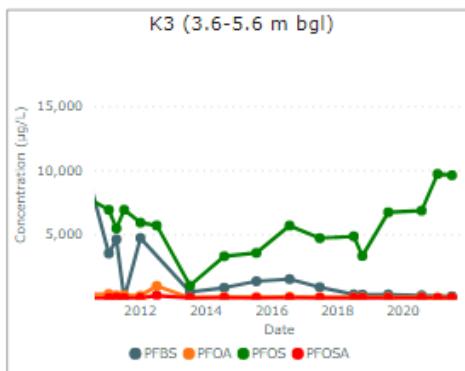
6. EERSTE INTERPRETATIE MEER RECENT BEKOMEN DATA (AUGUSTUS 2020-JULI 2021)

Voorliggend tussentijds verslag behandelt, in lijn met voorgaande tussentijds verslag, een periode van 3 jaar en meerbepaald de periode van augustus 2017 tot juli 2020. OVAM heeft ERM echter kort voor de uiterste datum voor het indienen van TTV 9 gevraagd om ook de meer recente resultaten welke ondertussen beschikbaar zijn mee op te nemen. Het is echter niet mogelijk in de beschikbare tijd een voldoende grondige evaluatie en volledige rapportage van deze data te uit te voeren, waardoor er geopteerd is om het volgende tussentijds verslag (TTV10) versneld in te dienen (januari 2022). De resultaten van de monitoringsrondes van oktober 2020, januari, april en juli 2021 zijn in afwachting hiervan hieronder in algemene zin besproken. Merk op dat een volledige en grondige evaluatie zal uitgevoerd worden in aanloop van het indienen van TTV10, wat er toe kan leiden dat onderstaande evaluatie dient aangepast te worden. Deze bespreking kan als gevolg niet beschouwd worden als onderdeel van de decretale rapportage van de lopende sanering.

6.1 Freatische aquifer

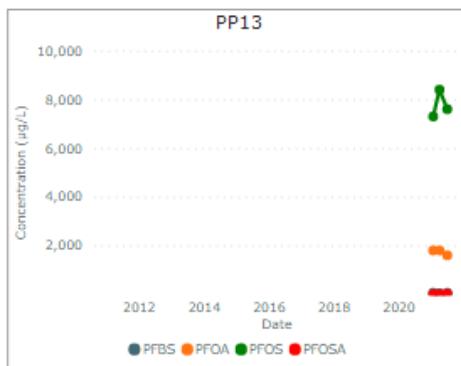
6.1.1 Gebouw 16

- De gemeten FC concentraties fluctueren, maar over het algemeen is een stabiele tot dalende trend aangetoond; en
- Ter hoogte van peilbuis K3 is een lichte stijging voor PFOS aangetoond. Deze peilbuis is gelegen nabij voormalige pompput PP01; er dient nauw opgevolgd te worden of in deze richting voldoende captatie is met de aanwezige pomppetten. Een peilbuis in meer noordoostelijke richting (P219 indien aanwezig) dient meegenomen te worden in het monitoringsprogramma. De monitoringsfrequentie van deze peilbuizen zal alvast tijdelijk verhoogd worden tot 4 maal per jaar.

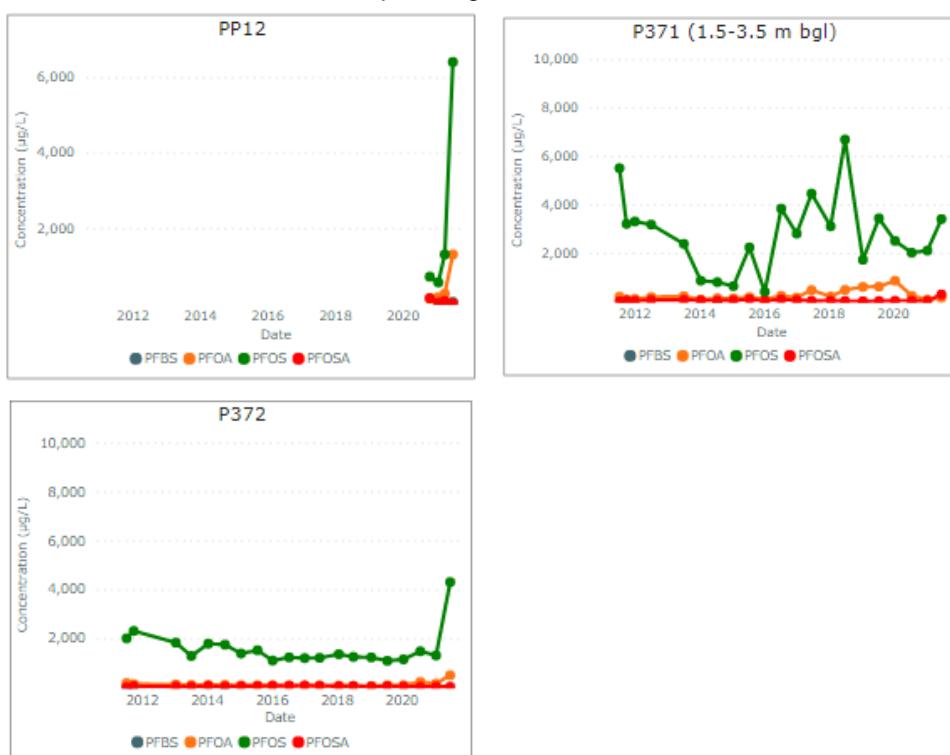


6.1.2 WWTP

- De gemeten FC concentraties fluctueren, maar over het algemeen is een stabiele tot dalende trend aangetoond;
- De gemeten FC concentraties in de nieuw geplaatste pompput PP13 zijn van dezelfde grootte-orde als voordien; concentraties in peilbuizen nabij deze pompput zijn eveneens stabiel tot dalend (P380, P381 en P382);



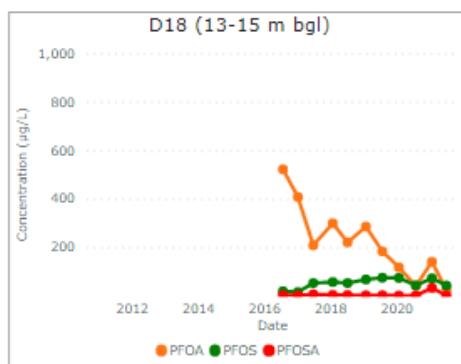
- De gemeten PFOS concentratie in nieuwe pompput PP12 is gestegen tot ca. 6400 µg/l; dit ligt in dezelfde grootte-orde als concentraties die reeds gemeten werden in de nabij gelegen peilbus P371. Ter hoogte van peilbus P371 fluctueert de PFOS concentratie, maar vertoont over het algemeen een stabiele trend.
 Ter hoogte van peilbus P372 (andere kant ondergrondse dijk nabij PP12) is wel een stijging voor PFOS aangetoond, van 1500 naar 4300 µg/l. Het betreft tot heden een eenmalig hogere concentratie. De PFOS concentratie ter hoogte van peilbus P372 dient verder opgevolgd te worden; een mogelijke invloed van de nieuwe pompput PP12 dient hierbij mee in beschouwing genomen te worden. Peilbus P372 zal bijgevolg eveneens bij de monitoring van oktober 2021 bemonsterd worden om dit verder op te volgen.



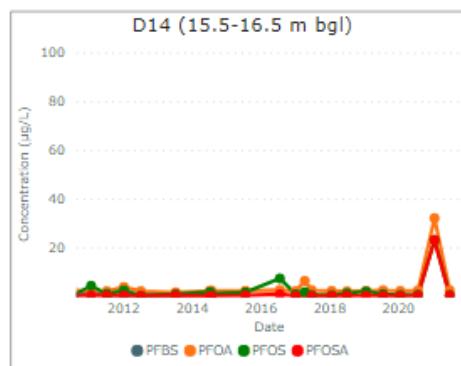
6.2 2^e aquifer

6.2.1 Gebouw 16

- De gemeten FC concentraties fluctueren, maar over het algemeen is een stabiele trend aangetoond. Ter hoogte van peilbuis D18 is een dalende trend voor PFOA aangetoond.

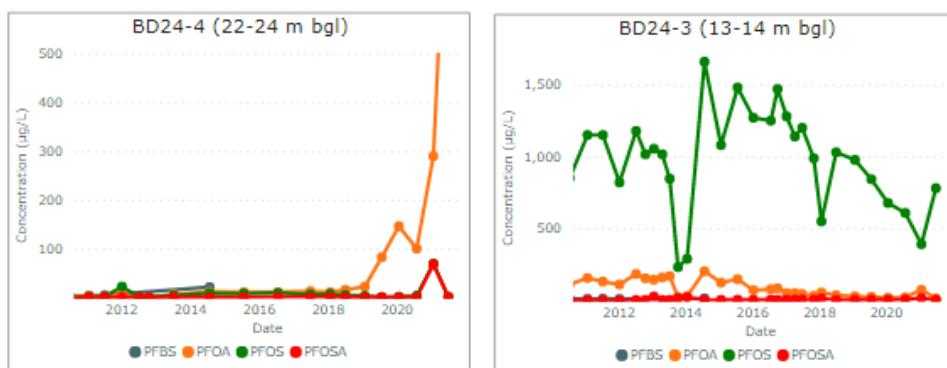


- De hogere concentraties gemeten ter hoogte van D14 (grens Mexico Natie) in januari 2021 zijn niet bevestigd tijdens de staalname van juli 2021.



6.2.2 WWTP

- De gemeten FC concentraties fluctueren, maar over het algemeen is een relatief stabiele tot dalende trend in enkele peilbuizen (o.a. P118a, P118b, D10, P321) aangetoond.
- Ter hoogte van peilbuis BD24-4 is met de staalname van juli 2021 de stijgende trend voor PFOA bevestigd (van 21 naar 930 µg/l). Peilbuis BD24-4 is aan de terreingrens, richting Blokkersdijk gelegen, met een filterdiepte van 22-24 m-mv; de concentratiestijging dient nauw opgevolgd te worden. De monitoringsfrequentie van deze peilbuis zal naar 4 maal per jaar verhoogd worden. Indien deze stijging verder bevestigd wordt, dient het verder in kaart brengen van de verontreiniging in deze zone geëvalueerd te worden. In de middeldiepe peilbuis BD24-3 op deze locatie, met filterstelling va 13-14 m-mv, is een andere ‘fingerprint’ aan FC parameters aanwezig.

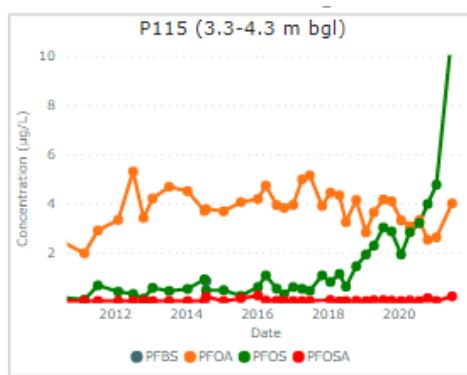


- De PFOS concentratie ter hoogte van peilbuis D11, in de zuidelijke hoek van de WWTP zone fluctueert, met mogelijk een licht stijgende trend. Ook voor deze peilbuis zal de monitoringsfrequentie tijdelijk verhoogd worden naar 4 maal per jaar, gezien pompput PP09 nabij deze locatie buiten gebruik gesteld is. Ook ter hoogte van peilbuis D16 is tijdens de monitoring van juli 2021 een hogere concentratie gemeten dan voordien, weliswaar eenmalig. Dit dient verder opgevolgd te worden; bij een bevestiging van deze verhoogde concentratie zal geëvalueerd worden of bijkomende acties nodig zijn.

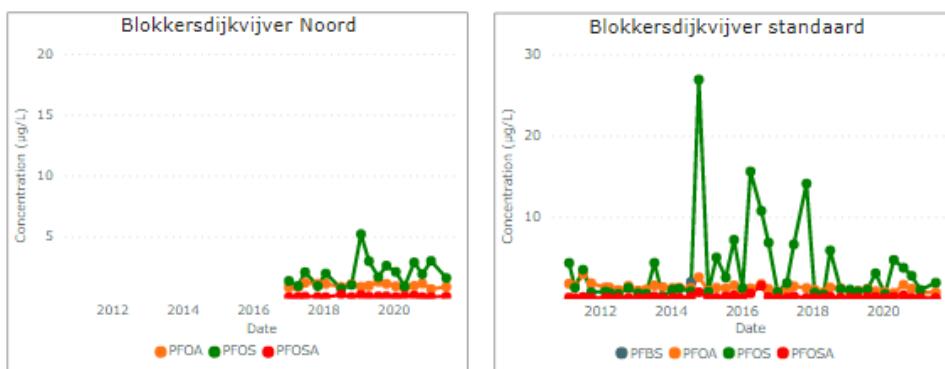


6.3 Blokkersdijk

- Ter hoogte van Blokkersdijk is nog geen nieuwe statistische analyse uitgevoerd met de resultaten tem juli 2021.
- In het algemeen liggen de recent gemeten concentraties in lijn met de voorgaande resultaten, met uitzondering van de PFOS concentratie ter hoogte van peilbuis P115; de eerder aangetoonde statistisch stijgende concentratietrend wordt ook bevestigd met de meer recente resultaten; een PFOS concentratie van 11 $\mu\text{g/l}$ is gemeten in juli 2021.
 De PFOS concentratie ter hoogte van de andere peilbuizen in het 3M pad ligt rond of lager dan 5 $\mu\text{g/l}$ tijdens de laatste monitoringsronden, hoewel deze peilbuizen in het verleden soms ook enkele uitschieters (max. ca. 40 $\mu\text{g/l}$) kenden wat betreft de concentratie aan PFOS.
- Mogelijk is dit gerelateerd aan de oude riolering die ter hoogte van het 3M pad gelegen is, hoewel dit niet eenduidig vast te stellen is. 3M zal van start gaan met het uitwerken van een plan van aanpak om deze oude riolering te verwijderen. De evolutie van de concentratie dient naar aanleiding hiervan opgevolgd te worden.
 Als de concentratiestijging verder bevestigd wordt, dient het verder in kaart brengen van de verontreiniging in deze zone geëvalueerd te worden. De peilbuis wordt reeds 4 maal per jaar bemonsterd, zoals eveneens Blokkersdijkvijver. Dit dient behouden te blijven.



- De gemeten PFAS concentraties in de 3M vijver en de Blokkersdijk vijver liggen lager dan 5 µg/l; de concentraties liggen in lijn met voorgaande metingen.



6.4 Palingbeek en Tophatgracht

De meer recente analyseresultaten geven geen opmerkelijke afwijkende concentratietrends weer. Absoluut gezien zijn relatief lage concentraties tijdens de laatste monitoringsperioden gemeten in vergelijking met vorige metingen.

6.5 Vuilvrachtberekening

De totale vuilvracht naar de Schelde is, rekening houdend met de meest recente resultaten, nog niet berekend. Een verdere evaluatie hiervan zal in het volgend tussentijds rapport opgenomen worden.

6.6 Grondwater- en stoftransportmodel

Voor de 3M site was reeds een grondwatermodel beschikbaar. Momenteel wordt dit grondwatermodel geupdated en uitgebreid naar een stoftransportmodel. Dit zal in de nabije toekomst een belangrijke tool zijn voor het beheer van de verontreiniging on site, maar ook voor de simulatie van externe factoren op de aanwezige verontreiniging en een verdere optimalisatie van de saneringsaanpak.

7. SCHADEGEVALLEN

In de periode van deze tussentijdse rapportage hebben twee schadegevallen plaatsgevonden.

7.1 Lek IBC-container ter hoogte van gebouw 2

In augustus 2017 is op het 3M terrein ten zuidoosten van gebouw 2 een lek vastgesteld in een IBC. Dit schadegeval is reeds gerapporteerd in het periodiek oriénterend bodemonderzoek van 2017 dat bij OVAM is ingediend en zal hier niet opnieuw besproken worden.

7.2 Hydraulische olie ter hoogte van constructiewerken project CS17

Op 10 december 2018 is op het 3M terrein ten noordoosten van gebouw 16, ter hoogte van de constructiewerken van het nieuwe gebouw 036 (project CS17), een lek vastgesteld in een drukleiding met hydraulische olie van een graafmachine. Hierdoor is ongeveer 10 liter hydraulische olie op een onverharde ondergrond terecht gekomen. Het incident is op 14 december 2018 gemeld aan de OVAM en de milieu-inspectie (melding aan OVAM dd. 14-12-2018; OVAM reactie dd. 20-12-2018 met referentie BB-LB2-KB-20180725215).

Figuur 7.1 Locatie schadegeval hydraulische olie



Het vrijgekomen product is deels opgevangen in een lekbak en deels afgegraven en opgeslagen in een cubitainer ($1m^3$) na het strooien van absorberende korrels. Na de herstelling van de leiding op 11 december 2018, kon de machine verplaatst worden. Op 12 december 2018 is de mogelijk geïmpacteerde grond verder afgegraven ($3 \times 3 m$) tot een diepte van 20 cm.

Er zijn stalen genomen na de ontgraving, van de bodem van de ontgravingsput, monsters van de wanden en een aantal preventieve ondiepe stalen van de bodem 1 meter buiten het ontgravingsvak. De boorwerkzaamheden en bemonsteringen zijn uitgevoerd door ERM conform de meest recente CMA-procedures. De stalen zijn genomen met gebruik van een edelmanboor. De samenstelling van het opgeboorde materiaal betreft schelpenhoudend, grof, lichtbruin zand. De stalen zijn geanalyseerd op droge stof en minerale olie door het erkend laboratorium Al-West B.V.

In de bodemstalen is geen concentratie aan minerale olie gemeten hoger dan de richtwaarde. In de diepe controlesmonsters onder de bodem van de ontgravingsput benaderde de gemeten concentratie minerale olie de streefwaarde (50 mg/kg ds). Gelet op het oppervlakkige karakter van de ontgraving

en de gunstige resultaten in het diepe controlemuster is de uitvoering van grondwateronderzoek niet noodzakelijk geacht. Aangezien in de controleslagen van de ontgravingssput geen overschrijdingen van de richtwaarde zijn gemeten, kan besloten worden dat de doelstelling van de schadebeperkende maatregelen gehaald is. Bijgevolg is geoordeeld dat er geen verdere maatregelen of een beschrijvend bodemonderzoek nodig zijn.

OVAM is akkoord gegaan met de conclusie van het rapport. Het evaluatierapport en het akkoord van OVAM zijn opgenomen in Bijlage 10.

8. TWEEDE FASE BODEMSANERINGSPROJECT

Na afronding van de Oosterweelwerken op Linkeroever is voorzien om het beschrijvend bodemonderzoek met focus op grondwater te actualiseren, waarin de nieuwe omgevingssituatie weerspiegeld zal worden. Deze actualisatie zal de basis vormen voor het uitwerken van een tweede gefaseerde saneringsaanpak voor de grondwaterverontreiniging.

In afwachting van een bodemsaneringsproject wordt de organofluorimpact ter hoogte van de zuidelijke perceelsgrens en de Palingbeek opgevolgd door middel van een periodieke monitoring. Vanzodra de activiteiten in deze zone het toelaten, zal in deze zone eveneens het monitoringsnetwerk voor de opvolging van de FC concentraties in het grondwater terug uitgebreid worden.

9. SAMENVATTING

In opdracht van 3M Belgium bvba (3M) voert Environmental Resources Management - ERM nv (ERM) als erkend bodemsaneringsdeskundige de milieukundige begeleiding uit van de saneringswerken op het terrein van 3M gelegen aan de Canadastraat 11 te Zwijndrecht.

Voorliggend verslag betreft het 9^{de} tussentijds verslag (TTV9) van de saneringswerken, zoals beschreven in het door de OVAM conform verklaarde bodemsaneringsproject (Eerste gefaseerd BSP, Arcadis, Ref. 11/003460, oktober 2008) en de acht voorgaande tussentijdse verslagen. Het betreft de saneringswerken op het deel productiezone, voormalige slibbekkens/waterzuiveringsinstallatie, natuurgebied Blokkersdijk en de 2de aquifer. De besproken saneringsperiode loopt van augustus 2017 tot en met juli 2020. Het doel van het eerste gefaseerd bodemsaneringsproject is om de vastgestelde grond- en grondwaterverontreinigingen te monitoren, te beheersen en, in zoverre ook technisch/financieel mogelijk, te reduceren en hierbij de verspreiding van de verontreiniging buiten de terreingrenzen te verminderen.

De volgende werken hebben plaatsgevonden gedurende de periode van augustus 2017 tot en met juli 2020:

- Opvolging en onderhoud van het grondwatersaneringssysteem in twee bronzones (gebouw 16 en WWTP zone). Het systeem omvat momenteel acht onttrekkingsputten (PP02-04-05-06-07-08-10-11), maar zal vanaf augustus 2020 nog twee bijkomende extractieputten (PP12 en PP13) omvatten ter vervanging van PP09. Het voornaamste doel van de sanering is om de horizontale en verticale verspreiding van de verontreiniging ter hoogte van de bronzones van gebouw 16 en de WWTP tegen te gaan. Er worden driemaandelijks stalen genomen van de pompputten om de efficiëntie van het saneringssysteem op te volgen;
- Periodieke grondwatermonitoring ter hoogte van de twee kernzones (gebouw 16 en WWTP), de 2^e aquifer, de zuidelijke perceelsgrens, het tankenpark (peilbuizen P18 en P28) en het natuurgebied Blokkersdijk; de periodieke monitoring van de kwaliteit van het oppervlaktewater van de Palingbeek, Tophatgracht, 3M vijver en Blokkersdijkvijver; periodieke monitoring van de collectorput en het effluent van de waterzuivering;
- Het berekenen van gemiddelde PFOS-vuilvracht die in de Schelde geloosd wordt via de regenwaterriolering, de bedrijfsafvalwaterzuivering en de grachten (Palingbeek en Tophatgracht);
- Het vergelijken van de analyseresultaten van de waterstalen geanalyseerd door het 3M Environmental Laboratorium en de dupliaatstalen geanalyseerd door SGS;
- Opvolging van de vooruitgang van de Oosterweelwerken, aangezien deze werken in belangrijke mate de timing en uitwerking van het 2^e gefaseerd bodemsaneringsproject bepalen;
- Herevaluatie statistische analyse op basis van meest recente analyseresultaten;
- Driemaandelijkse opmeting van de grondwaterstanden om de invloed van de seizonale fluctuaties en de pompputten op te volgen;
- Visuele check van de grondhopen verontreinigd met kwik;
- Begeleiden van de mitigerende maatregelen genomen naar aanleiding van het incident van 10 december 2018 waarbij ten noordoosten van gebouw 16, ter hoogte van de constructiewerken van het nieuwe gebouw 036 (project CS17), een lek vastgesteld werd in een drukleiding met hydraulische olie van een graafmachine.

Uit de saneringswerken en grondwaterstaalnames uitgevoerd in de voorbije periode (augustus 2017-juli 2020) kunnen volgende conclusies getrokken worden en worden volgende aanbevelingen gedaan:

- Tussen augustus 2017 en juli 2020 is ongeveer 31.100 m³ verontreinigd grondwater onttrokken. In desbetreffende periode is ongeveer 143 kg FC's verwijderd. In totaal is sinds juli 2011 circa 656 kg FC (berekend voor PFOS, PFOA en PFHS) verwijderd;

- Over het algemeen zijn de concentraties in de peilbuizen en pompputten stabiel wat erop duidt dat de verontreiniging globaal gezien gecapteerd wordt door het P&T systeem;
- Algemeen heeft het P&T systeem gedurende de periode augustus 2017 tot juli 2020 een relatief constante uptime gekend;
- De 10.000 µg/L contour voor de som van FC-concentraties in de eerste aquifer is in omvang afgenoem sinds de start van de sanering;
- Aan de saneringsdoelstelling voor de 2^e aquifer is eveneens voldaan: de gemeten concentraties in het grondwater van de 2^e aquifer ter hoogte van de bronzones zijn lager dan het vijfvoud van de concentraties bij de opstart van de sanering (juli 2011); Ter hoogte van de terreingrenzen zijn de concentraties aan FC-verbindingen lager dan 10% van de oplosbaarheid van de FC parameters;
- Uit de statistische evaluatie volgt dat er geen significant stijgende trend voor de PFOS concentratie ter hoogte van de Blokkersdijkvijver is aangetoond;
- De statistische evaluatie toont voor de PFOS-concentratie ter hoogte van peilbuizen L22, L31, L4, P114bis en P116 een significant dalende trend aan en voor peilbuis P115 een significant stijgende trend. Gelet op de dalende trend in de overige peilbuizen en de absolute concentraties ter hoogte van peilbuis P115 wordt gesteld dat er momenteel geen noodzaak is voor bijkomende saneringsacties in de Blokkersdijkzone, of voor een hogere bemonsteringsfrequentie van het oppervlaktewater. Er wordt echter wel aangeraden om P115 driemaandelijks te blijven monitoren;
- Voor de periode augustus 2017 tot en met juli 2020 bedraagt de berekende PFOS-vuilvracht naar de Schelde gemiddeld 198 g/dag. De berekende vuilvracht overschrijdt de PNEC-waarde zoals vastgelegd in het BSP van 2008 (370 g/dag) niet. Er dient opgemerkt te worden dat deze PNEC waarde in het kader van het lopende BBO geherevalueerd zal worden, rekening houdend met de nieuwe inzichten rond FC's.
- De PFOS-concentraties in het water van de collector put zijn sinds februari 2020, na installatie van het actief koolfilter systeem, aanzienlijk gedaald: van gemiddeld 190 µg/l in de periode juli 2017-januari 2020 tot gemiddeld 65 µg/l in de periode februari – juli 2020; Een verdere verbetering van de behandelingssappen is voorzien in de komende periode;
- De gemiddelde PFOS-concentratie in het effluent van de WWTP over de rapportage periode bedraagt 19 µg/L. 3M heeft nog verscheidene projecten lopende om de FC concentraties in het effluent verder te verlagen;
- De saneringsacties en periodieke monitoring dienen verder gezet te worden zoals samengevat in Tabel 5.1. De PFOS-concentratie trend in de Blokkersdijk peilbuizen, de 3M vijver en de Blokkersdijkvijver dient eveneens verder opgevolgd te worden;
- Twee nieuwe pompputten (PP12 en PP13) zullen in augustus 2020 geïnstalleerd worden ter vervanging van PP09. Aan de hand van verschillende pomptesten zal in 2021 ook het effect van een alternatief pompsysteem op de saneringsinstallatie onderzocht worden;
- Door de Oosterweelwerken en het bouwen van het nieuw productiegebouw 036 (project CS17) op de site van 3M, zijn verschillende peilbuizen buiten gebruik gesteld. Na afloop van de werken is het aangeraden om een aantal peilbuizen opnieuw te herplaatsen om de monitoring opnieuw optimaal uit te kunnen voeren;
- Bijkomend onderzoek naar de oorzaak, de omvang en de ernst van de verhoogde concentraties aan minerale olie ter hoogte van peilbuis P18 dient uitgevoerd te worden en zal uitgewerkt worden in een beschrijvend bodemonderzoek;
- De visuele inspectie van de grondhopen verontreinigd met kwik in augustus 2018 bevestigt dat de afdekking nog in goede staat is. Een volgende visuele inspectie dient in 2023 uitgevoerd te worden;

- In afwachting van het tweede gefaseerd bodemsaneringsproject worden de FC-concentraties ter hoogte van de zuidelijke perceelsgrens opgevolgd door middel van regelmatige grondwaterstaalnames in de nog aanwezige peilbuizen. Algemeen kan gesteld worden dat de gemeten concentraties FC-verbindingen in grondwater ter hoogte van de zuidelijke perceelsgrens in dezelfde lijn liggen als de concentraties gemeten tijdens voorgaande bemonsteringen. De resultaten duiden erop dat het onttrekkingssysteem, geïnstalleerd ter hoogte van de WWTP bronzone, de migratie van de verontreiniging naar de zuidelijke perceelsgrens beperkt. Wanneer er meer duidelijkheid is over de impact van de werken in het kader van de Oosterweelverbinding op deze zone, zal een saneringsaanpak voor de FC impact uitgewerkt worden in een 2^e gefaseerd bodemsaneringsproject. Van zodra de activiteiten in deze zone het toelaten, zal in deze zone eveneens het monitoringsnetwerk voor de opvolging van de FC concentraties in het grondwater terug uitgebreid worden; en
- In de periode augustus 2017- juli 2020 hebben zich twee milieu-incidenten voorgedaan op de site van 3M. Voor beide incidenten tonen de analyseresultaten geen impact naar de bodem in deze zone, en kan besloten worden dat de doelstelling van de schadebeperkende maatregelen gehaald zijn. Bijgevolg zijn er geen verdere acties vereist.

Er zijn geen grote wijzigingen ten opzichte van wat oorspronkelijk is opgenomen en vergund in het eerste gefaseerd bodemsaneringsproject.

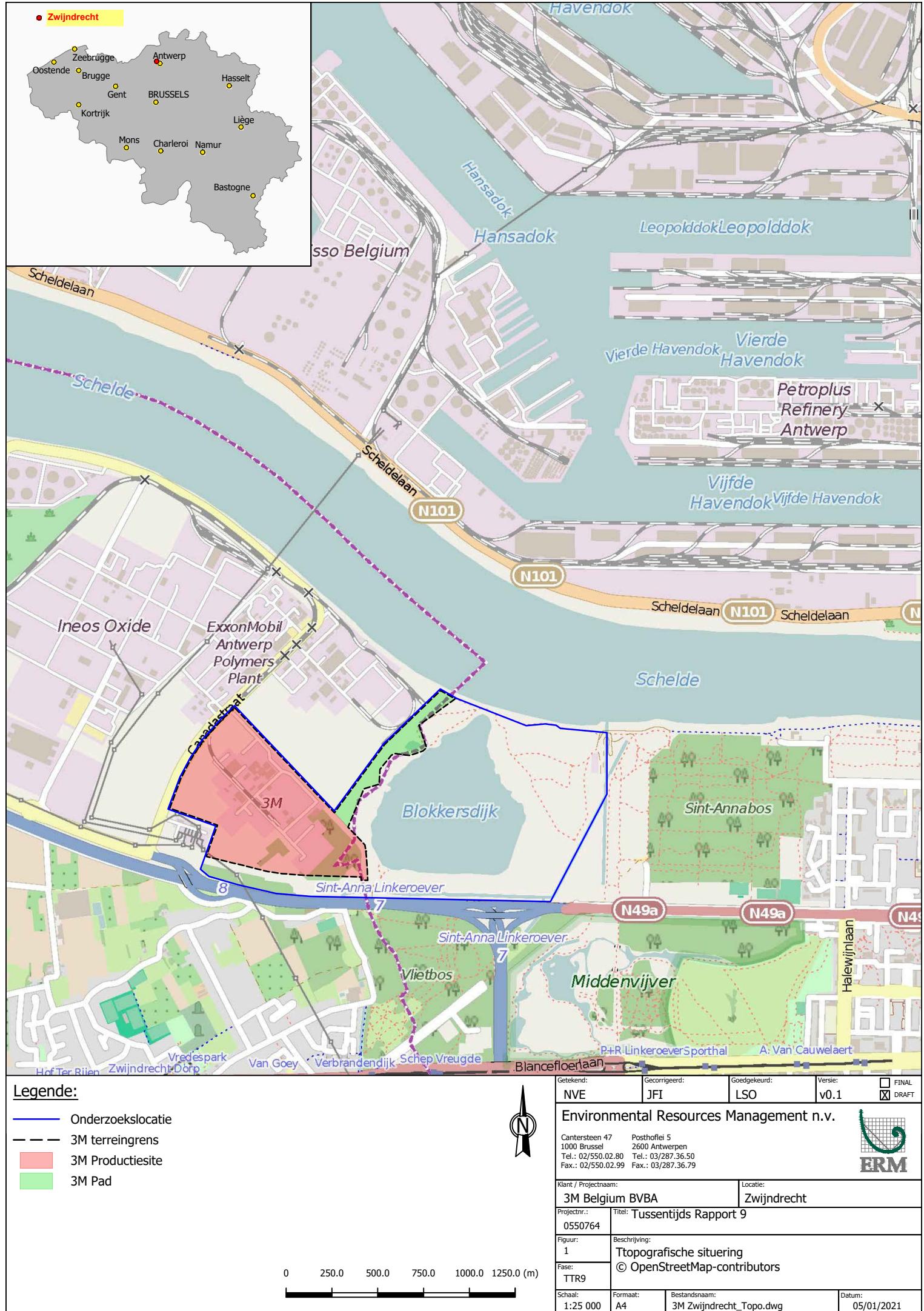
De volgende werken zullen in het kader van het bodemsaneringsproject verder gezet worden op de site van 3M:

- Voortzetten van de grondwateronttrekking in de twee bronzones en optimalisatie van het systeem via pomptesten;
- Monitoring grondwater- en oppervlaktewaterconcentraties conform frequentie vermeld in Tabel 5.1;
- Driemaandelijkse opvolging van de grondwaterpeilen;
- Berekening van de PFOS-vuilvracht die via de collector put, het effluent van de WWTP en de Palingbeek en Tophatgracht in de Schelde terechtkomt;
- Jaarlijkse statistische evaluatie van de PFOS-concentratie trend in het grondwater en oppervlaktewater van Blokkersdijk en de 3M vijver; en
- Visuele check van de grondhopen verontreinigd met kwik.

KAARTEN

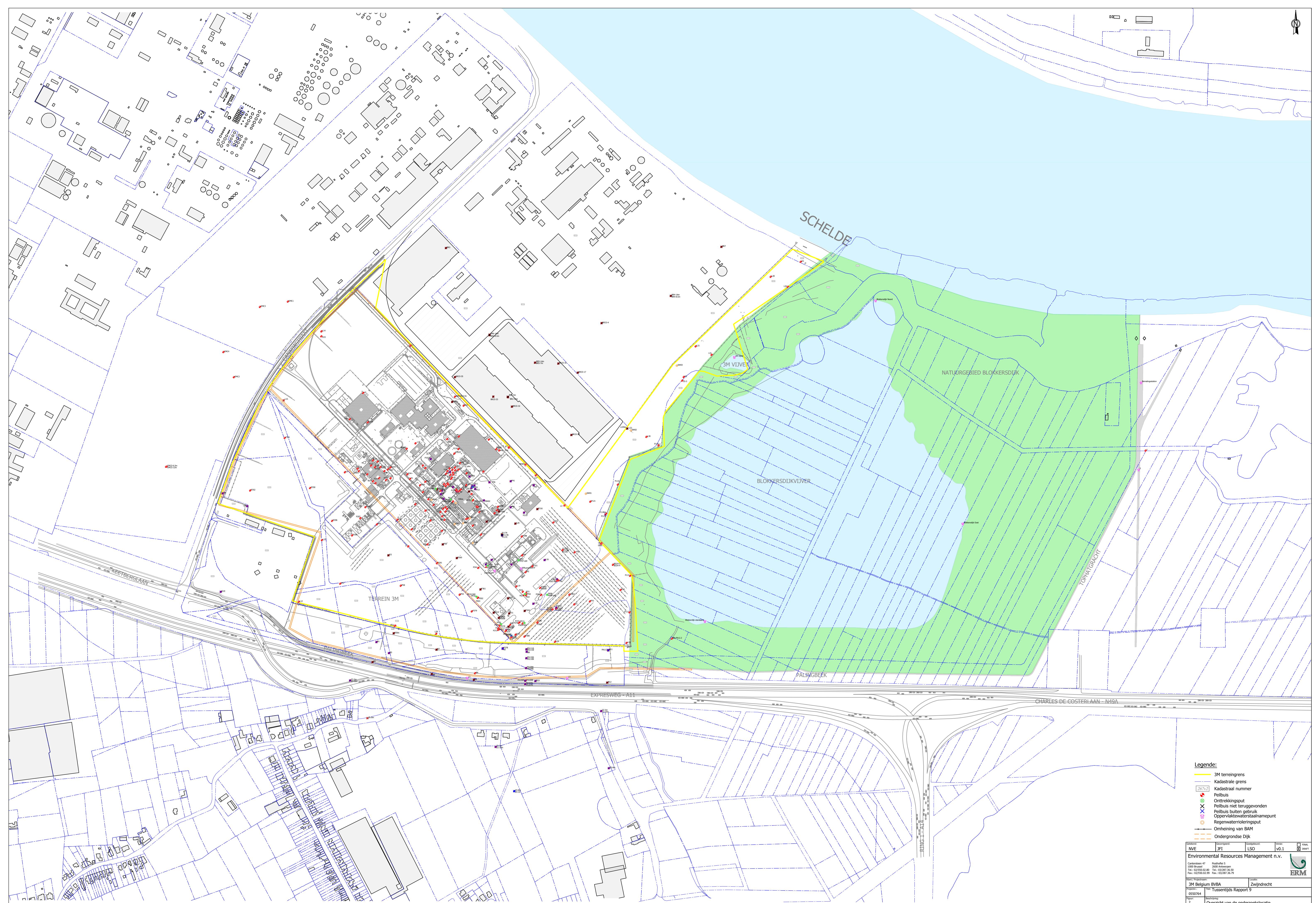
KAART 1

**SITUERING ONDERZOEKSLOCATIE OP TOPOGRAFISCHE
KAART**

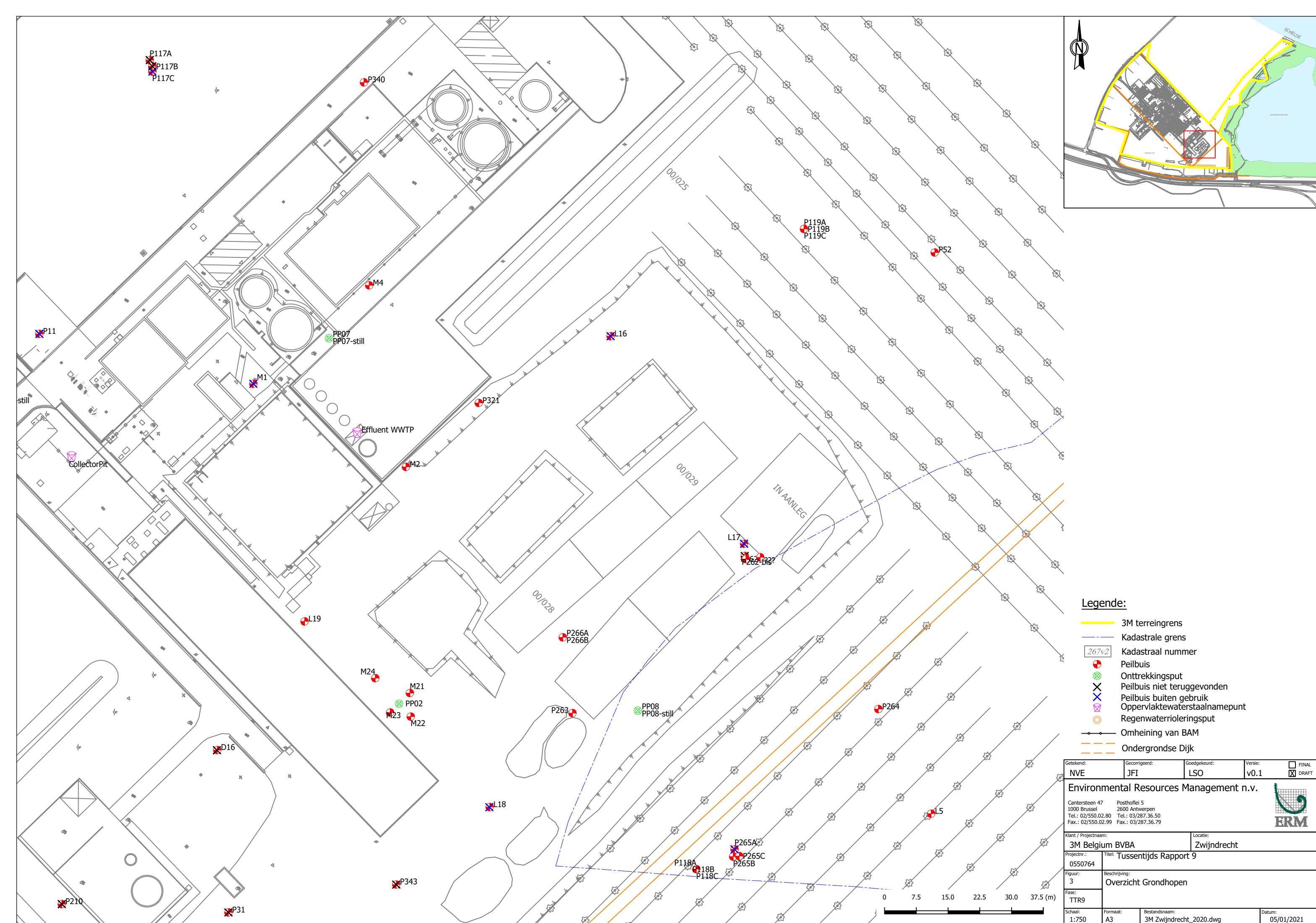


KAART 2

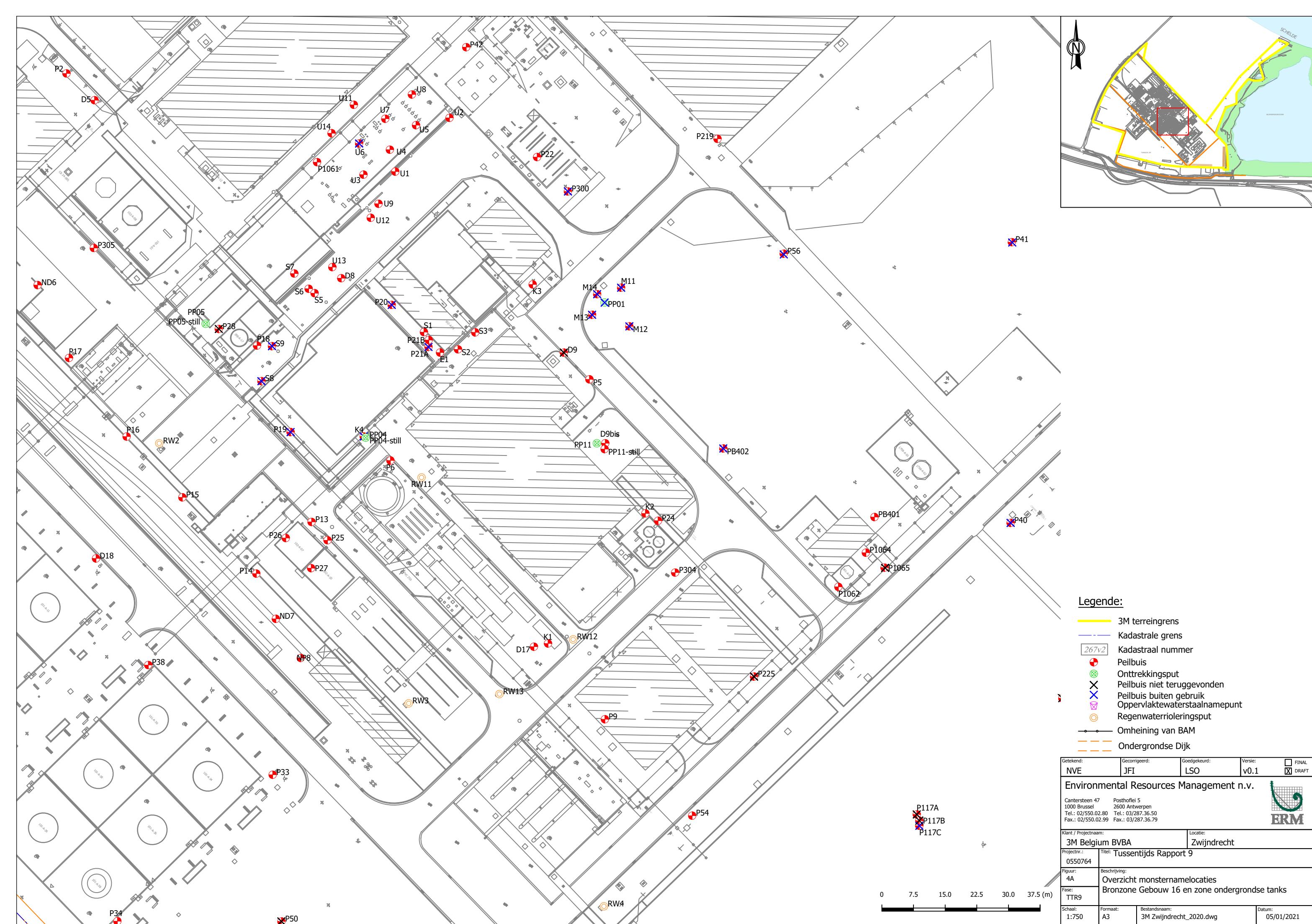
OVERZICHTSPLAN ONDERZOEKSLOCATIE

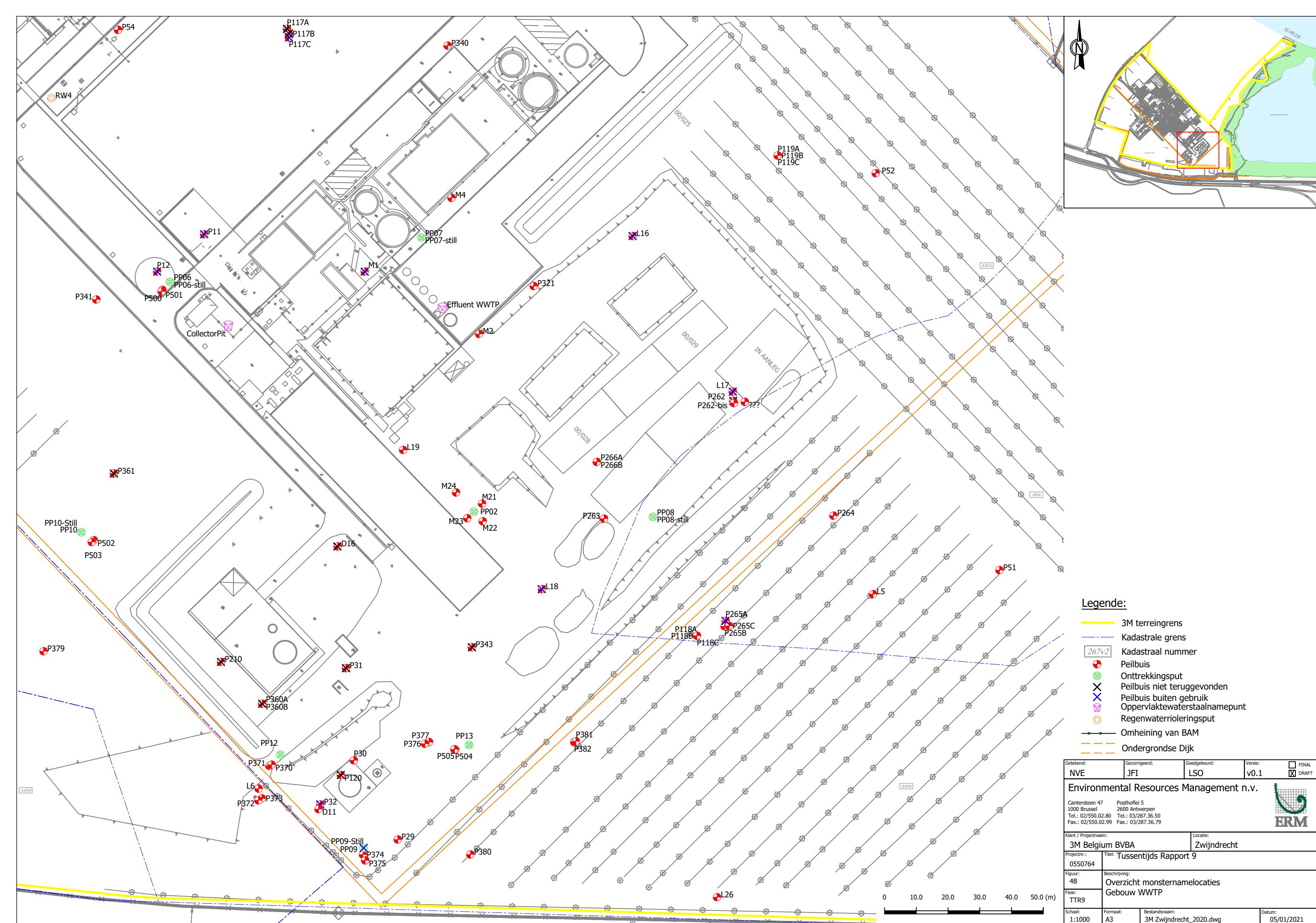


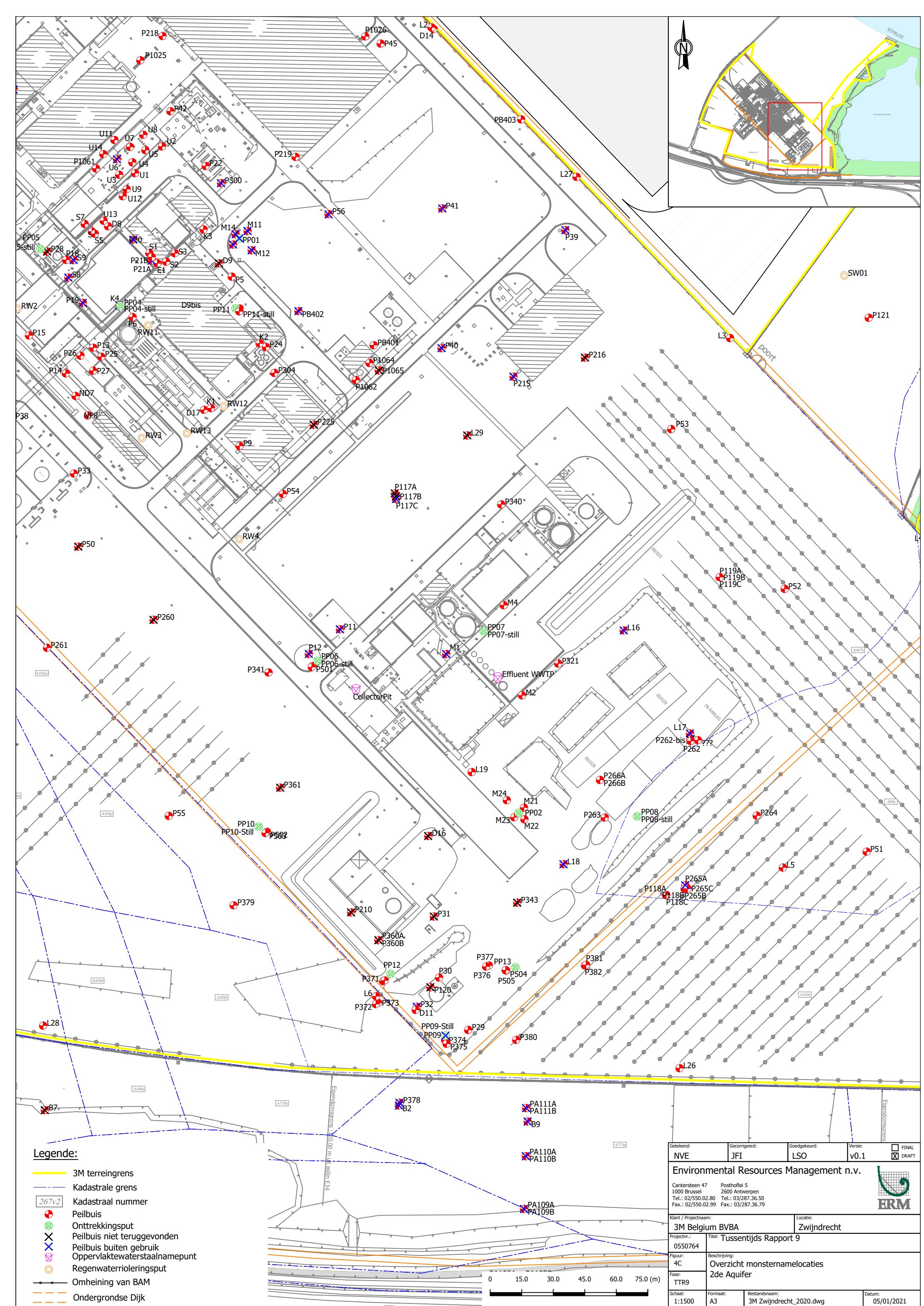
KAART 3 DETAILPLAN LIGGING GRONDHOPEN

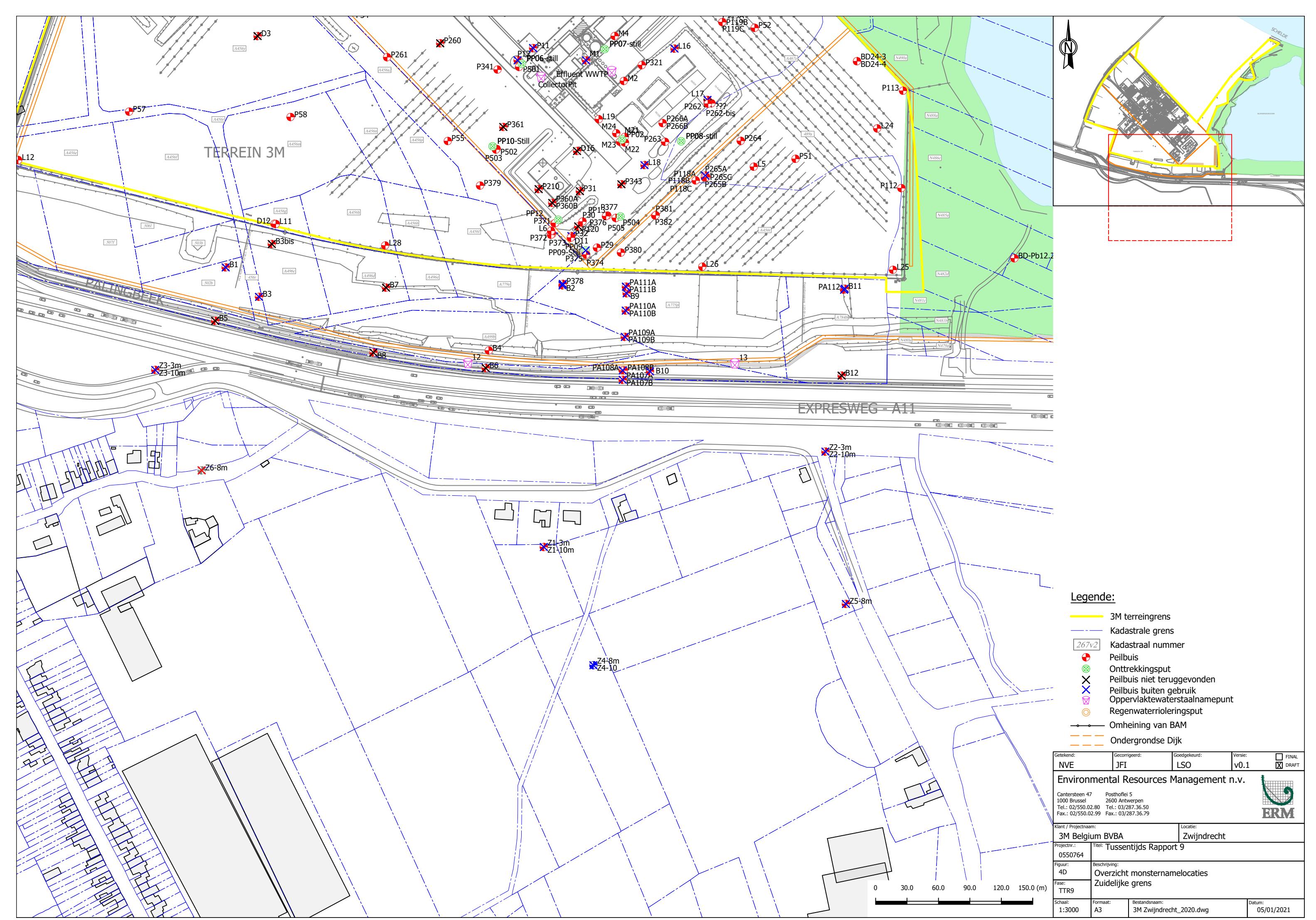


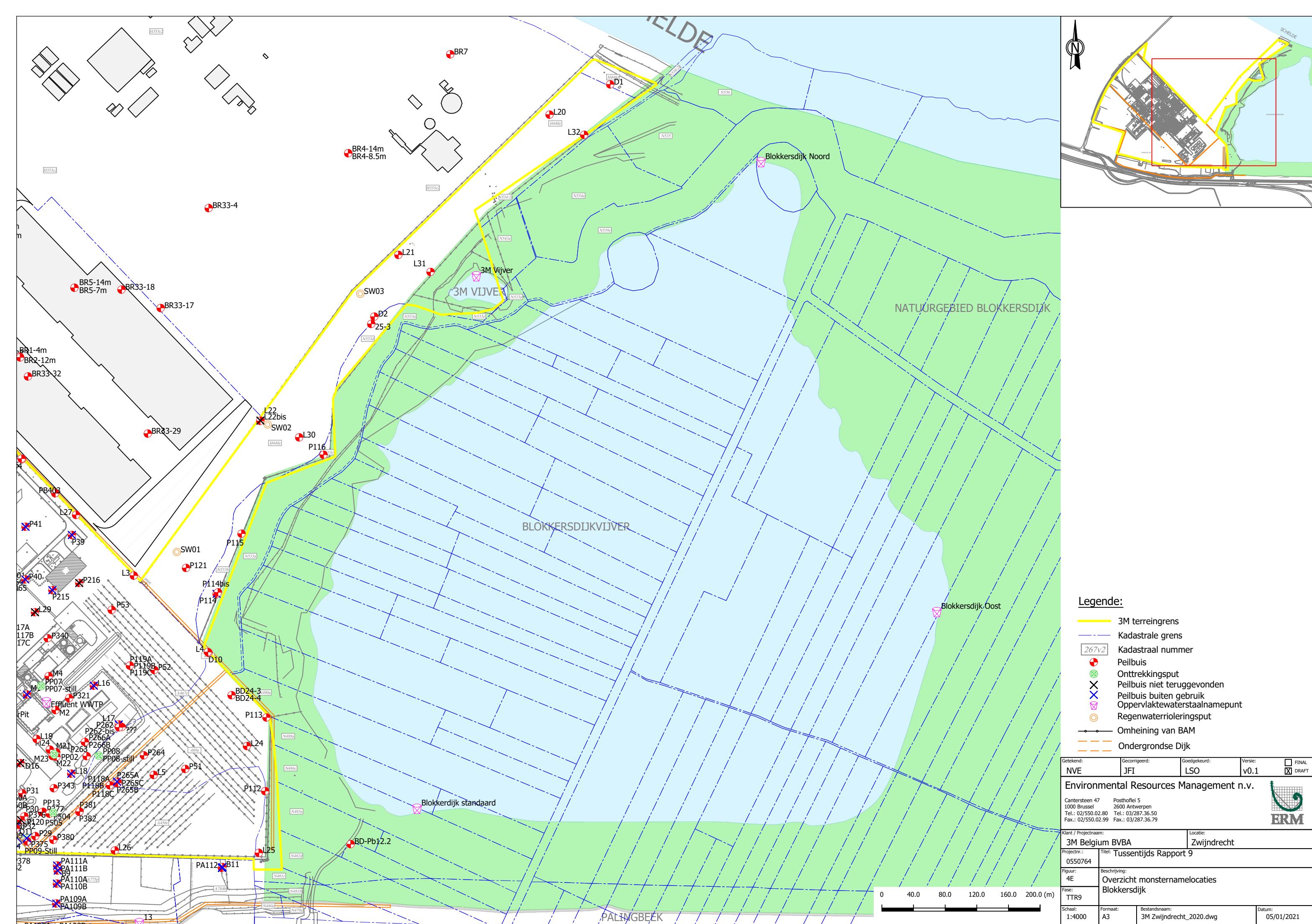
KAART 4 DETAILPLANNEN MONTERNAMELOCATIES

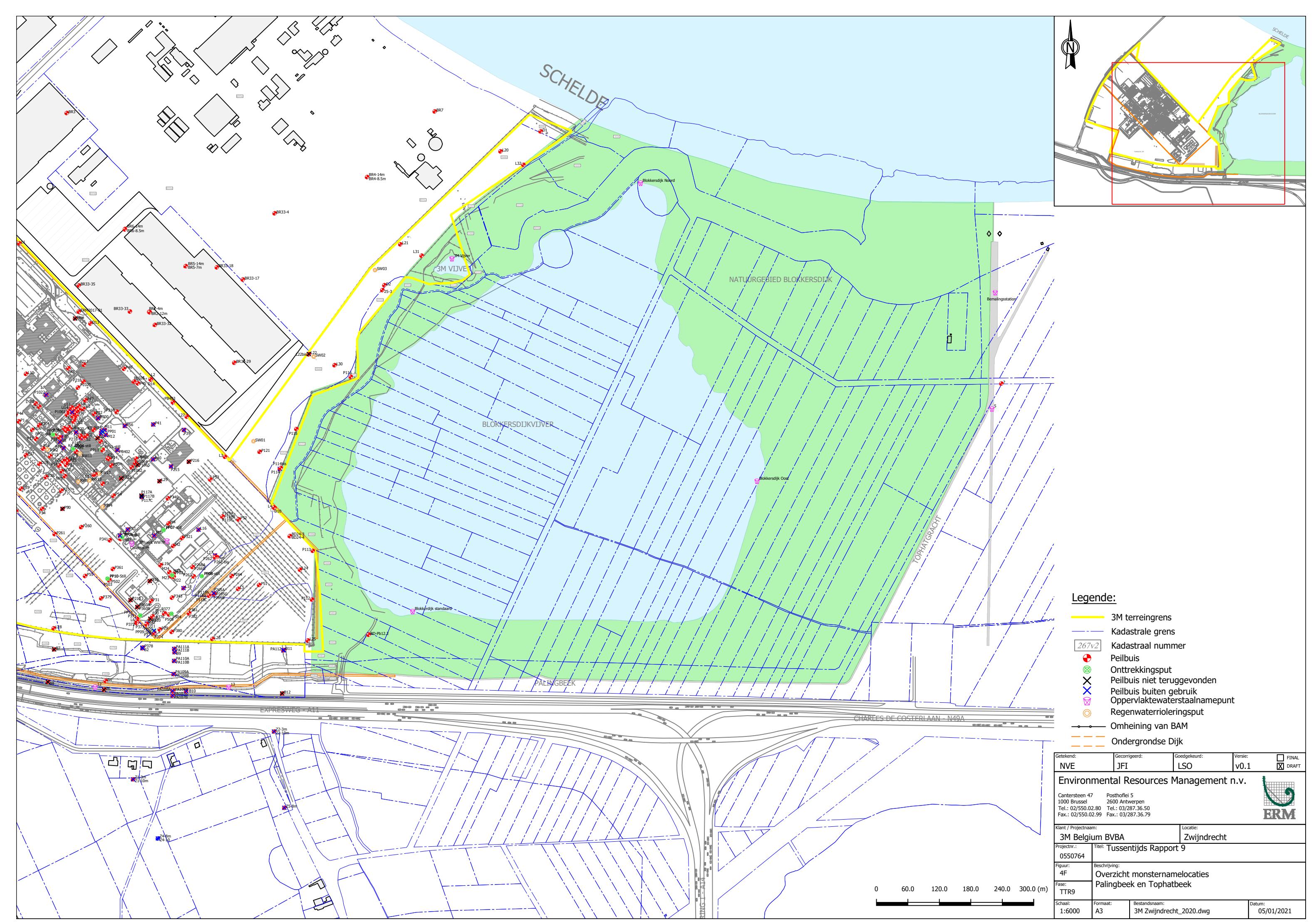


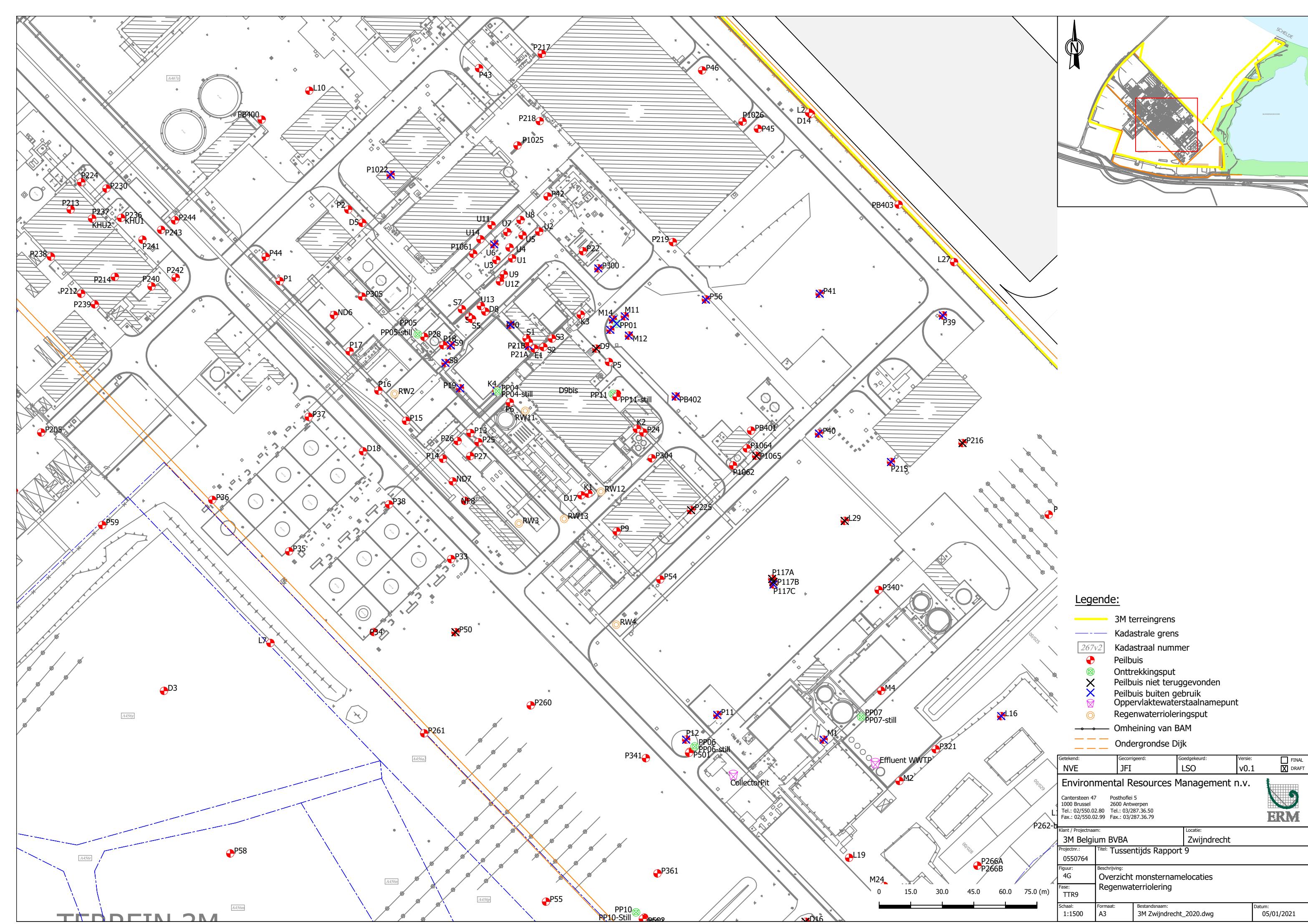




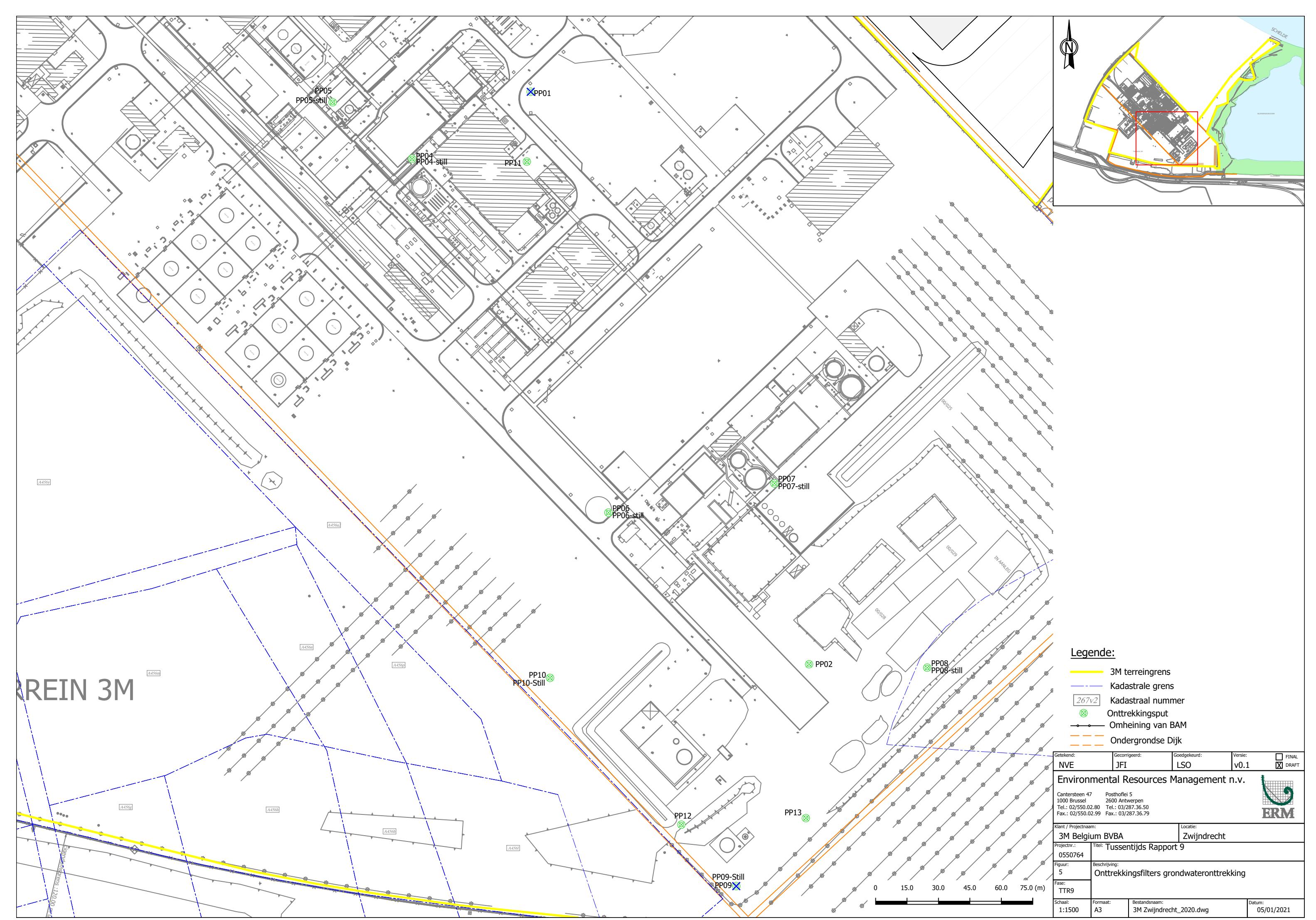








KAART 5 ONTTREKKINGSFILTERS GRONDWATERONTTREKKING



BIJLAGEN

BIJLAGE 1 OVAM COMMUNICATIE

STATIONSSTRAAT 110
2800 MECHELEN

Aangetekend met ontvangstbewijs 2009500832

TEL. 015 284 284
FAX 015 203 275

3M Belgium NV

WWW.OVAM.BE

Haven 1005, Canadalaan 11
2070 ZWIJDRECHT

Uw Bericht Van :

Afdeling : Bodembeheer

Uw Kenmerk :

Dienst : Bodemonderzoek en -sanering Oost

Bijlagen : 1 bundel

Contactpersoon : Dominique Suys [REDACTED]

Mechelen :

Ons Kenmerk : BB-O-DS/2009500832 (Dossiernummer: 732)

09 FEB. 2009

Kennisgeving van het conformiteitsattest voor het gefaseerd bodemsaneringsproject

Geachte heer,
Geachte mevrouw,

Op 12 november 2008 ontving de OVAM het rapport van het gefaseerd bodemsaneringsproject met als titel 'Bodemsaneringsproject 3M Belgium NV, Haven 1005, Canadalaan 11 te Zwijndrecht - 11/003460' dat op 29 oktober 2008 werd opgemaakt door Arcadis Belgium nv.

De OVAM verklaart het gefaseerd bodemsaneringsproject **conform** aan de bepalingen van het Bodemdecreet. Als bijlage bij deze brief vindt u het conformiteitsattest.

Overeenkomstig artikel 50, §2 van het Bodemdecreet zal de OVAM aan het college van burgemeester en schepenen het schriftelijke verzoek bezorgen om binnen de tien dagen na ontvangst van deze brief de bekendmaking uit te voeren.

Nulheffing

Overeenkomstig artikel 47 van het decreet van 2 juli 1981 betreffende de voorkoming en het beheer van afvalstoffen is het bedrag van de milieuhelling vastgesteld op 0 EUR per ton. Dit tarief van milieuhelling is geldig voor afvalstoffen afkomstig van bodemsaneringsoperaties, voor het storten op een daar toe vergunde stortplaats of het verbranden of meeverbranden in een daartoe vergunde inrichting van afvalstoffen. Het tarief kan slechts toegekend worden na advies van de OVAM en in die gevallen dat andere saneringswijzen onredelijk hoge kosten met zich mee brengen of onmogelijk zijn.

Indien dit van toepassing is voor dit bodemsaneringsproject kan een verzoek worden gericht tot toepassing van bovenvermeld bedrag aan milieuhelling. Dit verzoek met de noodzakelijke bijlagen moet worden gericht aan:

OVAM
de heer Filip De Naeyer
Diensthoofd
Stationsstraat 110
2800 MECHELEN

Het bedrag aan milieuhelling vastgesteld op 0 EUR per ton, kan alleen toegepast worden na de schriftelijke en feitelijke goedkeuring door de OVAM van bovenvermeld verzoek.

Beroep

Op grond van artikel 55 van het Bodemdecreet kunt u overeenkomstig artikel 146 tot en met 152 van het Bodemdecreet bij de Vlaamse Regering beroep aantekenen tegen dit besluit.

Bij het beroepschrift wordt, op straffe van onontvankelijkheid, een afschrift van dit besluit gevoegd.

Om ontvankelijk te zijn moet het beroep binnen een termijn van **dertig** dagen na ontvangst van deze kennisgeving, bij de Vlaamse Regering worden ingediend. U stuurt daarvoor een aangetekende brief met ontvangstbewijs naar het onderstaande adres of geeft uw brief tegen ontvangstbewijs af.

Departement Leefmilieu, Natuur en Energie
Afdeling Algemene Zaken, Communicatie en Juridische dienst
Juridische dienst
Koning Albert II-laan 20 bus 8
B-1000 BRUSSEL.

Wij vragen u vriendelijk het **dossiernummer 732** te vermelden telkens als u contact opneemt met de OVAM.

Hoogachtend,

Eddy Van Dyck
Afdelingshoofd

cc: Arcadis Belgium nv, Clara Snellingsstraat 27, 2100, Deurne - Antwerpen

Conformiteitsattest gefaseerd bodemsaneringsproject



Contactpersoon:Dominique Suys

Referentie:CBSP-nr-4177

09 FEB. 2009

1 Identificatie van het gefaseerd bodemsaneringsproject

Informatie over het gefaseerd bodemsaneringsproject:

- dossiernummer OVAM:732
- titel van het gefaseerd bodemsaneringsproject:Bodemsaneringsproject 3M Belgium NV, Haven 1005, Canadastraat 11 te Zwijndrecht - 11/003460
- opgesteld door: Arcadis Belgium nv
- in opdracht van: 3M Belgium NV
- datum van het gefaseerd bodemsaneringsproject: 29 oktober 2008
- ontvangstdatum van het gefaseerd bodemsaneringsproject: 12 november 2008
- Dit gefaseerd bodemsaneringsproject heeft betrekking op het deel productiezone en zone voormalige slibbekkens / waterzuiveringsinstallatie, het natuurreservaat Blokkersdijk en de 2de aquifer.

Informatie over de locatie:

- straat, huisnummer: HAVEN 1005 CANADA STRAAT 11
- postnummer, gemeente: 2070 ZWIJNDRECHT
- kadastrale gegevens van de betrokken gronden:

Gemeentenummer en afdeling	Sectie	Grondnummer
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 T
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0467 D
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	H	0448 C
11813 ANTWERPEN 13 AFD	N	0489 A
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0454 C
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 A 2
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 C
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 E
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 F
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 G
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 H
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 K
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 L
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 M
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 N
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 P
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 R
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 V
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 W
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 Y
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 Z

11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0496 C
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0496 D
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0496 E
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0499 B
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0502 C
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0511 A
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0769 K
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0775 P
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0775 R
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0779 E
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0782 B
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0784 B
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0785 B
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0786 A
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0786/02 G
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0786/02 H
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0789/02 K
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0789/02 L
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	Canadastraat
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	N49
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	Neerstraat
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	H	0353 Z
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	H	0448 D
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	H	0325 E
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	H	0505 D
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11813 ANTWERPEN 13 AFD	N	0481 A
11813 ANTWERPEN 13 AFD	N	0482 B
11813 ANTWERPEN 13 AFD	N	0483 A
11813 ANTWERPEN 13 AFD	N	0484
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11813 ANTWERPEN 13 AFD	N	0496
11813 ANTWERPEN 13 AFD	N	0497
11813 ANTWERPEN 13 AFD	N	0498
11813 ANTWERPEN 13 AFD	N	0499
11813 ANTWERPEN 13 AFD	N	0500
11813 ANTWERPEN 13 AFD	N	0501
11813 ANTWERPEN 13 AFD	N	0502
11813 ANTWERPEN 13 AFD	N	0503

11813 ANTWERPEN 13 AFD	N	0504
11813 ANTWERPEN 13 AFD	N	0505
11813 ANTWERPEN 13 AFD	N	0506
11813 ANTWERPEN 13 AFD	N	0507
11813 ANTWERPEN 13 AFD	N	0508
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11813 ANTWERPEN 13 AFD	N	0510
11813 ANTWERPEN 13 AFD	N	0511
11813 ANTWERPEN 13 AFD	N	0512
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11813 ANTWERPEN 13 AFD	N	0525
11813 ANTWERPEN 13 AFD	N	0526
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11813 ANTWERPEN 13 AFD	N	0533 C
11813 ANTWERPEN 13 AFD	N	0533 D
11813 ANTWERPEN 13 AFD	N	0533 E
11813 ANTWERPEN 13 AFD	N	0534 A
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11813 ANTWERPEN 13 AFD	N	0543 A
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11813 ANTWERPEN 13 AFD	N	0576
11813 ANTWERPEN 13 AFD	N	0577
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11813 ANTWERPEN 13 AFD	N	0579
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11813 ANTWERPEN 13 AFD	N	0614 A
11813 ANTWERPEN 13 AFD	N	0615
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11813 ANTWERPEN 13 AFD	N	0617 A
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11813 ANTWERPEN 13 AFD	N	0620 A
11813 ANTWERPEN 13 AFD	N	0621 A
11813 ANTWERPEN 13 AFD	N	0622
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11813 ANTWERPEN 13 AFD	N	0624 A
11813 ANTWERPEN 13 AFD	N	0625 A
11813 ANTWERPEN 13 AFD	N	0626 A
11813 ANTWERPEN 13 AFD	N	0627 B
11813 ANTWERPEN 13 AFD	N	0628 E
11813 ANTWERPEN 13 AFD	N	0629 D
11813 ANTWERPEN 13 AFD	N	0630 C
11813 ANTWERPEN 13 AFD	N	0632 A
11813 ANTWERPEN 13 AFD	N	0633 B
11813 ANTWERPEN 13 AFD	N	0634 A
11813 ANTWERPEN 13 AFD	N	0636 A
11813 ANTWERPEN 13 AFD	N	0638 A
11813 ANTWERPEN 13 AFD	N	0640 A
11813 ANTWERPEN 13 AFD	N	0641 A
11813 ANTWERPEN 13 AFD	N	0642 C
11813 ANTWERPEN 13 AFD	N	Blockerdweirweg
11813 ANTWERPEN 13 AFD	N	Rotbeek

- Op de volgende gronden vinden werkzaamheden plaats die noodzakelijk zijn om de bodemsanering uit te voeren:

Gemeente / Afdeling	Sectie	Perceelnummer	Oppervlakte
ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0467 D	32 ha 20 a 68 ca
ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	H	0448 C	5 ha 72 a 93 ca
ANTWERPEN 13 AFD	N	0489 A	2 ha 41 a 50 ca
ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 A 2	0 ha 97 a 53 ca
ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 R	1 ha 3 a 62 ca
ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0456 T	2 ha 24 a 58 ca
ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0496 C	0 ha 53 a 20 ca
ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0496 D	0 ha 47 a 67 ca
ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0775 P	1 ha 72 a 35 ca
ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0784 B	0 ha 99 a 35 ca
ANTWERPEN 13 AFD	N	0533 B	0 ha 37 a 50 ca
ANTWERPEN 13 AFD	N	0533 C	0 ha 7 a 38 ca
ANTWERPEN 13 AFD	N	0490 A	1 ha 74 a 0 ca
ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0786 H 02	0 ha 20 a 15 ca

- Andere dan te saneren percelen:

Gemeente / Afdeling	Sectie	Perceelnummer	Oppervlakte
ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0448 D	-
ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0717 C	-

ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0720	-
ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	Palingbeek	-
ANTWERPEN 13 AFD	N	Tophatgracht	-

Informatie over de geplande werkzaamheden:

- De historische grondwaterverontreiniging met organofluorverbindingen (vnl. PFOS en PFOA) op de 3M site is zowel aanwezig in de eerste als in de tweede aquifer.
- De algemene doelstelling van de sanering bestaat erin de verontreiniging met organofluorverbindingen in de bronzones te beheersen en te verwijderen, en verspreiding van organofluorverbindingen buiten de terreingrenzen te verminderen.
- Omdat de hoogste organofluorconcentraties aanwezig zijn ter hoogte van de productiezone en ter hoogte van de waterzuiveringsinstallatie (locatie van de voormalige slibbekkens) wordt ter hoogte van beide zones een grondwateronttrekkingssysteem opgebouwd.
- De waterkwaliteit van de Blokkersdijkvijver en de 3M vijver en het grondwater dat naar de vijver stroomt, zal regelmatig worden gecontroleerd. Indien een statistisch relevante stijgende PFOS concentratie wordt vastgesteld in de Blokkersdijkvijver zullen er actieve maatregelen toegepast moeten worden.
- Een deel van het verontreinigd grondwater komt in de regenwaterriolering terecht omwille van mogelijke aansluitingen van grondwaterdrains op deze riolering en lekken in de riolering. 3M zal een actief koolfiltereenheid plaatsen op de regenwaterriolering zodat minder vervuiling in de Schelde terechtkomt.
- Gezien de onzekerheden met betrekking tot de werken aan de Palingbeek in het kader van de Oosterweelverbinding, zal in een volgend gefaseerd bodemsaneringsproject de organofluorimpact op het zuidelijk 3M terreingedeelte en de Palingbeek behandeld worden.
- De historische grondwaterverontreiniging met vluchige aromaten (vnl. xylenen) ter hoogte van de ondergrondse tanks in de productiezone zal gebeuren door het zuurstofgehalte in de ondergrond te verhogen, waardoor de natuurlijke afbraak van deze stoffen zal versneld worden.
- Op het terrein is eveneens een volledig geïsoleerde grondhoop aanwezig met verhoogde kwik- en organofluorconcentraties. Omdat er geen risico's uitgaan van deze grond en deze grond ook niet verwerkt kan worden, zal deze grond op de huidige locatie blijven liggen. De afdichting zal op regelmatige basis gecontroleerd worden.

2 Uiteenzetting

Op grond van artikel 50, §1 van het Bodemdecreet spreekt de OVAM zich uit over de conformiteit van het gefaseerd bodemsaneringsproject met de bepalingen van dit decreet.

Bij de beoordeling van de conformiteit van het gefaseerd bodemsaneringsproject heeft de OVAM zich gebaseerd op het volgende:

- Het Bodemdecreet (artikel 47 tot en met 54, inzonderheid op artikel 50, §1);
- het besluit van de Vlaamse Regering van 14 december 2007 houdende vaststelling van het Vlaams reglement betreffende de bodemsanering en de bodembescherming (hierna te noemen 'Vlarebo') (artikel 77 tot en met 89, inzonderheid op artikel 87 tot en met 89);
- de standaardprocedure 'Bodemsaneringsproject';
- de codes van goede praktijk, zoals uitgewerkt door de OVAM;
- het gefaseerd bodemsaneringsproject met de titel "Bodemsaneringsproject 3M Belgium NV, Haven 1005, Canadastraat 11 te Zwijndrecht - 11/003460", opgesteld onder leiding van de bodemsaneringsdeskundige Arcadis Belgium nv in opdracht van 3M Belgium NV en aan de OVAM betekend op 12 november 2008,
- het beschrijvend bodemonderzoek met als titel Beschrijvend bodemonderzoek, 3M, Haven 1005, Canadastraat 11, 2070 Zwijndrecht + addendum dd. 25.08.2006 opgesteld door de erkende

bodemsaneringsdeskundige Arcadis Gedas nv op 30 juni 2006. Het besluit van de OVAM van 5 oktober 2006 waarbij het beschrijvend bodemonderzoek conform werd verklaard;

Ontvankelijk en vollediggefaseerd bodemsaneringsproject

De OVAM heeft het gefaseerd bodemsaneringsproject ontvankelijk en volledig bevonden.

Kennisgeving

Op 26 november 2008 werden de eigenaars en gebruikers van de gronden waarop werkzaamheden noodzakelijk zijn op de hoogte gebracht dat een ontvankelijk en volledig gefaseerd bodemsaneringsproject werd ingediend en van hun mogelijkheid om eventuele bezwaren of opmerkingen aan de OVAM mee te delen.

Advies en openbaar onderzoek

De OVAM heeft via aangetekende brief van 26 november 2008 aan de onderstaande besturen en overheden gevraagd advies uit te brengen over het bovengenoemde gefaseerd bodemsaneringsproject:

- College van Burgemeester en Schepenen, van en te 2070 ZWIJNDRECHT
- College van Burgemeester en Schepenen, van en te 2000 ANTWERPEN
- Agentschap R-O Vlaanderen, Afdeling R-O Antwerpen Lange Kievitstraat 111-113 bus 52 2018 Antwerpen
- Vlaamse Milieumaatschappij, Afdeling Operationeel Waterbeheer Lange Kievitstraat 111-113 bus 64 2018 Antwerpen
- Vlaamse overheid departement Leefmilieu, Afdeling Milieuvergunningen Lange Kievitstraat 111-113 bus 61 2018 Antwerpen
- Vlaamse Milieumaatschappij, Alfons Van De Maelestraat 96 9320 Erembodegem
- Agentschap voor Natuur en Bos, Lange Kievitstraat 111-113 bus 63 2018 Antwerpen

Op 26 november 2008 werd er op verzoek van de OVAM een openbaar onderzoek georganiseerd door de gemeente ZWIJNDRECHT en door de gemeente ANTWERPEN gedurende de periode van 4 december 2008 tot en met 2 januari 2009. Er werden geen bezwaren rechtstreeks aan de OVAM of in het kader van het openbaar onderzoek ingediend.

De onderstaande overheden of besturen hebben nagelaten om binnen de daarvoor voorziene termijn advies te geven:

- Vlaamse Milieumaatschappij, Afdeling Operationeel Waterbeheer Lange Kievitstraat 111-113 bus 64 2018 Antwerpen

Aangezien er geen advies verleend werd binnen de daarvoor voorziene termijn, wordt aangenomen dat een gunstig advies werd uitgebracht en wordt de procedure voortgezet.

De onderstaande overheden of besturen hebben gunstig advies verleend:

- College van Burgemeester en Schepenen, van en te 2070 ZWIJNDRECHT
- College van Burgemeester en Schepenen, van en te 2000 ANTWERPEN

- Agentschap R-O Vlaanderen, Afdeling R-O Antwerpen Lange Kievitstraat 111-113 bus 52 2018 Antwerpen
- Vlaamse overheid departement Leefmilieu, Afdeling Milieuvergunningen Lange Kievitstraat 111-113 bus 61 2018 Antwerpen
- Vlaamse Milieumaatschappij, Alfons Van De Maelestraat 96 9320 Erembodegem
- Agentschap voor Natuur en Bos, Lange Kievitstraat 111-113 bus 63 2018 Antwerpen

De bezwaren en opmerkingen uit de tijdig verstrekte adviezen worden als volgt door de OVAM geëvalueerd:

- Alle wettelijke maatregelen om de milieuveiligheid en de arbeidsveiligheid te verzekeren bij de uitvoering van de bodemsaneringswerken, en de maatregelen zoals beschreven in Achilles (veiligheid, gezondheid en milieupreventiesysteem voor on-site bodemsaneringswerken) moeten strikt worden gevuld.
- Tijdens de sanering moeten regelmatig analyses worden uitgevoerd en ter beschikking gehouden van de controllerende overheden.
- De voorziene grondwateronttrekking past in een bodemsanering met als doel de aanwezige bodemverontreiniging te saneren. De duur van de grondwateronttrekking is slechts richtinggevend en afhankelijk van het verloop van de bodemsanering. Als uit de monitoring gedurende de bodemsaneringswerken blijkt dat de saneringsobjectieven niet worden gehaald binnen de vooropgestelde termijnen, moeten de saneringswerken worden voortgezet.
- Het effluent van de grondwaterzuiveringsinstallatie dat geloosd wordt in het oppervlaktewater, moet voldoen aan de lozingsnormen zoals opgenomen in de vigerende milieuvergunning van 3M van 20 maart 2008.
- De start en het einde van elke fase van de grondwatersanering, moet worden gemeld aan de VMM op het volgende centrale e-mailadres: bodemsanering@VMM.be. Vermeld daarbij de naam van het gefaseerd bodemsaneringsproject, de gemeente, de start- of einddatum en bemaling of sanering.
- Het influent van de waterzuiveringsinstallatie moet periodiek worden gecontroleerd. Als de bodemsaneringsdeskundige op basis van een voldoende aantal representatieve analyses over een voldoende lange periode kan aantonen dat er een stabiele eindsituatie werd verkregen kan de grondwaterzuiveringsinstallatie worden verwijderd.
- De VMM heeft volgende bedenking : "Dat wat de organofluorverontreiniging ter hoogte van het natuurreervaat Blokkersdijk betreft recent onderzoek heeft uitgewezen (Universiteit Antwerpen) dat PFOS (perfluorooctaan sulfaat) werd aangetroffen in vogeleieren en -veren. Deze studie stelde vast dat "de PFOS-concentraties in de eieren van drie vogelsoorten de hoogste waren die ooit werden gemeten bij wilde dieren" ; Dat de voorgestelde maatregelen dan ook ondermaats zijn gezien de grote kwetsbaarheid van dit gebied ; Dat de vaststelling dat er geen negatief effect is waar te nemen gezien aan de vooropstelling (aantal soorten en aantal broedjaren) wordt voldaan, wetenschappelijk zwak is ; Dat zich hier een veel dieper wetenschappelijke studie opdringt teneinde na te gaan welke gevolgen deze hoge concentraties aan PFOS bij dieren (inzonder vogels) hebben, nu en in de toekomst en wat de mogelijke saneringsremedies zijn ; Dat voorgesteld wordt deze studie op te maken in een termijn van 3 jaar na bekomen van de conformverklaring van de bodemsanering. Een exemplaar van deze studie te bezorgen aan VMM, OVAM, AMV en ANB.
- Het agentschap voor Natuur en Bos heeft een opmerking in dezelfde richting : "ANB pleit voor één of andere vorm van biomonitoring om na te gaan of er door accumulatie geen steeds hogere concentraties aan PFOS in de lokale voedselketen geraken, zelfs bij een stabiele concentratie in het oppervlaktewater. Monitoring van de aantallen en soorten van de vogels gebeurt reeds, en wordt hiermee niet bedoeld. Wel blijft dus voor ANB wenselijk te meten of er geen verdere accumulatie optreedt in het ecosysteem, hetzij via concentraties in algen, onderwatervegetatie, ongewervelden en vissen. De frequentie hoeft ook niet jaarlijks te zijn."

De OVAM is van mening dat het aangewezen is dat de verschillende betrokken partijen de relevantie en de opzet van dergelijke wetenschappelijke studie bespreken. Er moet onder meer uitgemaakt worden hoe dergelijke studie er zou moeten uitzien, op welke factoren dan gemeten

moet worden, op welke targetsoorten gefocust moet worden, hoe bepaald moet worden of een bepaalde fluorconcentratie een risico inhoudt, en welke gevolgen aan de resultaten van de studie gegeven worden. Voor 1 maart 2010 dient de opzet van de studie aan de OVAM bezorgd te worden.

- De VMM heeft verder nog volgende opmerking : "Dat aangaande het voorstel van beheer van de grondhoop met verhoogde kwik- en organofluorconcentraties de bedenking wordt gemaakt of dit vanuit milieustandpunt duurzaam is. Immers wat kan er gebeuren mocht het bedrijf ooit de site verlaten? Geniet het niet de voorkeur deze zwaar verontreinigde grondhoop af te voeren naar een gecontroleerde en speciaal ingerichte stortsite waar ook het percolatiewater op de juiste manier wordt behandeld."

Zoals in het bodemsaneringsproject opgenomen zijn er duidelijke aanwijzingen dat reiniging van de gronden niet zal leiden tot doeltreffende resultaten. Afvoer van gronden impliceert om deze reden opslag op een klasse I stortplaats. Deze mogelijkheid wordt echter niet als BATNEEC beschouwd. De relatief beperkte kennis met betrekking tot fluorcomponenten bij derden en de afwezigheid van basismilieukwaliteitsnormen in Vlaanderen kunnen mogelijk aanleiding geven tot een foutieve inschatting en een potentiële schade als gevolg van het storten. Recente grondanalyses van de grondhoop met verhoogde kwik- en organofluorconcentraties hebben ook uitgewezen dat de bodemsaneringsnormen voor PFOS, PFOA en kwik niet overschreden worden. Omwille van deze redenen wordt de voorkeur gegeven aan beheer op de 3M-site. De grondhoop is voorzien van een onder- en bovenafdek. Er is in het project voorgesteld om de afdek op regelmatige basis te controleren met het oog op erosie en de vaststelling van eventuele noodzakelijke herstelwerkzaamheden.

- Het Agentschap Ruimtelijke Ordening Antwerpen merkt volgende op : "De bodemsanering heeft mogelijk voor een klein deel betrekking op het aanpalende natuurreervaat Blokkersdijk en het landschap 'Blokkersdijk' dat beschermd werd bij het Koninklijk Besluit van 12/05/1980. Gelet op deze reliëfwijziging en gelet op het feit dat de bodemsanering een belangrijke wijziging van de waterhuishoudingskanalen in het beschermd landschap 'Blokkersdijk' te weeg brengt, zal er voor deze stedenbouwkundige vergunningsplichtige werken een afzonderlijke aanvraag moeten worden ingediend. Aangezien er activiteiten en handelingen uitgevoerd worden in het beschermd landschap die verboden zijn, zullen deze werken voorgelegd moeten worden aan het Agentschap R-O Vlaanderen Onroerend Erfgoed-Monumenten. Bij de beoordeling van die werken zal er tevens worden onderzocht of dit project MER-plichtig is. Bovendien ligt de bodemsanering binnen de afbakening van het VEN 1^{ste} fase en heeft de aanvraag invloed op de speciale beschermingszone in het kader van de vogelrichtlijn. Bij de stedenbouwkundige vergunningsaanvraag dient dit project voorgelegd te worden aan het Agentschap voor Natuur en Bos. Tevens zal er advies moeten worden gevraagd aan de Provincie – Dienst Water, vanwege dat de aanvraag paalt aan de Palingbeek (2^{de} categorie)."

Als back-up scenario wordt ter hoogte van Blokkersdijk een beheersing van de verontreiniging voorzien door middel van onttrekking op horizontale drains ter hoogte van het 3M pad. Het gezuiverde grondwater wordt geherinfiltreerd via een open gracht om een negatief effect op het oppervlaktewaterpeil in de Blokkersdijk tegen te gaan. Op deze manier zal eenzelfde hoeveelheid water in Blokkersdijk terechtkomen. De reliëfwijziging heeft dus net tot doel om geen effect op de waterhuishouding in Blokkersdijk te veroorzaken.

De back-upvariant wordt uitgevoerd op eigendom van 3M, meer bepaald de verbindingsweg tussen de 3M hoofd-site en de Schelde. De administratieve grenzen van het natuurreervaat (Vogelrichtlijngebied) liggen eenduidig meer naar het oosten. Er valt geen eigendom van 3M binnen het natuurreervaat.

Op basis van het K.B. van 12 mei 1980 vallen 3 percelen van 3M binnen het 'beschermd landschap Blokkersdijk'. Er zijn geen indicaties dat de reliëfwijziging op deze percelen plaatsvindt en bijgevolg binnen het 'beschermd landschap Blokkersdijk' wordt doorgevoerd:

- Sectie N, perceel 533c: dit perceel is opgenomen in het bodemsaneringsproject als 'te saneren perceel waarop werken op plaatsvinden'. Op dit perceel is peilput P116 gelegen welke opgenomen is in het monitoringsprogramma. De werken in het kader van de back-upvariant worden niet uitgevoerd binnen dit kadastraal perceel;

- Sectie N, perceel 533e: dit perceel is eveneens betrokken in het bodemsaneringsproject. Werken worden op dit perceel niet uitgevoerd. Het betreft het perceel waarop de 3M vijver gelegen is;
- Sectie H, deel van perceel 448b: perceel 448b bestaat momenteel niet meer en er kan niet uitgezocht worden met welk huidig perceel of deel van perceel dit momenteel overeenkomt. Echter op basis van onderstaande kaart met aanduiding van de grenzen van het 'beschermd landschap Blokkersdijk' (bron: www.geovlaanderen.be) kan gesteld worden dat de werken in het kader van de back-up variant buiten het beschermd landschap gelegen zijn.

In het kader van het bodemsaneringsproject werd nagegaan of MER-plichtige activiteiten werden uitgevoerd. Op de voorgestelde saneringswerken is geen MER-plicht van toepassing. Wel was een passende beoordeling noodzakelijk door de nabije ligging van het natuurreservaat Blokkersdijk en deze werd ook uitgevoerd.

In deze fase is er eveneens geen aanvraag noodzakelijk bij de Provincie-Dienst Water, gezien de Palingbeek niet beïnvloed wordt door de voorgestelde werken. De onttrekking in de bronzones bevindt zich op grote afstand van de Palingbeek en piloottesten hebben eveneens uitgewezen dat de invloedsstraal minimaal is. De Provincie-Dienst Water zal echter wel geïnformeerd moeten worden bij opmaak van het gefaseerd bodemsaneringsproject dat werken voorziet in de nabijheid van de Palingbeek.

- Het gemeentelijk Havenbedrijf heeft volgende opmerking "Betreffende het eerste gefaseerd bodemsaneringsproject van 3M wordt opgemerkt dat er aan de vuilvracht (PFOS concentratie) "influx naar de Schelde" vanuit de palingbeek voorlopig niets wordt gedaan omdat de bestemming van deze zone in het kader van de Oosterweelverbinding nog niet gekend is. Het Havenbedrijf acht het aangewezen dat ook deze PFOS vuilvracht in deze fase reeds aangepakt wordt, ongeacht de uiteindelijke bestemming van de zone."

Momenteel zijn er geen risico's verbonden aan de verhoogde organofluorconcentraties in de Palingbeek. Gezien de Palingbeek in industriegebied gelegen is en deze geen natuurfunctie heeft, is de ecotoxicologische veilige concentratie (PNEC) van weinig belang. Rekening houdende met de verschillende influxen van PFOS in de Schelde (waaronder ook een gedeelte van de Palingbeek) wordt de ecotoxicologisch veilige PFOS-dagvracht voor de Schelde niet overschreden.

Aangezien het een historische verontreiniging betreft moeten er cfr het bodemdecreet vanuit risicogebaseerd standpunt niet onmiddellijk maatregelen voor de Palingbeek genomen worden. In het kader van de Oosterweelverbinding wordt de Palingbeek waarschijnlijk verlegd en krijgt ze een ecocorridorfunctie. Op dat moment kan er niet uitgesloten worden dat er risico's verbonden zijn aan de fluorimpact en dient overeenkomstig het bodemdecreet overgegaan te worden naar een BATNEEC-sanering.

Door de onzekerheden met betrekking tot de werken aan de Palingbeek in het kader van de Oosterweelverbinding kan momenteel geen finale saneringsvariant voorgesteld worden. Een gefaseerd bodemsaneringsproject zal bijgevolg opgesteld worden nadat de werken aan de Palingbeek zijn uitgevoerd en de organofluorimpact in de nieuwe Palingbeek gekend is.

Naast de bovenstaande opmerkingen heeft de OVAM de volgende aanvullende opmerkingen:

- Aangezien het een bedrijf in exploitatie betreft, zijn de mogelijke saneringstechnieken beperkt. De OVAM wil hierbij als randvoorwaarde stellen dat de in uitvoering zijnde saneringstechnologie op jaarlijkse basis geherevalueerd moet worden. De evolutie in nieuwe saneringstechnieken moet opgevolgd worden. Indien er een overdracht of sluiting gepland is, is een herevaluatie van de saneringstechniek eveneens noodzakelijk. Indien infrastructuurwerken op de site gepland zijn, dient bekeken te worden of andere actieve saneringsmaatregelen zoals ontgraving mogelijk zijn.
- Bij de uitvoering van de sanering dient rekening gehouden te worden met de aanwezigheid van de andere aanwezige verontreinigingen in het grondwater.
- De monitoringspeilbuizen dienen met een bepaalde frequentie op een uitgebreider parameterpakket (bv met TFA, ...) geanalyseerd te worden. Daarnaast dienen de nodige peilmetingen te gebeuren teneinde aan te tonen dat er geen relevante impact is op het oppervlaktewaterniveau in Blokkersdijk.

- Indien in afwachting van de opstelling van het tweede gefaseerd bodemsaneringsproject op basis van de monitoringsronden een verhoging van de fluorconcentraties in de Palingbeek of in de Z-peilputten wordt vastgesteld, dient nagegaan te worden welke maatregelen hiervoor genomen zullen worden.
- Volgens het project is de triggerfactor voor overgang naar een actieve back-up variant ter hoogte van Blokkersdijk gebaseerd op een vastgestelde statistisch stijgende trend in de PFOS-concentraties in de Blokkersdijkvijver. De OVAM is van mening dat als een statistische stijging vastgesteld wordt in de grondwaterconcentraties stroomopwaarts van Blokkersdijk, er niet afgewacht moet worden of dit een invloed zal hebben op de oppervlakewaterkwaliteit van Blokkersdijk. Overleg is op dat moment nodig om na te gaan of en welke actieve maatregelen toegepast moeten worden.
- Indien op basis van de analyseresultaten van de 2de aquifer een significante verhoging van de fluorconcentraties in de 2de aquifer wordt vastgesteld, dient de deskundige af te wegen welke actieve maatregelen aanvullend noodzakelijk zijn.
- De OVAM kan akkoord gaan met de voorgestelde herinfiltratieregels op voorwaarde dat het om een gesloten systeem gaat waarop de nodige controle wordt uitgevoerd.

Parameter	herinfiltratieregel
PFOS	1,5 µg/l
PFOA	Drinkwaternorm (1)
PFHS	Drinkwaternorm (1)
PFOSA	Drinkwaternorm (1)

(1) Opmerking: de herinfiltratieregel is de drinkwaternorm. Indien evenwel de terugsaneerwaarde strenger is, is dit de herinfiltratieregel. De herinfiltratiowaarde mag tevens niet hoger liggen dan de op dit ogenblik aangetroffen concentraties in de herinfiltratiezone.

- De eigen bedrijfsafvalwaterzuiveringsinstallatie van 3M zal aangewend worden voor de zuivering van het onttrokken grondwater uit de kernzone. Op de regenwaterriolering worden twee in serie geschakelde actief koolfilters geplaatst. Het behandelde regenwater wordt samen met het effluent van het bedrijfsafvalwater geloosd in de Schelde. De lozingsnormen en debieten moeten hierbij voldoen aan de vigerende milieuvergunning van 3M van 20 maart 2008. De influent- en effluentstromen van zowel onttrokken grondwater, bedrijfsafvalwater als regenwater dienen apart bemonsterd te worden, zowel naar volume als concentratie toe.
- De deskundige dient tijdens de sanering op regelmatige basis na te gaan of de configuratie van de onttrekkingsfilters geoptimaliseerd dient te worden.

Op basis van de bovenstaande evaluatie van de bezwaren en opmerkingen bestaat er geen aanleiding tot het opleggen van wijzigingen aan of aanvullingen op het gefaseerd bodemsaneringsproject.

Watertoets

Bij haar beoordeling heeft de OVAM rekening gehouden met de bepalingen van het decreet van 18 juli 2003 betreffende het integraal waterbeleid en het besluit van de Vlaamse regering van 20 juli 2006 tot vaststelling van nadere regels voor de toepassing van de watertoets, inzonderheid op artikel 4 over de waterparagraaf.

De geplande bodemsaneringswerken hebben als doel de kwaliteit van het watersysteem te verbeteren. Hiervoor moet een hoeveelheid verontreinigd grondwater worden onttrokken en na zuivering worden geloosd in het oppervlaktewater. Het volume te onttrekken grondwater is evenredig

met de aanwezige verontreiniging in het grondwater. De ontrekking is beperkt tot de freatische laag en de grondwateronttrekking is louter curatief van aard.

In het kader van de back-upvariant ter hoogte van Blokkersdijk wordt het gezuiverde grondwater gherinfiltreerd. Gelet op de strenge herinfiltratieregels en het feit dat het onttrokken grondwater opnieuw wordt gherinfiltreerd, worden geen ongunstige effecten verwacht op de aanwezige grondwaterkwaliteit.

De vigerende milieuvergunning van 3M voorziet de toe te passen lozingsnormen zodat de kwaliteit van het geloosde water in overeenstemming is met de kwaliteitseisen voor het uiteindelijke ontvangende oppervlaktewater.

Bepalingen van de bodemsaneringswerken

De onderstaande documenten en verwijzingen geven het kader weer voor de uitvoering van de bodemsaneringswerken:

- De standaardprocedure 'Bodemsaneringswerken, Eindevaluatieonderzoek en Nazorg' en de verschillende codes van goede praktijk creëren enerzijds een kader voor de opvolging, controle en rapportage van bodemsaneringswerken en bevatten anderzijds een aantal praktische richtlijnen voor de opvolging van de specifieke saneringstechnieken.
- Bodemsaneringswerken moeten worden uitgevoerd in overeenstemming met de regels opgenomen in het meest recente Achillespreventiesysteem.
- De leden van de Ondernemersvereniging Bodemsaneerders (OVB) moeten de voorschriften van de milieubeleidsovereenkomst van 9 juni 2004 betreffende de invoering van een milieuzorgsysteem in het kader van bodemsaneringswerken toepassen.
- De leden van de Ondernemersvereniging Bodemsaneerders (OVB) moeten de voorschriften van het Achilleszorgsysteem, dat een uitbreiding is van het Achillespreventiesysteem, toepassen.

De bodemsaneringswerken moeten worden uitgevoerd met oog voor de bescherming van mens en milieu en de verwezenlijking van een goede plaatselijke aanleg.

3 Besluit

Artikel 1 - Conformiteit

De OVAM verklaart dat het gefaseerd bodemsaneringsproject met de titel "Bodemsaneringsproject 3M Belgium NV, Haven 1005, Canadastraat 11 te Zwijndrecht - 11/003460", opgesteld onder leiding van de bodemsaneringsdeskundige Arcadis Belgium nv in opdracht van 3M Belgium NV en aan de OVAM betekend op 12 november 2008, conform aan de bepalingen van het Bodemdecreet is.

Art. 2 – Voorwaarden voor de uitvoering van de bodemsaneringswerken

Met het oog op de bescherming van mens en milieu en de verwezenlijking van een goede plaatselijke aanleg, legt de OVAM de volgende voorwaarden op aan de uitvoering van de bodemsaneringswerken:

- De bodemsaneringswerken moeten worden uitgevoerd overeenkomstig de standaardprocedure 'Bodemsaneringswerken, Eindevaluatieonderzoek en Nazorg' en de codes van goede praktijk.
- Bij eventuele calamiteiten moet de verantwoordelijke of de bij contract aangestelde verantwoordelijke van de bodemsaneringswerken, de bevoegde instanties en de OVAM onmiddellijk verwittigen.
- De meet- en controle-infrastructuur moet toegankelijk zijn voor de bevoegde toezichthoudende ambtenaren of een daartoe aangestelde onafhankelijke deskundige. Om de controle van deze installatie door de OVAM of door de andere bevoegde instantie mogelijk te maken, moet steeds

een sleutel van de installatie ter plaatse beschikbaar zijn. Ingeval de werf of inrichting waar de bodemsanering plaatsvindt niet permanent wordt bemand, moet voor de aanvang van de werkzaamheden schriftelijk de naam van een contactpersoon die steeds over de nodige sleutels beschikt, en zijn telefoonnummer, aan de OVAM worden meegeleid.

- De verantwoordelijke of bij contract aangestelde verantwoordelijke van de bodemsaneringswerken moet op eenvoudig verzoek van de toezichthoudende ambtenaren of een door hen aangestelde onafhankelijke deskundige alle nodige gegevens verstrekken om een degelijke evaluatie en controle van de voortgang van de werken te kunnen uitvoeren.
- Alle wettelijke maatregelen om de milieuveiligheid en de arbeidsveiligheid te verzekeren bij de uitvoering van de bodemsaneringswerken en de maatregelen zoals beschreven in Achilles, moeten strikt worden gevolgd,
- Het verloop en de resultaten van de bodemsaneringswerken moet jaarlijks aan de OVAM worden gerapporteerd volgens de standaardprocedure 'Bodemsaneringswerken, Eindevaluatieonderzoek en Nazorg'.
- De grondwateronttrekking is gekoppeld aan de strikte naleving van de eigen milieuvergunningsvoorraarden van 3M.
- Het effluent van de grondwaterzuiveringsinstallatie dat geloosd wordt in het oppervlaktewater, moet voldoen aan de lozingsnormen zoals opgenomen in de vigerende milieuvergunning van 3M van 20 maart 2008.
- De start en het einde van elke fase van de grondwatersanering, moet worden gemeld aan de VMM op het volgende centrale e-mailadres: bodemsanering@VMM.be. Vermeld daarbij de naam, de gemeente, de start- of einddatum en bemaling of sanering.
- De monitoring van de peilbuizen bij het beëindigen van de saneringswerken moet gebeuren nadat de evenwichtsituatie in de bodem is bereikt. Ter controle van de efficiëntie van de saneringsmaatregelen moeten minimaal de peilbuizen stroomafwaarts worden gecontroleerd alsook de peilbuizen met de hoogste concentratie aan verontreinigende stoffen, in de kern van de verontreiniging.
- De saneringswerken mogen pas worden stilgelegd na het bereiken van de vooropgestelde doelstellingen, of na onderling overleg met de OVAM, rekening houdend met de efficiëntie van de saneringstechniek (BATNEEC-principe) en de kwaliteit van het vaste deel van de aarde en van het grondwater. De vooropgestelde termijn van de bodemsanering is niet bindend en moet, gebaseerd op de bodemkwaliteit, op eenvoudig verzoek van de OVAM worden voortgezet.
- De uitvoering van bodemsaneringswerken is gebonden aan de regels die zijn opgenomen in het meest recente Achillespreventiesysteem. Dat betekent dat de opdrachtgever de bodemsaneerder, voor aanvang van de bodemsaneringswerken, een certificatie-instelling moet aanstellen. Op die manier kan worden nagegaan of de bodemsaneerder het Achillespreventiesysteem naleeft. Indien de bodemsaneerder lid is van de OVB heeft de hij een milieubeleidsovereenkomst getekend en is hij gebonden aan het Achilleszorgsysteem. In dat geval is het niet nodig dat er een certificatie-instelling wordt aangeschreven. De audit in het kader van het Achillespreventiesysteem moet worden uitgevoerd overeenkomstig de bepalingen van deel B van het Achillespreventiesysteem.
- De OVAM is van mening dat het aangewezen is om op korte termijn met de verschillende betrokken partijen de relevantie en de opzet van dergelijke wetenschappelijke studie te bespreken. Er moet onder meer uitgemaakt worden hoe dergelijke studie er zou moeten uitzien, op welke factoren dan gemeten moet worden, op welke targetsoorten gefocust moet worden, hoe bepaald moet worden of een bepaalde fluorconcentratie een risico inhoudt, en welke gevolgen aan de resultaten van de studie gegeven worden.
- Aangezien het een bedrijf in exploitatie betreft, zijn de mogelijke saneringstechnieken beperkt. De OVAM wil hierbij als randvoorwaarde stellen dat de in uitvoering zijnde saneringstechnologie op jaarlijkse basis geherevalueerd moet worden. De evolutie in nieuwe saneringstechnieken moet opgevolgd worden. Indien er een overdracht of sluiting gepland is, is een herevaluatie van de saneringstechniek eveneens noodzakelijk. Indien infrastructuurwerken op de site gepland zijn, dient bekeken te worden of andere actieve saneringsmaatregelen zoals ontgraving mogelijk zijn.
- Bij de uitvoering van de sanering dient rekening gehouden te worden met de aanwezigheid van de andere aanwezige verontreinigingen in het grondwater.
- De monitoringspeilbuizen dienen met een bepaalde frequentie op een uitgebreider parameterpakket (bv met TFA, ...) geanalyseerd te worden.

- Indien in afwachting van de opstelling van het tweede gefaseerd bodemsaneringsproject op basis van de monitoringsronden een verhoging van de fluorconcentraties in de Palingbeek of in de Z-peilputten wordt vastgesteld, dient nagegaan te worden welke maatregelen hiervoor genomen zullen worden.
- Volgens het project is de triggerfactor voor overgang naar een actieve back-up variant ter hoogte van Blokkersdijk gebaseerd op een vastgestelde statistisch stijgende trend in de PFOS-concentraties in de Blokkersdijkvijver. De OVAM is van mening dat als een statistische stijging vastgesteld wordt in de grondwaterconcentraties stroomopwaarts van Blokkersdijk, er niet afgewacht moet worden of dit een invloed zal hebben op de oppervlakewaterkwaliteit van Blokkersdijk. Overleg is op dat moment nodig om na te gaan of en welke actieve maatregelen toegepast moeten worden.
- Indien op basis van de analyseresultaten van de 2de aquifer een significante verhoging van de fluorconcentraties in de 2de aquifer wordt vastgesteld, dient de deskundige af te wegen welke actieve maatregelen aanvullend noodzakelijk zijn.
- herinfiltatienormen (op voorwaarde dat het om een gesloten systeem gaat waarop de nodige controle wordt uitgevoerd):

Parameter	herinfiltatienorm
PFOS	1,5 µg/l
PFOA	Drinkwaternorm (1)
PFHS	Drinkwaternorm (1)
PFOSA	Drinkwaternorm (1)

(1) Opmerking: de herinfiltatienorm is de drinkwaternorm. Indien evenwel de terugsaneerwaarde strenger is, is dit de herinfiltatienorm. De herinfiltatiewaarde mag tevens niet hoger liggen dan de op dit ogenblik aangetroffen concentraties in de herinfiltatiezone.

- De eigen bedrijfsafvalwaterzuiveringsinstallatie van 3M zal aangewend worden voor de zuivering van het ontrokken grondwater uit de kernzone. Op de regenwateriolering worden twee in serie geschakelde actief koolfilters geplaatst. Het behandelde regenwater wordt samen met het effluent van het bedrijfsafvalwater geloosd in de Schelde. De lozingsnormen en debieten moeten hierbij voldoen aan de vigerende milieuvvergunning van 3M van 20 maart 2008. De influent- en effluentstromen van zowel ontrokken grondwater, bedrijfsafvalwater als regenwater dienen apart bemonsterd te worden, zowel naar volume als concentratie toe.
- De deskundige dient tijdens de sanering op regelmatige basis na te gaan of de configuratie van de onttrekkingsfilters geoptimaliseerd dient te worden.

Art. 3 – Termijn aanvang van de bodemsaneringswerken

De bodemsaneringswerken zoals omschreven in voorliggend gefaseerd bodemsaneringsproject moeten worden aangevat **vóór 1 januari 2010**.

Overeenkomstig de standaardprocedure ‘Bodemsaneringswerken, Eindevaluatieonderzoek en Nazorg’ moet het volledig ingevulde kwaliteitsplan (inclusief bijlagen) minimaal acht dagen **vóór** de startvergadering aan de OVAM worden bezorgd.

te Mechelen,
09 FEB. 2009



Eddy Van Dyck
Afdelingshoofd

Nanda Hermes

Subject: FW: Verzoek tot akkoord kleine wijziging Bodemsaneringsproject 3M Zwijndrecht
(Dossiernr. 732)

From: Filip Collet [REDACTED]
Sent: Friday 9 March 2018 15:38
To: Charlotte Tack <REDACTED>
Cc: Nicole Cauberghe <REDACTED>; Nynke De Schutter <REDACTED>
Subject: [EXTERNAL] Re: Verzoek tot akkoord kleine wijziging Bodemsaneringsproject 3M Zwijndrecht (Dossiernr. 732)

Geachte mevrouw Tack,

gezien de te verwachten situatie, is het wenselijk om pompput PP09 buiten gebruik te stellen en een nieuwe te voorzien op een deel van het terrein waar in de nabije toekomst geen werken verwacht worden. Een iets noordelijker gelegen locatie is daarvoor aangewezen.

Met vriendelijke groeten,

Filip Collet | Afdeling Bodembeheer
Team Particulieren
OVAM | Stationsstraat 110 - 2800 Mechelen
T 015 284 573 | F 015 284 408
E [REDACTED] | www.ovam.be

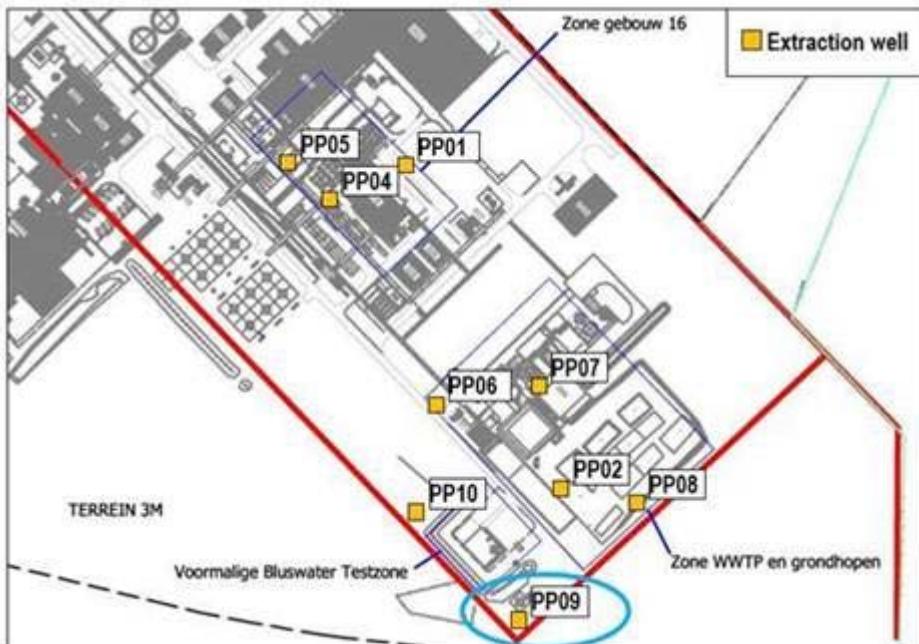
Samen maken we morgen mooier
Heeft u onze nieuwsbrief al gelezen? Schrijf u in op www.ovam.be/nieuwsbrief

Van: "Charlotte Tack" <REDACTED>
Aan: "Filip Collet" <REDACTED>, "Filip Collet" <REDACTED>
Cc: "Nicole Cauberghe" <REDACTED>, "Nynke De Schutter"
<REDACTED>
Verzonden: Vrijdag 2 maart 2018 16:42:00
Onderwerp: Verzoek tot akkoord kleine wijziging Bodemsaneringsproject 3M Zwijndrecht
(Dossiernr. 732)

Geachte heer Collet,
Beste Filip,

Met deze email willen wij je op de hoogte brengen van een mogelijke kleine wijziging van het bodemsaneringsproject (Fase 1) dat lopende is op het terrein van 3M te Zwijndrecht.

Zoals je weet, zal er mogelijk op het terrein van 3M, langs de zuidelijke perceelsgrens, een veiligheidsberm worden aangelegd. De exacte ligging en dimensie van de berm zijn nog niet gekend, er is ook nog geen vergunningsaanvraag ingediend. Concreet zou dit betekenen dat de veiligheidsberm ter hoogte van de meest zuidelijke punt van het terrein, op pompput PP09 komt te liggen waardoor deze onbereikbaar wordt. Verder zullen ook een aantal peilbuizen verloren gaan door de aanleg van de berm.



In overleg met onze bodemsaneringsdeskundige ERM zouden we daarom willen voorstellen om pompput PP09 buiten gebruik te stellen en meer noordwaarts op de 3M site een nieuwe pompput te voorzien (exakte ligging is nog te bepalen). De peilbuizen die verloren zullen gaan, zullen vooraf geïnventariseerd worden en daarna buitengebruik gesteld worden. Op basis van de inventarisatie zal beslist worden welke peilbuizen vervangen dienen te worden.

Graag hadden wij via deze email jouw goedkeuring ontvangen om pompput 09 te verplaatsen zoals hiervoor is beschreven en de peilbuizen buitengebruik te stellen (en indien noodzakelijk te vervangen). De details van de werken zullen opgenomen worden in het eerst volgende tussentijds verslag (na uitvoering) dat aan OVAM wordt overgemaakt.

Wij gaan ervanuit dat als wij binnen 14 dagen geen reactie ontvangen, je akkoord bent met de voorgestelde kleine wijziging.

Mocht je nog vragen of bemerkingen hebben, aarzel dan niet om met ons contact op te nemen.

Vriendelijke groeten,

3M Science.
Applied to Life.™

Charlotte Tack | EHS Engineer
3M MRD Manufacturing site
3M Belgium bvba/sprl, Haven 1005, Canadalaan 11, 2070 Zwijndrecht | België
Office: +32 3 250 7753
[REDACTED]



Disclaimer: [Link replaced by security measures](#), If needed for business purposes open a

Disclaimer: [Link replaced by security measures, If needed for business purposes open a ticket to ww_it-security-dlp-infra](#)

3M Note: This message is from an [EXTERNAL] sender.

If you suspect this message is malicious or spam, please click on the "Report Phishing - PhishMe" icon within the Outlook Ribbon to report it for evaluation, and do NOT open any attachments or click on any links. If you are using OWA, a handheld device, or do not see the icon, please follow the instructions below:

Click [here](#) to report this email as spam

Memo

Aan	OVAM – Karen Uyt den Houwen
Van	3M (Charlotte Tack) en ERM (Nanda Hermes)
Datum	19 februari 2019
Referentie	M005-0451640-V1.docx
Betreft	3M Zwijndrecht (OVAM dossier nr. 732) Aanpassing monitoringsschema betreffende lopende sanering

Beste mevrouw Uyt den Houwen,

Op 28 september 2018 vond overleg plaats over de vooruitgang van het lopende (eerste gefaseerd) bodemsaneringsproject voor de FC grondwaterverontreiniging op het terrein van 3M te Zwijndrecht. Het overleg is georganiseerd om OVAM op de hoogte te houden van de vorderingen van de lopende sanering en de volgende personen waren aanwezig: Karen Uyt den Houwen (OVAM), Nynke de Schutter en Nicole Cauberghe (3M) en, Nanda Hermes en Lieselotte Sorgeloos (ERM).

Tijdens het overleg heeft 3M een algemene voorstelling gegeven over de activiteiten en de site van 3M te Zwijndrecht. Vervolgens zijn door ERM de monitoringsresultaten van de periode augustus 2016-juli 2017 besproken. De monitoringsresultaten van deze periode zijn ook opgenomen in het laatste, 8^{ste} tussentijds rapport (TTR8), dat in augustus 2018 bij OVAM is ingediend.

Tot op heden was met OVAM overeengekomen om de monitoringsresultaten jaarlijks te rapporteren in een tussentijds rapport. Hoewel de FC concentraties in het grondwater kunnen fluctueren, vertonen deze ter hoogte van de meeste monitoringspeilbuizen een stabiele tot dalende concentratietrend. De FC contour (10.000 µg/l) van het grondwater in de eerste aquifer is ter hoogte van de beide bronzones in omvang afgenummerd doorheen de tijd. Gezien deze positieve evolutie van de lopende sanering, is tijdens het overleg voorgesteld om de frequentie waarmee tussentijds aan OVAM gerapporteerd wordt, af te bouwen. Deze kleine wijziging is door OVAM reeds bevestigd in haar akkoord van TTV8: in haar schrijven van 26 september 2018 (ref. BB-IKB-KUDH-20180544871) geeft OVAM aan dat het volgende tussentijds rapport ingediend dient te worden voor 25 september 2021, dus na drie jaar monitoring. Een jaarlijkse interne rapportage met bespreking van de monitoringsresultaten zal behouden blijven. Indien er belangrijke wijzigingen plaatsvinden in het monitoringsschema, zullen deze tussentijds, via een kort schrijven aan OVAM gecommuniceerd worden.

Samengaand met de aanpassing van de rapportagefrequentie is tijdens het overleg van 28 september 2018 aan OVAM voorgesteld om de dupliaat-analyses, die uitgevoerd worden door het door OVAM erkend laboratorium SGS en die dienen als verificatie van de analyses uitgevoerd door het 3M Environmental laboratorium in de USA, af te bouwen. Momenteel worden alle grondwaterstalen op FCs in het 3M Environmental laboratorium geanalyseerd. Aangezien dit geen door OVAM erkend labo is, is in oktober 2013 met OVAM afgesproken dat van elke meetlocatie minstens één grondwaterstaal jaarlijks een dupliaat geanalyseerd wordt.

door SGS. De volgende aanpassing van het analyseschema wordt voorgesteld voor de monitoringsronden vanaf januari 2019:

- Alle waterstalen die in het kader van de saneringsmonitoring worden genomen, zullen nog steeds geanalyseerd worden in het 3M Environmental laboratorium in de USA;
- Alle stalen die nodig zijn voor de PNEC evaluatie, meer bepaald de waterstalen van het effluent van de bedrijfswaterzuivering, het regenwater van de collectorput en het oppervlaktewater ter hoogte van het bemaalingsstation, zullen vier (4) maal per jaar geverifieerd worden door dupicaatanalyses die door SGS uitgevoerd worden;
- De oppervlaktewaterstalen van de Blokkersdijkvijver en 3M vijver, en de grondwaterstalen van de monitoringspeilbuizen ter hoogte van het 3M pad (aanpalend aan het natuurreervaat Blokkersdijk) zullen één (1) maal per jaar geverifieerd worden door dupicaatanalyses door SGS;
- De grondwaterstalen van de monitoringspeilbuizen ter hoogte van de twee bronzones (gebouw 16 en zone bedrijfswaterzuivering), zowel van de eerste als de tweede aquifer, zullen één (1) maal in de drie (3) jaar geverifieerd worden door dupicaatanalyses die door SGS uitgevoerd worden;
- De grondwaterstalen van de extractieputten zullen één (1) maal per jaar geverifieerd worden door SGS door dupicaatanalyses.

Verder is voorzien dat, indien er tijdens een monitoringsronde afwijkende resultaten worden gemeten, geëvalueerd zal worden of een heranalyse dan wel een bijkomende dupicaat staalname tijdens de volgende monitoring noodzakelijk is.

Mocht u nog vragen of bemerkingen hebben, aarzel niet om contact op te nemen met ERM (Nanda Hermes; ██████████). Indien wij tegen 22 maart 2019 geen tegenbericht ontvangen, gaan we ervan uit dat u akkoord ben met het aangepaste monitoringsschema.

Met vriendelijke groet,



Nanda Hermes
Principal Consultant ERM nv

Julie Fichefet

From: Nanda Hermes
Sent: dinsdag 19 februari 2019 9:34
To: Karen Uyt den houwen (██████████)
Cc: Nynke De Schutter (██████████); Charlotte Tack; Mattias Verbeeck;
Lieselotte Sorgeloos
Subject: 3M Zwijndrecht (OVAM dossier nr. 732) - Aanpassing monitoringsschema
betreffende lopende sanering
Attachments: M005-0451640-V1.pdf

Follow Up Flag: Follow up
Flag Status: Flagged

Beste Karen,

Zoals overeengekomen tijdens ons overleg vorig jaar september sturen we je hierbij ons voorstel voor een aangepast monitoringsschema voor de lopende grondwatersanering op het terrein van 3M Zwijndrecht. Deze wijziging kadert in de aanpassing van de rapportagefrequentie van jaarlijks naar driejaarlijks die reeds door OVAM bevestigd is in haar akkoord van achtste tussentijds rapport – TTV8 (ref. BB-IKB-KUDH-20180544871, dd. 26 september 2018).

In bijgevoegd voorstel zijn de dupliaat-analyses, uitgevoerd door SGS, afgebouwd rekening houdend met het doel van de genomen waterstalen.

Wij hopen dat OVAM akkoord kan gaan met deze kleine wijziging van het saneringsproject.

Mocht u nog vragen of bemerkingen hebben,aarzel niet om contact op te nemen met mij.

Indien wij tegen 22 maart 2019 geen tegenbericht ontvangen, gaan we ervan uit dat je akkoord ben met het aangepaste monitoringsschema.

Kind regards | Met vriendelijke groet,

Nanda Hermes
Principal Consultant

Out of Office on Wednesday | Niet op kantoor op woensdag

ERM

Posthoflei 5 B6 | 2600 Berchem | Belgium

M +32 473 974159

E ████████ W www.erm.com



ISO 9001 Quality Management	ISO 14001 Environmental Management	OHSAS 18001 Occupational Health & Safety Management
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Take a look at [ERM Sustainability Report 2017](#) and [ERM Foundation Annual Review 2017](#)

BIJLAGE 2

MILIEUDAGBOEKEN PP01-PP09-PP11



DAILY BOOK ENVIRONMENTAL SUPERVISION

Date: 3-6/12/2018

Project ID: 0451640 – 3M Belgium
OVAM ID: 732

Site Location	Weather
Canadastraat 11 2070 Zwijndrecht (Belgium)	Cloudy, 5-15°C

Distribution list	
3M	Nynke De Schutter (██████████), Charlotte Tack (██████████)
Weston Solutions	Dave Cairns (██████████)
ERM	Mattias Verbeeck (██████████), Erik Boeckx (██████████), Nanda Hermes (██████████), Julie Fichefet (██████████) Lieselotte Sorgeloos (██████████)
Envisan	Evert Blomme (██████████), Jeroen Van Den Bossche ██████████

Ongoing remediation:

Containment of the Fluor Chemicals (FC's) impact in groundwater in the source areas via groundwater extraction (nine extraction wells - PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 and PP10). Extracted groundwater is discharged to the site waste water treatment plant (WWTP).

Because PP01 is located within a new construction site, this well needs to be relocated. It is agreed that the relocate well PP01 is called PP11

Site visit – Status

December 3, 2018

After the general site setup, the drilling location and the trench are cleared with a vacuumtruck (Janssens).

3M has installed a rack to connect the effluent to the chemical sewer.

December 4, 2018

Bouten/Geotron is on site with a pulse drilling rig for the installation of the new well. The well is installed at a depth of 6,5 m-gl. Diameter of the well is 160 mm. The drilling diameter is 324 mm. The filter sand grain size is of 0.4-0.8 mm. Because Geotron didn't bring enough sand, it is planned to complete the well finishing the day after.

During the well installation, Envisan installed the piping and prepared to connect to the chemical sewer.

December 5, 2018

Bouten/Geotron completes the installation of the well with filter sand and Mycoliet (300). Zaniboo is on site to adjust the concrete housing of the pumping well with some additional holes.

December 6, 2018

Envisan installs the concrete housing on the well with the electric board and connects the piping with electric weldings. The piping is fixed in the pipe rack and ends inside the chemical sewer. 3M will connect the power supply.

Health & Safety	
No incidents or unsafe acts/conditions have been observed.	
Environmental Observation	
<u>Field observations</u> None	
<u>Sampling and Analysis</u> None	
<u>Photolog</u> <u>Clearance drilling hole</u> 	<u>Clearance trench</u> 
<u>Bouten/Geotron pulse drilling</u> 	<u>Inox well with sandcatcher</u> 

Zaniboo adjusting the concret housing



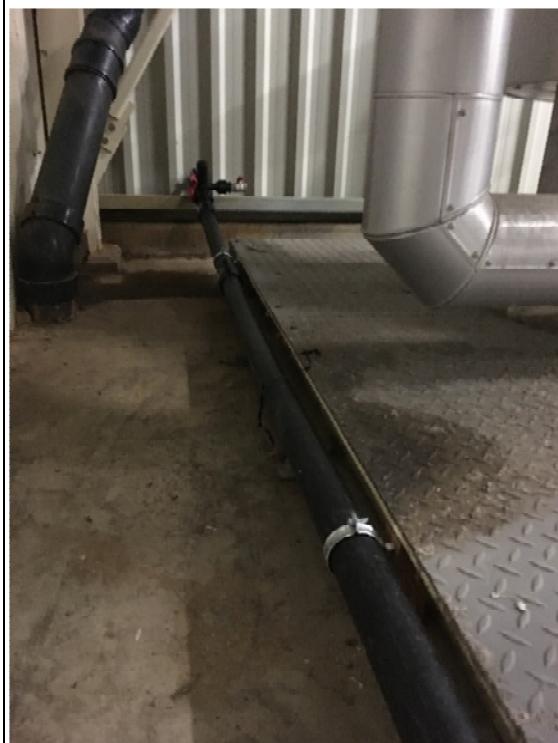
Well finishing



Pipe connection in building



Pipe passage inside building



Connection of well piping



PP11



**For the certified Soil expert
(bodemsaneringsdeskundige)**

Name and Signature

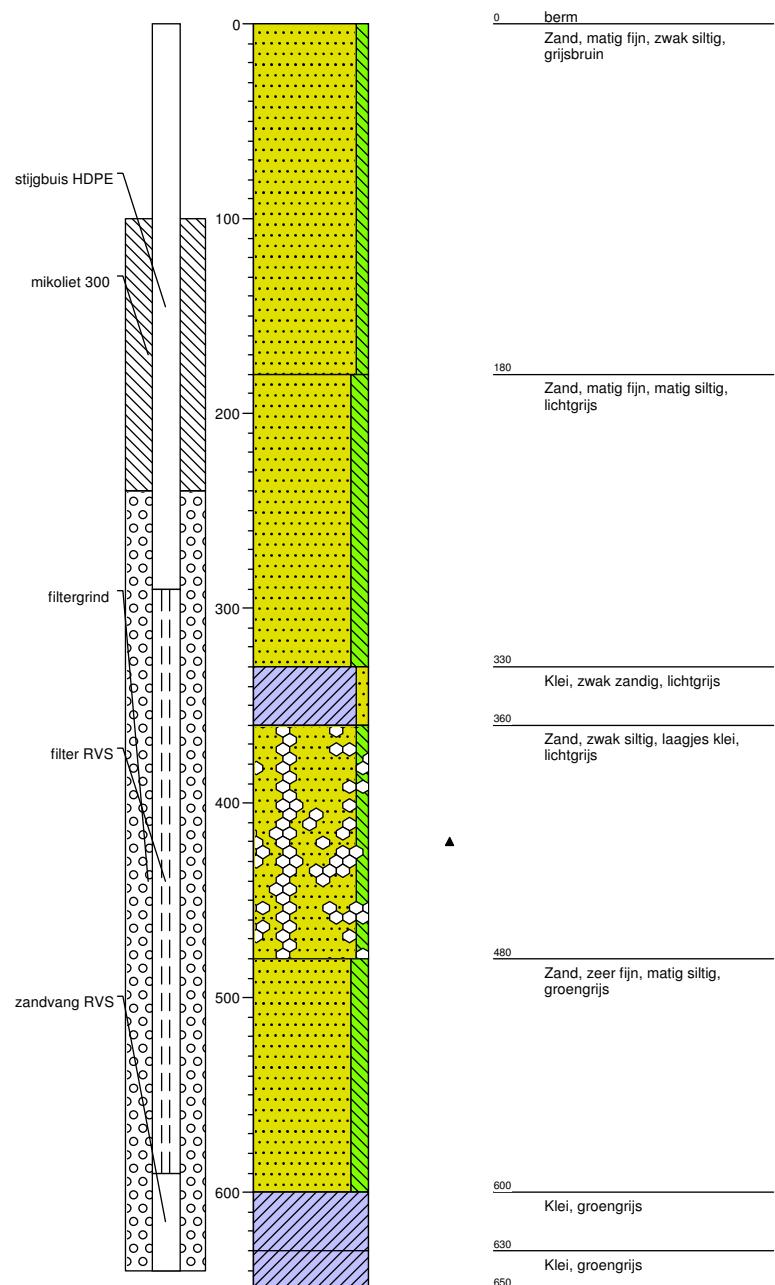
A handwritten signature in black ink, appearing to read "Erik Boeckx".

Erik Boeckx

Boring: 1

Datum: 04-12-2018

Boormeester: T. Nas



	DAILY BOOK ENVIRONMENTAL SUPERVISION CLEANING OF EXTRACTION WELLS, PUMPS AND PIPING AT THE 3M SITE, ZWIJNDRECHT <u>Date:</u> 03-07-2019 until 16-07-2019 <u>Project number:</u> 0499795 <u>Client:</u> 3M Belgium bvba
---	--

Location	(Sub)contractors involved
Canadastraat 11 2070 Zwijndrecht (BE)	Envisan nv All Clean bvba

Distribution list	
3M	Nynke De Schutter (██████████), Wouter Paque (██████████) and Sam Van Beneden (██████████)
Weston Solutions	Dave Cairns (██████████)
ERM	Nanda Hermes (██████████), Mattias Verbeeck (██████████), Nor Farina Nadzif (██████████), Julie Fichefet (██████████), Erik Boeckx (██████████)
Envisan	Evert Blomme (██████████)

Activities	
<u>Introduction:</u>	A groundwater extraction system consisting of nine extraction wells is operational at the 3M site in Zwijndrecht (BE). The aim of the system is to remediate groundwater impacted with Fluor Chemicals (FC's) and to mitigate contaminant migration. The impacted groundwater extracted by the remedial system is treated by the wastewater treatment plant of the site.
The extraction wells and pumps are periodically cleaned to optimize performance of the remedial system. Cleaning is required because of silting and rusting of wells and pumps.	
Between the 3 rd and 16 th of July 2019, cleaning and maintenance activities were executed by the remedial contractor Envisan and their subcontractor, the industrial cleaning company All Clean under the environmental supervision of ERM. This document summarizes the actions performed.	
<u>Wednesday July 3rd, 2019 (07:00-13:00)</u>	On Wednesday, Envisan and ERM prepared the extraction wells for cleaning by shutting down the installation, opening and dismantling all 9 wells and related piping. After dismantling, the pumps were taken to the Envisan workshop for technical inspection and revision. A report will be written by Envisan on the condition of the pumps. At PP05, the presence of a hard black precipitation on the pump, the transducer and whitin the piping is (again) identified.
<u>Activities performed:</u>	

- Shut down of the groundwater extraction installation;
- The electric box in the container is sealed following Lock Out/Tag Out -procedure; and
- Opening and dismantling of the wells (PP02, PP04, PP05, PP06, PP07, PP08, PP09, PP10 and PP11) in order to transport the pumps to Envisan's technical workshop for revision.

Thursday July 4th, 2019 (8:00-15:00)

On Thursday, ERM, Envisan and All Clean cleaned and flushed all (subsurface) pipes of all extraction wells, except PP09. As PP09 will be abandoned due to Oosterweel related construction activities, this well was decommissioned with bentonite by Envisan. PP09 will be relocated later this year. All Clean came on site with two vacuum trucks to execute these works. The cleaning was performed using high pressured water, resulting in a big amount of sediment removal. At the end of the day, water and sludge captured by the vacuum trucks was disposed in dewatering containers on site. ERM and Envisan added Boresaver (2kg) and Iron Clean Liquid (2L) to all wells, except PP09.

Activities performed:

- Envisan decommissioned PP09;
- Envisan dismantled the piping network inside the container;
- All Clean cleaned and flushed the dismantled pipes in the container; and
- All Clean removed sediments and depositions from inside the extraction wells and pipes. Flush water from extraction wells PP02, PP07, PP08 and PP10 is collected by the vacuum truck at the container. Flush water of PP04, PP05, PP06 and PP11 is directly discharged into the 3M chemical sewer.
- ERM and Envisan added Boresaver and Iron Clean Liquid to flush and clean all extraction wells, except PP09.

Thursday July 11th, 2019 (07:00-17:00)

The preliminary results of the pump tests performed by Envisan in their technical workshop indicated that 4 of the 8 pumps were still in good condition and could be re-installed. The motor of the other 4 pumps needed to be replaced. Only 7 of the 8 revised pumps arrived on site on Thursday. Therefore one of the spare pumps in the container needed to be used.

On Thursday, Envisan, ERM and All Clean came on site with one vacuum truck to remove the Boresaver product and Iron Clean Liquid from the extraction wells. Envisan re-installed the piping infrastructure inside the container, the flowmeters and re-installed the revised pumps around building 16 (PP04, PP05 and PP11) and some of the pumps around the WWTP (PP06 and PP08).

ERM measured the total depth of the extraction wells with an interface meter in order to determine the removed layer of sediment (see table below).

All flowmeters were reset and the pumps of PP04 and PP11 were restarted. PP05 was re-installed but not restarted because there are ongoing construction works around that area. Since some of the pumps around the WWTP were not installed yet, the pumps operated from the container were not restarted.

At the end of the day, water and sludge captured by the vacuum truck was disposed in 3M dewatering containers on site.

Activities performed :

- Removal of cleaning chemicals and cleaning of 8 extraction wells;
- Piping infrastructure of extraction system inside container re-installed;
- Depth measurement of all extraction wells; and

- Re-installation of 5 revised pumps;
- Restart of PP04 and PP11.

Friday July 12th, 2019 (07:00-15:00)

Envisan and ERM installed the 2 remaining revised pumps and 1 spare pump (PP02, PP07 and PP10). The spare pump was tested but was not working, therefore the second spare pump of the container was used. There are no spare pumps left in the container anymore. The spare pump was put inside well PP02.

ERM tried to restart the system but noticed leaks at the piping outside of the container and inside well PP06. Envisan came back in the evening to fix the leaks. Due to a miscommunication, only the pipes inside the container were checked and not outside of the container. The leak inside PP06 was fixed.

Activities performed :

- Re-installation of the 2 remaining revised pumps;
- Re-installation of 1 spare pump;
- 1 spare pump broken;
- No spare pumps left in the container anymore.

Monday July 15th, 2019

Envisan fixed the leaking pipes outside of the container.

Tuesday July 16th, 2019 (10:00-12:00)

ERM restarted the rest of the extraction system (PP02, PP06, PP07 and PP10), except PP08. When PP08 was turned on, no water was arriving in the container and no flow was registered. Envisan will be on site to check and fix this one of the following days (to be communicated by Envisan).

Activities performed :

- Restart of the remaining pumps (PP02, PP06, PP07 and PP10), except PP08.

Table 1. Measured extraction well depths following the July 2019 chemical cleaning campaign

Extraction well	Measured Depth (meter below top of well pipe)	Sediments accumulation (cm)
PP02	5.76	0
PP04	5.74	0
PP05	5.10	0
PP06	4.75	0
PP07	5.63	0
PP08	7.00	0
PP09	/	n.a.
PP10	6.49	0
PP11	6.75	0

The extraction system was (partly) restarted after installation of the 8 revised pumps. Below a summary of the extraction well status after cleaning.

Table 2. Summary of remediation system (July 2019)

Extraction well	Pump in place (y/n)	Reset total volume (y/n)	Operational (y/n)
PP02	Y	Y	Y
PP04	Y	Y	Y
PP05*	Y	Y	N
PP06	Y	Y	Y

PP07	Y	Y	Y	
PP08**	Y	Y	N	
PP09*	N	N	N	
PP10	Y	Y	Y	
PP11	Y	Y	Y	

* PP05 is turned off during the construction works.

** PP08 is turned off until revision by Envisan.

*** PP09 has been abandoned and decommissioned.

Annexes

- Photo log

For the Environmental Consultant

(bodemsaneringsdeskundige)

Name and Signature

Julie Fichefet

For the Client

Name and Signature

Photo log

Picture 1: Lock out/tag out



Picture 2: Labelling pumps



Picture 3: Black precipitation PP05



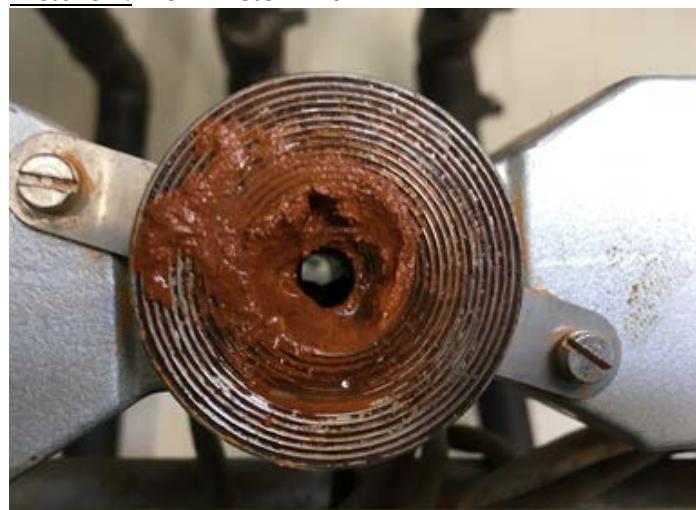
Picture 4: Black precipitation inside connection PP05



Picture 6: Non return valve PP06



Picture 7: Flow meter PP07



Picture 8: Cleaning of PP10 with vacuum trucks



Picture 9: Disconnected pipes inside container



Picture 10: Reconnected pipes and flow meters inside container



Picture 11: Cleaned non return valves PP08



Picture 12: Removal of Boresaver and Iron Clean liquid from PP07



Picture 13: Discharge of water/sludge at 3M



Picture 14: PP09 decommissioned





DAILY BOOK ENVIRONMENTAL SUPERVISION

Date: 11/10/2019

Project ID: 0458595 – 3M Belgium
OVAM ID: 732

Site Location	Weather
Canadastraat 11 2070 Zwijndrecht (Belgium)	Rain, 12°C

Distribution list	
3M	Nynke De Schutter (██████████), Charlotte Tack (██████████)
ERM	Nanda Hermes (██████████), Mattias Verbeeck (██████████), Nor Farina Nadzif (██████████), Julie Fichefet (██████████), Erik Boeckx (██████████)

Present				
Name	Function	Firm	Hour in	Hour out
Julie Fichefet	Project consultant	ERM	8:00	15:00
Erik Boeckx	Project consultant	ERM	8:00	15:00
Peter Janssens	Geoprobe drillings	BP ²	8:00	15:00
Alex Cleiren	Geoprobe drillings	BP ²	8:00	15:00

Replacement of PP09
To determine the locations for the new pumping well(s) that will replace PP09 at the Southern border of the 3M-site, BP ² performed 6 mechanical drillings with Geoprobe. The goal is to collect samples for grainsize analyses (AI West) and to find out the exact depth of the clay layer, which will define the depth of the well(s).

Figure 1: Drilling locations (overview)

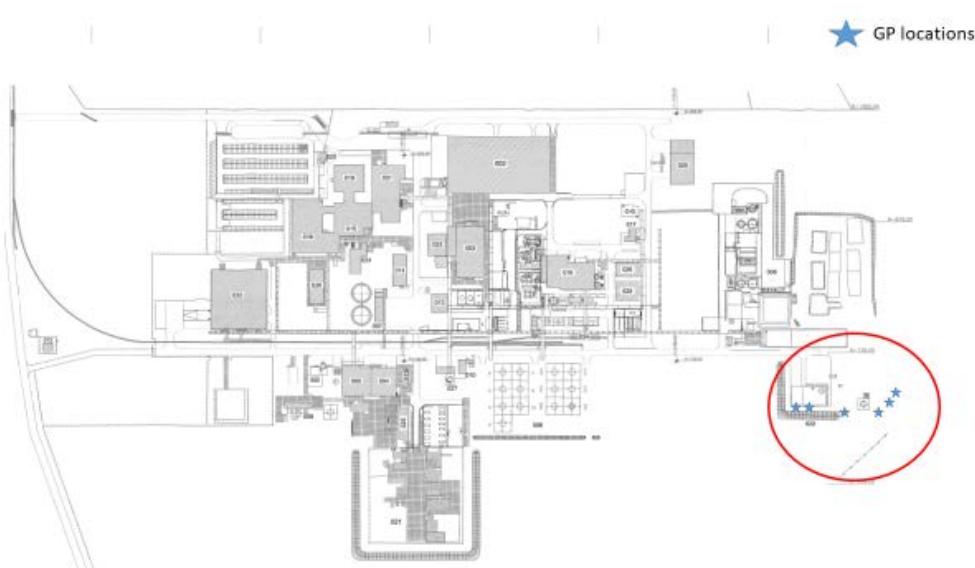
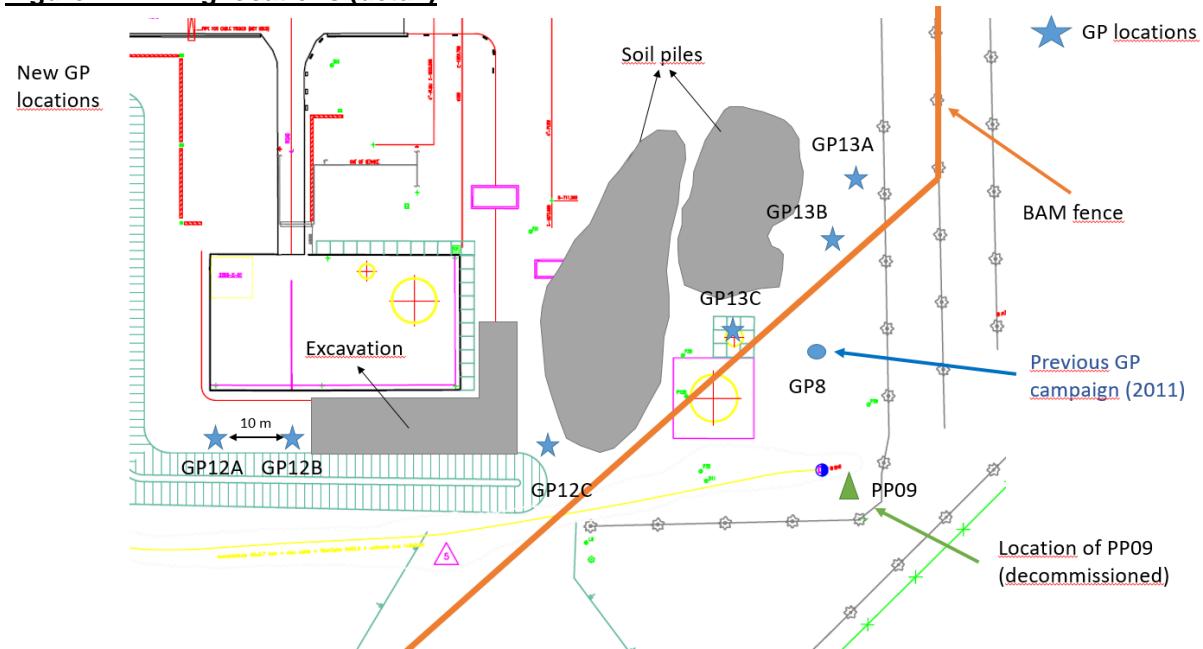


Figure 2: Drilling locations (detail)



Health & Safety

No incidents or unsafe acts/conditions have been observed.

Environmental Observation

Field observations

AT GP12A, between 2,0 and 3,5 m-gl a divergent odour (oil) and colour (black) was observed. Extra samples were taken for analyses on TPH, BTEXN and volatile oil.

Sampling and Analysis

Analyses on grain size (21) and TPH, BTEXN and volatile oil (3).

Additional remarks

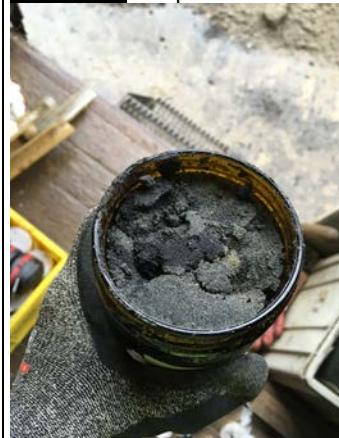
None

Photolog

Picture 1: Drilling GP12A



Picture 2: Sample from GP12A (2,0-2,5-gl)

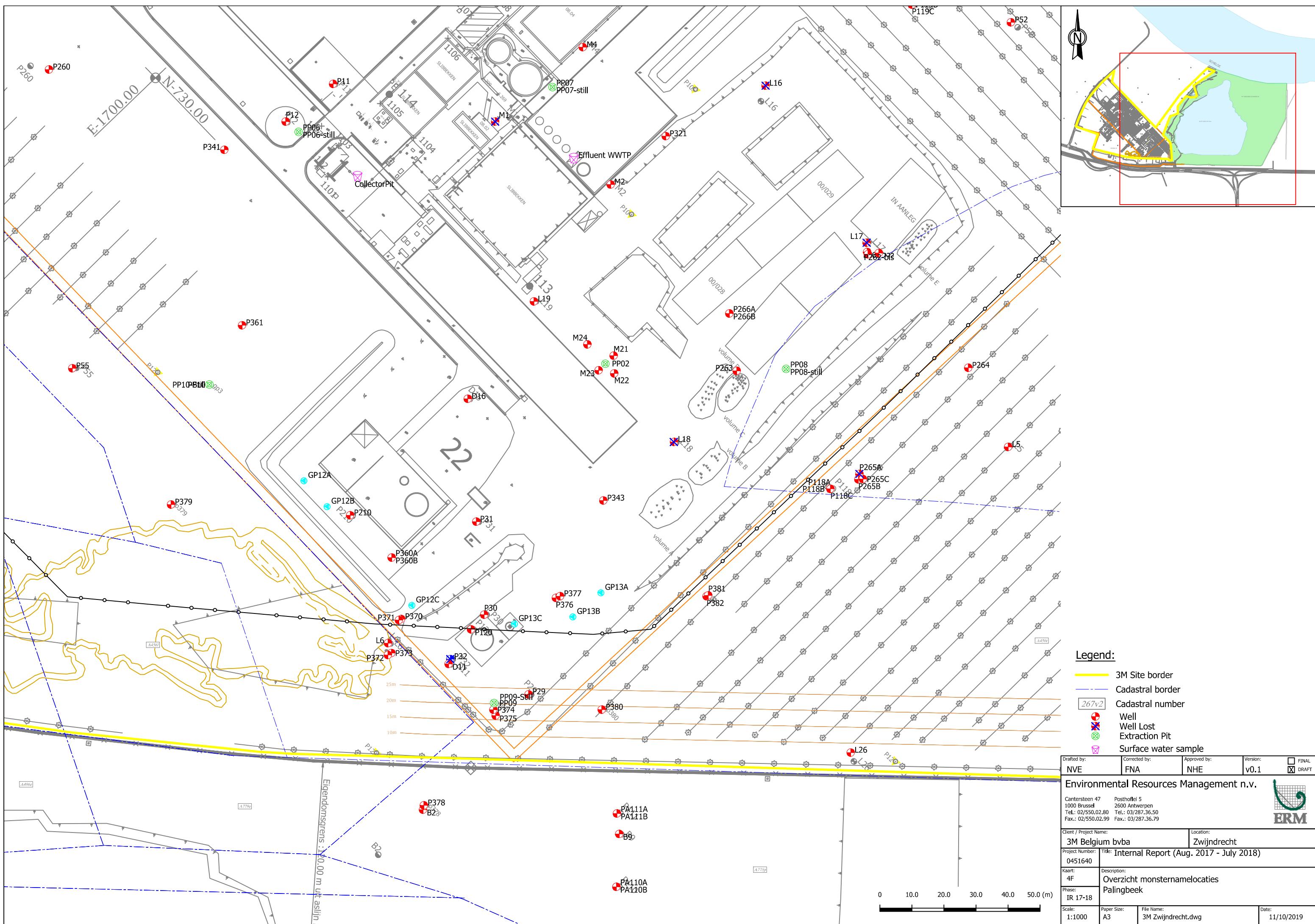


For the certified Soil expert
(bodemsaneringsdeskundige)

Name and Signature



Erik Boeckx





Code: BGP12A

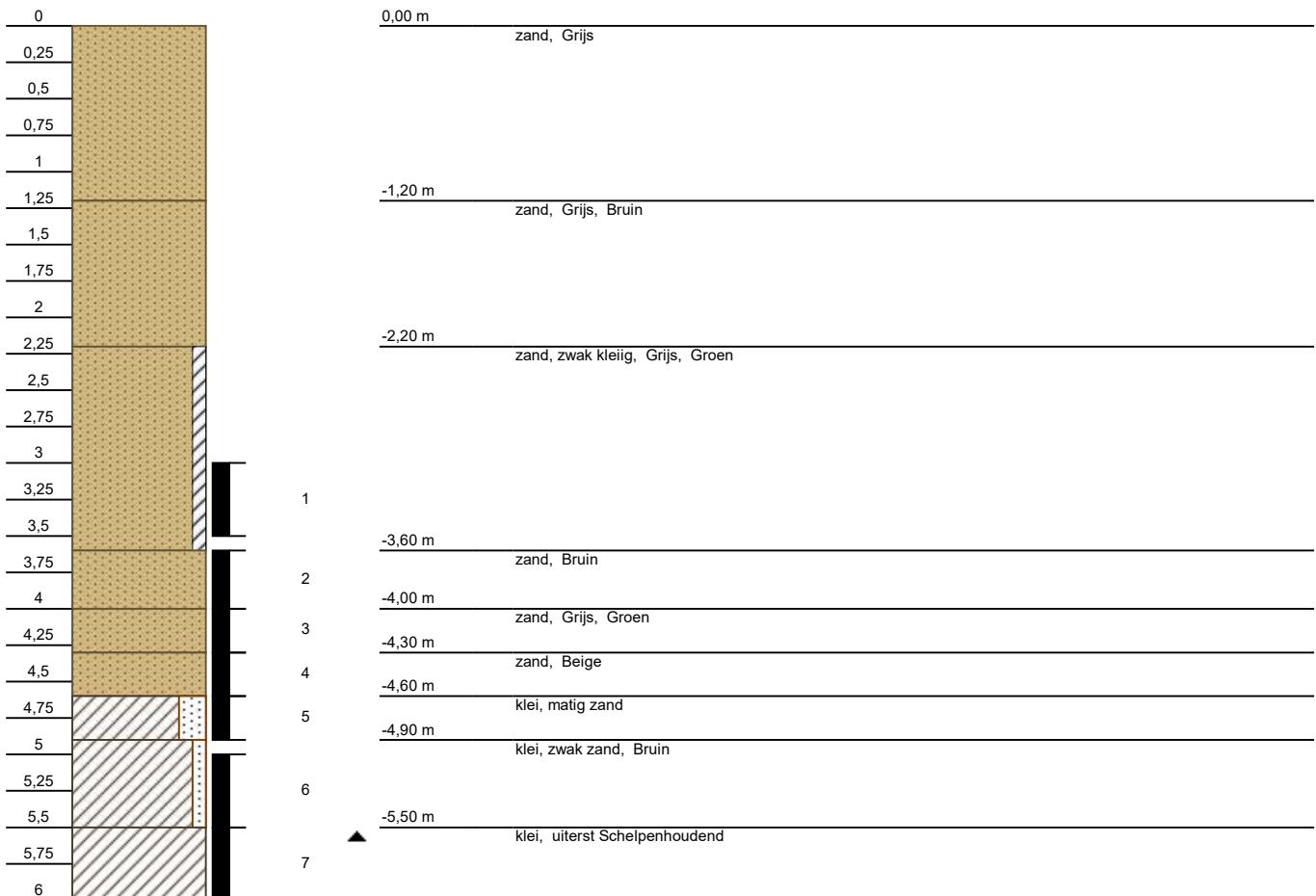
Beschrijving:

Datum: 4/10/2019

Z-Maaiveld: m

Grondwaterstand: m Diepte: 6 m

Terreinophoging: Gestaakt:



Projectcode: 0499795

Datum:

Naam:

BSD:

Projectnaam: 3M Zwijndrecht

Type: Onbekend

Geboord door:



Code: BGP12B

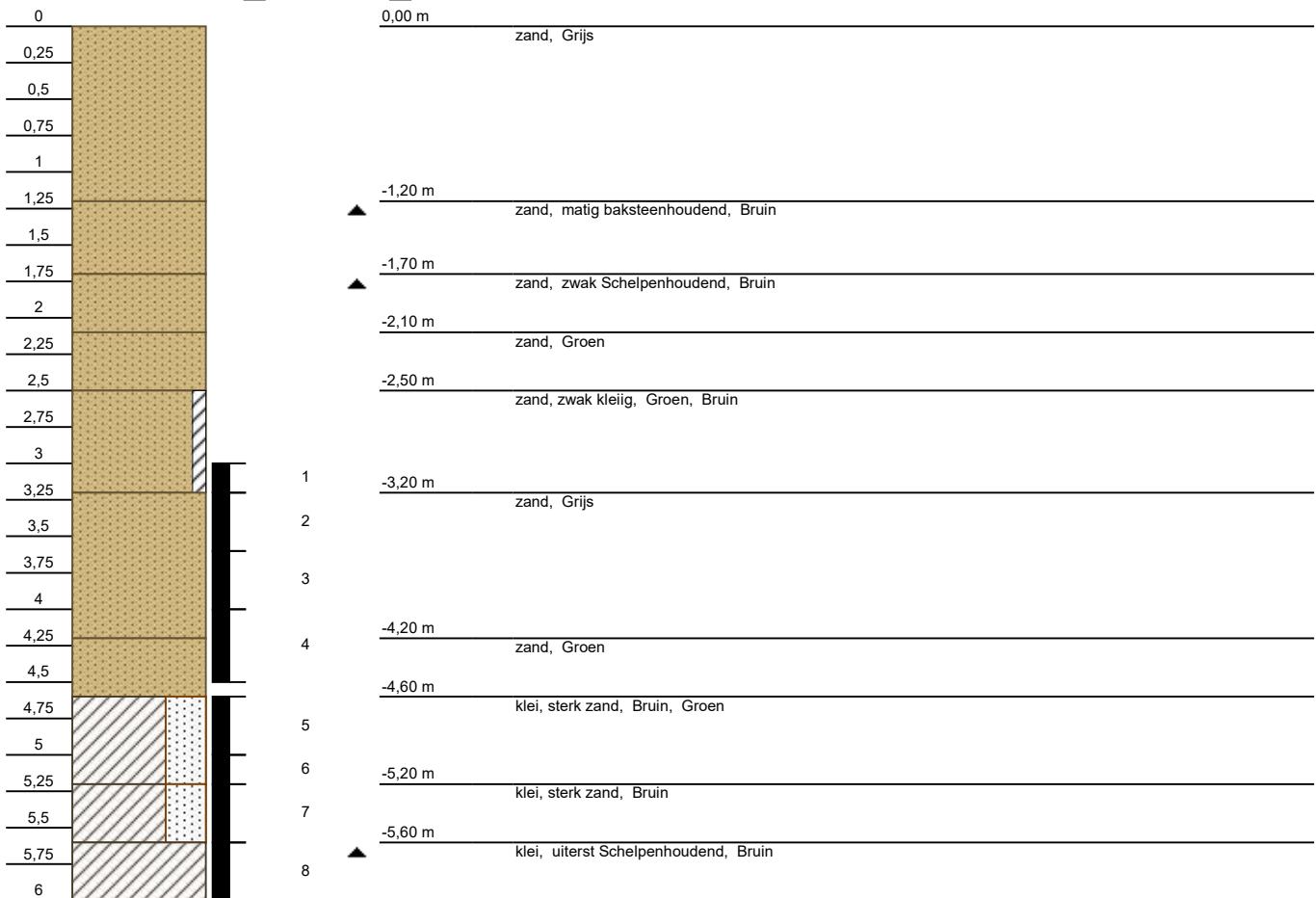
Beschrijving:

Datum: 4/10/2019

Z-Maaiveld: m

Grondwaterstand: m Diepte: 6 m

Terreinophoging: Gestaakt:



Projectcode: 0499795

Datum:

Naam:

BSD:

Projectnaam: 3M Zwijndrecht

Type: Onbekend

Geboord door:



Code: BGP12C

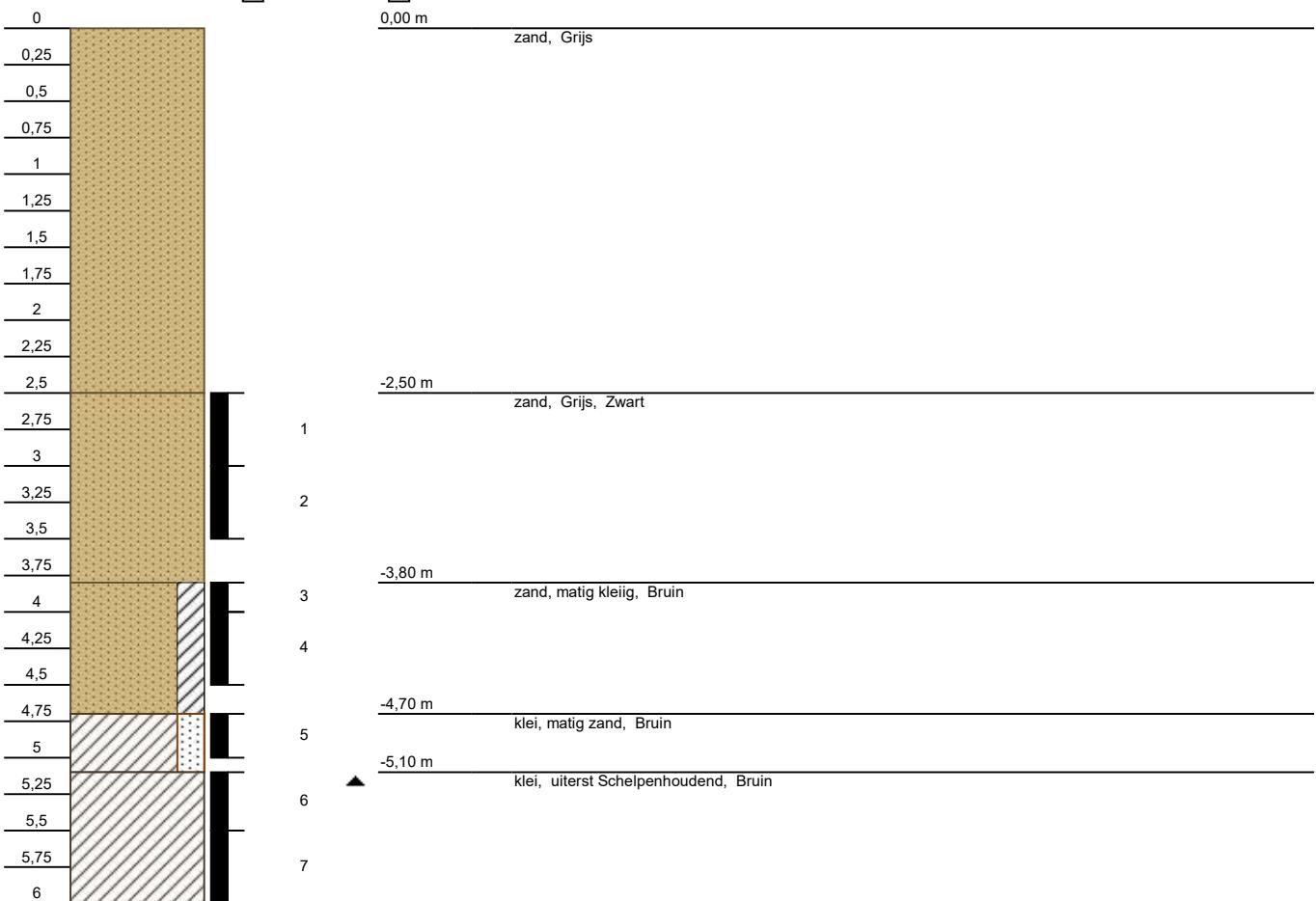
Beschrijving:

Datum: 4/10/2019

Z-Maaiveld: m

Grondwaterstand: m Diepte: 6 m

Terreinophoging: Gestaakt:



Projectcode: 0499795

Datum:

Naam:

BSD:

Projectnaam: 3M Zwijndrecht

Type: Onbekend

Geboord door:



Code: BGP13A

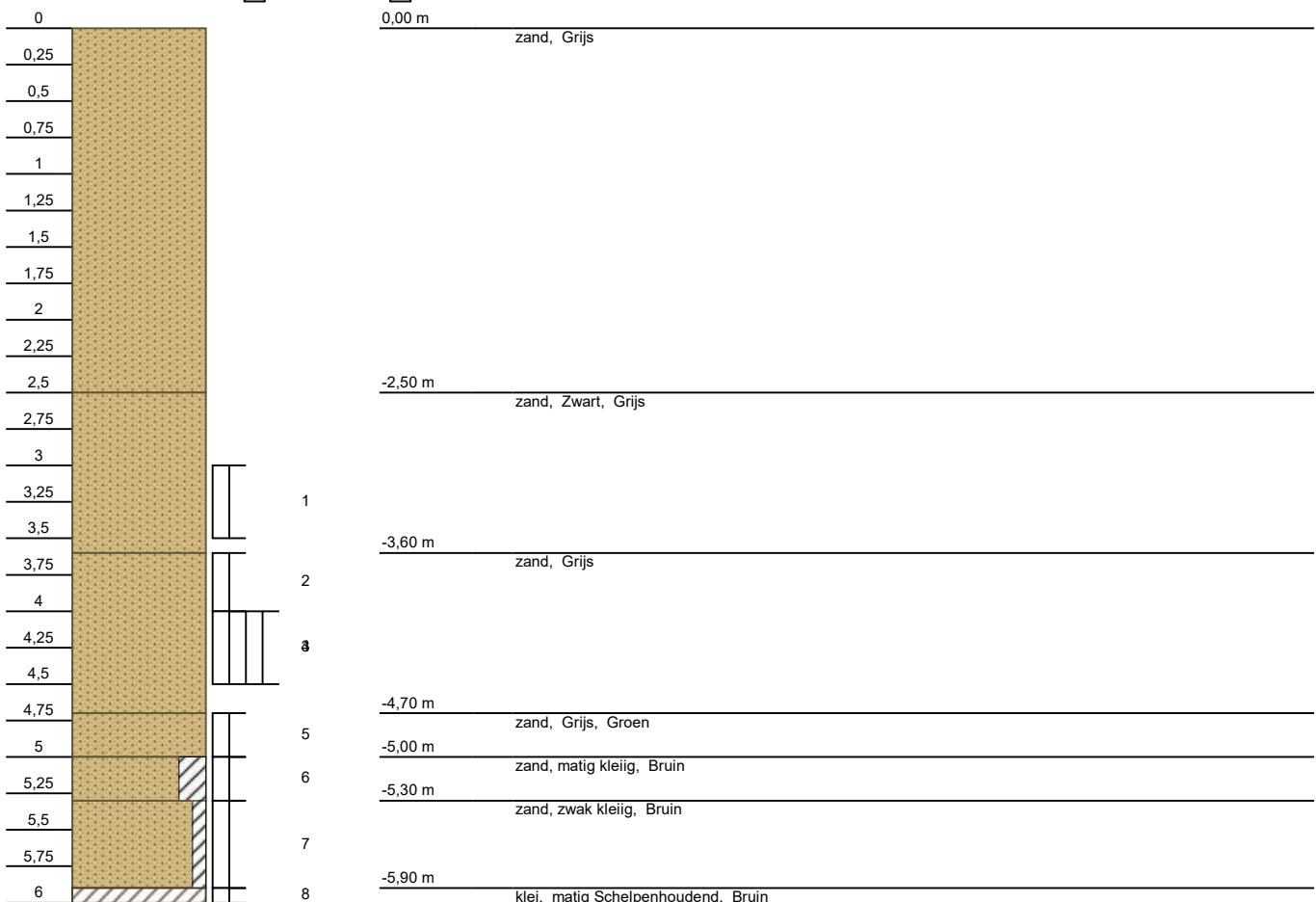
Beschrijving:

Datum: 4/10/2019

Z-Maaiveld: m

Grondwaterstand: m Diepte: 6 m

Terreinophoging: Gestaakt:



Projectcode: 0499795

Datum:

Naam:

BSD:

Projectnaam: 3M Zwijndrecht

Type: Onbekend

Geboord door:



Code: BGP13B

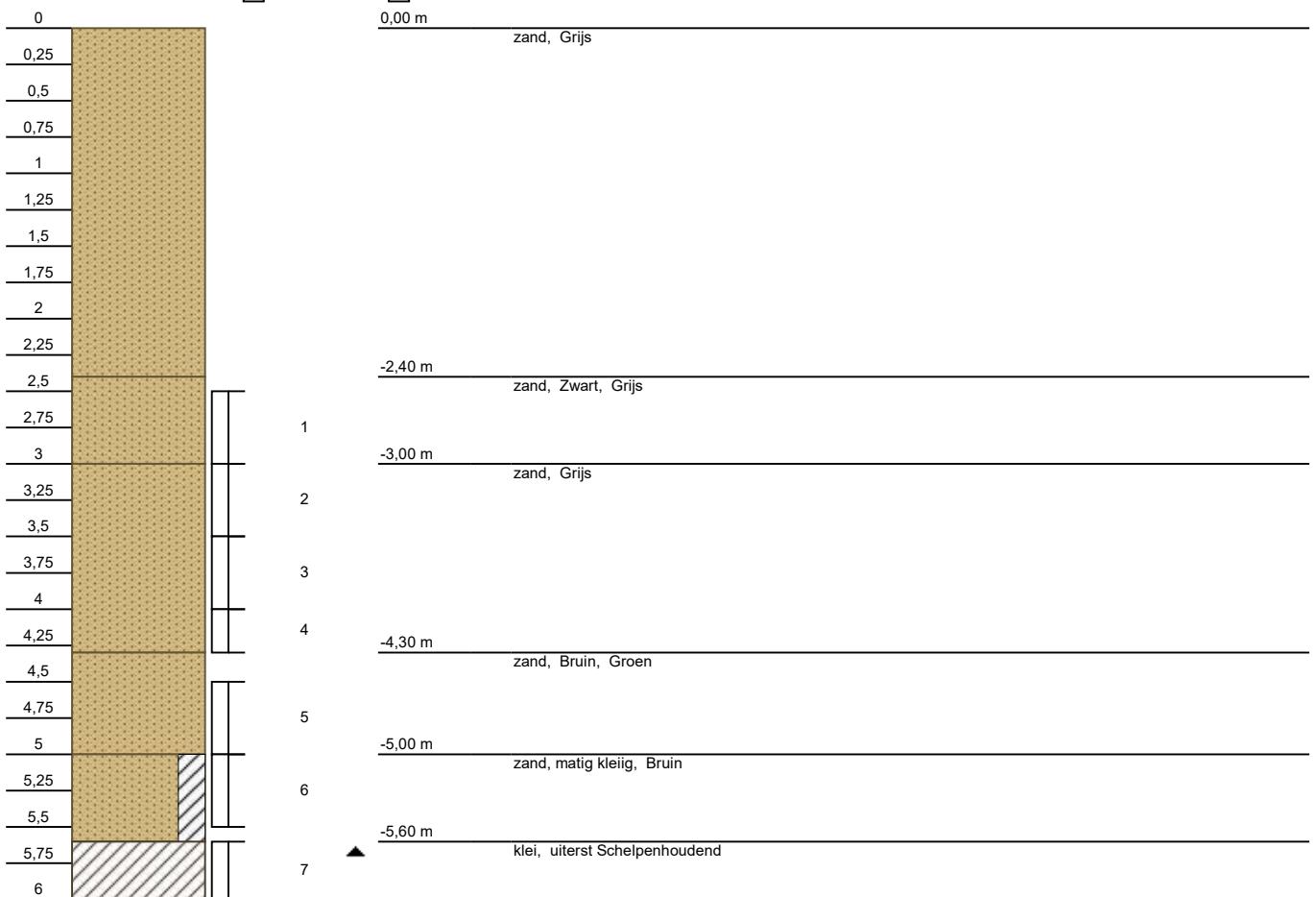
Beschrijving:

Datum: 4/10/2019

Z-Maaiveld: m

Grondwaterstand: m Diepte: 6 m

Terreinophoging: Gestaakt:



Projectcode: 0499795

Datum:

Naam:

BSD:

Projectnaam: 3M Zwijndrecht

Type: Onbekend

Geboord door:



Code: BGP13C

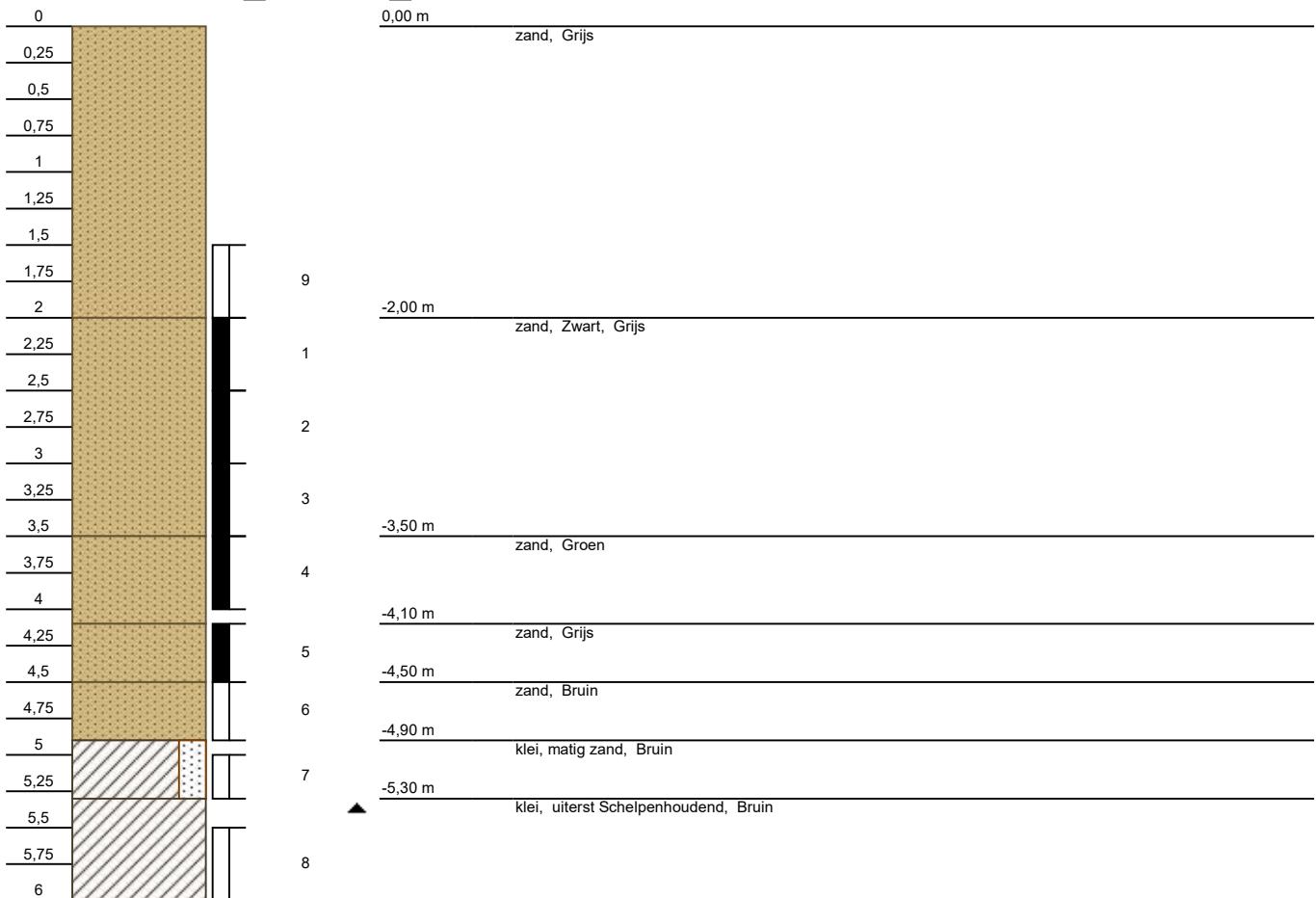
Beschrijving:

Datum: 4/10/2019

Z-Maaiveld: m

Grondwaterstand: m Diepte: 6 m

Terreinophoging: Gestaakt:



Projectcode: 0499795

Datum:

Naam:

BSD:

Projectnaam: 3M Zwijndrecht

Type: Onbekend

Geboord door:

AL-West B.V.

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Environmental Resource Management N.V. Belgium
Julie Fichefet
Cantersteen 47
1000 BRUSSEL
BELGIQUE

Datum 15.10.2019
Relatienr. 35005802
Opdrachtnr. 888434

ANALYSERAPPORT

Opdracht 888434 Bodem / Eluaat

Opdrachtgever 35005802 Environmental Resource Management N.V. Belgium
Uw referentie 0499795 3M SOW2019
Opdrachtacceptatie 07.10.19
Monsternemer Opdrachtgever

Geachte heer, mevrouw,

Hierbij zenden wij u de resultaten van het door u aangevraagde laboratoriumonderzoek.
De analyses zijn, tenzij anders vermeld, geaccrediteerd volgens NEN-EN-ISO/IEC 17025 en uitgevoerd overeenkomstig de onderzoeksmethoden die worden genoemd in de meest actuele versie van onze verrichtingenlijst van de Raad voor Accreditatie, accreditatienummer L005.

AL-West is erkend volgens VLAREL als laboratorium voor het uitvoeren van analyses in bodem, grondwater en afvalstoffen door de OVAM. In het rapport staat aangegeven welke analyses onder deze erkenning zijn uitgevoerd.

De parameter-specifieke meetonzekerheid en informatie over de berekeningsmethode zijn op aanvraag beschikbaar.

Dit rapport mag alleen in zijn geheel worden gereproduceerd. Indien u nog vragen heeft of aanvullende informatie wenst, verzoeken wij u om contact op te nemen met Klantenservice.

Wij vertrouwen erop u met de toegezonden informatie van dienst te zijn.

Met vriendelijke groet,

AL-West B.V. Dhr. Wouter Wanders, Tel. [REDACTED]
Klantenservice

AL-West B.V.

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Opdracht 888434 Bodem / Eluaat

Monsternr.	Monstername	Monsteromschrijving
423000	04.10.2019	BGP12A (300-350)
423001	04.10.2019	BGP12A (360-400)
423002	04.10.2019	BGP12A (430-460)
423003	04.10.2019	BGP12B (320-360)
423004	04.10.2019	BGP12B (400-450)

Eenheid	423000 BGP12A (300-350)	423001 BGP12A (360-400)	423002 BGP12A (430-460)	423003 BGP12B (320-360)	423004 BGP12B (400-450)
---------	----------------------------	----------------------------	----------------------------	----------------------------	----------------------------

Algemene monstervoorbehandeling

R3 Gewicht zeefffractie >4mm	%	<0,1	<0,1	<0,1	<0,1	<0,1
R3 Droge stof	%	80,8	82,8	83,4	82,7	81,3

Fracties (pipet)

Fractie < 2 µm	% md	5,4	3,5	4,0	2,7	2,1
Fractie < 16 µm	% md	10	6,5	4,6	5,6	2,6
Fractie < 32 µm	% md	18	11	5,1	9,3	2,7
Fractie < 50 µm	% md	26	20	5,5	20	3,2
Fractie < 63 µm	% md	29	31	5,8	27	3,6
Fractie < 125 µm	% md	66	62	18	65	32
Fractie < 250 µm	% md	97	98	78	97	96
Fractie < 500 µm	% md	99	99	99	99	99
Fractie < 1000 µm	% md	99	99	100	99	99
Fractie < 2000 µm	% md	99	100	100	100	99
Fractie < 2 µm	% Ds	5,3	3,4	4,0	2,7	2,1
Fractie < 16 µm	% Ds	9,9	6,3	4,6	5,4	2,7
Fractie > 2000 µm	% Ds	0,11 *	0,16 *	0,21 *	0,33 *	0,16 *

Klassiek Chemische Analyses

R3 Gloeiverlies (organische stof)	% Ds	1,3	0,7	0,7	0,7	0,4
Calciet (CaCO3)	% Ds	<1,0 *	<1,0 *	<1,0 *	<1,0 *	<1,0 *

Aromaten

R3 Benzeen	mg/kg Ds	--	--	--	--	--
R3 Tolueen	mg/kg Ds	--	--	--	--	--
R3 Ethylbenzeen	mg/kg Ds	--	--	--	--	--
R3 m,p-Xyleen	mg/kg Ds	--	--	--	--	--
R3 o-Xyleen	mg/kg Ds	--	--	--	--	--
Naftaleen	mg/kg Ds	--	--	--	--	--
R3 Som Xylenen	mg/kg Ds	--	--	--	--	--

Minerale olie (CMA)

R3 Koolwaterstoffractie C10-C40	mg/kg Ds	--	--	--	--	--
R3 Koolwaterstoffractie C10-C12	mg/kg Ds	--	--	--	--	--
R3 Koolwaterstoffractie C12-C20	mg/kg Ds	--	--	--	--	--
R3 Koolwaterstoffractie C20-C30	mg/kg Ds	--	--	--	--	--
R3 Koolwaterstoffractie C30-C40	mg/kg Ds	--	--	--	--	--

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Opdracht 888434 Bodem / Eluaat

Monsternr.	Monstername	Monsteromschrijving
423005	04.10.2019	BGP12B (460-500)
423006	04.10.2019	BGP12C (300-350)
423007	04.10.2019	BGP12C (400-450)
423008	04.10.2019	BGP12C (470-500)
423009	04.10.2019	BGP13A (360-400)

Eenheid	423005 BGP12B (460-500)	423006 BGP12C (300-350)	423007 BGP12C (400-450)	423008 BGP12C (470-500)	423009 BGP13A (360-400)
---------	----------------------------	----------------------------	----------------------------	----------------------------	----------------------------

Algemene monstervoorbehandeling

R3 Gewicht zeefffractie >4mm	%	<0,1	<0,1	<0,1	<0,1	<0,1
R3 Droge stof	%	81,0	78,6	81,0	78,4	80,8

Fracties (pipet)

Fractie < 2 µm	% md	16	1,4	7,2	20	3,0
Fractie < 16 µm	% md	19	2,1	8,5	28	4,0
Fractie < 32 µm	% md	19	2,5	9,2	29	1,7
Fractie < 50 µm	% md	20	3,5	9,7	30	7,1
Fractie < 63 µm	% md	21	8,3	10	30	8,0
Fractie < 125 µm	% md	34	52	31	43	36
Fractie < 250 µm	% md	86	74	84	90	98
Fractie < 500 µm	% md	99	100	98	99	99
Fractie < 1000 µm	% md	100	100	99	100	99
Fractie < 2000 µm	% md	100	100	100	100	99
Fractie < 2 µm	% Ds	15	1,3	7,2	20	3,0
Fractie < 16 µm	% Ds	18	2,0	8,5	27	4,0
Fractie > 2000 µm	% Ds	1,1 *	0,11 *	0,40 *	0,76 *	0,30 *

Klassiek Chemische Analyses

R3 Gloeiverlies (organische stof)	% Ds	1,7	0,7	0,7	1,8	0,8
Calciet (CaCO ₃)	% Ds	<1,0 *	4,5 *	<1,0 *	<1,0 *	<1,0 *

Aromaten

R3 Benzeen	mg/kg Ds	--	--	--	--	--
R3 Tolueen	mg/kg Ds	--	--	--	--	--
R3 Ethylbenzeen	mg/kg Ds	--	--	--	--	--
R3 m,p-Xyleen	mg/kg Ds	--	--	--	--	--
R3 o-Xyleen	mg/kg Ds	--	--	--	--	--
Naftaleen	mg/kg Ds	--	--	--	--	--
R3 Som Xylenen	mg/kg Ds	--	--	--	--	--

Minerale olie (CMA)

R3 Koolwaterstoffractie C10-C40	mg/kg Ds	--	--	--	--	--
R3 Koolwaterstoffractie C10-C12	mg/kg Ds	--	--	--	--	--
R3 Koolwaterstoffractie C12-C20	mg/kg Ds	--	--	--	--	--
R3 Koolwaterstoffractie C20-C30	mg/kg Ds	--	--	--	--	--
R3 Koolwaterstoffractie C30-C40	mg/kg Ds	--	--	--	--	--

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Opdracht 888434 Bodem / Eluaat

Monsternr.	Monstername	Monsteromschrijving
423010	04.10.2019	BGP13A (470-500)
423011	04.10.2019	BGP13A (530-590)
423012	04.10.2019	BGP13B (300-350)
423013	04.10.2019	BGP13B (400-430)
423014	04.10.2019	BGP13B (500-550)

Eenheid	423010 BGP13A (470-500)	423011 BGP13A (530-590)	423012 BGP13B (300-350)	423013 BGP13B (400-430)	423014 BGP13B (500-550)
---------	----------------------------	----------------------------	----------------------------	----------------------------	----------------------------

Algemene monstervoorbehandeling

R3 Gewicht zeefffractie >4mm	%	<0,1	<0,1	<0,1	<0,1	<0,1
R3 Droge stof	%	80,8	74,3	80,7	78,9	81,2

Fracties (pipet)

Fractie < 2 µm	% md	7,4	21	6,0	3,5	23
Fractie < 16 µm	% md	8,5	27	8,7	4,2	30
Fractie < 32 µm	% md	9,0	34	13	5,5	33
Fractie < 50 µm	% md	9,6	30	16	8,1	34
Fractie < 63 µm	% md	10	31	20	10	34
Fractie < 125 µm	% md	47	39	56	42	43
Fractie < 250 µm	% md	98	92	95	96	85
Fractie < 500 µm	% md	99	99	98	99	98
Fractie < 1000 µm	% md	100	100	99	99	100
Fractie < 2000 µm	% md	100	100	99	99	100
Fractie < 2 µm	% Ds	7,3	20	5,8	3,4	23
Fractie < 16 µm	% Ds	8,5	26	8,5	4,1	30
Fractie > 2000 µm	% Ds	0,26 *	0,83 *	0,20 *	<0,10 *	0,92 *

Klassiek Chemische Analyses

R3 Gloeiverlies (organische stof)	% Ds	1,1	2,2	1,1	1,3	1,6
Calciet (CaCO ₃)	% Ds	<1,0 *	<1,0 *	<1,0 *	<1,0 *	<1,0 *

Aromaten

R3 Benzeen	mg/kg Ds	--	--	--	--	--
R3 Tolueen	mg/kg Ds	--	--	--	--	--
R3 Ethylbenzeen	mg/kg Ds	--	--	--	--	--
R3 m,p-Xyleen	mg/kg Ds	--	--	--	--	--
R3 o-Xyleen	mg/kg Ds	--	--	--	--	--
Naftaleen	mg/kg Ds	--	--	--	--	--
R3 Som Xylenen	mg/kg Ds	--	--	--	--	--

Minerale olie (CMA)

R3 Koolwaterstoffractie C10-C40	mg/kg Ds	--	--	--	--	--
R3 Koolwaterstoffractie C10-C12	mg/kg Ds	--	--	--	--	--
R3 Koolwaterstoffractie C12-C20	mg/kg Ds	--	--	--	--	--
R3 Koolwaterstoffractie C20-C30	mg/kg Ds	--	--	--	--	--
R3 Koolwaterstoffractie C30-C40	mg/kg Ds	--	--	--	--	--

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Opdracht 888434 Bodem / Eluaat

Monsternr.	Monstername	Monsteromschrijving
423015	04.10.2019	BGP13C (200-250)
423016	04.10.2019	BGP13C (300-350)
423017	04.10.2019	BGP13C (350-400)
423018	04.10.2019	BGP13C (410-450)
423019	04.10.2019	BGP13C (500-530)

Eenheid	423015 BGP13C (200-250)	423016 BGP13C (300-350)	423017 BGP13C (350-400)	423018 BGP13C (410-450)	423019 BGP13C (500-530)
---------	----------------------------	----------------------------	----------------------------	----------------------------	----------------------------

Algemene monstervoorbehandeling

R3 Gewicht zeefffractie >4mm	%	<0,1	<0,1	<0,1	<0,1	<0,1
R3 Droge stof	%	75,5	82,1	82,3	82,4	72,8

Fracties (pipet)

Fractie < 2 µm	% md	--	<0,50	--	1,2	17
Fractie < 16 µm	% md	--	0,79	--	1,6	24
Fractie < 32 µm	% md	--	0,79	--	1,8	24
Fractie < 50 µm	% md	--	1,0	--	3,9	27
Fractie < 63 µm	% md	--	1,1	--	4,2	27
Fractie < 125 µm	% md	--	12	--	22	39
Fractie < 250 µm	% md	--	88	--	79	91
Fractie < 500 µm	% md	--	99	--	99	99
Fractie < 1000 µm	% md	--	100	--	99	99
Fractie < 2000 µm	% md	--	100	--	99	100
Fractie < 2 µm	% Ds	--	<0,50	--	1,2	17
Fractie < 16 µm	% Ds	--	0,76	--	1,6	24
Fractie > 2000 µm	% Ds	--	1,2 *	--	<0,10 *	16 *

Klassiek Chemische Analyses

R3 Gloeiverlies (organische stof)	% Ds	--	0,4	--	0,2	1,8
Calciet (CaCO3)	% Ds	--	2,8 *	--	<1,0 *	<1,0 *

Aromaten

R3 Benzeen	mg/kg Ds	<0,05	--	<0,05	--	--
R3 Tolueen	mg/kg Ds	<0,05	--	<0,05	--	--
R3 Ethylbenzeen	mg/kg Ds	<0,05	--	<0,05	--	--
R3 m,p-Xyleen	mg/kg Ds	<0,10	--	<0,10	--	--
R3 o-Xyleen	mg/kg Ds	<0,050	--	<0,050	--	--
Naftaleen	mg/kg Ds	<0,10	--	<0,10	--	--
R3 Som Xylenen	mg/kg Ds	n.a.	--	n.a.	--	--

Minerale olie (CMA)

R3 Koolwaterstoffractie C10-C40	mg/kg Ds	<50 *	--	<50 *	--	--
R3 Koolwaterstoffractie C10-C12	mg/kg Ds	<8 *	--	<8 *	--	--
R3 Koolwaterstoffractie C12-C20	mg/kg Ds	<12 *	--	<12 *	--	--
R3 Koolwaterstoffractie C20-C30	mg/kg Ds	<15 *	--	<15 *	--	--
R3 Koolwaterstoffractie C30-C40	mg/kg Ds	<15 *	--	<15 *	--	--

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Opdracht 888434 Bodem / Eluaat

Monsternr.	Monstername	Monsteromschrijving
423020	04.10.2019	BGP13C (150-200)

Eenheid **423020**
BGP13C (150-200)

Algemene monstervoorbehandeling

R3 Gewicht zeefffractie >4mm	%	<0,1
R3 Droge stof	%	90,6

Fracties (pipet)

Fractie < 2 µm	% md	--
Fractie < 16 µm	% md	--
Fractie < 32 µm	% md	--
Fractie < 50 µm	% md	--
Fractie < 63 µm	% md	--
Fractie < 125 µm	% md	--
Fractie < 250 µm	% md	--
Fractie < 500 µm	% md	--
Fractie < 1000 µm	% md	--
Fractie < 2000 µm	% md	--
Fractie < 2 µm	% Ds	--
Fractie < 16 µm	% Ds	--
Fractie > 2000 µm	% Ds	--

Klassiek Chemische Analyses

R3 Gloeiverlies (organische stof)	% Ds	--
Calciet (CaCO ₃)	% Ds	--

Aromaten

R3 Benzeen	mg/kg Ds	<0,05
R3 Tolueen	mg/kg Ds	<0,05
R3 Ethylbenzeen	mg/kg Ds	<0,05
R3 m,p-Xyleen	mg/kg Ds	<0,10
R3 o-Xyleen	mg/kg Ds	<0,050
Naftaleen	mg/kg Ds	<0,10
R3 Som Xylenen	mg/kg Ds	n.a.

Minerale olie (CMA)

R3 Koolwaterstoffractie C10-C40	mg/kg Ds	<50 *
R3 Koolwaterstoffractie C10-C12	mg/kg Ds	<8 *
R3 Koolwaterstoffractie C12-C20	mg/kg Ds	<12 *
R3 Koolwaterstoffractie C20-C30	mg/kg Ds	<15 *
R3 Koolwaterstoffractie C30-C40	mg/kg Ds	<15 *

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Opdracht 888434 Bodem / Eluaat

Eenheid	423000 BGP12A (300-350)	423001 BGP12A (360-400)	423002 BGP12A (430-460)	423003 BGP12B (320-360)	423004 BGP12B (400-450)
---------	----------------------------	----------------------------	----------------------------	----------------------------	----------------------------

Vluchtige verbindingen

VKF C6-C10	mg/kg Ds	--	--	--	--
------------	----------	----	----	----	----

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Opdracht 888434 Bodem / Eluaat

Eenheid	423005 BGP12B (460-500)	423006 BGP12C (300-350)	423007 BGP12C (400-450)	423008 BGP12C (470-500)	423009 BGP13A (360-400)
---------	----------------------------	----------------------------	----------------------------	----------------------------	----------------------------

Vluchtige verbindingen

VKF C6-C10	mg/kg Ds	--	--	--	--
------------	----------	----	----	----	----

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Opdracht 888434 Bodem / Eluaat

Eenheid	423010 BGP13A (470-500)	423011 BGP13A (530-590)	423012 BGP13B (300-350)	423013 BGP13B (400-430)	423014 BGP13B (500-550)
---------	----------------------------	----------------------------	----------------------------	----------------------------	----------------------------

Vluchtige verbindingen

VKF C6-C10	mg/kg Ds	--	--	--	--
------------	----------	----	----	----	----

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Opdracht 888434 Bodem / Eluaat

Eenheid	423015 BGP13C (200-250)	423016 BGP13C (300-350)	423017 BGP13C (350-400)	423018 BGP13C (410-450)	423019 BGP13C (500-530)
---------	----------------------------	----------------------------	----------------------------	----------------------------	----------------------------

Vluchtige verbindingen

VKF C6-C10	mg/kg Ds	4,0	--	<1,0	--	--
------------	----------	-----	----	------	----	----

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Opdracht 888434 Bodem / Eluaat

Eenheid **423020**
BGP13C (150-200)

Vluchtige verbindingen

VKF C6-C10	mg/kg Ds	1,9
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R3) Erkend volgens OVAM

Verklaring:< of n.a. betekent dat het gehalte van de component lager is dan de rapportagegrens.

Toelichting

- 423015 Er werd geen geschikte recipient geleverd voor de analyse van vluchtige organisch componenten.
423017 Er werd geen geschikte recipient geleverd voor de analyse van vluchtige organisch componenten.
423020 Er werd geen geschikte recipient geleverd voor de analyse van vluchtige organisch componenten.

Begin van de analyses: 08.10.2019

Einde van de analyses: 15.10.2019

De onderzoeksresultaten hebben alleen betrekking op het aangeleverde monstermateriaal. Monsters met onbekende herkomst kunnen slechts beperkt gecontroleerd worden op plausibiliteit.

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Klantenservice

Toegepaste methoden

CMA/2/II/A.1: Droge stof

CMA/2/II/A.2: Gloeiverlies (organische stof)

CMA/3/E: Benzeen Tolueen Ethylbenzeen m,p-Xyleen o-Xyleen Naftaleen Som Xylenen VKF C6-C10

CMA/3/R1: Koolwaterstoffractie C10-C40 * Koolwaterstoffractie C10-C12 * Koolwaterstoffractie C12-C20 *
Koolwaterstoffractie C20-C30 * Koolwaterstoffractie C30-C40 *

CMA/5/B.3 ; CMA/5/B.4: Gewicht zeeffractie >4mm

conform NEN 5753: Fractie > 2000 µm *

conform NEN 5753: Fractie < 2 µm Fractie < 16 µm Fractie < 32 µm Fractie < 50 µm Fractie < 63 µm Fractie < 125 µm
Fractie < 250 µm Fractie < 500 µm Fractie < 1000 µm Fractie < 2000 µm Fractie < 2 µm Fractie < 16 µm

conform NEN-ISO 10693: Calciet (CaCO₃) *

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Bijlage bij Opdrachtnr. 888434

CONSERVING, CONSERVINGSTERMIJN EN VERPAKKING

Er zijn verschillen met de richtlijnen geconstateerd die mogelijk de betrouwbaarheid van de resultaten van onderstaande monsters of analyses beïnvloeden.

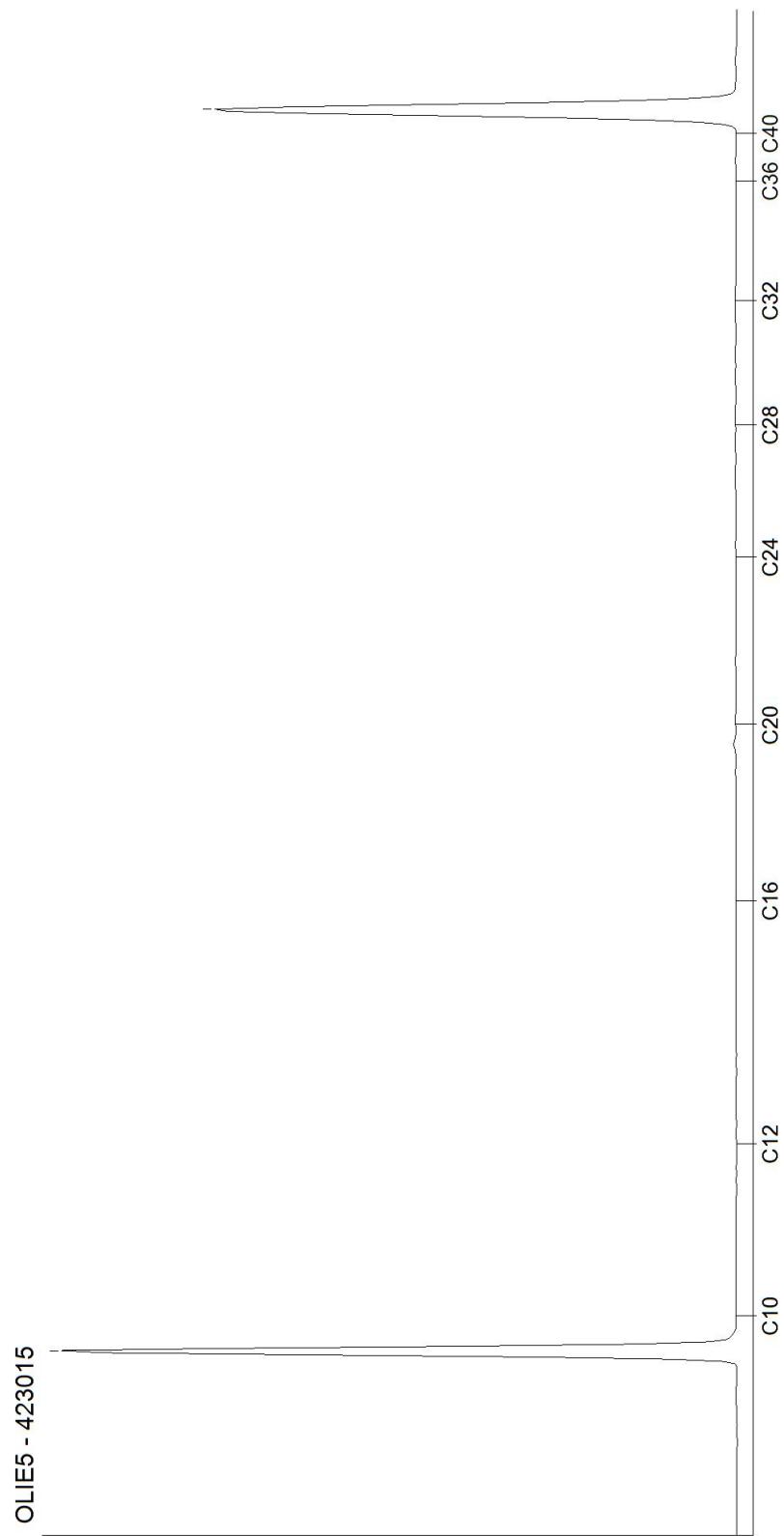
- 423015 Geen aparte monsterhouder voor de vluchtlige verbindingen aangeleverd.
- 423017 Geen aparte monsterhouder voor de vluchtlige verbindingen aangeleverd.
- 423020 Geen aparte monsterhouder voor de vluchtlige verbindingen aangeleverd.

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CHROMATOGRAM for Order No. 888434, Analysis No. 423015, created at 14.10.2019 12:58:51

Monsteromschrijving: BGP13C (200-250)



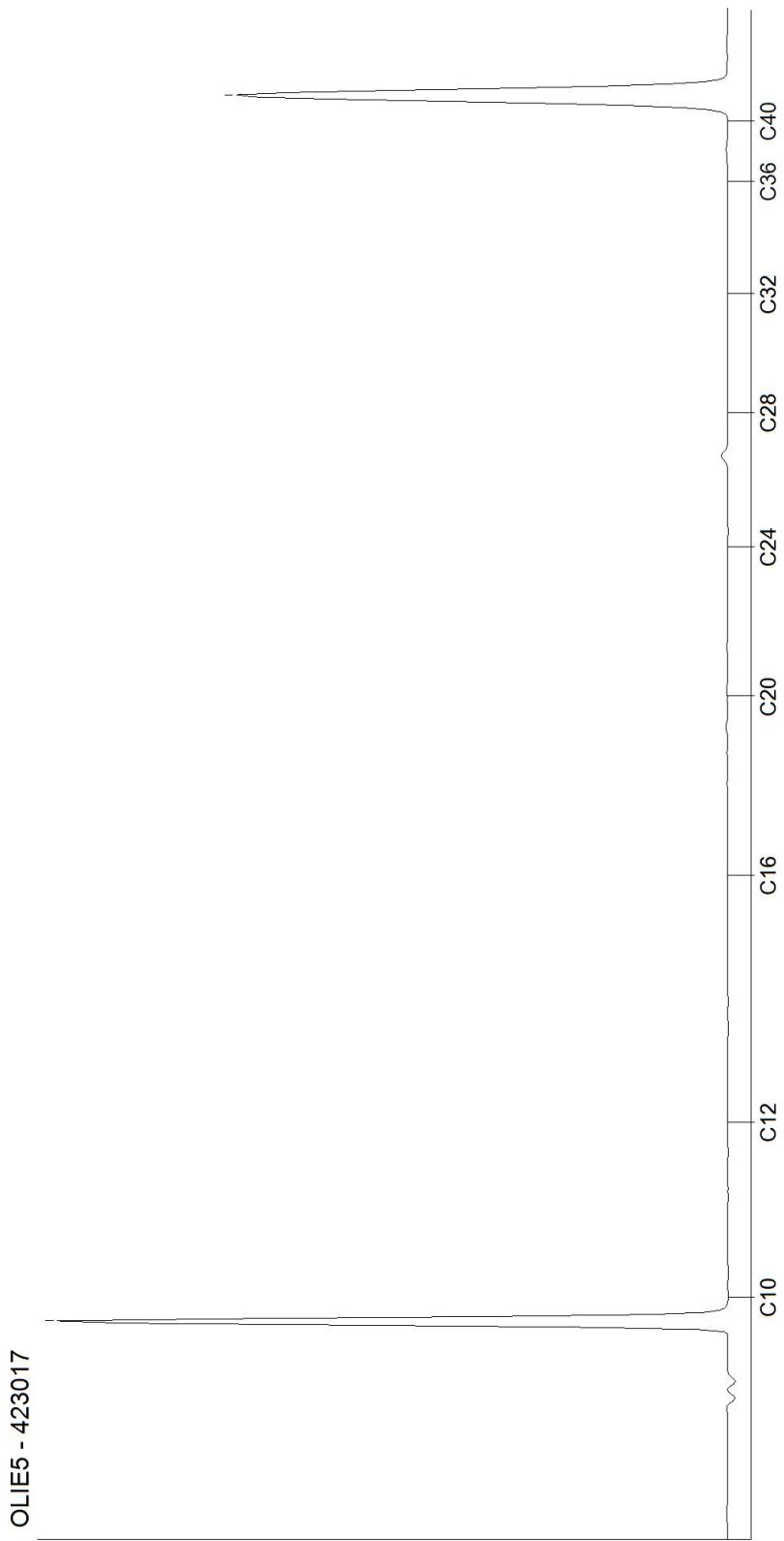
Blad 1 van 3

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CHROMATOGRAM for Order No. 888434, Analysis No. 423017, created at 15.10.2019 14:28:03

Monsteromschrijving: BGP13C (350-400)



Blad 2 van 3

Olie5 - 423017

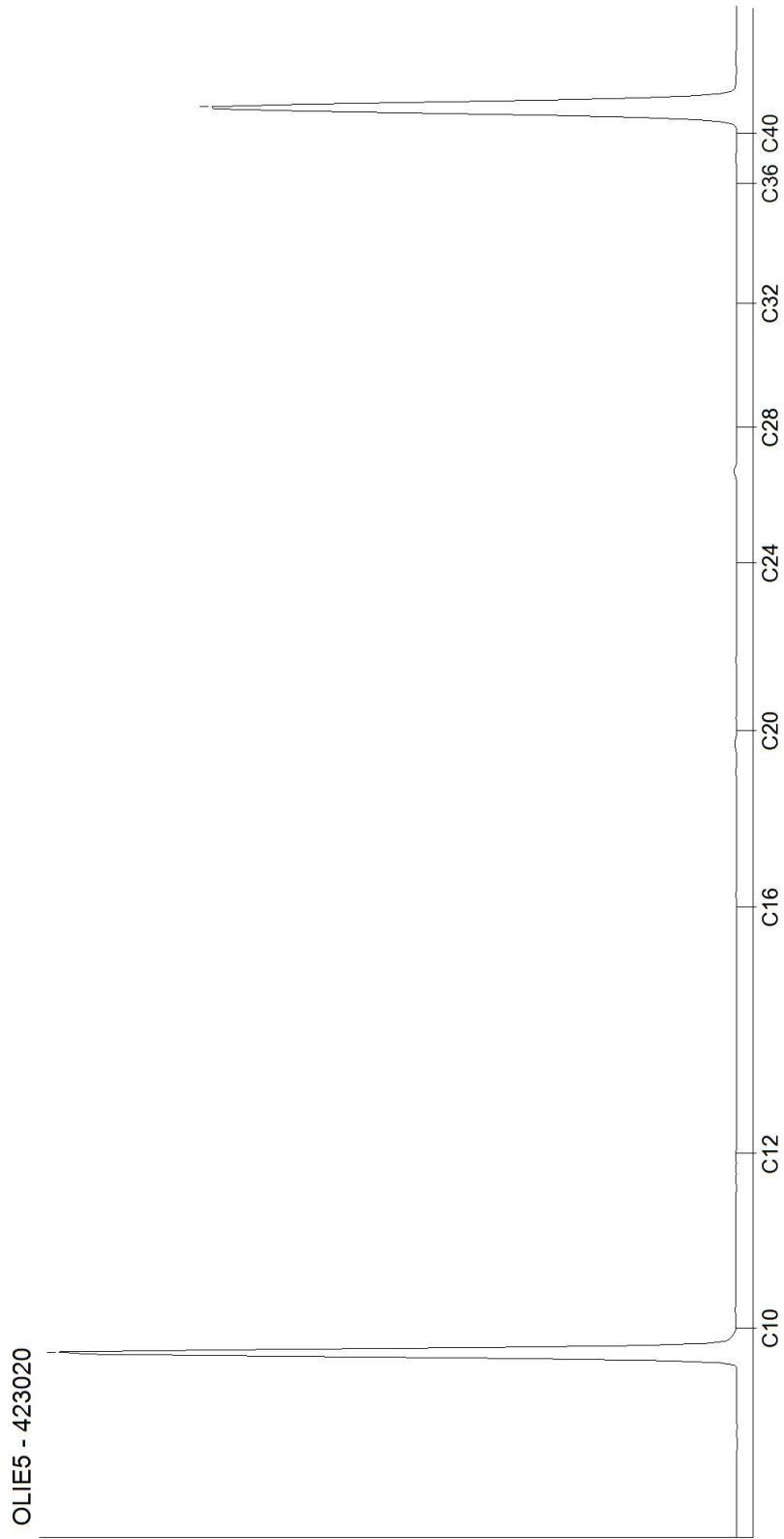
Kamer van Koophandel Directeur
Nr. 08110898 ppa. Marc van Gelder
VAT/BTW-ID-Nr.: Dr. Paul Wimmer
NL 811132559 B01

AL-West B.V.

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CHROMATOGRAM for Order No. 888434, Analysis No. 423020, created at 14.10.2019 12:58:51

Monsteromschrijving: BGP13C (150-200)



Blad 3 van 3

AL-West B.V.

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Environmental Resource Management N.V. Belgium
Julie Fichefet
Cantersteen 47
1000 BRUSSEL
BELGIQUE

Datum 17.10.2019
Relatienr. 35005802
Opdrachtnr. 888836

ANALYSERAPPORT

Opdracht 888836 Bodem / Eluaat

Opdrachtgever 35005802 Environmental Resource Management N.V. Belgium
Uw referentie 0499795 3M SOW2019
Opdrachtacceptatie 10.10.19
Monsternemer Opdrachtgever

Geachte heer, mevrouw,

Hierbij zenden wij u de resultaten van het door u aangevraagde laboratoriumonderzoek.
De analyses zijn, tenzij anders vermeld, geaccrediteerd volgens NEN-EN-ISO/IEC 17025 en uitgevoerd overeenkomstig de onderzoeksmethoden die worden genoemd in de meest actuele versie van onze verrichtingenlijst van de Raad voor Accreditatie, accreditatienummer L005.

AL-West is erkend volgens VLAREL als laboratorium voor het uitvoeren van analyses in bodem, grondwater en afvalstoffen door de OVAM. In het rapport staat aangegeven welke analyses onder deze erkenning zijn uitgevoerd.

De parameter-specifieke meetonzekerheid en informatie over de berekeningsmethode zijn op aanvraag beschikbaar.

Dit rapport mag alleen in zijn geheel worden gereproduceerd. Indien u nog vragen heeft of aanvullende informatie wenst, verzoeken wij u om contact op te nemen met Klantenservice.

Wij vertrouwen erop u met de toegezonden informatie van dienst te zijn.

Met vriendelijke groet,

AL-West B.V. Dhr. Wouter Wanders, Tel. [REDACTED]
Klantenservice

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Opdracht 888836 Bodem / Eluaat

Monsternr.	Monstername	Monsteromschrijving
424870	04.10.2019	BGP13C (200-250)
424871	04.10.2019	BGP13C (350-400)
424872	04.10.2019	BGP13C (150-200)

Eenheid	424870	424871	424872
BGP13C (200-250)		BGP13C (350-400)	BGP13C (150-200)

Algemene monstervoorbehandeling

R3 Gewicht zeeffracatie >4mm	%	<0,1	<0,1	<0,1
R3 Droge stof	%	79,4	80,4	90,4

Fracties (pipet)

R3 Fractie < 2 µm	%	4,4 *	7,9 *	3,5 *
-------------------	---	-------	-------	-------

Klassiek Chemische Analyses

R3 Organisch koolstof (OVAM)	g/kg Ds	2,0 *	<1,0 *	<1,0 *
R3 Organische Stof (OVAM)	% Ds	0,34 *	<0,10 * x)	<0,10 * x)

x) Gehaltes beneden de rapportagegrens zijn niet mee ingegeven.

R3) Erkend volgens OVAM

Verklaring:<" of n.a. betekent dat het gehalte van de component lager is dan de rapportagegrens.

Begin van de analyses: 10.10.2019

Einde van de analyses: 17.10.2019

De onderzoeksresultaten hebben alleen betrekking op het aangeleverde monstermateriaal . Monsters met onbekende herkomst kunnen slechts beperkt gecontroleerd worden op plausibiliteit .

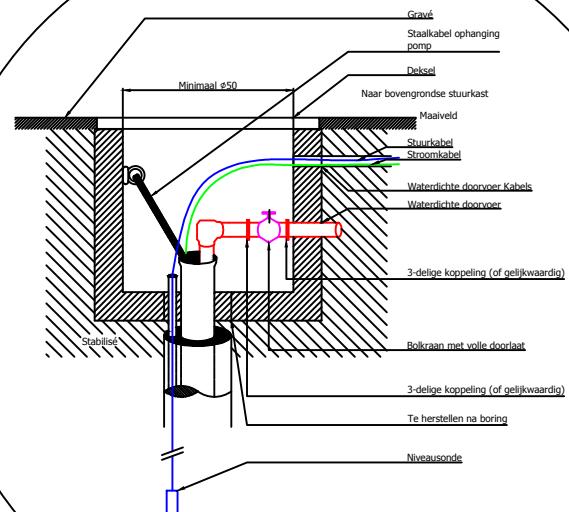
AL-West B.V. Dhr. Wouter Wanders, Tel. +31/570788115
Klantenservice

Toegepaste methoden

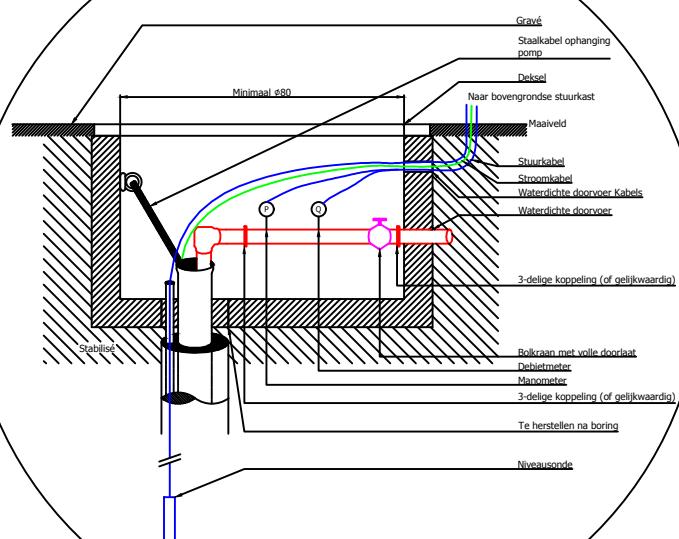
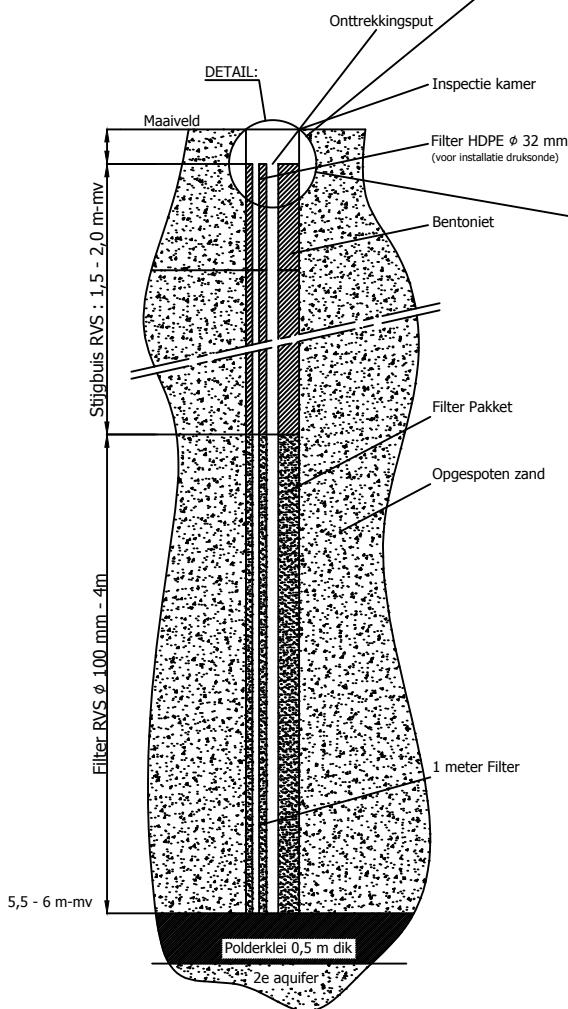
- CMA/2/II/A.1: Droge stof
CMA/2/II/A.6: Fractie < 2 µm *
CMA/2/II/A.7: Organisch koolstof (OVAM) * Organische Stof (OVAM) *
CMA/5/B.3 ; CMA/5/B.4: Gewicht zeeffracatie >4mm

BIJLAGE 3

**SCHEMATISCHE WEERGAVE POMPPUTTEN
INSPECTIEKAMER**



Zone WWTP



Zone gebouw 16

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:
PPR	RMA	NHE	v0.1
Environmental Resources Management n.v.			
Cantersteen 47 1000 Brussel Tel.: 02/550.02.80 Fax.: 02/550.02.99	Posthoflei 5 2600 Antwerpen Tel.: 03/287.36.50 Fax.: 03/287.36.79	Place de l'Université 16 1348 Louvain-la-Neuve Tel.: 010/48.35.39 Fax.: 010/48.35.36	<input type="checkbox"/> FINAL <input checked="" type="checkbox"/> DRAFT
Klant / Projectnaam: 3M Belgium bvba	Locatie: Zwijndrecht		
Projectnr.: 0341743	Titel: 7de Tussentijds verslag Bodemsaneringswerken		
Kaart: 10	Beschrijving: Doorsnede Pompput en inspectiekamer		
Fase: TTV-7			
Schaal: /-	Formaat: A4	Bestandsnaam: 3M-Doorsneden.dwg	Datum: 22/12/2016



BIJLAGE 4 MILIEUDAGBOEKEN EN RAPPORTEN CLEANINGS



Purazur

Water Treatment Solutions

Rapport

van RLO
onderwerp 4592 3M Zwijndrecht: Cleaningwerken September 2017
datum 08/10/2017
pagina's 1/8

Cleaningwerken onttrekkingsinstallatie September 2017 te 3M Zwijndrecht

Documentstatus

Actie	Naam	Functie	Handtekening
Auteur	Reinout Van Loon	Werfleider	



Purazur

Water Treatment Solutions

1. Cleaningwerken September 2017

1.1. Visuele controle en reiniging pompen

1.1.1. Pompen bij aankomst

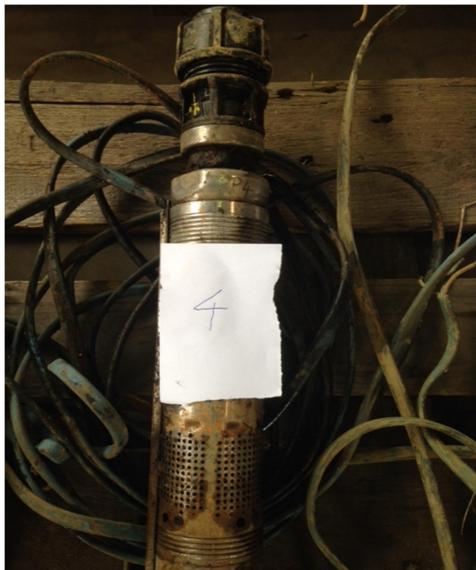
1.1.1.1. PP1



1.1.1.2. PP2



1.1.1.3. PP4



1.1.1.4. PP5



1.1.1.5. PP6



1.1.1.6. PP7



1.1.1.7. PP8



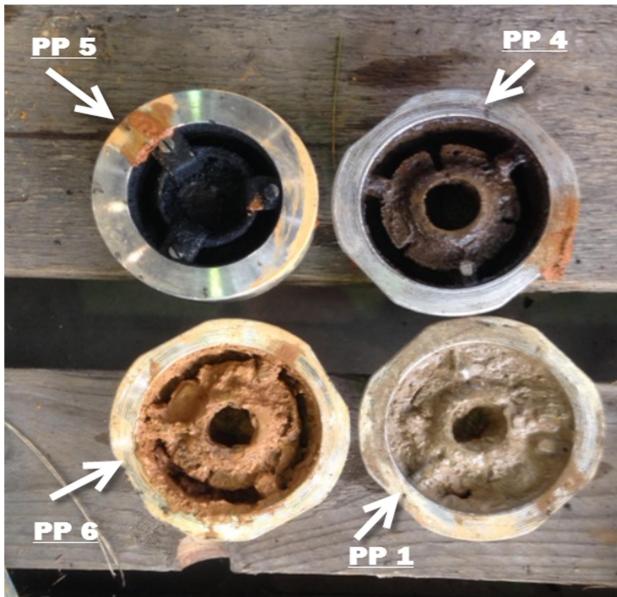
1.1.1.8. PP9



1.1.1.9. PP10



1.1.1.10. Terugslagkleppen PP1, PP4, PP5 en PP6





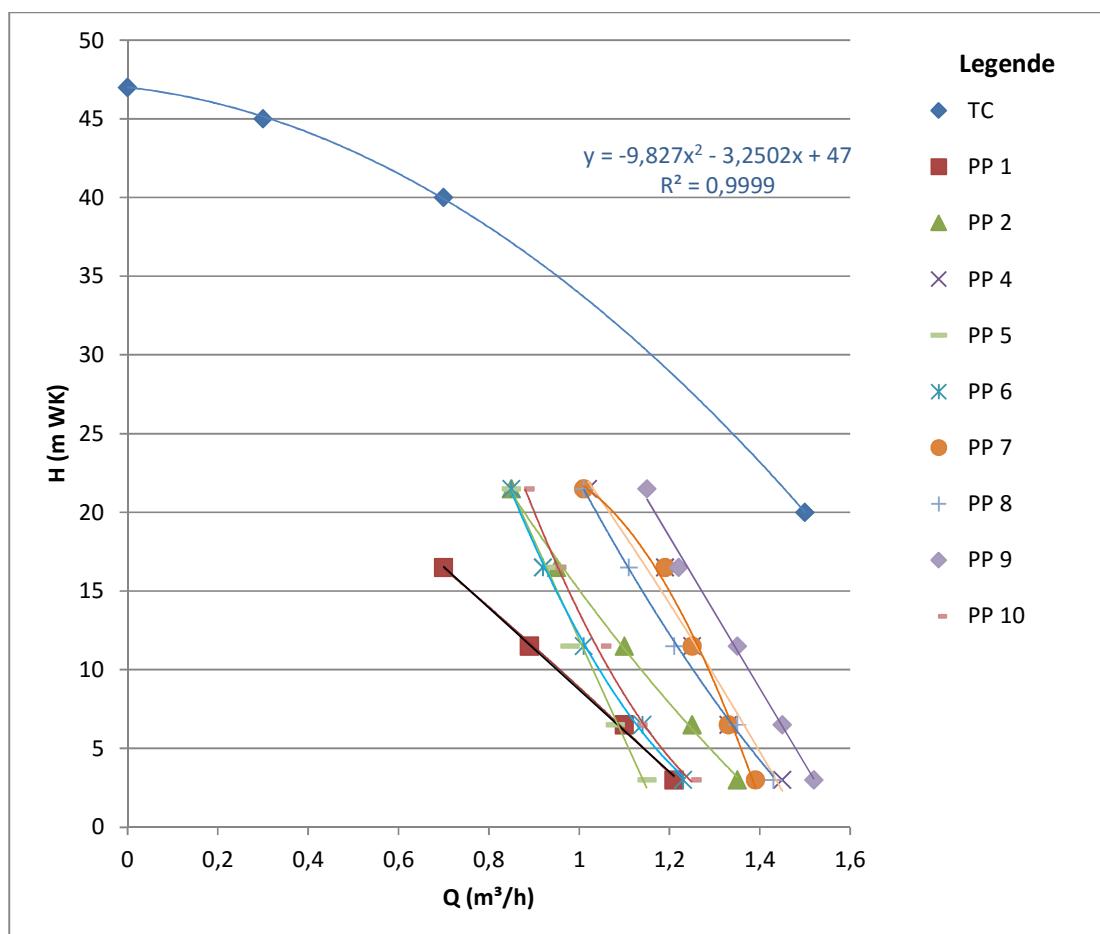
1.2. Test pompen

Na een eerste reiniging van de pompen, werden deze terug samengebouwd en hydraulisch getest. Uit deze hydraulische testen bleek dat alle motorgedeeltes en waaierhuizen nog operationeel konden zijn maar de bijhorende drukken en debieten niet in overeenstemming waren met de theoretische werkingsgrafieken van deze pompen. Daarom werden de pompen een 2^{de} maal gereinigd door elke pomp een half uur in een lichte zuuroplossing (HCl) te laten circuleren.

Na deze 2^{de} reiniging kon vastgesteld worden dat alle pompen een normale werkingsgrafiek vertoonde en deze bijgevolg alle terug werden gemonteerd op site. Bij PP7 werd bijkomend nog een kabeldoorvoer vervangen daar deze defect was.

De terugslagkleppen van PP1, PP4, PP5 en PP6 werden na reiniging alle terug gemonteerd in de desbetreffende onttrekkingsputten.

Werkingsgrafieken na een eerste reiniging:

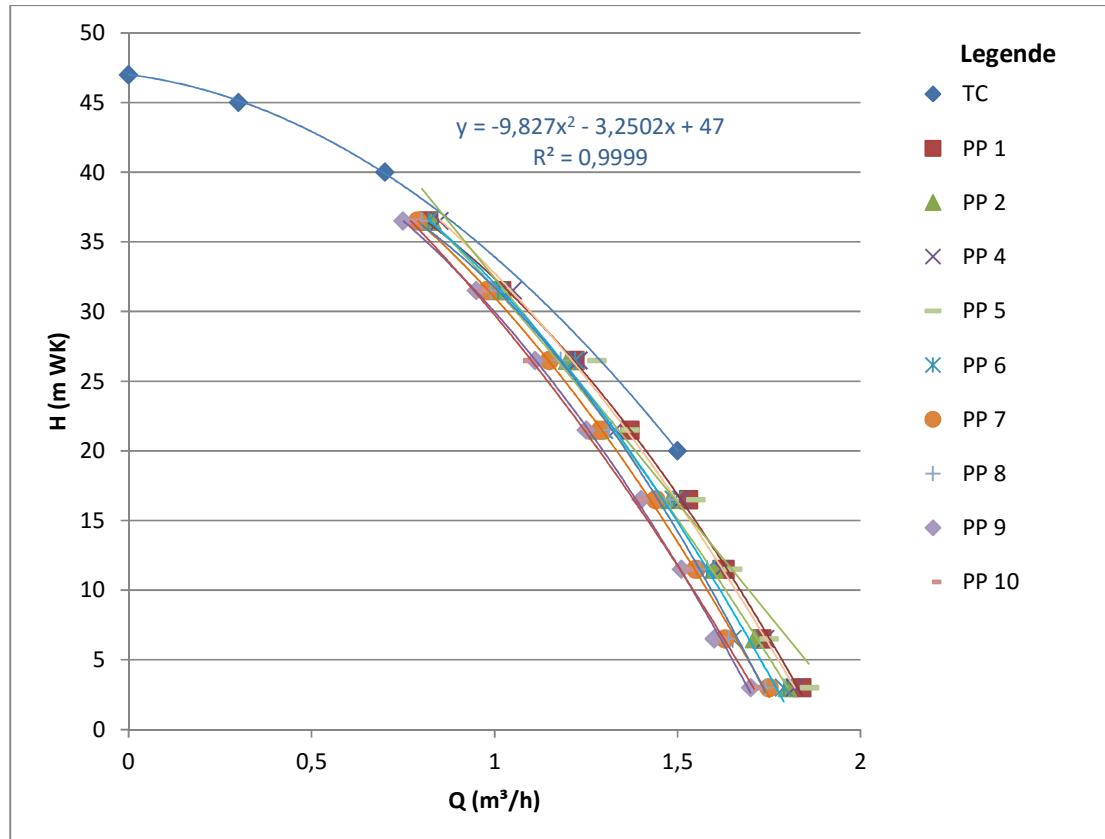




Purazur

Water Treatment Solutions

Werkingsgrafieken na de 2^{de} reiniging



Rapport

van RLO
onderwerp 4592 3M Zwijndrecht: Cleaningwerken Maart 2018
datum 09/04/2018
pagina's 1/8

Cleaningwerken onttrekkingsinstallatie Maart 2018 te 3M Zwijndrecht

Documentstatus

Actie	Naam	Functie	Handtekening
Auteur	Reinout Van Loon	Lead Operations & Maintenance Engineer	

1. Cleaningwerken Maart 2018

1.1. Visuele controle en reiniging pompen

1.1.1. Pompen bij aankomst

1.1.1.1. PP1



1.1.1.2. PP2



1.1.1.3. PP4



1.1.1.4. PP5



1.1.1.5. PP6



1.1.1.6. PP7

Geen foto beschikbaar

1.1.1.7. PP8



1.1.1.8. PP9



1.1.1.9. PP10



1.1.1.10. Terugslagkleppen PP1, PP4, PP5 en PP6





1.2. Test pompen

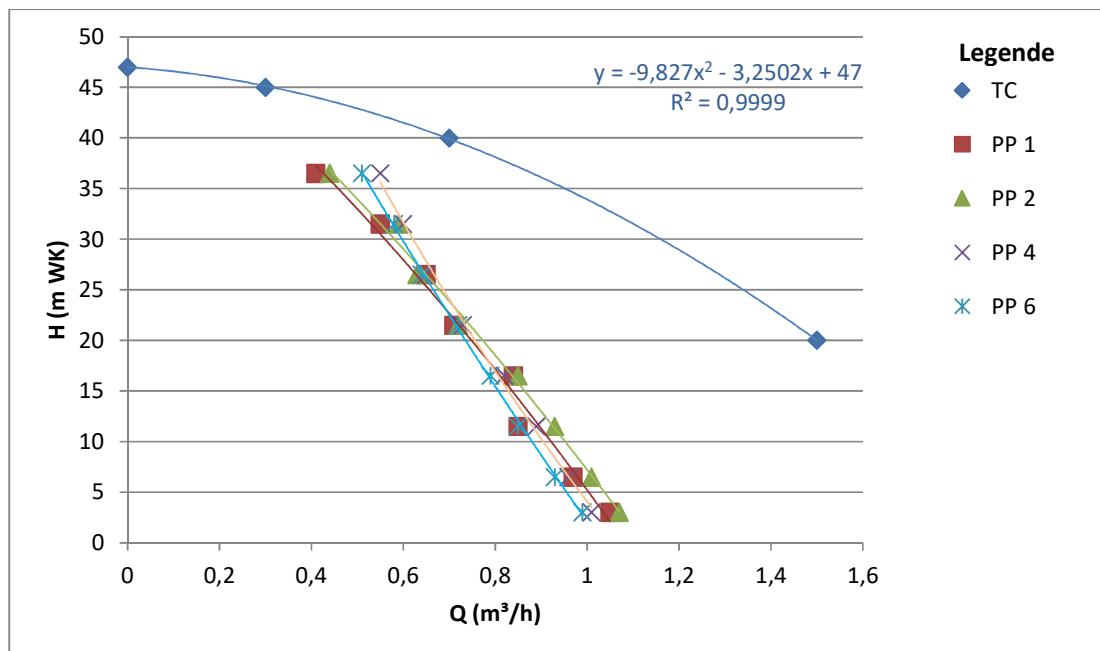
Na een eerste reiniging van de pompen, werden deze terug samengebouwd en hydraulisch getest. Uit deze hydraulische testen bleek dat alle motorgedeeltes en waaierhuizen nog operationeel konden zijn maar de bijhorende drukken en debieten niet in overeenstemming waren met de theoretische werkingsgrafieken van deze pompen. Daarom werden de pompen een 2^{de} maal gereinigd door elke pomp een half uur in een lichte zuuroplossing (HCl) te laten circuleren.

Na deze 2^{de} reiniging kon vastgesteld worden dat alle pompen een normale werkingsgrafiek vertoonde en deze bijgevolg alle terug werden gemonteerd op site.

De terugslagkleppen van PP1, PP4, PP5 en PP6 werden na reiniging alle terug gemonteerd in de desbetreffende onttrekkingsputten.

Werkingsgrafieken na een eerste reiniging:

PP 1,2,4,5 & 6:

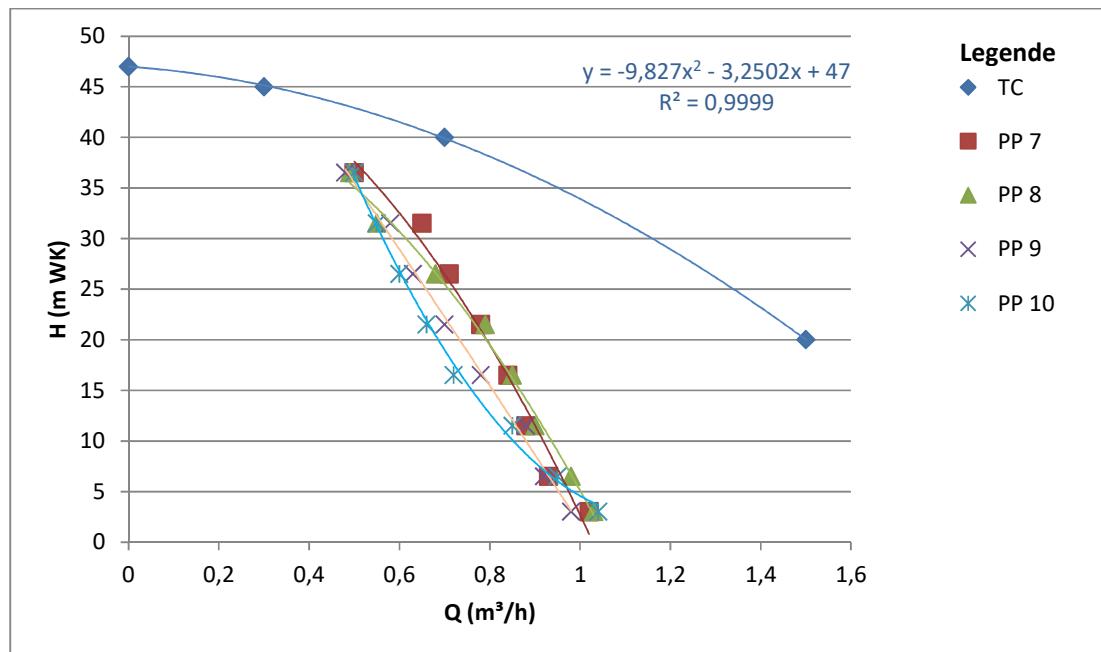




Purazur

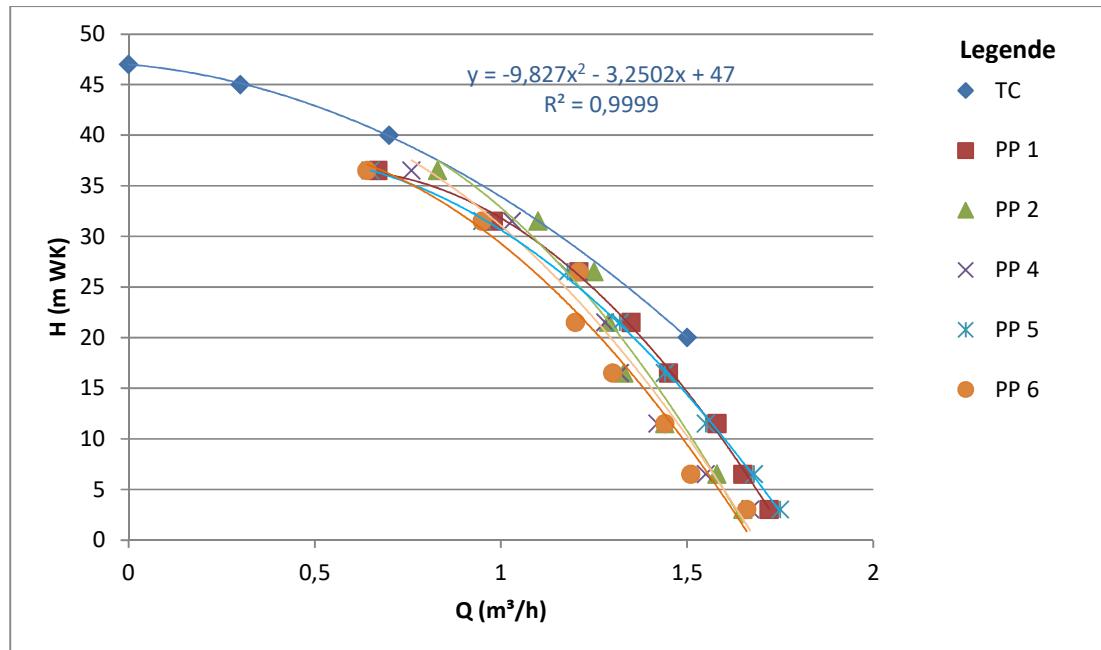
Water Treatment Solutions

PP 7,8,9 & 10:



Werkingsgrafieken na de 2^{de} reiniging

PP 1,2,4,5 & 6:

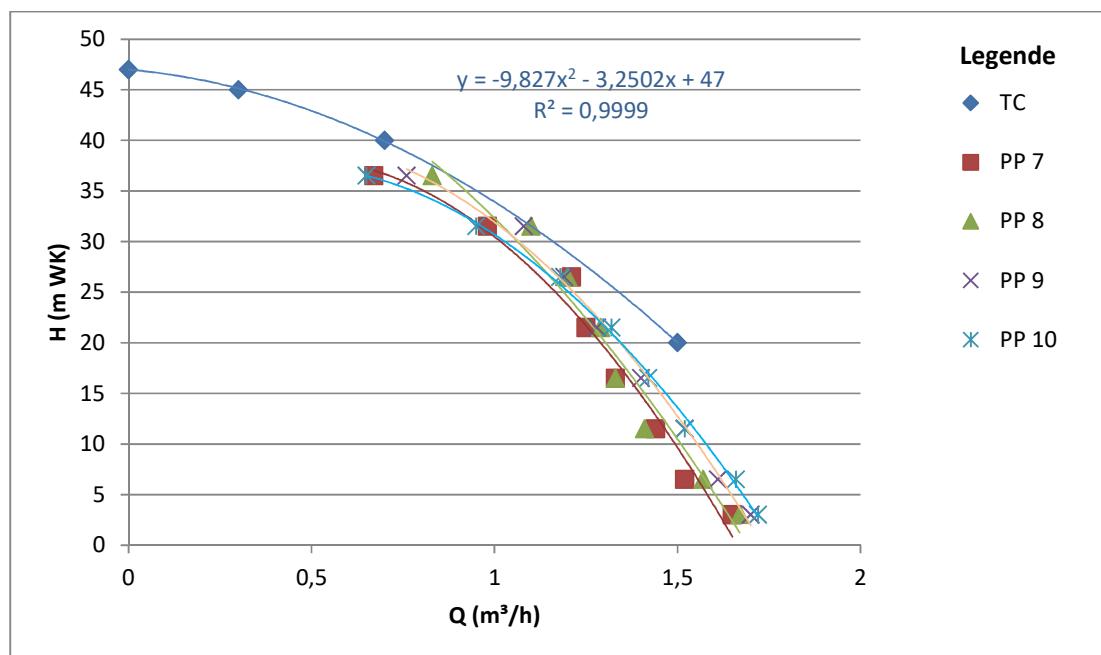




Purazur

Water Treatment Solutions

PP 7,8,9 &10:





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ENVIRONMENTAL SOLUTIONS

Rapport

Chemische cleaning 09/01/2020

Site: 3M Zwijndrecht



Titel document:

RAPPORT CHEMISCHE CLEANING 09-01-2020

Project:

ONTTREKKINGSINSTALLATIE 3M ZWIJDRECHT

Opgemaakt door Kris Dendoncker



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ENVIRONMENTAL SOLUTIONS

Rapport

Chemische cleaning 09/01/2020

Site: 3M Zwijndrecht

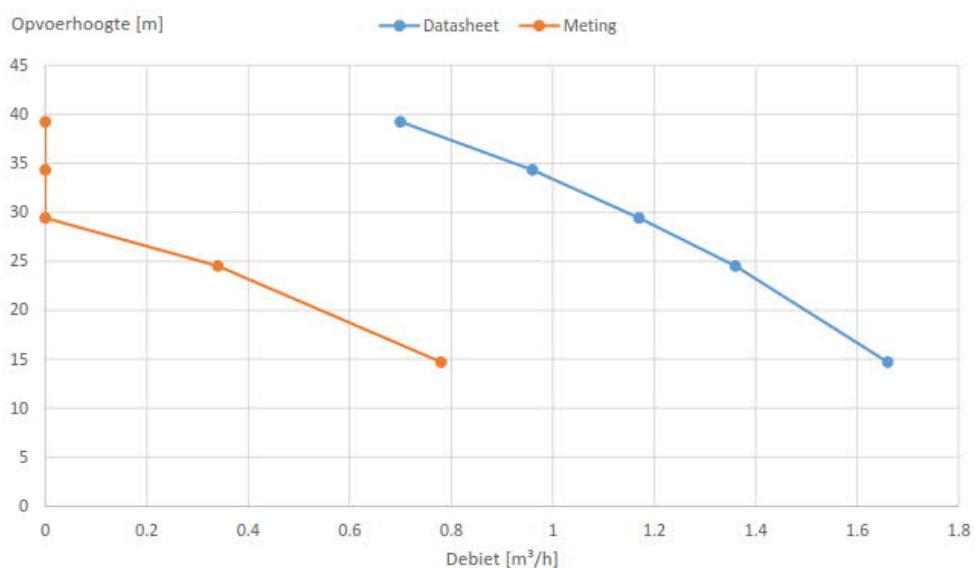
1 PP 2



**Pomptest WO 21970101**

Afdeling: Envisan

Datum: 08.01.2020 10:53:01



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Punt 3	1.17	3	29	0	3	29
Punt 4	0.96	3.5	34	0	3.5	34
Punt 5	0.7	4	39	0	4	39

Figure 1: PP 2 voor cleaning

De pomptest van pomp 2 geeft duidelijk een verminderd pompvermogen aan. De maximale opvoerhoogte kan door de verstopping niet meer gehaald worden. Bij lage opvoerhoogtes is het debiet slechts 25 % van de theoretische waarde.



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2 PP 4



De motor van pomp 4 was stuk. Bijgevolg kon geen pompprestatiecurve worden opgesteld.



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Chemische cleaning 09/01/2020

Site: 3M Zwijndrecht

3 PP 5

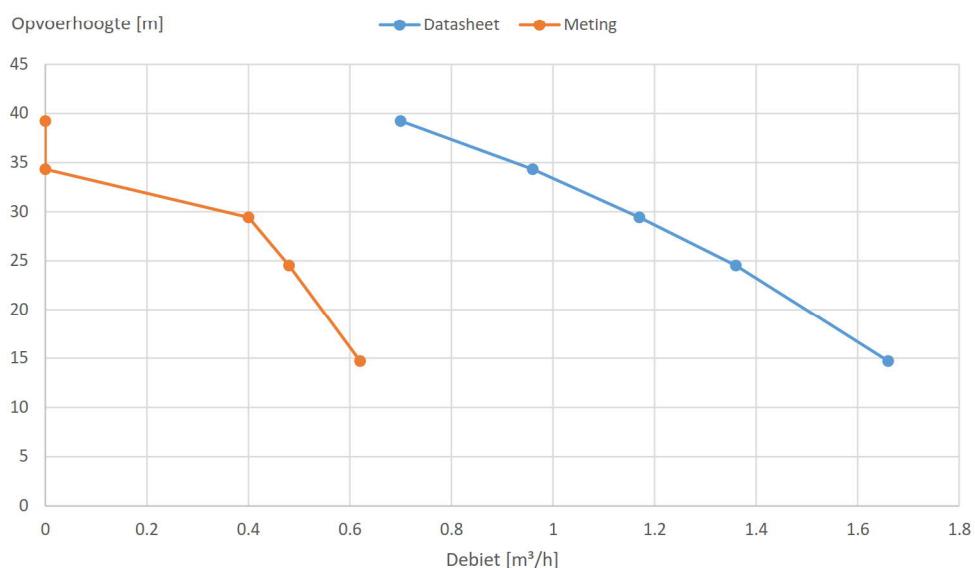




Pomptest WO 21970101

Afdeling: Envisan

Datum: 08.01.2020 09:29:19



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Punt 2	1.36	2.5	25	0.48	2.5	25
Punt 3	1.17	3	29	0.4	3	29
Punt 4	0.96	3.5	34	0	3.5	34
Punt 5	0.7	4	39	0	4	39

Figure 2: PP 5 voor cleaning

De pomphuis van pomp 5 was volledig versleten. Het maximale debiet met versleten waaiers bedroeg 0.62 m³/u waar de theoretische waarde 1.66 m³/u bedraagt.



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Chemische cleaning 09/01/2020

Site: 3M Zwijndrecht

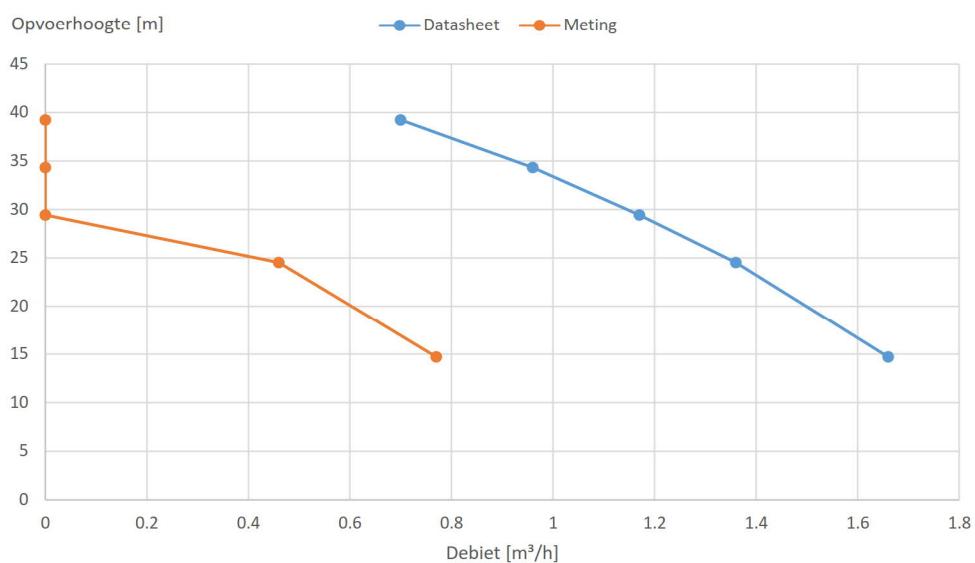
4 PP 6



**Pomptest WO 21970101**

Afdeling: Envisan

Datum: 08.01.2020 15:17:46



	Datasheet			Metting		
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Punt 2	1.36	2.5	25	0.46	2.5	25
Punt 3	1.17	3	29	0	3	29
Punt 4	0.96	3.5	34	0	3.5	34
Punt 5	0.7	4	39	0	4	39

Figure 3: PP 6 voor cleaning

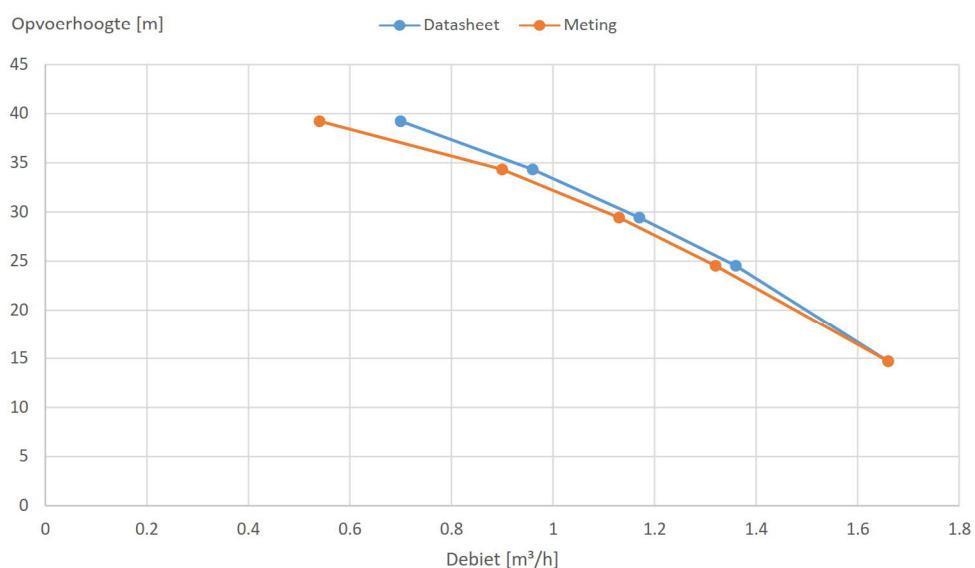
De pomptest van pomp 6 geeft duidelijk een verminderd pompvermogen aan. De maximale opvoerhoogte kan door de verstopping niet meer gehaald worden. Bij lage opvoerhoogtes is het debiet slechts 34 % van de theoretische waarde. Na cleaning bereikt de pomp terug zijn maximale capaciteit.



Pomptest WO 21970101

Afdeling: Envisan

Datum: 09.01.2020 16:08:55



	Datasheet			Meting		
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Punt 2	1.36	2.5	25	1.32	2.5	25
Punt 3	1.17	3	29	1.13	3	29
Punt 4	0.96	3.5	34	0.9	3.5	34
Punt 5	0.7	4	39	0.54	4	39

Figure 4: PP 6 na cleaning



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Site: 3M Zwijndrecht

5 PP 7





Pomptest WO 21970101

Afdeling: Envisan

Datum: 09.01.2020 08:48:23

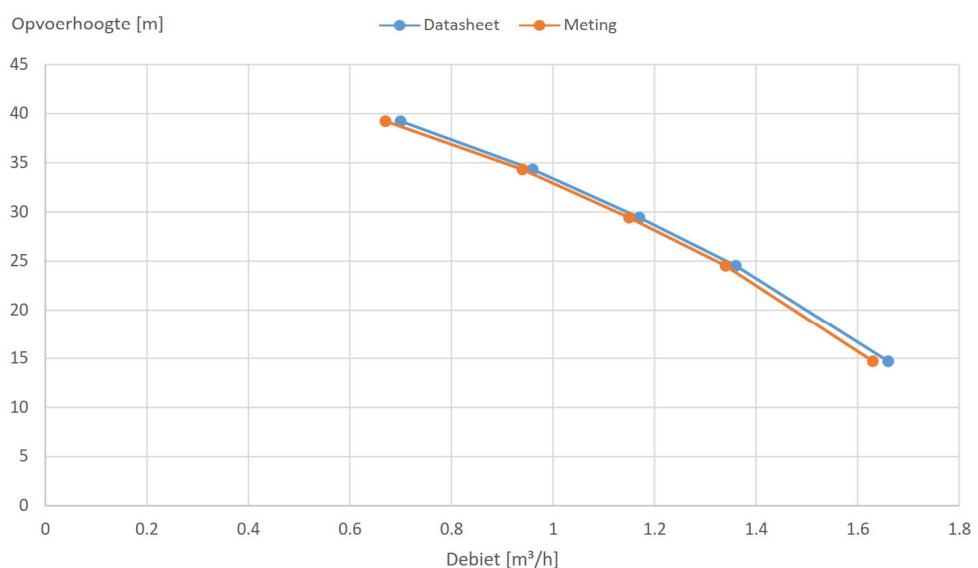


Figure 5: PP 7 voor cleaning

 Envisan <small>ENVIRONMENTAL SOLUTIONS</small>	Rapport Chemische cleaning 09/01/2020	Site: 3M Zwijndrecht
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Pomptest WO 21970101

Afdeling: Envisan

Datum: 09.01.2020 14:59:04

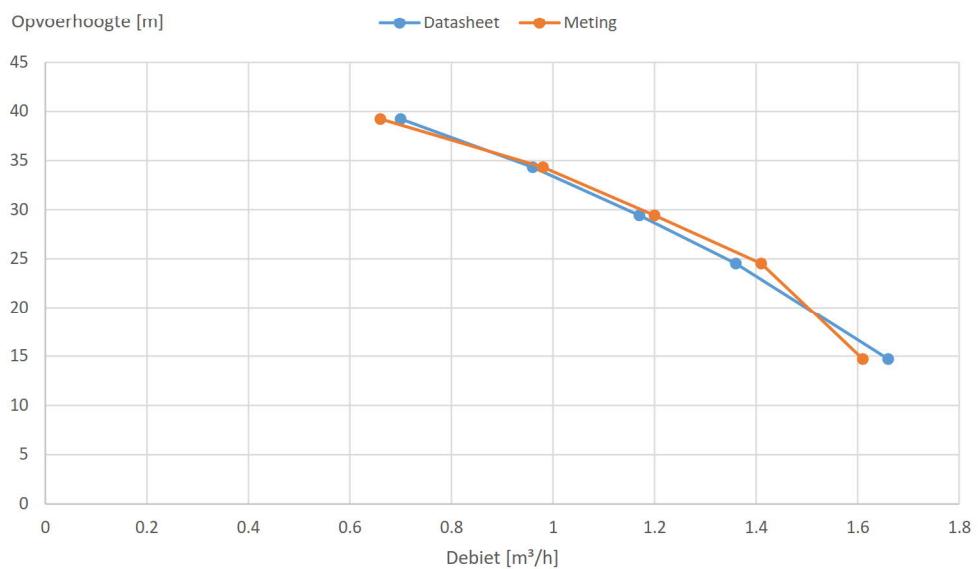


Figure 6: PP 7 na cleaning

Uit de pompprestatiecurven van pomp 7 blijkt dat pomp 7 zowel voor als na de cleaning zijn theoretische capaciteit bereikt.



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6 PP 8

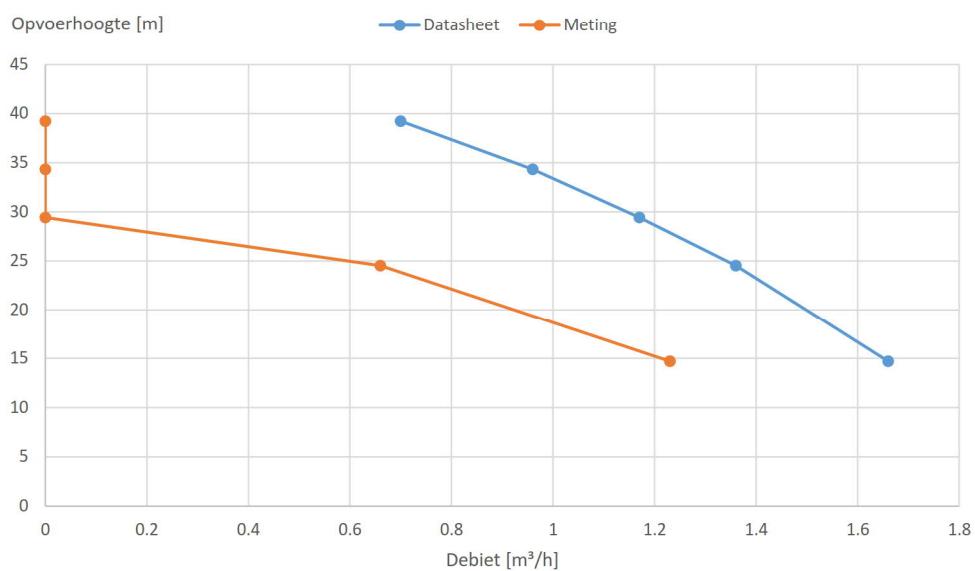




Pomptest WO 21970101

Afdeling: Envisan

Datum: 09.01.2020 10:02:32



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Punt 2	1.36	2.5	25	0.66	2.5	25
Punt 3	1.17	3	29	0	3	29
Punt 4	0.96	3.5	34	0	3.5	34
Punt 5	0.7	4	39	0	4	39

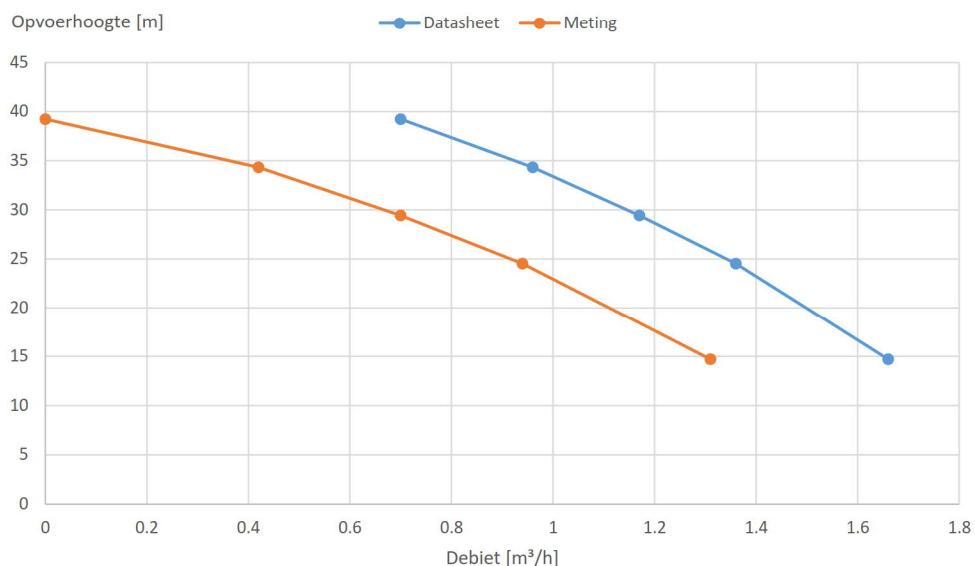
Figure 7: PP 8 voor cleaning



Pomptest WO 21970101

Afdeling: Envisan

Datum: 13.01.2020 10:57:02



	Datasheet			Meting		
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Punt 1	1.66	1.5	15	1.31	1.5	15
Punt 2	1.36	2.5	25	0.94	2.5	25
Punt 3	1.17	3	29	0.7	3	29
Punt 4	0.96	3.5	34	0.42	3.5	34
Punt 5	0.7	4	39	0	4	39

Figure 8: PP 8 na cleaning

De pomptest van pomp 8 geeft duidelijk een verminderd pompvermogen aan. De maximale opvoerhoogte kan door de verstopping niet meer gehaald worden. Bij lage opvoerhoogtes is het debiet slechts 49 % van de theoretische waarde. Na cleaning bereikt de pomp slechts ca. 65% van zijn theoretische capaciteit.



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Chemische cleaning 09/01/2020

Site: 3M Zwijndrecht

7 PP 10





Pomptest WO 21970101

Afdeling: Envisan

Datum: 10.01.2020 08:36:14

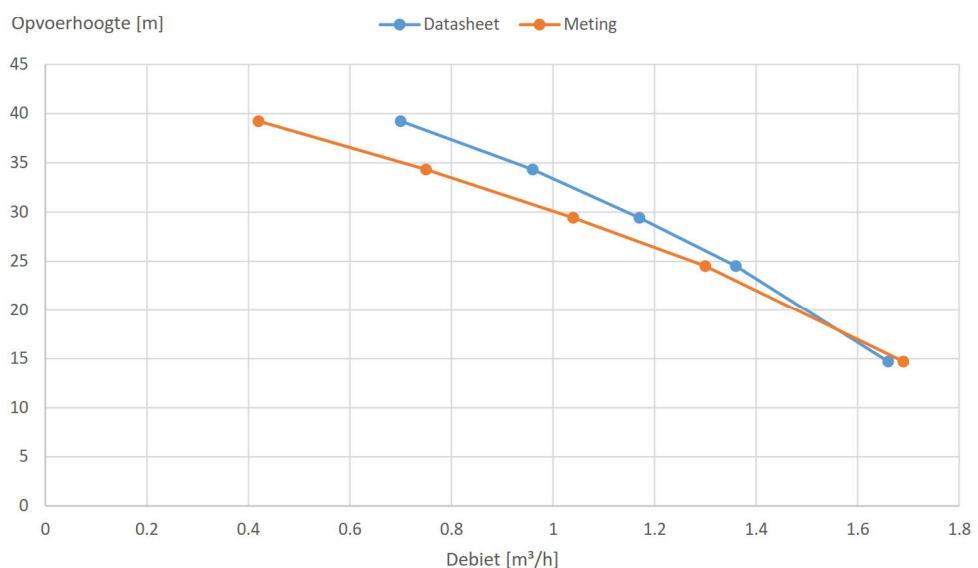


Figure 9: PP 10 na cleaning

Voor de cleaning zat de pomp volledig verstopt en kon geen pompprestatiecurve worden opgesteld. Na de cleaning bereikt de pomp terug zijn theoretische capaciteit.



Envisan
ENVIRONMENTAL SOLUTIONS

Rapport

Chemische cleaning 09/01/2020

Site: 3M Zwijndrecht

8 PP 11

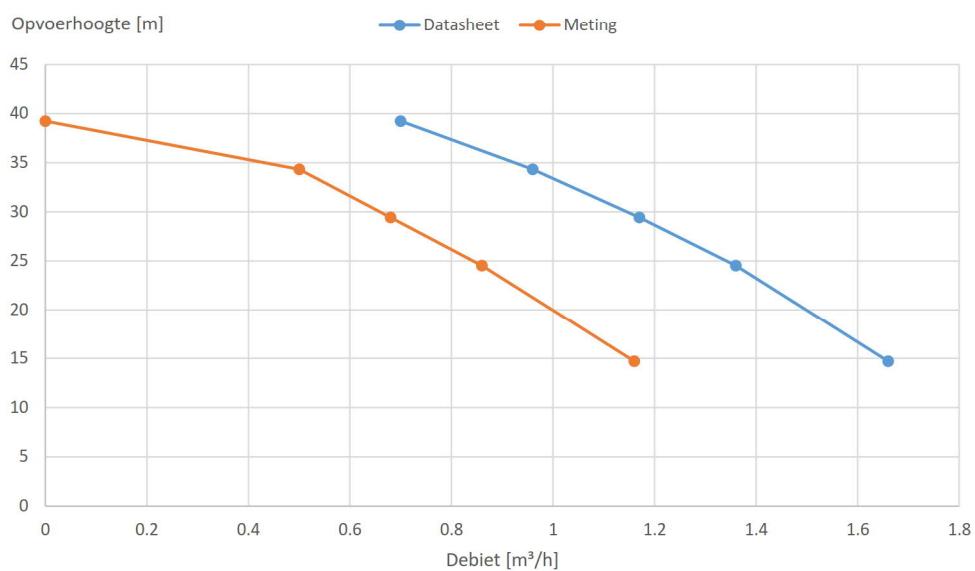




Pomptest WO 21970101

Afdeling: Envisan

Datum: 10.01.2020 11:27:59



	Datasheet			Meting		
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Punt 1	1.66	1.5	15	1.16	1.5	15
Punt 2	1.36	2.5	25	0.86	2.5	25
Punt 3	1.17	3	29	0.68	3	29
Punt 4	0.96	3.5	34	0.5	3.5	34
Punt 5	0.7	4	39	0	4	39

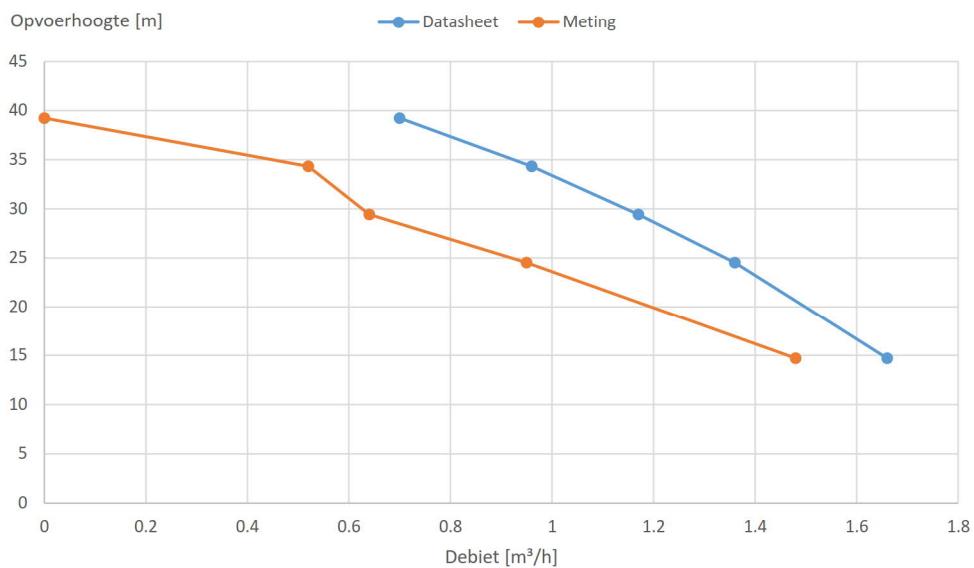
Figure 10: PP 11 voor cleaning

 Envisan <small>ENVIRONMENTAL SOLUTIONS</small>	Rapport Chemische cleaning 09/01/2020	Site: 3M Zwijndrecht
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Pomptest WO 21970101

Afdeling: Envisan

Datum: 10.01.2020 15:46:27



	Datasheet			Meting		
	Debit [m³]	Druk [bar]	Opvoerhoogte [m]	Debit [m³]	Druk [bar]	Opvoerhoogte [m]
Punt 1	1.66	1.5	15	1.48	1.5	15
Punt 2	1.36	2.5	25	0.95	2.5	25
Punt 3	1.17	3	29	0.64	3	29
Punt 4	0.96	3.5	34	0.52	3.5	34
Punt 5	0.7	4	39	0	4	39

Figure 11: PP 11 na cleaning

De pomptest van pomp 11 geeft duidelijk een verminderd pompvermogen aan. Bij lage opvoerhoogtes is het debiet slechts 70 % van de theoretische waarde. Na cleaning bereikt de pomp slechts ca. 75% van zijn theoretische capaciteit. Deze beperkte verbetering na cleaning duidt er op dat het pomphuis versleten is en binnenkort aan vervangen zal toe zijn.



Envisan
ENVIRONMENTAL SOLUTIONS

Rapport

Chemische cleaning 09/01/2020

Site: 3M Zwijndrecht



Titel document:

RAPPORT LIGHT CLEANING 21-04-2020

Project:

ONTTREKKINGSINSTALLATIE 3M ZWIJDRECHT

Opgemaakt door Kris Dendoncker



1 PP 2

De pomp geeft tot net voor de cleaning een acceptabel debiet van 18,8 l/min bij een overdruk van 0,1 bar. Na cleaning geeft de pomp een hoog debiet van 54,2 l/min bij een overdruk van 0,17 bar. We kunnen besluiten dat de pomp nog goed werkt en de cleaning zijn werk gedaan heeft.





2 PP 4

De pomp geeft tot net voor de cleaning een acceptabel debiet van 20,8 l/min bij een overdruk van 0,6 bar. Na cleaning geeft de pomp een debiet van 16,7 l/min bij een druk van 0,36 bar. We kunnen besluiten dat de pomp nog goed werkt en de cleaning zijn werk gedaan heeft. Er is wel een probleem met de profibus geeft wel een foutmelding. Gezien dit een deel van de sturing is, zal 3M dit moeten bekijken.





3 PP 5

De pomp geeft tot net voor de cleaning een zeer laag debiet van 1,12 l/min bij een overdruk van 0,14 bar. Na cleaning geeft de pomp een debiet van 7,5 l/min bij een druk van 0,2 bar. We kunnen besluiten dat de pomp beter werkt na de cleaning maar dat de waaiers in de pomp versleten zijn.



 Envisan <small>ENVIRONMENTAL SOLUTIONS</small>	Rapport Chemische cleaning 09/01/2020	Site: 3M Zwijndrecht
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4 PP 6

De pomp geeft tot net voor de cleaning een acceptabel maximaal debiet van 19,8 l/min bij een overdruk van 0,65 bar. Na cleaning geeft de pomp een debiet van 22,9 l/min bij een druk van 0,95 bar. We kunnen besluiten dat de pomp goed werkt na de cleaning maar dat de leidingen aangeladen zijn. Voorlopig is het debiet goed, maar er is ook drukopbouw door verstopping van de leidingen.



5 PP 7

Geen foto beschikbaar

De pomp geeft tot net voor de cleaning acceptabel maximaal debiet van 24,8 l/min bij een overdruk van 0,23 bar. Na cleaning geeft de pomp een debiet van 19,95 l/min bij een druk van 0,23 bar. We kunnen besluiten dat de pomp nog goed werkt en de cleaning zijn werk gedaan heeft.



6 PP 8

De pomp geeft tot net voor de cleaning een zeer laag maximaal debiet van 2,3 l/min bij een overdruk van 0,2 bar. Na cleaning geeft de pomp een debiet van 2,9 l/min bij een overdruk van 0,12 bar. Visueel wordt echter vast gesteld dat de leiding volledig is aangeslagen. Het lage debiet wordt dus veroorzaakt door de verstopte leiding.





7 PP 10

De pomp geeft tot net voor de cleaning een acceptabel maximaal debiet van 17,2 l/min bij een overdruk van 0,73 bar. Na cleaning geeft de pomp een debiet van 16,5 l/min bij een druk van 0,15 bar. We kunnen besluiten dat de pomp nog goed werkt en de cleaning zijn werk gedaan heeft. Er bevindt zich wel een klein lek aan de flenskoppeling van de pomp. Dit wordt bij een volgend onderhoud hersteld (als de stukken binnen zijn).





8 PP 11

De pomp krijgt wel stroom maar draait niet. De pomp werd meegenomen naar Aalst voor een revisie. In Aalst blijkt dat de waaiers volledig uitgesleten zijn. Ook blijkt er iets mis te zijn met de voeding van de pomp. Dit wordt verder nagekeken.





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ENVIRONMENTAL SOLUTIONS

Rapport

Chemische cleaning 07/07/2020

Site: 3M Zwijndrecht



Titel document:

RAPPORT CHEMISCHE CLEANING 07-07-2020

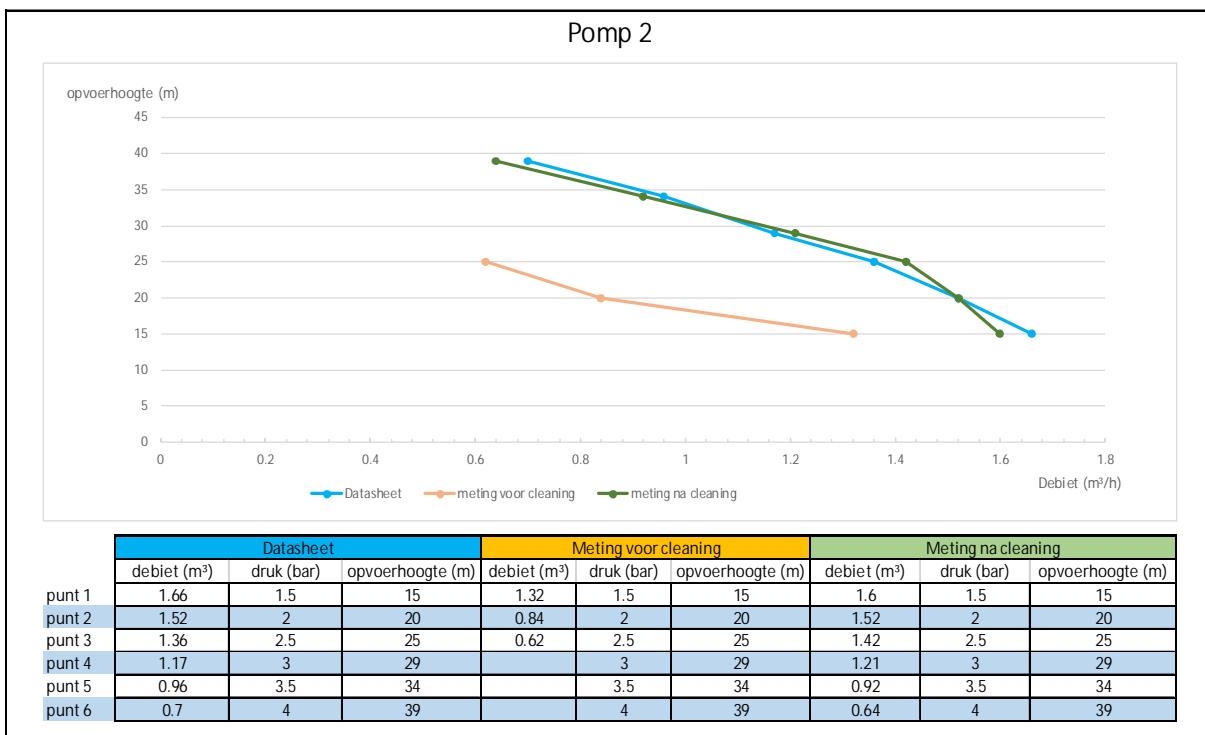
Project:

ONTTREKKINGSINSTALLATIE 3M ZWIJDRECHT

Opgemaakt door Kris Dendoncker

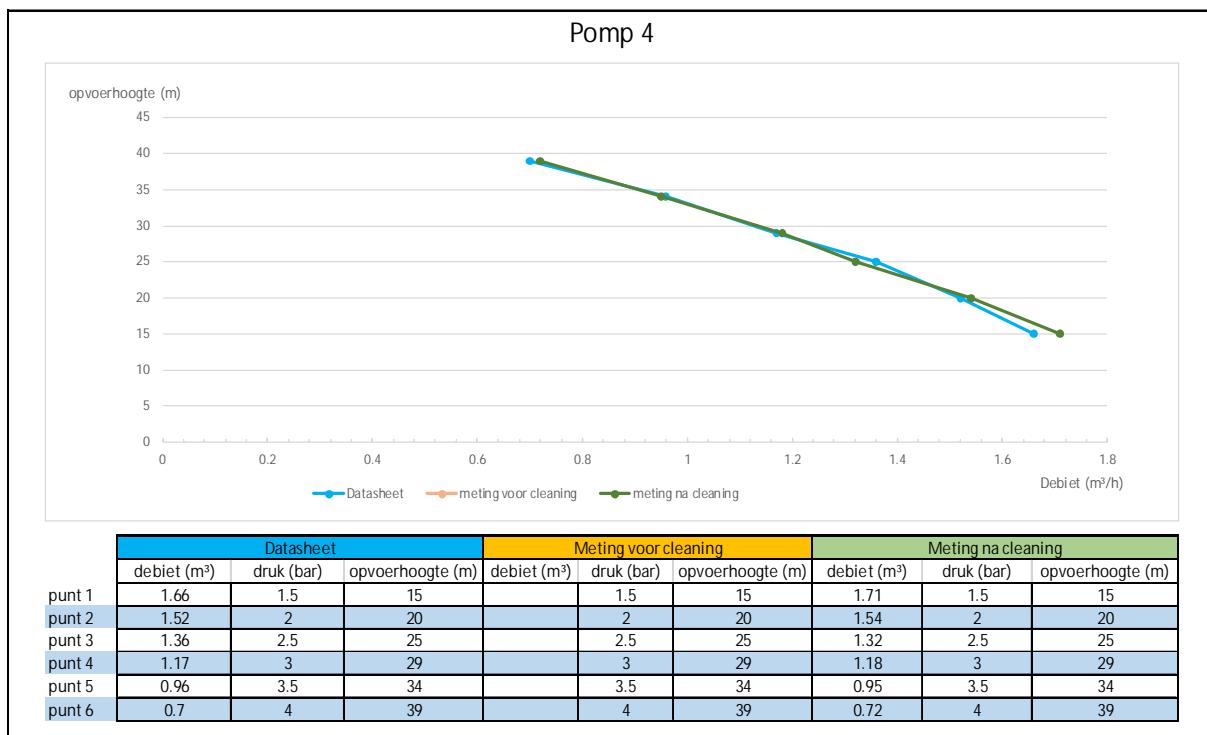


1 PP 2



Tabel 1: pomp 2

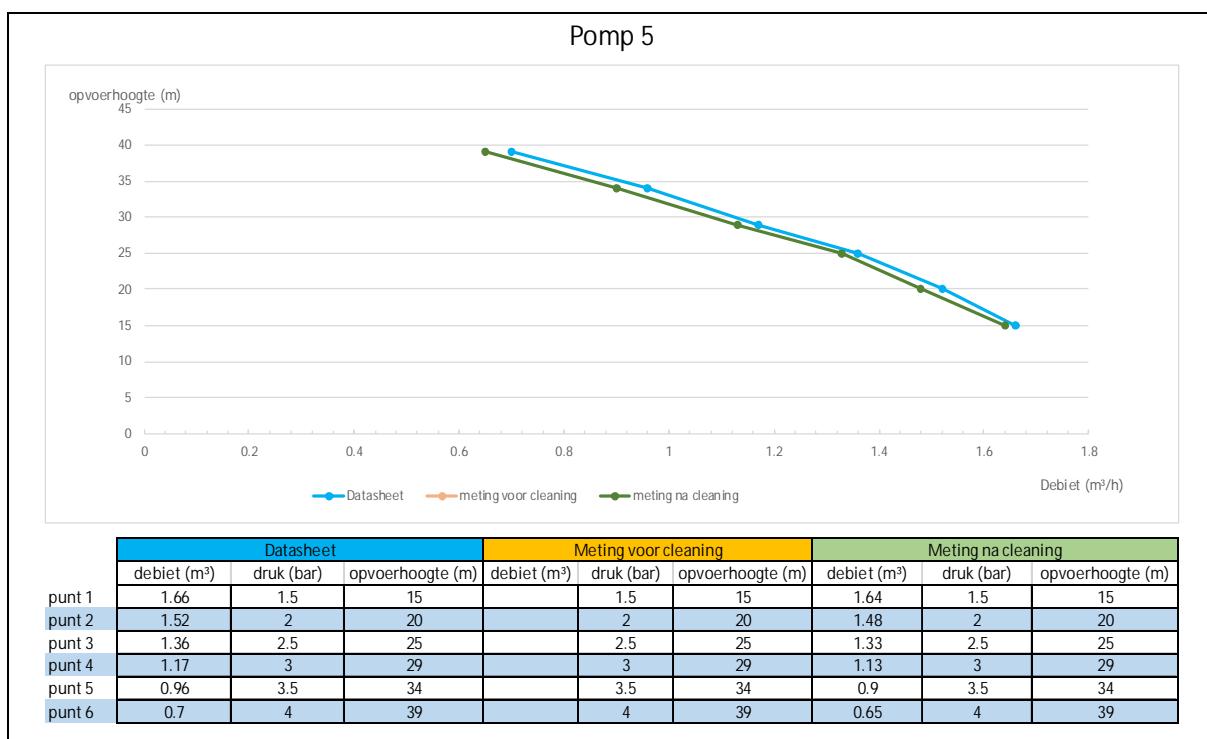
De pomptest van pomp 2 voor de cleaning geeft duidelijk een verminderd pompvermogen aan. De maximale opvoerhoogte kan door de verstopping niet meer gehaald worden. Bij lage opvoerhoogtes is het debiet slechts 79 % van de theoretische waarde. Na cleaning benaderd de pomp weer de theoretische debieten en opvoerhoogtes. De pomp is dus nog in uitstekende staat.

**2 PP 4****Tabel 2: Pomp 4**

Door de verontreiniging aan de pomp kon geen betrouwbare pomptest meer uitgevoerd worden. Na cleaning benaderd de pomp weer de theoretische debieten en opvoerhoogtes. De pomp is dus nog in uitstekende staat.



3 PP 5

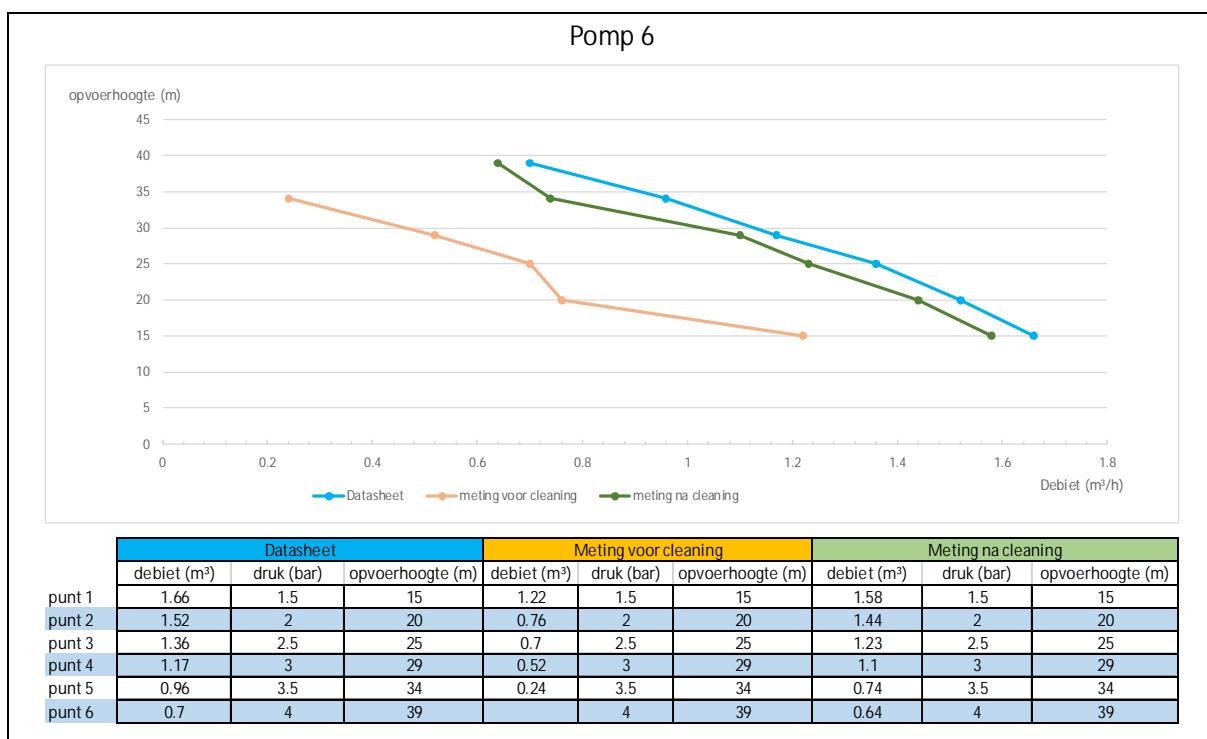


Tabel 3: pomp 5

Door de verontreiniging aan de pomp kon geen betrouwbare pomptest meer uitgevoerd worden. Na cleaning benaderd de pomp weer de theoretische debieten en opvoerhoogtes. De pomp is dus nog in uitstekende staat.



4 PP 6



Tabel 4: pomp 6

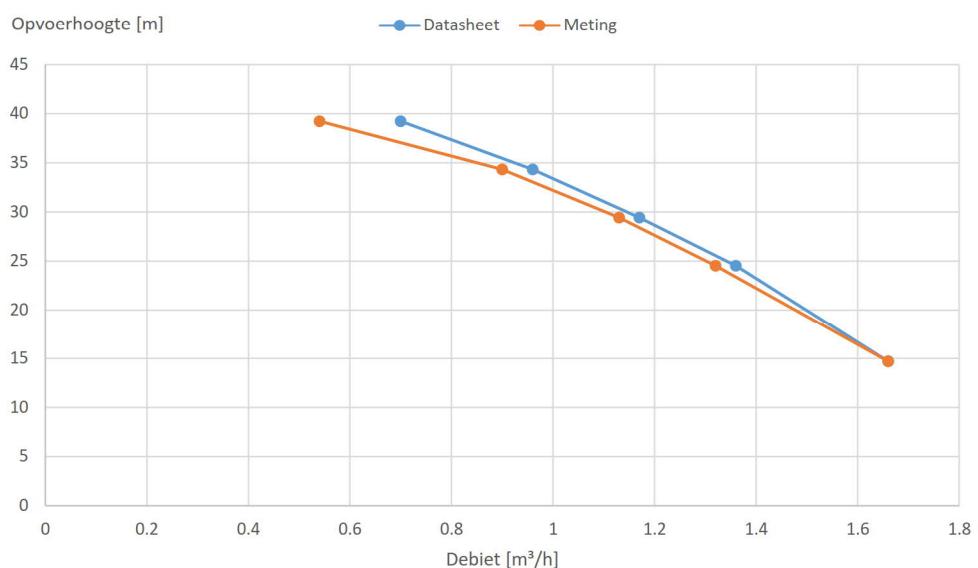
De pomptest van pomp 6 geeft duidelijk een verminderd pompvermogen aan. De maximale opvoerhoogte kan door de verstopping niet meer gehaald worden. Bij lage opvoerhoogtes is het debiet slechts 73 % van de theoretische waarde. Na cleaning benaderd de pomp ongeveer terug zijn theoretische waarden. De pomp is nog in degelijke staat.

 Envisan <small>ENVIRONMENTAL SOLUTIONS</small>	Rapport	Site: 3M Zwijndrecht
Chemische cleaning 07/07/2020		

Pomptest WO 21970101

Afdeling: Envisan

Datum: 09.01.2020 16:08:55

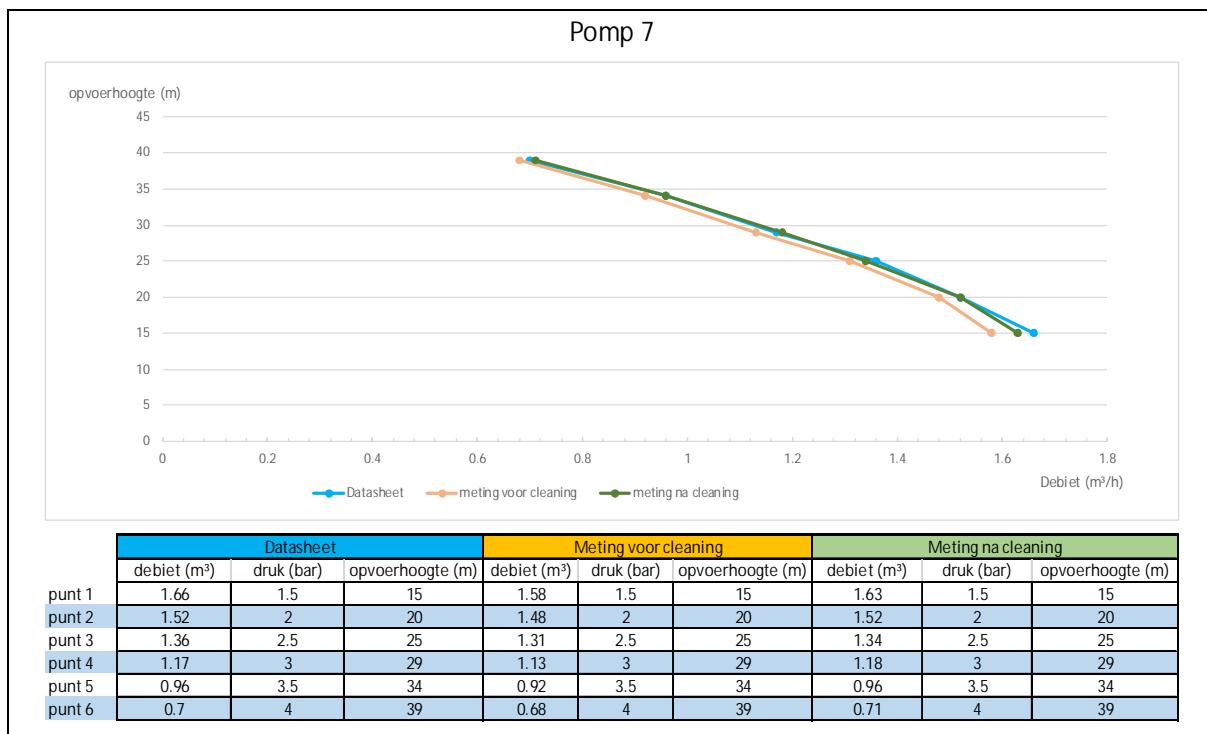


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Punt 1	1.66	1.5	15	1.66	1.5	15
Punt 2	1.36	2.5	25	1.32	2.5	25
Punt 3	1.17	3	29	1.13	3	29
Punt 4	0.96	3.5	34	0.9	3.5	34
Punt 5	0.7	4	39	0.54	4	39

Figure 1: PP 6 na cleaning



5 PP 7

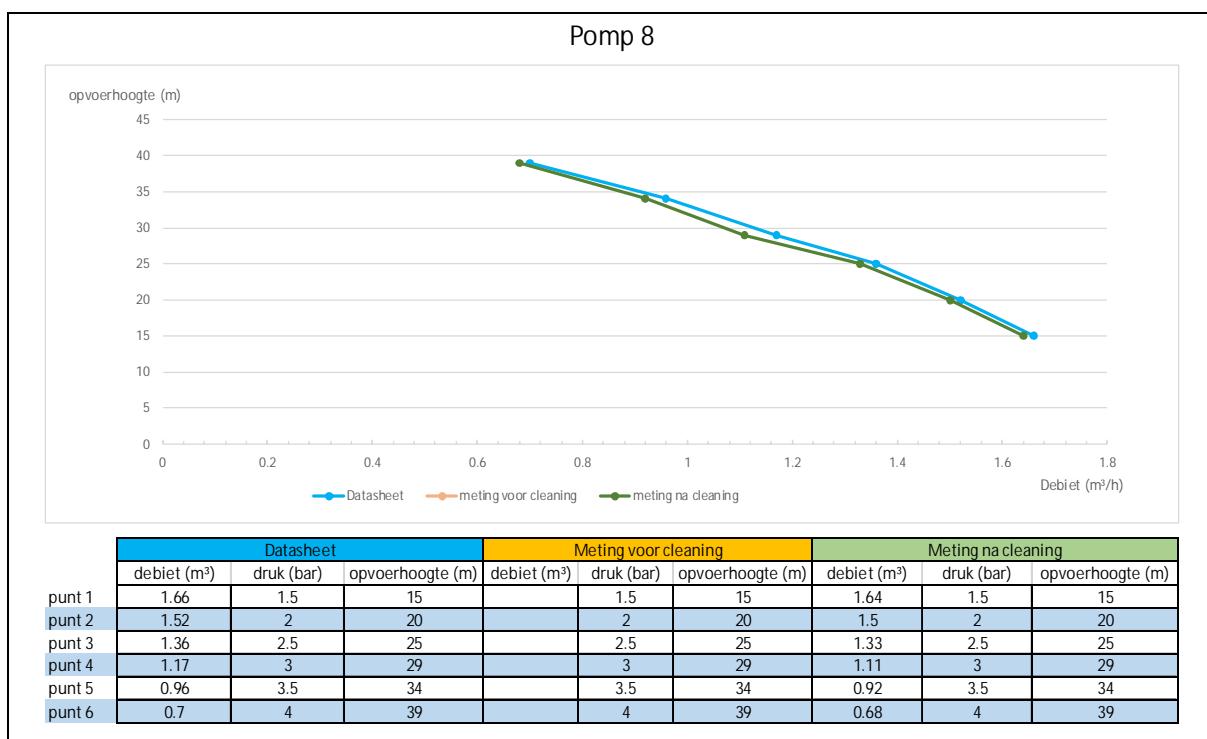


Tabel 5: pomp 7

Uit de pompprestatiecurven van pomp 7 blijkt dat pomp 7 zowel voor als na de cleaning zijn theoretische capaciteit bereikt. De pomp is nog in uitstekende staat.



6 PP 8

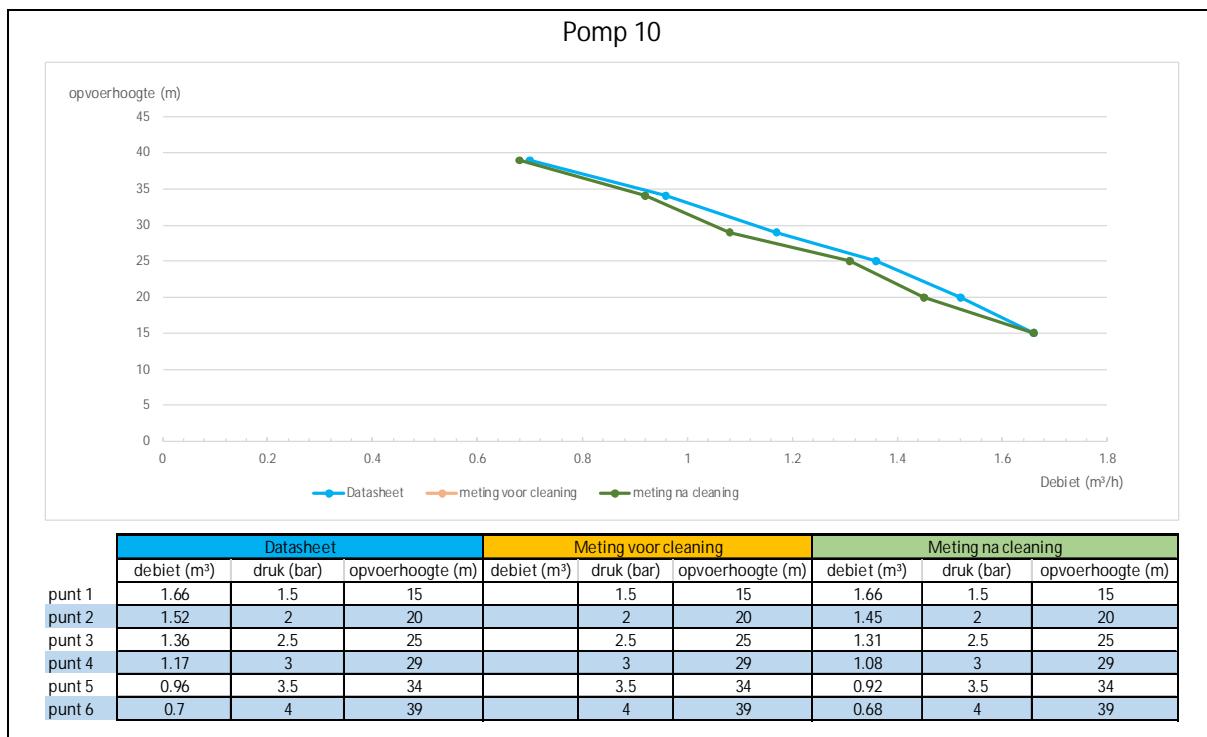


Tabel 6: pomp 8

Door de verontreiniging aan de pomp kon geen betrouwbare pomptest meer uitgevoerd worden. Na cleaning benaderd de pomp weer de theoretische debieten en opvoerhoogtes. De pomp is dus nog in uitstekende staat.

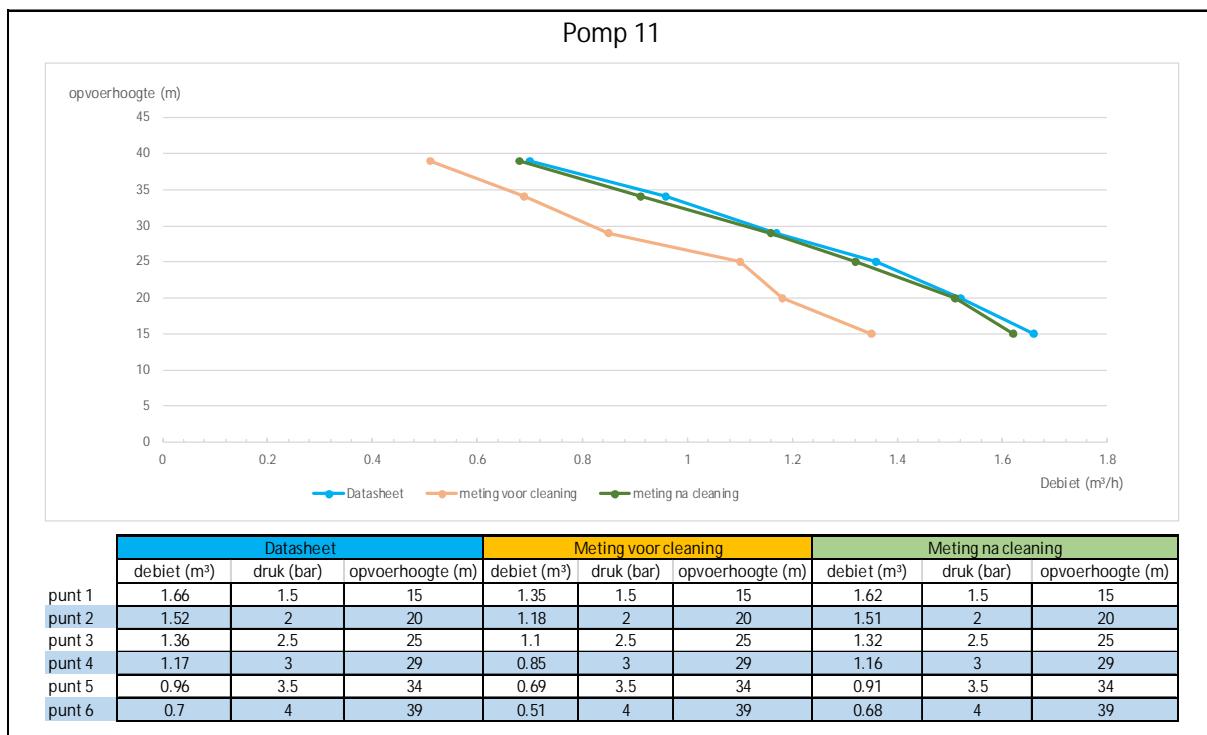


7 PP 10

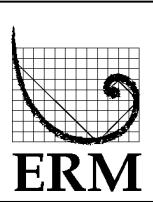


Tabel 7: pomp 10

Door de verontreiniging aan de pomp kon geen betrouwbare pomptest meer uitgevoerd worden. Na cleaning benaderd de pomp weer de theoretische debieten en opvoerhoogtes. De pomp is dus nog in uitstekende staat.

**8 PP 11****Tabel 8: pomp 11**

De pomptest van pomp 11 geeft duidelijk een verminderd pompvermogen aan. De maximale opvoerhoogte kan door de verstopping niet meer gehaald worden. Bij lage opvoerhoogtes is het debiet slechts 81 % van de theoretische waarde. Na cleaning benaderd de pomp weer de theoretische debieten en opvoerhoogtes. De pomp is dus nog in uitstekende staat.



DAILY BOOK ENVIRONMENTAL SUPERVISION

CLEANING OF EXTRACTION WELLS, PUMPS AND PIPING AT THE 3M SITE, ZWIJNDRECHT

Date: 5-9-2017 until 14-09-2017

Project number: 0398993

Client: 3M Belgium bvba

Location Canadastraat 11 2070 Zwijndrecht (BE)	Subcontractors involved Purazur nv Chris Vereecke & Zoon bvba
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Distribution list	
3M	Nynke De Schutter (██████████), Wouter Paque (██████████) and Sam Van Beneden (██████████)
Weston Solutions	Dave Cairns (██████████)
ERM	Erik Boeckx (██████████), Nanda Hermes (██████████), Mattias Verbeeck (██████████) and Tine Mandonx (██████████)
PURAZUR	Reinout Van Loon (██████████), Pieter Van der Mussele (██████████) and Erwin Mariën (██████████)

Activities	
<u>Introduction:</u>	A groundwater extraction system consisting of nine extraction wells is operational at the 3M site in Zwijndrecht (BE). The aim of the system is to remediate groundwater impacted with Fluor Chemicals (FC's) and to mitigate contaminant migration. The impacted groundwater extracted by the remedial system is treated by the wastewater treatment plant of the site.
	The extraction wells and pumps are periodically cleaned to optimize performance of the remedial system. Cleaning is required because of silting and rusting of wells and pumps.
	Between 5 and 14 September 2017, cleaning and maintenance activities were executed by the remedial contractor Purazur and their subcontractor, the industrial cleaning company Chris Vereecke & Zoon (Vereecke), under the environmental supervision of ERM. This document summarizes the actions performed.
<u>Tuesday 5 September (08:00-13:00)</u>	On Tuesday, Purazur prepared the extraction wells for cleaning by shutting down the installation, opening and dismantling 9 wells and piping. After dismantling, the pumps were taken to the workshop of Purazur for technical inspection and revision. A report will be written by Purazur on the condition of the pumps. At PP05, the presence of a hard black layer on the pump, the transducer and inside the piping is identified again.

The flowmeter of PP05 has been disconnected by 3M and taken for cleaning due to unreliable values on the display.

Activities performed:

- Shut down of the groundwater extraction installation;
- Disconnecting and cleaning of flow meter of PP05; and
- Opening and dismantling of the wells (PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 and PP10) in order to transport the pumps to Purazur's technical workshop for revision.

Wednesday 6 September (8:00-15:00)

On Wednesday, Purazur/Vereecke cleaned and flushed all (subsurface) pipes of the extraction system of the nine extractions wells. Vereecke came on site with two vacuum trucks to execute these works. The cleaning was performed using a jetting nozzle with an extension of 50 meter, resulting in a big amount of sediment removal. At the end of the day, water and sludge captured by the vacuum trucks was disposed in dewatering containers at the site.

Activities performed:

- Purazur dismantled the piping network inside the container;
- Cleaning and flushing of the dismantled pipes in the container;
- Removal of sediments and depositions from inside the extraction wells and pipes. Flush water of PP02, PP07, PP08, PP09 and PP10 is collected by the vacuum truck at the extraction wells. Flush water of PP01, PP04, PP05 and PP06 is directly discharged into the 3M chemical sewer.

Thursday 13 September (8:30-12:00)

ERM added Boresaver (2kg) and Iron Clean Liquid (2L) to the wells.

Activities performed:

- Adding Boresaver and Iron Clean Liquid to flush and clean extraction wells (by ERM).

Wednesday 14 September (8:00-16:00)

On Wednesday, Purazur/Vereecke came on site with one vacuum truck to remove the Boresaver product and Iron Clean Liquid from the extraction wells. Purazur reinstalled the piping-infrastructure inside the container and re-installed all revised pumps (PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 and PP10).

The preliminary results of the pump tests performed by Purazur in their technical workshop indicated that all pumps are still in good condition. Two pumps (PP02 and PP06) will most likely need a renewal of pump fans in the near future.

3M reinstalled the flow meter of PP05. After restarting the pump, the flow meter (bleu) did not respond. Needs to be checked again by 3M (as communicated).

ERM measured the total depth of the extraction wells with an interface meter in order to determine the removed layer of sediment (see table below).

All flowmeters were reset and the pumps restarted (except for PP05).

At the end of the day, water and sludge captured by the vacuum truck was disposed in dewatering containers at the site.

Table 1. Measured extraction well depths after the September 2017 chemical cleaning campaign.

Extraction well	Measured Depth (meter below top of well pipe)	Sediments accumulation (cm)
PP01	5.20	0
PP02	5.76	0
PP04	5.72	0
PP05	5.07	0
PP06	4.75	0
PP07	5.63	0
PP08	7.00	0
PP09	6.46	0
PP10	6.48	0

The extraction system was restarted after installation of the 9 revised pumps. Below a summary of the extraction well status after cleaning.

Table 2. Summary of remediation system (September 2017)

Extraction well	Pump in place (y/n)	Reset total volume (y/n)	Operational (y/n)	Q (l/min)
PP01	Y	Y	Y	6.0
PP02	Y	Y	Y	2.0
PP04	Y	Y	Y	1.0
PP05	Y	N	N	n.a
PP06	Y	Y	Y	6.0
PP07	Y	Y	Y	8.0
PP08	Y	Y	Y	7.0
PP09	Y	Y	Y	2.0
PP10	Y	Y	Y	8.0

Activities performed :

- Removal of cleaning product and cleaning of nine extraction wells;
- Piping infrastructure of extraction system inside container re-installed;
- Depth measurement of all 9 extraction wells;
- Re-installation of the 9 revised pumps; and
- Restart of the remedial extraction installation.

Annexes

- Photo log

For the Environmental Consultant <i>(bodemsaneringsdeskundige)</i> Name and Signature  Erik Boeckx	For the Client Name and Signature
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Photo log

Picture 1: Disconnected piping



Picture 2: Cleaning flow meters

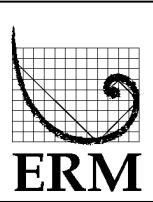


Picture 4: Cleaning well piping (PP10)



Picture 4: Flush water collection





DAILY BOOK ENVIRONMENTAL SUPERVISION

CLEANING OF EXTRACTION WELLS, PUMPS AND PIPING AT THE 3M SITE, ZWIJDRECHT

Date: 14/12/2017-15/12/2017

Project number: 0398993

Client: 3M Belgium bvba

Location Canadastraat 11 2070 Zwijndrecht (BE)	Subcontractors involved Purazur nv Chris Vereecke & Zoon bvba
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Distribution list	
3M	Nynke De Schutter (██████████), Wouter Paque (██████████) and Sam Van Beneden (██████████),
Weston Solutions	Dave Cairns (██████████)
ERM	Erik Boeckx (██████████), Nanda Herms (██████████), Mattias Verbeeck (██████████) and Elien Kemme (██████████)
PURAZUR	Reinout Van Loon (██████████), Erwin Mariën (██████████) and Pieter Van der Mussele (██████████)

Activities

Introduction:

A groundwater extraction system consisting of nine extraction wells is operational at the 3M site in Zwijndrecht (BE). The aim of the system is to remediate groundwater impacted with Fluor Chemicals (FC's) and to mitigate contaminant migration. The impacted groundwater extracted by the remedial system is treated by the wastewater treatment plant of the site.

The extraction wells and pumps are periodically cleaned to optimize performance of the remedial system. Cleaning is required because of silting and rusting of wells and pumps.

On December 14, 2017, cleaning and maintenance activities were executed by the remedial contractor Purazur and their subcontractor, the industrial cleaning company Chris Vereecke & Zoon (Vereecke), under the environmental supervision of ERM. This document summarizes the actions performed during this cleaning event.

Thursday 14 December (08:00-15:00)

Purazur prepared the extraction wells for cleaning by shutting down the installation, opening and dismantling 9 wells and piping. Vereecke cleaned and flushed all of the nine extractions wells. Vereecke came on site with one vacuum truck to execute these works. At the end of the day, water and sludge captured by the vacuum truck has been disposed in dewatering containers at the site.

A blackish hard precipitated product has been observed on the pump and piping of PP05 (see picture 1). This substance has been removed and sampled during the previous cleaning event and a sample has been transported to the 3M environmental lab. Analytical results indicated that the sample contained CaCO₃ and silicate. In October 2017, PP05 was shut off because of a blockage in the effluent piping. During this cleaning event, Vereecke unsuccessfully tried to clear the piping with high water pressure indicating a severe obstruction. As discussed, the next option is to use the spare piping.

At the end of the day the installation was restarted. All pumps work, except for PP05. At PP08, the flow rate remains low and could not be increased with the valve.

Activities performed:

- Shut down of the groundwater extraction installation;
- Opening and dismantling of the wells (PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 and PP10);
- Removal of sediments and depositions from inside the extraction wells and connecting pipes. Cleaning of pumps with high pressurized water.

Friday 15 December (10:00-12:00)

ERM measured the total depth of the extraction wells with an interface meter in order to determine the removed layer of sediment.

Table 1. Measured extraction well depths after the December 2017 cleaning campaign.

Extraction well	Measured Depth (meter below top of well pipe)	Sediments accumulation (cm)
PP01	5.20	0
PP02	5.76	0
PP04	5.72	0
PP05	5.07	0
PP06	4.75	0
PP07	5.63	0
PP08	7.00	0
PP09	6.46	0
PP10	6.48	0

The flow rates were adjusted. Below a summary of the extraction well status after cleaning.

Table 2. Summary of remediation system (March 2017)

Extraction well	Pump in place (y/n)	Reset total volume (y/n)	Operational (y/n)	Q (l/min)
PP01	Y	N	Y	5.0
PP02	Y	N	Y	2.0
PP04	Y	N	Y	2.0
PP05	Y	N	N	0.0
PP06	Y	N	Y	5.0
PP07	Y	N	Y	7.0
PP08	Y	N	Y	5.0
PP09	Y	N	Y	2.0
PP10	Y	N	Y	8.0

The flow rate of PP08 could not be increased with the valve. If these observations are

confirmed in the coming weeks, ERM suggest to take out the pump for revision in Purazur's workshop.

Activities performed :

- Depth measurement of extraction wells; and
- Settings adjustment of the remedial extraction installation.

Annexes

- Photo log

For the Environmental Consultant <i>(bodemsaneringsdeskundige)</i> Name and Signature  Erik Boeckx	For the Client Name and Signature
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Photo log

Picture 1: Black deposition at PP05



Picture 2: Check valve PP06



Picture 3: Pump PP09 before cleaning



Picture 4: Pump PP09 after cleaning



Picture 5: Cleaning well PP06





DAILY BOOK ENVIRONMENTAL SUPERVISION

CLEANING OF EXTRACTION WELLS, PUMPS AND PIPING AT THE 3M SITE, ZWIJNDRECHT

Date: 20/03/2018-30/03/2018

Project number: 0398993

Client: 3M Belgium bvba

Location Canadastraat 11 2070 Zwijndrecht (BE)	Subcontractors involved Purazur nv Chris Vereecke & Zoon bvba
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Distribution list	
3M	Nynke De Schutter (██████████), Wouter Paque ██████████ and Sam Van Beneden (██████████),
Weston Solutions	Dave Cairns (██████████)
ERM	Erik Boeckx (██████████), Nanda Herms (██████████), Mattias Verbeeck (██████████) and Elien Kemme (██████████)
PURAZUR	Reinout Van Loon (██████████), Erwin Mariën (██████████) and Pieter Van der Mussele ██████████

Activities

Introduction:

A groundwater extraction system consisting of nine extraction wells is operational at the 3M site in Zwijndrecht (BE). The aim of the system is to remediate groundwater impacted with Fluor Chemicals (FC's) and to mitigate contaminant migration. The impacted groundwater extracted by the remedial system is treated by the wastewater treatment plant of the site.

The extraction wells and pumps are periodically cleaned to optimize performance of the remedial system. Cleaning is required because of silting and rusting of wells and pumps.

Starting on March 20, 2018, cleaning and maintenance activities were executed by the remedial contractor Purazur and their subcontractor, the industrial cleaning company Chris Vereecke & Zoon (Vereecke), under the environmental supervision of ERM. This document summarizes the actions performed during this cleaning event.

Wednesday 20 March (7:30-12:00)

Purazur prepared the extraction wells for cleaning by shutting down the installation, opening and dismantling 9 wells and piping. After dismantling, the pumps were taken to the workshop of Purazur for technical inspection and revision. A report is written by Purazur on the

condition of the pumps. All pumps can be installed again.
At PP05, the presence of a hard black layer on the pump, the transducer and inside the piping is identified again. The PP05 flow meter has been disconnected and will be inspected and calibrated by 3M. Also inside this flow meter, a black layer was detected.

All piping and flow meters are disconnected and ready for cleaning.

Activities performed:

- Shut down of the groundwater extraction installation;
- Opening and dismantling of the wells (PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 and PP10);

Wednesday 28 March (8:00-16:30)

Vereecke cleaned and flushed all of the nine extractions wells, the connecting piping and flow meters. Vereecke came on site with two vacuum trucks to execute these works. At the end of the day, water and sludge captured by the vacuum truck has been disposed in dewatering containers at the site.

A blackish hard precipitated product has been observed again on the pump and piping of PP05 (see picture 3). This substance has been removed.

In the afternoon ERM adds chemicals and flushes all wells.

Activities performed:

- Removal of sediments and depositions from inside the extraction wells and connecting pipes;
- ERM adds chemicals in wells.

Friday 30 March (8:00-15:30)

Vereecke is on site to clean the pumping wells again and remove sediment and chemicals. At PP07 inflow of sand is observed. Purazur connects all pumps. At PP09, the flow meter and transducer indicate a negative value and remains off. 3M will be asked to check the electric connections of the components.

At the end of the day the installation was restarted. All pumps work, except for PP09. At PP05, the total flow still adds a negative value.

ERM measured the total depth of the extraction wells with an interface meter in order to determine the removed layer of sediment.

Table 1. Measured extraction well depths after the March 2018 cleaning campaign.

Extraction well	Measured Depth (meter below top of well pipe)	Sediments accumulation (cm)
PP01	5.20	0
PP02	5.76	0
PP04	5.72	0
PP05	5.07	0
PP06	4.75	0
PP07	5.63	0
PP08	7.00	0
PP09	6.46	0
PP10	6.48	0

The flow rates were adjusted. Below a summary of the extraction well status after cleaning.

Table 2. Summary of remediation system (March 2018)

Extraction well	Pump in place (y/n)	Reset total volume (y/n)	Operational (y/n)	Q (l/min)
PP01	Y	Y	Y	5.0
PP02	Y	Y	Y	2.0
PP04	Y	Y	Y	2.0
PP05	Y	Y	Y	2.0
PP06	Y	Y	Y	5.0
PP07	Y	Y	Y	8.0
PP08	Y	Y	Y	7.0
PP09	Y	Y	N	0.0
PP10	Y	Y	Y	8.0

Activities performed :

- Installation pumps;
- Depth measurement of extraction wells; and
- Settings adjustment of the remedial extraction installation.

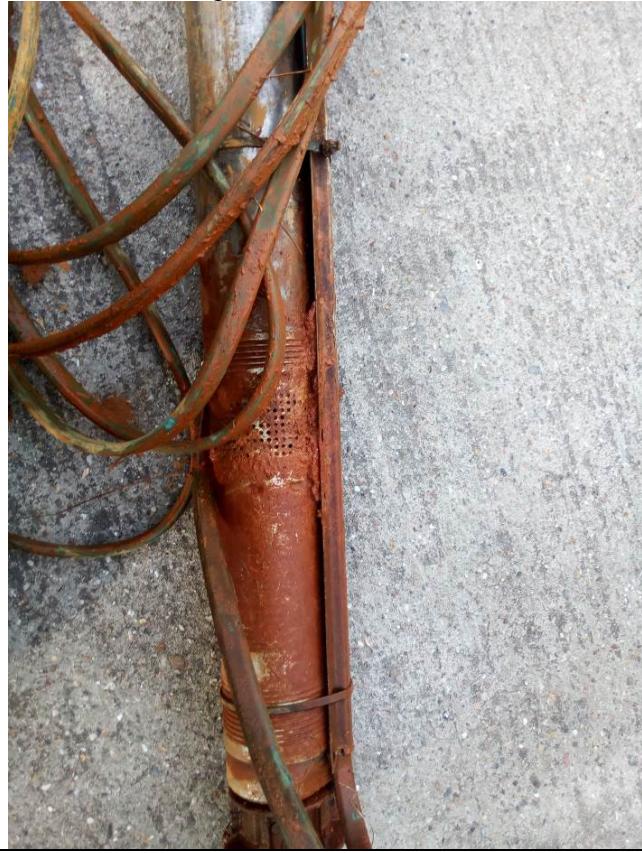
Annexes

- Photo log

For the Environmental Consultant <i>(bodemsaneringsdeskundige)</i> Name and Signature  Erik Boeckx	For the Client Name and Signature
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Photo log

Picture 1: Red deposition at PP10



Picture 2: Grey deposition at PP07



Picture 3: Black deposition at PP05



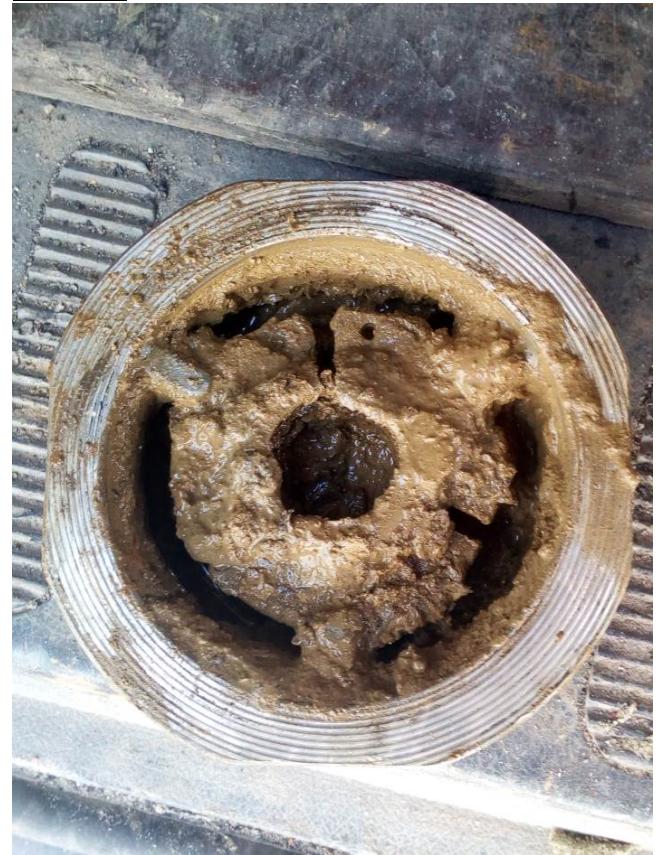
Picture 4: Flow meter PP05



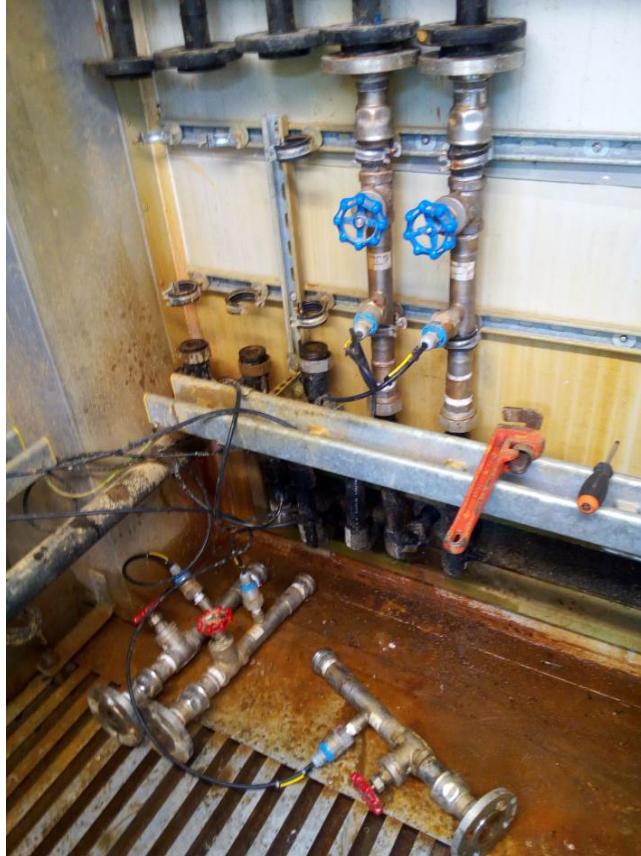
Picture 5: Connection PP10



Picture 6: Non return valve PP07



Picture 7: Preparation of pipe/valves cleaning



Picture 8: Cleaning connections



Picture 9: Cleaned flow meter PP05



Picture 10: Pipe cleaning with a sewer rat



Picture 11: Pipe cleaning



Picture 12: Levels and pressure start up





DAILY BOOK ENVIRONMENTAL SUPERVISION

CLEANING OF EXTRACTION WELLS, PUMPS AND PIPING AT THE 3M SITE, ZWIJNDRECHT

Date: 26-9-2018 until 04-10-2018

Project number: 0451640

Client: 3M Belgium bvba

Location	Subcontractors involved
Canadastraat 11 2070 Zwijndrecht (BE)	Purazur nv Chris Vereecke & Zoon bvba

Distribution list	
3M	Nynke De Schutter (██████████), Wouter Paque (██████████) and Sam Van Beneden (██████████)
Weston Solutions	Dave Cairns (██████████)
ERM	Erik Boeckx (██████████), Nanda Hermes (██████████), Mattias Verbeeck (██████████)
PURAZUR	Reinout Van Loon (██████████), Pieter Van der Mussele (██████████) and Erwin Mariën (██████████)

Activities
<p><u>Introduction:</u></p> <p>A groundwater extraction system consisting of nine extraction wells is operational at the 3M site in Zwijndrecht (BE). The aim of the system is to remediate groundwater impacted with Fluor Chemicals (FC's) and to mitigate contaminant migration. The impacted groundwater extracted by the remedial system is treated by the wastewater treatment plant of the site.</p> <p>The extraction wells and pumps are periodically cleaned to optimize performance of the remedial system. Cleaning is required because of silting and rusting of wells and pumps.</p> <p>Between 26 September 2018 and 04 October, cleaning and maintenance activities were executed by the remedial contractor Purazur and their subcontractor, the industrial cleaning company Chris Vereecke & Zoon (Vereecke), under the environmental supervision of ERM. This document summarizes the actions performed.</p>
<p><u>Wednesday September 26, 2018 (08:00-13:00)</u></p> <p>On Wednesday, Purazur prepared the extraction wells for cleaning by shutting down the installation, opening and dismantling all 9 wells and related piping. After dismantling, the pumps were taken to the workshop of Purazur for technical inspection and revision. A report will be written by Purazur on the condition of the pumps. At PP05, the presence of a hard black precipitation on the pump, the transducer and within the piping is identified again.</p>

Activities performed:

- Shut down of the groundwater extraction installation; and
- Opening and dismantling of the wells (PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 and PP10) in order to transport the pumps to Purazur's technical workshop for revision.

Thursday September 27, 2018 (8:00-15:00)

On Thursday, Purazur/Vereecke cleaned and flushed all (subsurface) pipes of the extraction system of the nine extractions wells. Vereecke came on site with two vacuum trucks to execute these works. The cleaning was performed using high pressured water, resulting in a big amount of sediment removal. At the end of the day, water and sludge captured by the vacuum trucks was disposed in dewatering containers at the site.

Activities performed:

- The electric box in the container is sealed following Lock Out/Tag Out -procedure;
- Purazur dismantled the piping network inside the container;
- Cleaning and flushing of the dismantled pipes in the container; and
- Removal of sediments and depositions from inside the extraction wells and pipes. Flush water from extraction wells PP02, PP07, PP08, PP09 and PP10 is collected by the vacuum truck at the container. Flush water of PP01, PP04, PP05 and PP06 is directly discharged into the 3M chemical sewer.

Monday October 1, 2018 (8:30-14:30)

ERM added Boresaver (2kg) and Iron Clean Liquid (2L) to all wells, except PP01. This well is located inside the construction area for a new building and will be decommissioned and relocated in the near future.

Activities performed:

- Adding Boresaver and Iron Clean Liquid to flush and clean all extraction wells, except for PP01 (by ERM).

Wednesday October 3, 2018 (8:00-16:00)

The preliminary results of the pump tests performed by Purazur in their technical workshop indicated that all pumps are still in good condition and could be re-installed.

On Wednesday, Purazur/Vereecke came on site with one vacuum truck to remove the Boresaver product and Iron Clean Liquid from the extraction wells. Purazur reinstalled the piping-infrastructure inside the container, the flowmeters and re-installed the revised pumps in the WWTC area (PP02, PP06, PP07, PP08, PP09 and PP10).

ERM measured the total depth of the extraction wells with an interface meter in order to determine the removed layer of sediment (see table below).

All flowmeters were reset and the pumps restarted, except PP01. The pump of PP01 is stored next to the spare pumps in the container.

At the end of the day, water and sludge captured by the vacuum truck was disposed in dewatering containers at the site.

Activities performed :

- Removal of cleaning chemicals and cleaning of 8 extraction wells;
- Piping infrastructure of extraction system inside container re-installed;
- Depth measurement of all extraction wells; and
- Re-installation of 6 revised pumps.

Thursday October 4, 2018 (8:00-12:00)

Purazur installs the remaining pumps and connects the flow meters.

After restarting the pump of PP05, the flow meter (blue) still does not respond and indicates a negative flow. Needs to be checked again by 3M (as communicated).

Activities performed :

- Because of low groundwaterlevels and to optimize the extraction, most of the start levels were adjusted in the 3M-PLC;
- Re-installation of the 2 remaining revised pumps; and
- Restart of the extraction system.

Table 1. Measured extraction well depths following the Sept. 2018 chemical cleaning campaign

Extraction well	Measured Depth (meter below top of well pipe)	Sediments accumulation (cm)
PP01	5.20	0
PP02	5.76	0
PP04	5.72	0
PP05	5.07	0
PP06	4.75	0
PP07	5.63	0
PP08	7.00	0
PP09	6.46	0
PP10	6.48	0

The extraction system was restarted after installation of the 8 revised pumps. Below a summary of the extraction well status after cleaning.

Table 2. Summary of remediation system (September 2018)

Extraction well	Pump in place (y/n)	Reset total volume (y/n)	Operational (y/n)	Q (l/min)
PP01	N	N	N	-
PP02	Y	Y	Y	0.9
PP04	Y	Y	Y	0.6
PP05	Y	N	Y	0.3
PP06	Y	Y	Y	4.4
PP07	Y	Y	Y	7.7
PP08	Y	Y	Y	6.3
PP09	Y	Y	Y	1.8
PP10	Y	Y	Y	7.2

Table 3. New settings pump-startlevel (September 2018)

Extraction well	Old start level	New start level
PP01	-	-
PP02	1.00	0.70
PP04	1.00	0.70
PP05	0.90	0.70
PP06	1.00	0.70
PP07	1.20	1.00
PP08	1.20	1.00
PP09	1.00	1.00
PP10	1.20	1.00

Annexes

- Photo log

For the Environmental Consultant

(bodemsaneringsdeskundige)

Name and Signature



Erik Boeckx

For the Client

Name and Signature

Photo log

Picture 1: Collecting pumps



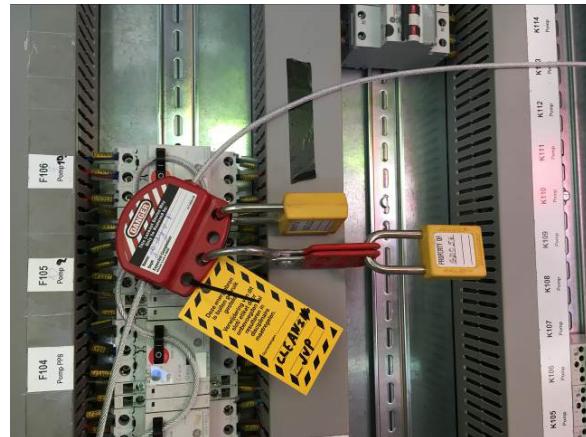
Picture 2: Black precipitation PP05



Picture 4: Black precipitation inside connection PP05



Picture 4: Lock out tag out



Picture 6: Non return valve PP06



Picture 7: Dismanteled piping container



Picture 8: Cleaning flow meters



Picture 9: Discharge of water/sludge at 3M





DAILY BOOK ENVIRONMENTAL SUPERVISION

CLEANING OF EXTRACTION WELLS, PUMPS AND PIPING AT THE 3M SITE, ZWIJNDRECHT

Date: 11-12-2018

Project number: 0451640

Client: 3M Belgium bvba

Location	Subcontractors involved
Canadastraat 11 2070 Zwijndrecht (BE)	Purazur nv Chris Vereecke & Zoon bvba

Distribution list	
3M	Nynke De Schutter (██████████), Wouter Paque (██████████) and Sam Van Beneden ██████████
Weston Solutions	Dave Cairns (██████████)
ERM	Erik Boeckx (██████████), Nanda Hermes (██████████), Mattias Verbeeck (██████████) and Julie Fichefet ██████████
PURAZUR	Reinout Van Loon (██████████) and Pieter Van der Mussele (██████████)

Activities
<u>Introduction:</u> A groundwater extraction system consisting of nine extraction wells is operational at the 3M site in Zwijndrecht (BE). The aim of the system is to remediate groundwater impacted with Fluor Chemicals (FC's) and to mitigate contaminant migration. The impacted groundwater extracted by the remedial system is treated by the wastewater treatment plant of the site.
The extraction wells and pumps are periodically cleaned to optimize performance of the remedial system. Cleaning is required because of silting and rusting of wells and pumps.
On 11 December 2018, cleaning and maintenance activities were executed by the remedial contractor Purazur and their subcontractor, the industrial cleaning company Chris Vereecke & Zoon (Vereecke), under the environmental supervision of ERM. This document summarizes the actions performed.
<u>Tuesday 11 December, 2018 (7:00-15:00)</u> On Tuesday, Purazur prepared the extraction wells for cleaning by shutting down the installation, opening and dismantling all 8 wells and related piping (all wells but PP01, to be relocated). At PP05, the presence of a hard black precipitation on the pump, the transducer and whitin the piping has been identified again. At PP08, the pump exit and the piping were nearly completely blocked by sludge. This could be removed with high pressured water.

Vereecke came on site with one vacuum truck to execute these works. The cleaning was performed using high pressured water, resulting in a big amount of sediment removal. At the end of the day, water and sludge captured by the vacuum trucks was disposed in dewatering containers at the site.

ERM measured the total depth of the extraction wells with an interface meter in order to determine the removed layer of sediment (see table below).

Table 1. Measured extraction well depths following the Dec. 2018 chemical cleaning campaign

Extraction well	Measured Depth (meter below top of well pipe)	Sediments accumulation (cm)
PP01	5.20	0
PP02	5.76	0
PP04	5.72	0
PP05	5.07	0
PP06	4.75	0
PP07	5.63	0
PP08	7.00	0
PP09	6.46	0
PP10	6.48	0

All flowmeters were reset and the pumps restarted, except PP01.

At the end of the day, water and sludge captured by the vacuum truck was disposed in dewatering containers at the site.

The extraction system was restarted after installation of the 8 pumps. Below a summary of the extraction well status after cleaning.

Table 2. Summary of remediation system (December 2018)

Extraction well	Pump in place (y/n)	Reset total volume (y/n)	Operational (y/n)	Q (l/min)
PP01	N	N	N	-
PP02	Y	Y	Y	0.9
PP04	Y	Y	Y	0.6
PP05	Y	Y	Y	0.3
PP06	Y	Y	Y	4.2
PP07	Y	Y	Y	7.5
PP08	Y	Y	Y	6.2
PP09	Y	Y	Y	1.8
PP10	Y	Y	Y	7.2

Table 3. New settings pump-startlevel (December 2018)

Extraction well	Old start level	New start level
PP01	-	-
PP02	1.00	0.70
PP04	1.00	0.70
PP05	0.90	0.70
PP06	1.00	0.70
PP07	1.20	1.00
PP08	1.20	1.00

PP09	1.00	1.00
PP10	1.20	1.00

Activities performed:

- Shut down of the groundwater extraction installation;
- The electric box in the container is sealed following Lock Out/Tag Out –procedure;
- Opening and dismantling of the wells (PP02, PP04, PP05, PP06, PP07, PP08, PP09 and PP10)
- Removal of sediments and depositions from inside the extraction wells and pipes. Flush water from the extraction wells is collected by the vacuum truck;
- Depth measurement of all extraction wells;
- Re-installation of all pumps; and
- Reset of the flow meters and restart.

Annexes

- Photo log

For the Environmental Consultant (bodemsaneringsdeskundige) Name and Signature  Erik Boeckx	For the Client Name and Signature
Photo log	
Picture 1: Rust at PP06 	Picture 2: Black precipitation PP05 

Picture 4: Black precipitation connection PP05



Picture 4: Connection PP08



Picture 6: PP08 before cleaning



Picture 7: PP08 after cleaning



Picture 8: Cleaning PP05



Picture 9: Cleaned PP04



	DAILY BOOK ENVIRONMENTAL SUPERVISION CLEANING OF EXTRACTION WELLS, PUMPS AND PIPING AT THE 3M SITE, ZWIJNDRECHT <u>Date:</u> 03-07-2019 until 16-07-2019 <u>Project number:</u> 0499795 <u>Client:</u> 3M Belgium bvba
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Location	(Sub)contractors involved
Canadastraat 11 2070 Zwijndrecht (BE)	Envisan nv All Clean bvba

Distribution list	
3M	Nynke De Schutter (██████████), Wouter Paque (██████████) and Sam Van Beneden (██████████)
Weston Solutions	Dave Cairns (██████████)
ERM	Nanda Hermes (██████████), Mattias Verbeeck (██████████), Nor Farina Nadzif (██████████), Julie Fichefet (██████████), Erik Boeckx (██████████)
Envisan	Evert Blomme (██████████)

Activities	
<u>Introduction:</u>	A groundwater extraction system consisting of nine extraction wells is operational at the 3M site in Zwijndrecht (BE). The aim of the system is to remediate groundwater impacted with Fluor Chemicals (FC's) and to mitigate contaminant migration. The impacted groundwater extracted by the remedial system is treated by the wastewater treatment plant of the site.
The extraction wells and pumps are periodically cleaned to optimize performance of the remedial system. Cleaning is required because of silting and rusting of wells and pumps.	
Between the 3 rd and 16 th of July 2019, cleaning and maintenance activities were executed by the remedial contractor Envisan and their subcontractor, the industrial cleaning company All Clean under the environmental supervision of ERM. This document summarizes the actions performed.	
<u>Wednesday July 3rd, 2019 (07:00-13:00)</u>	On Wednesday, Envisan and ERM prepared the extraction wells for cleaning by shutting down the installation, opening and dismantling all 9 wells and related piping. After dismantling, the pumps were taken to the Envisan workshop for technical inspection and revision. A report will be written by Envisan on the condition of the pumps. At PP05, the presence of a hard black precipitation on the pump, the transducer and whitin the piping is (again) identified.
<u>Activities performed:</u>	

- Shut down of the groundwater extraction installation;
- The electric box in the container is sealed following Lock Out/Tag Out -procedure; and
- Opening and dismantling of the wells (PP02, PP04, PP05, PP06, PP07, PP08, PP09, PP10 and PP11) in order to transport the pumps to Envisan's technical workshop for revision.

Thursday July 4th, 2019 (8:00-15:00)

On Thursday, ERM, Envisan and All Clean cleaned and flushed all (subsurface) pipes of all extraction wells, except PP09. As PP09 will be abandoned due to Oosterweel related construction activities, this well was decommissioned with bentonite by Envisan. PP09 will be relocated later this year. All Clean came on site with two vacuum trucks to execute these works. The cleaning was performed using high pressured water, resulting in a big amount of sediment removal. At the end of the day, water and sludge captured by the vacuum trucks was disposed in dewatering containers on site. ERM and Envisan added Boresaver (2kg) and Iron Clean Liquid (2L) to all wells, except PP09.

Activities performed:

- Envisan decommissioned PP09;
- Envisan dismantled the piping network inside the container;
- All Clean cleaned and flushed the dismantled pipes in the container; and
- All Clean removed sediments and depositions from inside the extraction wells and pipes. Flush water from extraction wells PP02, PP07, PP08 and PP10 is collected by the vacuum truck at the container. Flush water of PP04, PP05, PP06 and PP11 is directly discharged into the 3M chemical sewer.
- ERM and Envisan added Boresaver and Iron Clean Liquid to flush and clean all extraction wells, except PP09.

Thursday July 11th, 2019 (07:00-17:00)

The preliminary results of the pump tests performed by Envisan in their technical workshop indicated that 4 of the 8 pumps were still in good condition and could be re-installed. The motor of the other 4 pumps needed to be replaced. Only 7 of the 8 revised pumps arrived on site on Thursday. Therefore one of the spare pumps in the container needed to be used.

On Thursday, Envisan, ERM and All Clean came on site with one vacuum truck to remove the Boresaver product and Iron Clean Liquid from the extraction wells. Envisan re-installed the piping infrastructure inside the container, the flowmeters and re-installed the revised pumps around building 16 (PP04, PP05 and PP11) and some of the pumps around the WWTP (PP06 and PP08).

ERM measured the total depth of the extraction wells with an interface meter in order to determine the removed layer of sediment (see table below).

All flowmeters were reset and the pumps of PP04 and PP11 were restarted. PP05 was re-installed but not restarted because there are ongoing construction works around that area. Since some of the pumps around the WWTP were not installed yet, the pumps operated from the container were not restarted.

At the end of the day, water and sludge captured by the vacuum truck was disposed in 3M dewatering containers on site.

Activities performed :

- Removal of cleaning chemicals and cleaning of 8 extraction wells;
- Piping infrastructure of extraction system inside container re-installed;
- Depth measurement of all extraction wells; and

- Re-installation of 5 revised pumps;
- Restart of PP04 and PP11.

Friday July 12th, 2019 (07:00-15:00)

Envisan and ERM installed the 2 remaining revised pumps and 1 spare pump (PP02, PP07 and PP10). The spare pump was tested but was not working, therefore the second spare pump of the container was used. There are no spare pumps left in the container anymore. The spare pump was put inside well PP02.

ERM tried to restart the system but noticed leaks at the piping outside of the container and inside well PP06. Envisan came back in the evening to fix the leaks. Due to a miscommunication, only the pipes inside the container were checked and not outside of the container. The leak inside PP06 was fixed.

Activities performed :

- Re-installation of the 2 remaining revised pumps;
- Re-installation of 1 spare pump;
- 1 spare pump broken;
- No spare pumps left in the container anymore.

Monday July 15th, 2019

Envisan fixed the leaking pipes outside of the container.

Tuesday July 16th, 2019 (10:00-12:00)

ERM restarted the rest of the extraction system (PP02, PP06, PP07 and PP10), except PP08. When PP08 was turned on, no water was arriving in the container and no flow was registered. Envisan will be on site to check and fix this one of the following days (to be communicated by Envisan).

Activities performed :

- Restart of the remaining pumps (PP02, PP06, PP07 and PP10), except PP08.

Table 1. Measured extraction well depths following the July 2019 chemical cleaning campaign

Extraction well	Measured Depth (meter below top of well pipe)	Sediments accumulation (cm)
PP02	5.76	0
PP04	5.74	0
PP05	5.10	0
PP06	4.75	0
PP07	5.63	0
PP08	7.00	0
PP09	/	n.a.
PP10	6.49	0
PP11	6.75	0

The extraction system was (partly) restarted after installation of the 8 revised pumps. Below a summary of the extraction well status after cleaning.

Table 2. Summary of remediation system (July 2019)

Extraction well	Pump in place (y/n)	Reset total volume (y/n)	Operational (y/n)
PP02	Y	Y	Y
PP04	Y	Y	Y
PP05*	Y	Y	N
PP06	Y	Y	Y

PP07	Y	Y	Y	
PP08**	Y	Y	N	
PP09*	N	N	N	
PP10	Y	Y	Y	
PP11	Y	Y	Y	

* PP05 is turned off during the construction works.

** PP08 is turned off until revision by Envisan.

*** PP09 has been abandoned and decommissioned.

Annexes

- Photo log

For the Environmental Consultant

(bodemsaneringsdeskundige)

Name and Signature

Julie Fichefet

For the Client

Name and Signature

Photo log

Picture 1: Lock out/tag out



Picture 2: Labelling pumps



Picture 3: Black precipitation PP05



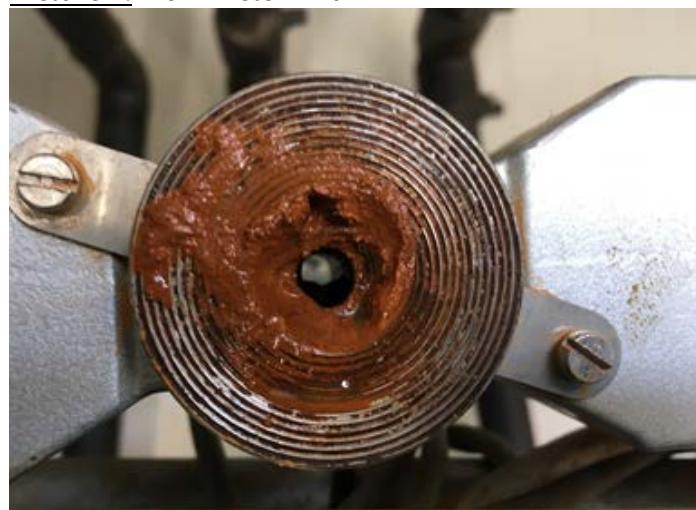
Picture 4: Black precipitation inside connection PP05



Picture 6: Non return valve PP06



Picture 7: Flow meter PP07



Picture 8: Cleaning of PP10 with vacuum trucks



Picture 9: Disconnected pipes inside container



Picture 10: Reconnected pipes and flow meters inside container



Picture 11: Cleaned non return valves PP08



Picture 12: Removal of Boresaver and Iron Clean liquid from PP07

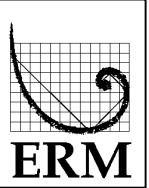


Picture 13: Discharge of water/sludge at 3M



Picture 14: PP09 decommissioned





DAILY BOOK ENVIRONMENTAL SUPERVISION

CLEANING OF EXTRACTION WELLS, PUMPS AND PIPING AT THE 3M SITE, ZWIJNDRECHT

Date: 09-10-2019

Project number: 0499795

Client: 3M Belgium bvba

Location	Subcontractors involved
Canadastraat 11 2070 Zwijndrecht (BE)	Envisan nv All Clean bvba

Distribution list	
3M	Nynke De Schutter (██████████), Wouter Paque (██████████) and Sam Van Beneden (██████████)
Weston Solutions	Dave Cairns (██████████)
ERM	Erik Boeckx (██████████), Nanda Hermes (██████████), Mattias Verbeeck (██████████) and Julie Fichefet (██████████)
Envisan	Evert Blomme (██████████), Kris Dendoncker (██████████)

Activities
<p><u>Introduction:</u></p> <p>A groundwater extraction system consisting of nine extraction wells is operational at the 3M site in Zwijndrecht (BE). The aim of the system is to remediate groundwater impacted with Fluor Chemicals (FC's) and to mitigate contaminant migration. The impacted groundwater extracted by the remedial system is treated by the wastewater treatment plant of the site.</p>
<p>The extraction wells and pumps are periodically cleaned to optimize performance of the remedial system. Cleaning is required because of silting and rusting of wells and pumps.</p>
<p>On 09 October 2019, cleaning and maintenance activities were executed by the remedial contractor Envisan and their subcontractor, the industrial cleaning company All Clean, under the environmental supervision of ERM. This document summarizes the actions performed.</p>
<p><u>Wednesday 09 Oktober 2019 (7:00-15:00)</u></p> <p>On Wednesday, Envisan prepared the extraction wells for cleaning by shutting down the installation, opening and dismantling all 8 wells and related piping. At PP05, there was no presence of a hard black precipitation on the pump. PP09 was not cleaned as it has been dismantled and will be relocated. All pumps and connecting piping and wells were cleaned.</p>
<p>All Clean came on site with one vacuum truck to execute these works. The cleaning was performed using high pressured water, resulting in a big amount of sediment removal. At the end of the day, water and sludge captured by the vacuum trucks was disposed in dewatering</p>

containers at the site.

ERM measured the total depth of the extraction wells with an interface meter in order to determine the removed layer of sediment (see table below).

Table 1. Measured extraction well depths following the Okt. 2019 cleaning campaign

Extraction well	Measured Depth (meter below top of well pipe)	Sediments accumulation (cm)
PP02	5.76	0
PP04	5.72	0
PP05	5.07	0
PP06	4.75	0
PP07	5.63	0
PP08	7.00	0
PP09*	-	-
PP10	6.48	0
PP11	6.75	0

*Well decommissioned

All the pumps restarted, except PP07, PP08 and PP09. Pumps PP07 and PP08 are broke and will be replaced in week 44.

At the end of the day, water and sludge captured by the vacuum truck was disposed in dewatering containers at the site.

The extraction system was restarted after installation of the 6 pumps. Below a summary of the extraction well status after cleaning.

Table 2. Summary of remediation system (Oktober 2019)

Extraction well	Pump in place (y/n)	Reset total volume (y/n)	Operational (y/n)	Q (m³/day)
PP02	Y	N	Y	0.8
PP04	Y	N	Y	0.6
PP05	Y	N	Y	0.2
PP06	Y	N	Y	3.9
PP07*	N	N	N	7.5
PP08*	N	N	N	5.5
PP09**	N	N	N	-
PP10	Y	N	Y	7.0
PP011	Y	N	Y	3.2

* PP07 and PP08 are off, until new pumps will be installed by Envisan (week 44).

** PP09 has been abandoned and decommissioned.

Activities performed:

- Shut down of the groundwater extraction installation;
- The electric box in the container is sealed following Lock Out/Tag Out -procedure;
- Opening and dismantling of the wells (PP02, PP04, PP05, PP06, PP07, PP08, PP10 and PP11)
- Removal of sediments and depositions from inside the extraction wells and pipes. Flush water from the extraction wells is collected by the vacuum truck;
- Depth measurement of all extraction wells;
- Re-installation of all pumps; and

- Restart.

Annexes

- Photo log

For the Environmental Consultant
(bodemsaneringsdeskundige)
 Name and Signature

Erik Boeckx

For the Client
 Name and Signature

Photo log

Picture 1: All Clean vacuumtruck



Picture 2: Cleaning of PP06 with high pressure



Picture 4: PP02 after cleaning



Picture 4: Cleaning of well PP07



Picture 6: PP04 after cleaning



Picture 7: PP05 before cleaning





DAILY BOOK ENVIRONMENTAL SUPERVISION

CLEANING OF EXTRACTION WELLS, PUMPS AND PIPING AT THE 3M SITE, ZWIJNDRECHT

Date: 08-01-2020 until 17-01-2020

Project number: 0499795

Client: 3M Belgium bvba

Location	(Sub)contractors involved
Canadastraat 11 2070 Zwijndrecht (BE)	Envisan nv All Clean bvba

Distribution list	
3M	Nynke De Schutter (██████████), Wouter Paque (██████████) and Sam Van Beneden (██████████)
Weston Solutions	Dave Cairns (██████████)
ERM	Nanda Hermes (██████████), Mattias Verbeeck (██████████), Lieselotte Sorgeloos (██████████), Julie Fichefet (██████████), Erik Boeckx (██████████)
Envisan	Evert Blomme (██████████) and Kris Dendoncker (██████████)

Activities
<p><u>Introduction:</u></p> <p>A groundwater extraction system consisting of eight (PP09 will be relocated in February 2020) extraction wells is operational at the 3M site in Zwijndrecht (BE). The aim of the system is to remediate groundwater impacted with Fluor Chemicals (FC's) and to mitigate contaminant migration. The impacted groundwater extracted by the remedial system is treated by the wastewater treatment plant of the site.</p>
<p>The extraction wells and pumps are periodically cleaned to optimize performance of the remedial system. Cleaning is required because of silting and rusting of wells and pumps.</p>
<p>Between the 8th and 17th of January 2020, cleaning and maintenance activities were executed by the remedial contractor Envisan and their subcontractor, the industrial cleaning company All Clean under the environmental supervision of ERM. This document summarizes the actions performed.</p>
<p><u>Wednesday January 8, 2020 (07:00-16:00)</u></p> <p>On Wednesday, Envisan prepared the extraction wells for cleaning by shutting down the installation, opening and dismantling all 8 functioning wells and related piping. After dismantling, the pumps were taken to the Envisan workshop for technical inspection and revision. A report will be written by Envisan describing pump conditions. At PP05, the usual hard black precipitation on the pump was observed again.</p>

Activities performed:

- Shut down of the groundwater extraction installation;
- The electric box in the container is sealed following Lock Out/Tag Out - procedure; and
- Opening and dismantling of the wells (PP02, PP04, PP05, PP06, PP07, PP08, PP10 and PP11) in order to transport the pumps to Envisan's technical workshop for revision.

Thursday January 9 , 2020 (7:00-16:00)

On Thursday, Envisan and All Clean cleaned and flushed all (subsurface) pipes of all extraction wells. Before this cleaning event, 3M did cleaning works of a drain pipe near PP07 with high pressured water. This created an underground connection towards PP07 resulting in brown sludge coming in the inspection pit and possibly inside the well. The sludge was removed from the well and pit during this cleaning event.

PP09 will be relocated later this year. The piping of PP09 was located and cut, close to the new location. The part of the piping that will be re-used for the new wells has been incorporated in today's cleaning activities. This pit is still open and the piping was not sealed off. All Clean came on site with two vacuum trucks to execute all cleaning works. The cleaning was performed using high pressured water, resulting in a big amount of sediment removal. The pumps and piping were cleaned with water on the grid of the chemical sewer. Flush water from extraction wells PP02, PP07, PP08 and PP10 is collected by the vacuum truck at the container. Flush water of PP04, PP05, PP06 and PP11 is directly discharged into the 3M chemical sewer. At the end of the day, water and sludge captured by the vacuum trucks was disposed in 3M dewatering containers on site. After a first cleaning, Envisan added Boresaver (2kg) and Iron Clean Liquid (2L) to all wells, except PP09.

Activities performed:

- Envisan manually dug up and cut the piping of PP09;
- Envisan dismantled the piping network inside the container;
- All Clean cleaned and flushed the dismantled pipes in the container;
- All Clean removed sediments and depositions from inside the extraction wells and pipes; and
- Envisan added Boresaver and Iron Clean Liquid to flush and clean all extraction wells, except PP09.

Friday January 17, 2020 (07:00-16:00)

The preliminary results of the pump tests performed by Envisan in their technical workshop indicated that all pumps were still in good condition and could be re-installed except for PP04 and PP05. The pump chamber of PP5 and the engine of PP04 was broke. The useful parts could be combined and are installed in PP05. One of the spare pumps in the container was used as the new pump for PP04.

On Friday, Envisan and All Clean came on site with one vacuum truck to remove the Boresaver product and Iron Clean Liquid from the extraction wells. Envisan re-installed the piping infrastructure inside the container, all flowmeters and re-installed the revised pumps in the wells around building 16 (PP04, PP05 and PP11) and around the WWTP (PP02, PP06, PP07, PP08 and PP10).

ERM measured the total depth of the extraction wells with an interface meter in order to determine the removed layer of sediment (see table below).

All installed pumps were restarted.

At the end of the day, water and sludge captured by the vacuum truck was disposed in 3M dewatering containers on site.

Activities performed :

- All-Clean removed cleaning chemicals at 8 extraction wells;
- Envisan re-installed piping infrastructure of extraction system inside container and the revised pumps.

Monday January 20, 2020 (14:00-15:00)

ERM reset all flowmeters and checks the performance of the extraction system.

Table 1. Measured extraction well depths following the January 2020 chemical cleaning campaign

Extraction well	Measured Depth (meter below top of well pipe)	Sediments accumulation (cm)
PP02	5.76	0
PP04	5.74	0
PP05	5.10	0
PP06	4.75	0
PP07	5.63	0
PP08	7.00	0
PP09	/	n.a.
PP10	6.49	0
PP11	6.75	0

The extraction system was restarted after installation of the 8 revised pumps. Below a summary of the extraction well status after cleaning.

Table 2. Summary of remediation system (January 2020)

Extraction well	Pump in place (y/n)	Reset total volume (y/n)	Operational (y/n)
PP02	Y	Y	Y
PP04	Y	Y	Y
PP05	Y	Y	Y
PP06	Y	Y	Y
PP07	Y	Y	Y
PP08	Y	Y	Y
PP09*	N	N	N
PP10	Y	Y	Y
PP11	Y	Y	Y

* PP09 has been abandoned and decommissioned.

Annexes

- Photo log

For the Environmental Consultant
(bodemsaneringsdeskundige)
Name and Signature

For the Client
Name and Signature

Photo log

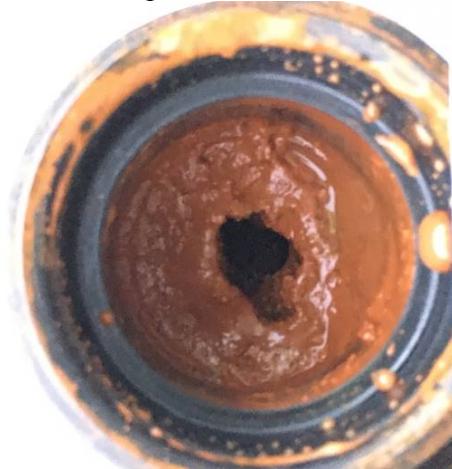
Picture 1: All Clean truck



Picture 2: Sludge at PP10



Picture 3: Pump connection PP10



Picture 4: Pump PP06 after spray off



Picture 5: Cleaned connections PP09 and PP10



Picture 6: Pump PP05 after spray off



Picture 7: Cleaning well PP08



Picture 8: Sludge on PP08



Picture 9: Sludge inside connection PP08



Picture 10: Cleaned non return valves container



Picture 11: Sludge inside PP07



Picture 12: Cleaning PP07



Picture 13: Locating piping PP09



Picture 14: Cut piping PP09





DAILY BOOK ENVIRONMENTAL SUPERVISION

CLEANING OF EXTRACTION WELLS, PUMPS AND PIPING AT THE 3M SITE, ZWIJNDRECHT

Date: 20-22/04/2020

Project number: 0550764

Client: 3M Belgium bvba

Location	Contractors involved
Canadastraat 11 2070 Zwijndrecht (BE)	Envisan nv All Clean bvba

Distribution list	
3M	Nynke De Schutter (██████████), Wouter Paque (██████████) and Sam Van Beneden (██████████)
Weston Solutions	Dave Cairns (██████████)
ERM	Lieselotte Sorgeloos (██████████), Erik Boeckx (██████████), Mattias Verbeeck (██████████) and Julie Fichefet (██████████)
Envisan	Evert Blomme (██████████) and Kris Dendoncker (██████████)

Activities
<p><u>Introduction:</u></p> <p>A groundwater extraction system consisting of nine extraction wells is operational at the 3M site in Zwijndrecht (BE). The aim of the system is to remediate groundwater impacted with Fluor Chemicals (FC's) and to mitigate contaminant migration. The impacted groundwater extracted by the remedial system is treated by the wastewater treatment plant of the site.</p>
<p>The extraction wells and pumps are periodically cleaned to optimize performance of the remedial system. Cleaning is required because of silting and rusting of wells and pumps.</p>
<p>From 20 April 2020 to 22 April 2020, cleaning and maintenance activities were executed by the remedial contractor Envisan and their subcontractor, the industrial cleaning company All Clean, under the environmental supervision of ERM. This document summarizes the actions performed during this cleaning event.</p>
<p><u>Monday 20 April 2020 (7:00-15:00)</u></p> <p>On Monday, Envisan prepared the extraction wells for cleaning by shutting down the installation, opening and dismantling all 8 operational wells, related piping and flowmeters. At PP05, there was presence of a hard black precipitation on the pump and flowmeter. PP09 was not cleaned as this well has been decommissioned, dismantled and to be relocated in the near future. All wells, pumps and connecting piping were cleaned. To increase the impact of the cleaning, Envisan cleaned the pumps (and PP05 flowmeter) with diluted hydrochloric acid.</p>

Tuesday 21 April 2020 (7:00-15:00)

All Clean came on site with one vacuum truck to execute today's cleaning activities. The cleaning was performed using high pressured water, resulting in a big amount of sediment removal. At the end of the day, water and sludge captured by the vacuum trucks was disposed in dewatering containers at the site.

PP09 was not cleaned as this well has been decommissioned, dismantled and to be relocated in the near future. All wells, pumps and connecting piping were cleaned.

After the cleaning event, ERM measured the total depth of the extraction wells with an interface meter in order to determine maximum sediment removal (see table below).

Table 1. Measured extraction well depths following the April '20 cleaning campaign

Extraction well	Measured Depth (meter below top of well pipe)	Sediments accumulation (cm)
PP02	5.76	0
PP04	5.72	0
PP05	5.07	0
PP06	4.75	0
PP07	5.63	0
PP08	7.00	0
PP09*	-	-
PP10	6.48	0
PP11	6.75	0

*Well decommissioned in July 2019

Wednesday 23 April 2020 (7:00-15:00)

All pumps were re-installed, except PP11 (motor block failure). The pump is taken to the Envisan workshop for further inspection and testing.

The extraction system was restarted after installation of the 7 pumps. Below a summary of the extraction well status following this cleaning event.

Table 2. Summary of remediation system (Oktober 2019)

Extraction well	Pump in place (y/n)	Reset total volume (y/n)	Operational (y/n)	Q (m³/day)
PP02	Y	N	Y	0.8
PP04	Y	N	Y	0.6
PP05	Y	N	Y	0.2
PP06	Y	N	Y	4.0
PP07	Y	N	Y	7.4
PP08	Y	N	Y	5.5
PP09*	N	N	N	-
PP10	Y	N	Y	6.9
PP11**	N	N	Y	-

* PP09 has been decommissioned In July 2019

** PP11 was taken by Envisan for check-up

Overview performed activities:

- Shut down of the groundwater extraction installation;
- The electric box in the container is sealed following Lock Out/Tag Out-procedure;
- Opening and dismantling of the wells (PP02, PP04, PP05, PP06, PP07, PP08, PP10 and PP11)
- All pumps were cleaned with diluted hydrochloric acid and high pressured water;
- Removal of sediments and depositions from inside the extraction wells and pipes. Flush water from the extraction wells is collected by the vacuum truck;
- Depth measurement of all extraction wells;
- Re-installation of all operational pumps (except PP11); and
- Restart of the groundwater extraction system.

Annexes

- Photo log

For the Environmental Consultant

(bodemsaneringsdeskundige)

Name and Signature

Erik Boeckx

For the Client

Name and Signature

Photo log

Picture 1: Pumps PP08 and PP10



Picture 2: PP04



Picture 4: PP05



Picture 4: PP06

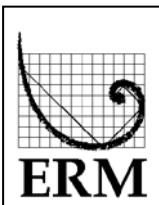


Picture 6: Pumps after cleaning



Picture 7: Cleaning well PP10





DAILY BOOK ENVIRONMENTAL SUPERVISION

CLEANING OF EXTRACTION WELLS, PUMPS AND PIPING AT THE 3M SITE, ZWIJNDRECHT

Date: 06-07-2020 until 09-07-2020

Project number: 0550764

Client: 3M Belgium bvba

Location	(Sub)contractors involved
Canadastraat 11 2070 Zwijndrecht (BE)	Envisan nv All Clean bvba

Distribution list	
3M	Nynke De Schutter (██████████), Charlotte Tack (██████████)
Weston Solutions	Dave Cairns (██████████)
ERM	Mattias Verbeeck (██████████), Lieselotte Sorgeloos (██████████), Julie Fichefet (██████████), Erik Boeckx (E██████████)
Envisan	Kris Dendoncker (██████████)

Activities
<p><u>Introduction:</u></p> <p>A groundwater extraction system consisting of eight extraction wells is operational at the 3M site in Zwijndrecht, Belgium: PP02, PP04, PP05, PP06, PP07, PP08, PP10 and PP11. PP09 was decommissioned in July 2019 and will be replaced by two new extraction wells PP12 and PP13, that will be placed in August 2020. The aim of the system is to remediate groundwater impacted with Fluor Chemicals (FC's) and to mitigate contaminant migration. The impacted groundwater extracted by the remedial system is treated by the wastewater treatment plant of the site.</p>
<p>The extraction wells and pumps are periodically cleaned to optimize performance of the remedial system. Cleaning is required because of silting and rusting of wells and pumps.</p>
<p>Between the 6th and 9th of July 2020, cleaning and maintenance activities were executed by the remedial contractor Envisan and their subcontractor, the industrial cleaning company All Clean under the environmental supervision of ERM. This document summarizes the actions performed during this cleaning event.</p>
<p><u>Monday January 6, 2020 (07:00-16:00)</u></p> <p>On Monday, Envisan prepared the extraction wells for cleaning by shutting down the installation, opening and dismantling the 7 functioning wells and related piping. PP11 has not been operational since March 13, 2020 because of a broken engine. The pump had already been removed prior to the cleaning works and has been at Envisan's atelier. Envisan has ordered 2 new pumps to replace the pump of PP11 and for the new well PP12.</p> <p>After dismantling, the pumps were taken to the Envisan workshop for technical inspection and</p>

revision. A report will be written by Envisan describing pump conditions. At PP05, the usual hard black precipitation on the pump was observed again. It was noticed that a connection between the piping and flowmeter was broken. Envisan does not have that type of connection and has to order new ones before being able to replace this connection.

Activities performed:

- Shut down of the groundwater extraction installation;
- The electric box in the container is sealed following Lock Out/Tag Out - procedure; and
- Opening and dismantling of the wells (PP02, PP04, PP05, PP06, PP07, PP08, and PP10) in order to transport the pumps to Envisan's technical workshop for revision.

Tuesday July 7, 2020 (7:00-16:00)

On Tuesday, Envisan and All Clean cleaned and flushed all (subsurface) pipes of all extraction wells. All Clean came on site with two vacuum trucks to execute all cleaning works. The cleaning was performed using high pressured water, resulting in a big amount of sediment removal.

The pumps and piping were cleaned with water on the grid of the chemical sewer. Flush water from extraction wells PP02, PP07, PP08 and PP10 was collected by the vacuum truck at the container. Flush water of PP04, PP05, PP06 and PP11 was directly discharged into the 3M chemical sewer. Envisan noticed that the pipes of PP08 were completely clogged when disconnecting them. The pump of PP08 had been switched off during ERM's last site visit (16th of June 2020) because it was assumed the pipe was clogged (high pressure and no flow registered). Envisan noticed during their site visit of the 29th of June 2020 that the fans of the pump of PP08 were completely worn off.

At the end of the day, water and sludge captured by the vacuum trucks was disposed in 3M dewatering containers on site. After the cleaning, Envisan added Boresaver (2kg) and Iron Clean Liquid (2L) to all wells.

Activities performed:

- Envisan dismantled the piping network inside the container;
- All Clean cleaned and flushed the dismantled pipes in the container;
- All Clean removed sediments and depositions from inside the extraction wells and pipes; and
- Envisan added Boresaver and Iron Clean Liquid to flush and clean all extraction wells.

Thursday July 9, 2020 (07:00-16:00)

The preliminary results of the pump tests performed by Envisan in their technical workshop indicated that all pumps were still in good condition and could be re-installed. The worn off fans of pump PP08 were replaced by the fans of the broken pump of PP11, so that this pump could be reinstalled as well. The new pump to replace the broken one in PP11, has not been delivered yet and will be placed once it has arrived.

On Thursday, Envisan and All Clean came on site with one vacuum truck to remove the Boresaver product and Iron Clean Liquid from the extraction wells. Envisan re-installed the piping infrastructure inside the container, all flowmeters and re-installed the revised pumps in the wells around building 16 (PP04, PP05 and PP11) and around the WWTP (PP02, PP06, PP07, PP08 and PP10).

ERM measured the total depth of the extraction wells with an interface meter in order to determine the removed layer of sediment (see table below).

All installed pumps were restarted.

At the end of the day, water and sludge captured by the vacuum truck was disposed in 3M

dewatering containers on site.

Table 1. Measured extraction well depths following the July 2020 chemical cleaning campaign

Extraction well	Measured Depth (meter below top of well pipe)	Sediments accumulation (cm)
PP02	5.78	0
PP04	5.75	0
PP05	5.14	0
PP06	4.75	0
PP07	5.70	0
PP08	7.04	0
PP09*	/	n.a.
PP10	6.52	0
PP11	6.80	0

* PP09 has been abandoned and decommissioned in July 2019.

The extraction system was restarted after installation of the 7 revised pumps. PP05 was not restarted because a connection was reported broken when the pump was removed. Envisan has ordered a new connection and is waiting for delivery. PP04 was not restarted due to a technical failure between the control room and the well. The well could be turned on manually but was not working when the system was reset to automatic in the control room. Envisan will contact 3M to see how this issue can be resolved. Below a summary of the extraction well status after cleaning.

Table 2. Summary of remediation system (July 2020)

Extraction well	Pump in place (y/n)	Reset total volume (y/n)	Operational (y/n)
PP02	Y	Y	Y
PP04	Y	Y	N
PP05	Y	N	N
PP06	Y	Y	Y
PP07	Y	Y	Y
PP08	Y	Y	Y
PP09*	N	N	N
PP10	Y	Y	Y
PP11	N	N	N

* PP09 has been abandoned and decommissioned in July 2019.

ERM reseted all flowmeters, except those from PP05 and PP11.

Activities performed :

- All-Clean removed cleaning chemicals at 8 extraction wells;
- Envisan re-installed piping infrastructure of extraction system inside container and the revised pumps; and
- ERM reseted all flowmeters, except for PP05 and PP11.

Annexes

- Photo log

For the Environmental Consultant

(bodemsaneringsdeskundige)

Name and Signature

Erik Boeckx

Julie Fichefet

For the Client

Name and Signature

Photo log

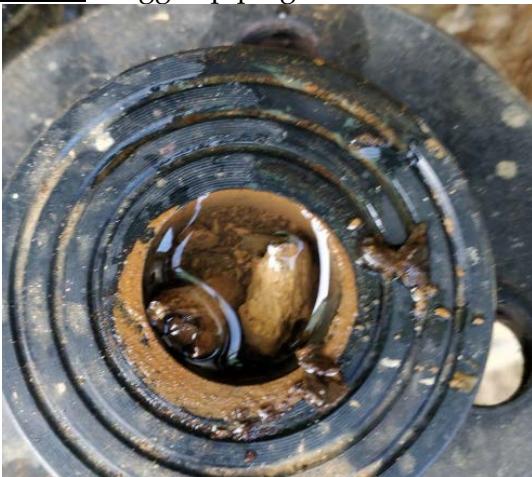
Picture 1: All Clean truck



Picture 2: Pump PP06, PP04 and PP05



Picture 3: Clogged piping PP08



Picture 4: Clogged connection PP08



Picture 5: Cleaning well PP06



Picture 6: Adding Boresaver to well PP07



Picture 7: Sludge inside connection PP06



Picture 8: Non return valve PP04



Picture 9: Non return valve PP04



Picture 10: Cleaned non return valve container



Picture 11: Pumps back from atelier Envisan



Picture 12: Reconnecting pipes at back of the container



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ANALYTICAL REPORT

IAC17-05284-002

3M Lab Request Number: E17-02076

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
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1. Introduction - Summary.

At the request (Lab Request Number: E17-02076) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, one water sample was collected on Nov 7th 2017 from 1 locations and analyzed for the following perfluorinated compounds:

- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$) (and its ^{13}C -labeled analogues)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$) (and its ^{13}C -labeled analogues)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$) (and its ^{13}C -labeled analogues)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$) (and its ^{13}C -labeled analogue)
- ^{13}C -labeled analogue of PFUdA ($C_4F_{21}^{13}C_6F_2^{13}COO^-$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared one sample container for each sampling location under the direction of Geert De Smet. Each empty container was marked with a "fill to here" line and was fortified with a surrogate recovery spike and an internal standard spike, prior to being sent to the field for sample collection.

Table 1 summarizes the sample results with their uncertainty. All PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$ unless noted otherwise. See Section 4 of the report for additional information on the method QC used to assess method uncertainty.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)			
	PFHS	PFOA	PFOS	FOSA
3M vijver	0.532	0.967	1.90	0.026 (a)

(a): The recovery of FOSA for 3M vijver is $\pm 65\%$.

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

The sample was collected in a polyethylene bottle prepared at the SGS Belgium NV, Division IAC Laboratory. Based on the concentrations as reported in previous reports, the bottle was spiked, prior to sample collection, in the laboratory with a known volume of a surrogate recovery solution ($^{13}\text{C}_2\text{-PFHS}$, $^{13}\text{C}_4\text{-PFOA}$, $^{13}\text{C}_4\text{-PFOS}$, $^{13}\text{C}_7\text{-PFUdA}$) and internal standard solution ($^{13}\text{C}_3\text{-PFHS}$, $^{13}\text{C}_8\text{-PFOA}$, $^{13}\text{C}_8\text{-PFOS}$, $^{13}\text{C}_8\text{-FOSA}$). Table 2 below details the sample collected and spikes added to the bottle.

Table 2. Sample Collection and Spike Information.

Sample Identification	Nominal Final Volume Collected (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Final Nominal Spike Concentration (ng/mL)	
				$^{13}\text{C-PFC-SS}$	$^{13}\text{C-PFC-IS}$
3M vijver	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0

Solution A = 1000 ng/mL (nominal) $^{13}\text{C-PFC}$ Surrogate Recovery Standards

Solution B = 1500 ng/mL (nominal) $^{13}\text{C-PFC}$ Internal Standards

2.2. Extraction.

All samples, calibration standards, and associated quality control samples were extracted using a modified procedure of ECO/AV/IAC/064 “Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)”. Briefly, an amount of sample (see table 3) was loaded, onto a pre-conditioned Waters tC18 solid phase extraction (SPE) cartridge (Sep-Pak, 1g, 6cc) using a vacuum manifold. The loaded SPE cartridges were then eluted with 5 mL of methanol using vacuum.

Table 3. Sample amount used.

Sample	Amount of Sample used (mL)	Concentration Factor
3M vijver	40	8
Field Trip Blank	40	8

2.3. Determination of suspended solids in water.

No suspended solids

2.4. Analysis.

All solutions and extracts were analyzed for the PFCs (PFHS, PFOA, PFOS, FOSA) and the surrogate recovery standards (¹³C₂-PFHS, ¹³C₄-PFOA, ¹³C₄-PFOS, ¹³C₇-PFUdA) and internal standards (¹³C₃-PFHS, ¹³C₈-PFOA, ¹³C₈-PFOS, ¹³C₈-FOSA) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetri C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C ₃ -PFHS	2.00 - 3.00	402.0 / 80.0	60	38	51
¹³ C ₂ -PFHS	2.30 - 3.30	403.0 / 84.0	60	38	51
¹³ C ₈ -PFOS	3.00 - 4.00	507.0 / 80.0	60	48	56
¹³ C ₄ -PFOA	2.30 - 3.50	417.0 / 372.0	100	11	14
¹³ C ₈ PFOA	2.30 - 3.50	421.0 / 376.0	60	11	14
PFHS	2.00 - 3.00	398.86 / 79.83	40	38	51
		398.86 / 98.79	40	35	51
PFOA	2.30 - 3.65	412.9 / 218.98	40	11	14
		412.9 / 368.87	40	11	14
PFOS	2.92 - 4.20	498.78 / 79.77	50	48	56
		498.78 / 98.73	50	39	56
¹³ C ₄ -PFOS	3.00 - 4.00	503.0 / 80.0	60	48	56
¹³ C ₇ -PFUdA	4.30 - 5.60	570.0 / 525.0	60	12	17
FOSA	4.00 - 5.60	497.8 / 77.82	125	34	44
¹³ C ₈ FOSA	4.30 - 5.70	506.0 / 78.0	60	34	44

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Extracted Calibration Standard.

Extracted calibration standards were prepared by spiking known amounts of stock solutions containing PFHS, PFOA, PFOS, FOSA and ^{13}C -labeled analogues into 40 mL of HPLC water. Each spiked water standard was then extracted in the same manner as the collected samples. A total of 12 spiked standards ranging from 0.005 ng/mL to 100 ng/mL (nominal) were prepared. Each curve point contains the mixture of internal standards at a nominal concentration of 1 ng/mL. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. The calibration curve will be generated by taking the ratio of the standard peak area counts over the internal standard peak area counts to fit the data for each analyte. Each extracted calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of $\pm 25\%$ ($\pm 30\%$ for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The 0.025ng/mL (nominal) reporting limit is a practical quantitation limit (PQL) required by the requester and it is possible that the samples contain target analytes at quantifiable concentrations below the PQL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within $\pm 25\%$, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by loading 40 mL of HPLC water onto a Waters tC18 solid phase extraction (SPE) cartridge (SEP-Pak, 1g, 6cc) and eluting with 5 mL of methanol using the same extraction procedure as the samples. Method blanks were prepared to evaluate the levels of background contamination in the overall extraction process (glassware, SPE cartridges, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carry over.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of HPLC water, spiked with the surrogate recovery standards and internal standards, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCSs)

Low (0.125 ng/mL nominal concentration) and high (2.5 ng/mL nominal concentration) lab control spikes were prepared and analyzed in duplicate. LCSs were prepared by spiking known amounts of the analytes and surrogates into 40 mL of HPLC water to produce the desired concentration. The spiked water samples were extracted and analyzed in the same manner as the samples.

All LCSs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

All LCSs produced recoveries within the method acceptance criteria of $\pm 15\%$ RPD for precision, except for PFOA LCSH.

Table 8 summarize the LCS recovery results.

Table 7. Lab Control Spike Results.

Extraction date	Description	Nominal Spike Level (ng/mL)	Percent Recovery							
			$^{13}\text{C}_4\text{-PFOA}$	$^{13}\text{C}_4\text{-PFOS}$	$^{13}\text{C}_2\text{-PFHS}$	$^{13}\text{C}_7\text{-PFuDA}$	PFHS	PFOA	PFOS	FOSA
21 November 2017	LCSL01	0.125	90.5	94.9	98.3	87.6	91.4	93.3	111	107
	LCSL02	0.125	101	101	90.5	90.1	89.9	87.5	98.1	108
	Average		95.7	98.0	94.4	88.9	90.7	90.4	105	107
	%RPD		11	6.3	8.3	2.8	1.7	6.4	13	1.2
	LCSH 01	2.5	87.1	109	106	113	88.2	88.5	99.8	108
	LCSH 02	2.5	100	97.3	108	104	94.4	111	106	111
	Average		93.6	103	107	108	91.3	100	103	110
	%RPD		14	11	1.9	8.4	6.8	22 (a)	5.7	3.0

(a) The recovery of the RPD fell outside the method acceptance criterion of $\pm 15\%$.

3.6. Surrogates.

Surrogate recovery standards were added to all samples to evaluate overall method performance.

3.7. Internal Standards.

Internal standards were added to all samples to calculate the concentration of PFCs in the samples by using internal standard calibration.

3.8. Equations.

Recovery.

$$\text{Recovery (\%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

Tabel 8 and 9 summarizes the results for the sample locations.

Table 8. Sample Results PFHS and PFOA.

	PFHS ng/ml	$^{13}\text{C}_2\text{-PFHS}$ %Rec	PFOA ng/ml	$^{13}\text{C}_4\text{-PFOA}$ %Rec
Field Trip Blank	<0.025	93.2	<0.025	92.9
3M vijver	0.532	85.5	0.967	92.9

Table 9. Sample Results PFOS and FOSA.

	PFOS ng/ml	$^{13}\text{C}_4\text{-PFOS}$ %Rec	FOSA ng/ml	$^{13}\text{C}_7\text{-PFuDA}$ %Rec
Field Trip Blank	<0.025	99.0	<0.025	85.3
3M vijver	1.90	81.6	0.026	39.1 (a)

(a): The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130%.

All PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$, except for FOSA for the following sample locations:

(a): The recovery of FOSA for 3M vijver is $\pm 65\%$.

5. Conclusion.

- The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130% for one sample location.
- Lab control spike recoveries fell within the method acceptance criteria of 25%.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Tine Mandonx (ERM Belgium).

8. Signatures.

Geert De Smet,
Lab Operations Manager

Date December 6th, 2017



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date December 6th, 2017

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The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.



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ANALYTICAL REPORT

IAC17-05284-001

3M Lab Request Number: E17-02076

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

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All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction - Summary.

At the request (Lab Request Number: E17-02076) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected between Nov 6th and Nov 7th, 2017 from 11 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOA ($C_6F_{13}^{13}CF_2^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOS ($C_7F_{15}^{13}CF_2SO_3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Geert De Smet. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles. Table 1 summarizes the sample results. All PFBS, PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
Effluent WWTP		3.63	3.40	3.51	<0.208
Bemalingsstation		35.0	51.1	57.1	<0.314
Collector put		12.6	47.0	141	5.74
L4		2.54	1.85	27.0	<0.314
P116		1.46	2.10	9.01	0.223
PP01	86.9	4957	49.3	1423	19.6
PP04	2780	202	664	3984	34.9
PP06	13.4	40.1	125	1025	22.5
PP08	50.0	177	433	3334	20.8
PP10	31.5	181	623	2618	9.03
13		17.7	63.2	173	<1.14

2. Methods – Analytical and Preparatory.**2.1. Sample collection.**

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
Effluent WWTP	100
Bemalingsstation	100
Collector put	100
L4	100
P116	100
PP01	100
PP04	100
PP06	100
PP08	100
PP10	100
13	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
Effluent WWTP	1.0	0.025	Solution A	1.0	-
Bemalingsstation	1.0	0.05	Solution A	2.0	-
Collector put	1.0	0.1	Solution A	5.0	-
L4	1.0	0.05	Solution A	2.0	-
P116	1.0	0.025	Solution A	1.0	-
PP01	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP04	1.0	0.2	Solution A	10.0	0.5mL extract + 10.0mL MeOH (*)
PP06	1.0	0.1	Solution A	10.0	1.0mL extract + 2.0mL MeOH (*)
PP08	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP10	1.0	0.1	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
13	1.0	0.1	Solution A	10.0	-
Field Trip Blank	1.0	0.1	Solution A	10.0	-

(*): MeOH:LCMS-water (60:40)
 Solution A = 1000 ng/mL (nominal) ¹³C-PFC Surrogate Standards

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetre C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
PFBS	2.08 - 2.50	298.93/79.82	40	28	45
		298.93/98.78	40	29	45
PFHS	2.45 - 3.05	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.80 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
¹³ C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ^{13}C -PFOA, PFOS, FOSA and ^{13}C -PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of $\pm 25\%$ ($\pm 30\%$ for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.0769 to 0.128 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.156 to 30.0 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within $\pm 25\%$, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by diluting 1 mL water with 10.0 mL MeOH:LCMS-water (60:40). Method blanks were prepared to evaluate the levels of background contamination in the overall dilution process (glassware, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carryover.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of LCMS-water, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCS)

One lab control spike was prepared and analyzed.

LCS was prepared by spiking known amounts of the analytes into 1.0 mL of LCMS-water to produce the desired concentration.

The spiked water sample was diluted with 10.0 mL MeOH:LCMS-water (60:40) and analyzed in the same manner as the samples.

The LCS produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy, except for ^{13}C -PFOS (recovery is 74.1)

Table 7 summarizes the LCS recovery results

Table 7. Lab Control Spike Results.

Dilution date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
21 November 2017	LCS1	80	116	94.6	89.9	109	95.2	86.5	75.1

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing PFBS, PFHS, PFOA, ^{13}C -PFOA, PFOS, FOSA and ^{13}C -PFOS (QC Lab Low and QC Lab High).

The QCs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 8 summarizes the QCs recovery results.

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
10 Oktober 2017	QC Lab Low inj1	10	114	88.4	119	124	103	89.8	125
	QC Lab High inj1	100	110	84.1	109	106	104	88.2	95.4
	QC Lab Low inj2	10	121	98.8	118	121	118	90.7	123
	QC Lab High inj2	100	122	88.4	100	108	102	81.3	108

3.7. Equations.Recovery.

$$\text{Recovery (\%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

The following tables summarize the results for the sample locations.

Table 9. Sample Results PFBS, PFHS, PFOA, PFOS and FOSA

	PFBS	PFHS	PFOA	PFOS	FOSA
	ng/ml	ng/ml	ng/ml	ng/ml	ng/ml
Effluent WWTP		3.63	3.40	3.51	<0.208
Bemalingsstation		35.0	51.1	57.1	<0.314
Collector put		12.6	47.0	141	5.74
L4		2.54	1.85	27.0	<0.314
P116		1.46	2.10	9.01	0.223
PP01	86.9	4957	49.3	1423	19.6
PP04	2780	202	664	3984	34.9
PP06	13.4	40.1	125	1025	22.5
PP08	50.0	177	433	3334	20.8
PP10	31.5	181	623	2618	9.03
13		17.7	63.2	173	<1.14
Field Trip Blank	<0.854	<1.42	<0.910	<1.38	<1.14

Table 10. Sample Results ^{13}C -PFOA and ^{13}C -PFOS

	^{13}C -PFOA		^{13}C -PFOS	
	ng/ml	% Rec	ng/ml	% Rec
Effluent WWTP	35.3	123	21.5	69.8 (*)
Bemalingsstation	73.0	127	43.7	70.9
Collector put	144	126	98.6	80.1
L4	73.5	128	56.1	91.2
P116	35.6	124	20.3	66.0 (*)
PP01	105	91.2	89.0	72.2
PP04	211	91.9	181	73.6
PP06	142	124	90.7	74
PP08	83.5	72.7	98.8	80.2
PP10	123	107	90.9	73.8
13	133	116	97.3	79.0
Field Trip Blank	137	120	106	86.2

(*): the recovery fell outside the method acceptance criterion of 70-130%.

5. Conclusion.

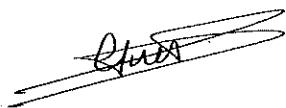
- The recovery of ¹³C-PFOA and ¹³C-PFOS fell within the method acceptance criterion of 70-130%, except for two sample locations.
- Lab control spike recoveries fell within the method acceptance criteria for all compounds, indicating that the analysis method is in control,
- QC Lab Standards recoveries fell within the method acceptance criteria for all compounds.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Tine Mandonx (ERM Belgium).

8. Signatures.

Geert De Smet,
Lab Operations Manager

Date December 6th, 2017



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date December 6th, 2017

I.A.C.
A Division of SGS Belgium NV

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The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.

Final Report

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

November 2017 Sampling

Laboratory Request Number: ISO17-14-04

Report Date – Date of Last Signature

Testing Laboratory

3M Environment, Health, and Safety
3M EHS Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Nicole Cauberghe
3M Belgium EHS Operations
3M Belgium; ZW018/0/33
Phone: [REDACTED]



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M EHS Laboratory

3M EHS Laboratory Technical Director: William K. Reagen, Ph.D.
3M Principal Analytical Investigator: Susan Wolf
Report Author: Chelsie Grochow

Analytical Report ISO17-14-04

Water Sample Analysis at 3M Antwerp, Belgium
November 2017 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M EHS Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. All samples were collected on November 6-7, 2017 and returned to the 3M EHS Laboratory on November 14, 2017 at ambient temperature. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS) (select locations only), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO17-14-04.

The 3M EHS Laboratory prepared sample containers for thirty-one sampling locations. Each sample set consisted of a field sample and field sample duplicate. Nine locations also included a target analyte field matrix spike. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with a mass-labeled internal standard mix and select sample bottles were fortified with surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS prior to being sent to the field for sample collection.

Samples were prepared and analyzed using method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Select internal standards were used to aid in the data quality objectives.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: 2nd Aquifer					
ISO17-14-04-001	BD24-3; Sample	31.2	29.7	982	4.86
ISO17-14-04-001-DUP	BD24-3; Sample Dup	32.0	28.9	993	3.74
		Average	31.6	29.3	988
		%RPD Sample/Sample Dup	2.5	2.7	4.30
ISO17-14-04-002	D09; Sample	182	2490	1100	17.3
ISO17-14-04-002-DUP	D09; Sample Dup	180	2440	1080	16.6
		Average	181	2470	1090
		%RPD Sample/Sample Dup	1.1	2.0	17.0
ISO17-14-04-003	P121; Sample	6.63	3.38	80.6	2.12
ISO17-14-04-003-DUP	P121; Sample Dup	6.75	3.52	83.4	2.24
		Average	6.69	3.45	82.0
		%RPD Sample/Sample Dup	1.8	4.1	2.18
ISO17-14-04-004	P321; Sample	2500	639	6060	0.190
ISO17-14-04-004-DUP	P321; Sample Dup	2590	649	6260	0.174
		Average	2550	644	6160
		%RPD Sample/Sample Dup	3.5	1.6	0.182
Zone: Blokkersdijk nature reserve					
ISO17-14-04-005	3M vijver; Sample	1.15	0.567	2.37	<0.0500
ISO17-14-04-005-DUP	3M vijver; Sample Dup	1.00	0.525	2.43	<0.0500
		Average	1.08	0.546	2.40
		%RPD Sample/Sample Dup	14	7.7	2.5
ISO17-14-04-006	Blokkersdijkvijver noord; Sample	1.20	0.659	0.993	<0.0500
ISO17-14-04-006-DUP	Blokkersdijkvijver noord; Sample Dup	1.07	0.569	0.897	0.0506
		Average	1.14	0.614	0.945
		%RPD Sample/Sample Dup	11	15	0.0506
ISO17-14-04-007	Blokkersdijkvijver - standard; Sample	1.32	1.20	17.0	0.0584
ISO17-14-04-007-DUP	Blokkersdijkvijver - standard; Sample Dup	1.18	1.02	11.1	0.0322
		Average	1.25 ⁽²⁾	1.11 ⁽²⁾	14.1 ⁽²⁾
		%RPD Sample/Sample Dup	11	16	0.0453
ISO17-14-04-008	L21; Sample	0.197	0.0920	4.00	0.0418
ISO17-14-04-008-DUP	L21; Sample Dup	0.183	0.0924	3.70	0.0410
		Average	0.190 ⁽²⁾	0.0922 ⁽²⁾	3.85 ⁽²⁾
		%RPD Sample/Sample Dup	7.4	0.43	0.0414
ISO17-14-04-009	L22; Sample	0.404	0.270	8.52	<0.0250
ISO17-14-04-009-DUP	L22; Sample Dup	0.388	0.252	8.30	0.0294
		Average	0.396 ⁽²⁾	0.261 ⁽²⁾	8.41 ⁽²⁾
		%RPD Sample/Sample Dup	4.0	6.9	0.0294

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 10%, PFBS ± 11%, PFHS ± 8.4%, and PFOS ± 13%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 8.9%, PFHS ± 10%, PFOS ± 13%, and PFOSA ± 12%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
- (4) Data uncertainty was expanded for PFOSA to ±38% based on FMS recovery. See section 4 for more information.
- (5) Data uncertainty was expanded based on surrogate recovery. See section 4 for more information.

Table 1 continued. Sample Results Summary ⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Blokkersdijk nature reserve					
ISO17-14-04-010	L31; Sample	0.634	0.524	7.95	0.0806
ISO17-14-04-010-DUP	L31; Sample Dup	0.734	0.585	7.96	0.0776
		Average	0.684	0.555	7.96
		%RPD Sample/Sample Dup	15	11	0.0791
ISO17-14-04-011	L4; Sample	1.85	1.96	28.6	0.214
ISO17-14-04-011-DUP	L4; Sample Dup	1.88	1.91	26.7	0.228
		Average	1.87	1.94	27.7
		%RPD Sample/Sample Dup	1.6	2.6	0.221
ISO17-14-04-012	P114bis; Sample	1.86	1.25	14.5	0.0462
ISO17-14-04-012-DUP	P114bis; Sample Dup	1.87	1.20	14.2	0.0552
		Average	1.87 ⁽²⁾	1.23 ⁽²⁾	14.4 ⁽²⁾
		%RPD Sample/Sample Dup	0.54	4.1	0.0507
ISO17-14-04-013	P115; Sample	3.96	1.93	1.05	<0.0500
ISO17-14-04-013-DUP	P115; Sample Dup	3.84	1.85	1.09	<0.0500
		Average	3.90	1.89	1.07
		%RPD Sample/Sample Dup	3.1	4.2	<0.0500
ISO17-14-04-014	P116; Sample	1.05	0.526	8.24	0.0958
ISO17-14-04-014-DUP	P116; Sample Dup	1.30	0.626	8.23	0.0938
		Average	1.18	0.576	8.24
		%RPD Sample/Sample Dup	21 ⁽³⁾	17	0.0948
					2.1
Zone: Effluent WWTP					
ISO17-14-04-015	Effluent WWTP; Sample	2.00	2.66	3.64	0.0708
ISO17-14-04-015-DUP	Effluent WWTP; Sample Dup	1.96	2.64	3.84	0.0558
		Average	1.98 ⁽²⁾	2.65 ⁽²⁾	3.74 ⁽²⁾
		%RPD Sample/Sample Dup	2.0	0.75	0.0633
					24 ⁽³⁾
Zone: Palingbeek & Tophatgracht					
ISO17-14-04-025	12; Sample	66.0	16.6	240	0.193
ISO17-14-04-025-DUP	12; Sample Dup	64.9	16.8	245	0.173
		Average	65.5	16.7	0.183
		%RPD Sample/Sample Dup	1.7	1.2	11
ISO17-14-04-026	13; Sample	66.8	24.2	200	0.250
ISO17-14-04-026-DUP	13; Sample Dup	59.5	18.4	196	0.214
		Average	63.2	21.3	0.232
		%RPD Sample/Sample Dup	12	27 ⁽³⁾	16
ISO17-14-04-027	5; Sample	38.8	34.0	67.6	0.254
ISO17-14-04-027-DUP	5; Sample Dup	38.8	34.6	66.9	0.388
		Average	38.8 ⁽²⁾	34.3 ⁽²⁾	0.321
		%RPD Sample/Sample Dup	0.0	1.7	42 ⁽³⁾

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 10%, PFBS \pm 11%, PFHS \pm 8.4%, and PFOS \pm 13%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 8.9%, PFHS \pm 10%, PFOS \pm 13%, and PFOSA \pm 12%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) Data uncertainty was expanded for PFOSA to \pm 38% based on FMS recovery. See section 4 for more information.
- (5) Data uncertainty was expanded based on surrogate recovery. See section 4 for more information.

Table 1 continued. Sample Results Summary ⁽¹⁾

		Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Palingbeek & Tophatgracht						
ISO17-14-04-028	Bemalingsstation; Sample	40.2	35.1	56.7	0.334	
ISO17-14-04-028-DUP	Bemalingsstation; Sample Dup	39.4	35.1	56.4	0.342	
Average		39.8	35.1	56.6	0.338	
%RPD Sample/Sample Dup		2.0	0.0	0.53	2.4	
Zone: Sewer						
ISO17-14-04-029	Collector put; Sample	43.9	16.7	157	16.8	
ISO17-14-04-029-DUP	Collector put; Sample Dup	42.1	16.4	161	11.6	
Average		43.0	16.6	159	14.2	
%RPD Sample/Sample Dup		4.2	1.8	2.5	37 ⁽³⁾	
Zone: Source area – WWTP						
ISO17-14-04-031	P263; Sample	1300	443	7530	23.6	
ISO17-14-04-031-DUP	P263; Sample Dup	1320	440	7380	23.0	
Average		1310	442	7460	23.3 ⁽⁴⁾	
%RPD Sample/Sample Dup		1.5	0.68	2.0	2.6	
		Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Extractions wells P&T						
ISO17-14-04-016	PP01; Sample	53.4	75.8	10600	2060	30.4
ISO17-14-04-016-DUP	PP01; Sample Dup	52.3	74.4	10500	2080	23.8
Average		52.9	75.1	10600 ⁽⁵⁾	2070	27.1
%RPD Sample/Sample Dup		2.1	1.9	0.95	0.97	24 ⁽³⁾
ISO17-14-04-017	PP02; Sample	399	32.0	4500	28300	33.4
ISO17-14-04-017-DUP	PP02; Sample Dup	393	32.5	4510	26100	41.0
Average		396	32.3	4510	27200	37.2
%RPD Sample/Sample Dup		1.5	1.6	0.22	8.1	20
ISO17-14-04-018	PP04; Sample	718	2830	326	6160	56.2
ISO17-14-04-018-DUP	PP04; Sample Dup	714	2840	321	6100	56.2
Average		716	2840	324	6130	56.2
%RPD Sample/Sample Dup		0.56	0.35	1.5	0.98	0.0
ISO17-14-04-019	PP05; Sample	2210	20000	5320	758	37.2
ISO17-14-04-019-DUP	PP05; Sample Dup	2300	20900	5620	793	42.4
Average		2260	20500	5470	776	39.8
%RPD Sample/Sample Dup		4.0	4.4	5.5	4.5	13
ISO17-14-04-020	PP06; Sample	100	13.2	39.3	988	70.8
ISO17-14-04-020-DUP	PP06; Sample Dup	99.7	12.9	38.5	1080	70.6
Average		99.9	13.1	38.9	1030	70.7
%RPD Sample/Sample Dup		0.30	2.3	2.1	8.9	0.28

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 10%, PFBS \pm 11%, PFHS \pm 8.4%, and PFOS \pm 13%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 8.9%, PFHS \pm 10%, PFOS \pm 13%, and PFOSA \pm 12%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) Data uncertainty was expanded for PFOSA to \pm 38% based on FMS recovery. See section 4 for more information.
- (5) Data uncertainty was expanded based on surrogate recovery. See section 4 for more information.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source area - Building 16						
ISO17-14-04-021	PP07; Sample	217	76.5	69.3	1680	67.0
ISO17-14-04-021-DUP	PP07; Sample Dup	221	74.4	68.2	1640	70.8
		Average	219	75.5	68.8	1660
		%RPD Sample/Sample Dup	1.8	2.8	1.6	2.4
ISO17-14-04-022	PP08; Sample	596	42.2	295	6840	50.0
ISO17-14-04-022-DUP	PP08; Sample Dup	578	42.1	298	6900	52.0
		Average	587	42.2	297	6870
		%RPD Sample/Sample Dup	3.1	0.24	1.0	0.87
ISO17-14-04-023	PP09; Sample	625	20.1	314	2230	17.9
ISO17-14-04-023-DUP	PP09; Sample Dup	639	18.7	314	2400	12.5
		Average	632	19.4	314	2320
		%RPD Sample/Sample Dup	2.2	7.2	0.0	7.3
ISO17-14-04-024	PP10; Sample	545	15.6	196	3350	16.5
ISO17-14-04-024-DUP	PP10; Sample Dup	527	15.9	191	3160	16.1
		Average	536	15.8	194	3260
		%RPD Sample/Sample Dup	3.4	1.9	2.6	5.8
ISO17-14-04-030	P21B; Sample	9140	6630	10300	65500	4.00
ISO17-14-04-030-DUP	P21B; Sample Dup	9690	6730	10600	69600	3.98
		Average	9410	6680	10500	67600 ⁽⁵⁾
		%RPD Sample/Sample Dup	5.8	1.5	2.9	6.1

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 10%, PFBS \pm 11%, PFHS \pm 8.4%, and PFOS \pm 13%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 8.9%, PFHS \pm 10%, PFOS \pm 13%, and PFOSA \pm 12%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) Data uncertainty was expanded for PFOSA to \pm 38% based on FMS recovery. See section 4 for more information.
- (5) Data uncertainty was expanded based on surrogate recovery. See section 4 for more information.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis".

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluoroctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluoroctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected on November 6-7, 2017 in Nalgene™ (high-density polyethylene) bottles prepared at the 3M EHS Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. All sample locations were spiked with a mass-labeled internal standard mix at a nominal concentration of 1 ng/mL and select samples were fortified with surrogate recovery standards (SRSs) at a nominal concentration of 0.1 ng/mL prior to sample collection. Samples were received on November 14, 2017.

2.3 Sample Preparation

Select samples were prepared for PFOA, PFHS, PFOS and PFBS (select locations only) using a direct injection by methanol dilution method. Samples that required a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples that required a 1:100 dilution were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol. Samples that required a 1:500 dilution were prepared by diluting 0.02 mL of a well-mixed sample with 10.0 mL of methanol. Samples that required a 1:1000 dilution were prepared by first diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol, followed by a second dilution prepared by diluting 1 mL of the diluted sample into 9 mL of methanol. Diluted samples and LCSs were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

Sample locations Blokkersdijkvijver – standard, L21, L22, P114bis, Effluent WWTP, 5, and Travel Blank sample and FMS Low were analyzed for PFOA, PFHS, and PFOS by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples along with the calibration standards and method blanks.

All samples were prepared for PFOSA by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The laboratory control samples were then diluted with methanol in the same manner.

2.4 Analysis

All samples and quality control samples were analyzed for the target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). SRSs were added and included in the analysis of these samples. Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.2. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

The following analytical runs were used to report the results herein:

12/8/17 (ETS Kirk) External Standard Calibration Analysis:

- All sample locations were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported **except** for the following: Blokkersdijkvijver – standard, L21, L22, P114bis, Effluent WWTP, and Travel Blank sample and FMS Low (PFOA, PFHS, PFOS), 5 (PFOA, PFOS), PP01 (PFHS), PP04 (PFBS), and P21B (PFOS).

12/12/17 (ETS Kirk) External Standard Calibration Analysis:

- Sample locations PP01 (PFHS), PP04 (PFBS), and P21B (PFOS).

12/13/17 (ETS Tesla) Internal Standard Calibration Analysis:

- All sample locations were analyzed for PFOSA. All sample results were reported except for the following: Blokkersdijkvijver – standard, L21, L22, P114bis, Effluent WWTP, and Travel Blank sample.

12/15/17 (ETS Kirk) Internal Standard Calibration Analysis:

- Sample locations Blokkersdijkvijver – standard, L21, L22, P114bis, Effluent WWTP, and Travel Blank sample (PFOA, PFHS, PFOS, PFOSA), 5 (PFOA, PFHS), and Travel Blank FMS Low (PFOA, PFHS, PFOS).

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk	ETS Tesla
Liquid Chromatograph	Agilent 1260	Agilent 1260
Analysis Method	ETS-8-044.3	ETS-8-044.3
Analysis Date	12/8/17, 12/12/17, 12/15/17	12/13/17
Guard column	Prism RP (2.1 mm X 50 mm), 5 μ	Prism RP (2.1 mm X 50 mm), 5 μ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ
Injection Volume	2 or 5 μ L	5 μ L
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 6500+
Ion Source	Turbo Spray	Turbo Spray
Polarity	Negative	Negative
Software	Analyst 1.6.3	Analyst 1.6.3

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis				
Step Number	Total Time (min)	Flow Rate (μ L/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0.00	300	90.0	10.0
1	0.50	300	90.0	10.0
2	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard	Mass Transition Q1/Q3
PFOA	413/369	$[^{13}\text{C}_8]\text{-PFOA}$	421/376
	413/219		
	413/169		
PFBS	299/99	NA	NA
	299/80		
PFHS	399/99	$[^{13}\text{C}_3]\text{-PFHS}$	402/80
	399/80		
PFOS	499/99	$[^{13}\text{C}_8]\text{-PFOS}$	507/80
	499/80		
	499/130		
PFOSA	498/78	$[^{13}\text{C}_8]\text{-PFOSA}$	506/78
$[^{13}\text{C}_4]\text{-PFOA}$	417/372	$[^{13}\text{C}_8]\text{-PFOA}$	421/376
$[^{13}\text{C}_4]\text{-PFOS}$	503/80	$[^{13}\text{C}_8]\text{-PFOS}$	507/80

NA = Not Applicable
The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

3 Data Analysis

3.1 Calibration

12/8/17 and 12/12/17 Analysis (External Standard Calibration): Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed on 12/8/17. On 12/12/17, calibration standards ranging from 0.25 ng/mL to 100 ng/mL (nominal) were analyzed. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL to 25 ng/mL (nominal) or 0.25 ng/mL to 25 ng/mL (nominal) on 12/12/17. A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

12/15/17 Analysis (Internal Standard Calibration): Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of twelve calibration standards ranging from 0.0125 ng/mL to 25 ng/mL (nominal) were analyzed. The calibration standards also contained surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

12/13/17 Analysis of PFOSA (Internal Standard Calibration): Samples were analyzed for PFOSA against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into laboratory reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 1.0 ng/mL. Calibration standards ranging from 0.025 ng/mL.

to 200 ng/mL (nominal) were analyzed. Prior to analysis, the calibration standards were diluted 2x by removing a 0.4 mL aliquot and diluting it with 0.4 mL of methanol. A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of $100\pm 25\%$ ($100\pm 30\%$ for the lowest curve point) were met for all analytes. The correlation coefficient (r) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.3 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the Table 6 below.

Table 6. LOQ

Analyte	LOQ, ng/mL ⁽¹⁾ 12/8/17 Analysis	LOQ, ng/mL ⁽¹⁾ 12/12/17 Analysis	LOQ, ng/mL ⁽²⁾ 12/13/17 Analysis	LOQ, ng/mL ⁽²⁾ 12/15/17 Analysis
PFOA	0.0192	NA	NA	0.0240
PFBS	0.100	0.250	NA	NA
PFHS	0.0200	0.250	NA	0.0250
PFOS	0.0185	0.232	NA	0.0464
PFOSA	NA	NA	0.0500	0.0250

NA = Not Applicable

(1) A dilution factor was not applied to the LOQ.

(2) A dilution factor of 2 was applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of $100\pm 25\%$.

3.5 Blanks

Two types of blanks were prepared and analyzed with the samples: method/solvent blanks and field/trip blanks. Each blank result was reviewed and used to evaluate method performance. The method blanks were used to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. LCSs were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. Surrogates [$^{13}\text{C}_4$]-PFOA and [$^{13}\text{C}_4$]-PFOS were added post dilution when analyzed by external standard. The results in Table 7 for these LCSs are reported with the dilution factor applied.

Method ETS-8-044.3 states that the average recovery of LCSs at each spiking level must be within 80%-120% with a RSD ≤20%. All LCS samples met criteria with the following exceptions:

- 12/8/17: One low-level LCS (LCS-171208-3) was contaminated with PFBS giving a recovery of 323%. The LCS was excluded from the sample set average and RSD calculations and not used to determine data uncertainty.
- 12/12/17: Low-level and mid-level LCSs had an average recovery of 121% for [¹³C₄]-PFOS.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report. The following calculations were used to generate data in Table 7.

$$\text{LCS Percent Recovery} = \frac{\text{Calculated Concentration}}{\text{Spike Concentration}} * 100\%$$

$$\text{LCS% RSD} = \frac{\text{standard deviation LCS replicates}}{\text{average LCS recovery}} * 100\%$$

Table 7. Laboratory Control Spike Results.

Lab ID	PFOA (Linear + Branched)			PFBS		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-171208-1	100	95.4	95.4	100	105	105
LCS-171208-2	100	98.9	98.9	100	95.9	95.9
LCS-171208-3	100	97.4	97.4	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾
Average ± %RSD	97.2% ± 1.8%			100% ± 9.1%		
LCS-171208-4	5000	4980	99.6	5000	5170	103
LCS-171208-5	5000	4920	98.4	5000	5170	103
LCS-171208-6	5000	4780	95.6	5000	4870	97.4
Average ± %RSD	97.9% ± 2.1%			101% ± 3.4%		
LCS-171208-7	35000	31400	89.7	35000	33500	95.7
LCS-171208-8	35000	31900	91.1	35000	34400	98.3
LCS-171208-9	35000	32100	91.7	35000	33500	95.7
Average ± %RSD	90.9% ± 1.1%			96.6% ± 1.5%		

Lab ID	PFHS			PFOS (Linear + Branched)		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-171208-1	100	97.9	97.9	100	81.3	81.3
LCS-171208-2	100	89.4	89.4	100	74.3	74.3
LCS-171208-3	100	95.4	95.4	100	89.4	89.4
Average ± %RSD	94.2% ± 4.6%			81.7% ± 9.3%		
LCS-171208-4	5000	5070	101	5000	4860	97.2
LCS-171208-5	5000	5000	100	5000	4670	93.4
LCS-171208-6	5000	4990	99.8	5000	4670	93.4
Average ± %RSD	100% ± 0.87%			94.7% ± 2.3%		
LCS-171208-7	35000	34900	99.7	35000	34200	97.7
LCS-171208-8	35000	33700	96.3	35000	35000	100
LCS-171208-9	35000	34700	99.1	35000	34200	97.7
Average ± %RSD	98.4% ± 1.9%			98.5% ± 1.3%		

NA = Not Applicable

(1) LCS not reported.

(2) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 12/8/17	[¹³ C ₄]-PFOA			[¹³ C ₄]-PFOS		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-171208-1	0.199	0.219	110	0.190	0.205	108
LCS-171208-2	0.199	0.232	117	0.190	0.205	108
LCS-171208-3	0.199	0.236	118	0.190	0.207	109
Average ± %RSD	115% ± 3.8%			108% ± 0.53%		
LCS-171208-4	1.99	2.11	106	1.90	2.10	111
LCS-171208-5	1.99	2.21	111	1.90	2.08	109
LCS-171208-6	1.99	2.13	107	1.90	2.05	108
Average ± %RSD	108% ± 2.4%			109% ± 1.4%		

ETS-8-044.3 External Calibration Analyzed 12/12/17	PFBS			PFHS		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-171212-1	1000	954	95.4	1000	974	97.4
LCS-171212-2	1000	956	95.6	1000	974	97.4
LCS-171212-3	1000	1000	100	1000	1020	102
Average ± %RSD	97.0% ± 2.7%			98.9% ± 2.7%		
LCS-171212-4	25000	25200	101	25000	25100	100
LCS-171212-5	25000	25200	101	25000	25400	102
LCS-171212-6	25000	25400	102	25000	24900	99.6
Average ± %RSD	101% ± 0.46%			101% ± 1.0%		
LCS-171212-7	70000	70800	101	70000	72700	104
LCS-171212-8	70000	69000	98.6	70000	70600	101
LCS-171212-9	70000	69300	99.0	70000	72800	104
Average ± %RSD	99.6% ± 1.4%			103% ± 1.7%		

NA = Not Applicable

(1) LCS not reported.

(2) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 12/12/17	PFOS (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-171212-1	1000	962	96.2
LCS-171212-2	1000	913	91.3
LCS-171212-3	1000	994	99.4
Average ± %RSD	95.6% ± 4.3%		
LCS-171212-4	25000	24500	98.0
LCS-171212-5	25000	24200	96.8
LCS-171212-6	25000	24200	96.8
Average ± %RSD	97.2% ± 0.71%		
LCS-171212-7	70000	74000	106
LCS-171212-8	70000	70300	100
LCS-171212-9	70000	72400	103
Average ± %RSD	103% ± 2.6%		

ETS-8-044.3 External Calibration Analyzed 12/12/17	[¹³ C ₂]-PFOS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-171212-1	0.478	0.562	118
LCS-171212-2	0.478	0.579	121
LCS-171212-3	0.478	0.592	124
Average ± %RSD	121% ± 2.5%⁽²⁾		
LCS-171212-4	4.78	5.64	118
LCS-171212-5	4.78	5.83	122
LCS-171212-6	4.78	5.90	123
Average ± %RSD	121% ± 2.2%⁽²⁾		

NA = Not Applicable

(1) LCS not reported.

(2) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 1213/17	PFOSA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-171213-1	0.199	0.212	107
LCS-171213-2	0.199	0.210	106
LCS-171213-3	0.199	0.206	104
Average ± %RSD	105% ± 1.5%		
LCS-171213-4	19.9	19.5	98.0
LCS-171213-5	19.9	19.3	97.0
LCS-171213-6	19.9	19.1	96.0
Average ± %RSD	97.0% ± 1.0%		
LCS-171213-7	139	150	108
LCS-171213-8	139	151	109
LCS-171213-9	139	152	109
Average ± %RSD	109% ± 0.66%		

ETS-8-044.3 Internal Calibration Analyzed 12/15/17	PFOA (Linear + Branched)			PFHS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-171215-1	0.190	0.182	95.8	0.198	0.206	104
LCS-171215-2	0.190	0.182	95.8	0.198	0.198	100
LCS-171215-3	0.190	0.186	97.9	0.198	0.208	105
Average ± %RSD	96.5% ± 1.3%			103% ± 2.6%		
LCS-171215-4	1.90	1.94	102	1.98	1.98	100
LCS-171215-5	1.90	1.91	101	1.98	1.94	98.0
LCS-171215-6	1.90	1.86	97.9	1.98	1.98	100
Average ± %RSD	100% ± 2.1%			99.3% ± 1.2%		
LCS-171215-7	33.2	31.8	95.8	34.7	32.4	93.4
LCS-171215-8	33.2	30.4	91.6	34.7	31.0	89.3
LCS-171215-9	33.2	31.2	94.0	34.7	32.0	92.2
Average ± %RSD	93.8% ± 2.3%			91.6% ± 2.3%		

NA = Not Applicable

(1) LCS not reported.

(2) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

Lab ID	PFOS (Linear + Branched)			PFOSA		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-171215-1	0.184	0.199	108	0.198	0.189	95.5
LCS-171215-2	0.184	0.195	106	0.198	0.195	98.5
LCS-171215-3	0.184	0.200	109	0.198	0.198	100
Average ± %RSD	108% ± 1.3%			98.0% ± 2.4%		
LCS-171215-4	1.84	1.87	102	1.98	1.96	99.0
LCS-171215-5	1.84	1.82	98.9	1.98	1.95	98.5
LCS-171215-6	1.84	1.84	100	1.98	1.92	97.0
Average ± %RSD	100% ± 1.4%			98.1% ± 1.1%		
LCS-171215-7	32.2	30.0	93.2	34.7	37.4	108
LCS-171215-8	32.2	30.2	93.8	34.7	36.8	106
LCS-171215-9	32.2	30.8	95.7	34.7	38.6	111
Average ± %RSD	94.2% ± 1.4%			108% ± 2.4%		

Lab ID	[¹³ C ₄]-PFOA			[¹³ C ₄]-PFOS		
	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-171215-1	0.198	0.200	101	0.189	0.195	103
LCS-171215-2	0.198	0.192	97.0	0.189	0.190	101
LCS-171215-3	0.198	0.202	102	0.189	0.197	104
Average ± %RSD	100% ± 2.7%			103% ± 1.9%		
LCS-171215-4	1.98	1.93	97.5	1.89	1.86	98.4
LCS-171215-5	1.98	1.96	99.0	1.89	1.89	100
LCS-171215-6	1.98	1.90	96.0	1.89	1.84	97.4
Average ± %RSD	97.5% ± 1.6%			98.6% ± 1.4%		

NA = Not Applicable

(1) LCS not reported.

(2) LCSs did not meet acceptance criteria of 100 ± 20%.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.4. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in Table 8 below.

Table 8. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	4.99	±10%
PFBS	External	5.40	±11%
PFHS	External	4.19	±8.4%
PFOS	External	6.32	±13%
PFOA	Internal	4.44	±8.9%
PFHS	Internal	5.02	±10%
PFOS	Internal	6.63	±13%
PFOSA	Internal	6.13	±12%

3.8 Field Matrix Spikes (FMS)

Target analyte field matrix spike samples were collected for select sampling points to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions at 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOA and PFOS. The standards used for the preparation of the field matrix spiking solutions greater than 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, select sample bottles contained stable isotope surrogate recovery spikes of [¹³C₄]-PFOA and [¹³C₄]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles prior to sample collection. The [¹³C₄]-PFOA was selected to represent the C8 perfluorocarboxylic acid. The [¹³C₄]-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

The following calculation was used to generate data in section 4 of the report.

$$FMS\ Recovery = \frac{Sample\ Concentration\ of\ FMS - Average\ Concentration\ (Field\ Sample\ & Field\ Sample\ Dup)}{Spike\ Concentration} \times 100\%$$

Table 9. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
Blokkersdijjkvijver – standard, P115, Effluent WWTP	FMS	2.40	2.50	2.50	2.32	2.50
PP06	FMS	100	100	100	100	100
13	FMS	202	203	203	202	2.50
P263	FMS	1030	25.0	1030	1030	25.0
D09, PP04	FMS	2030	25.0	2030	2030	25.0
P321	FMS	2520	20.0	2520	2520	20.0
Trip Blank	Low	2.40	2.50	2.50	2.32	2.50
	High	2520	20.0	2520	2520	20.0

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of $\pm 30\%$, demonstrate that the method is appropriate for the given matrix.

The method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. At times, the spike level exceeded the recommended upper limit of 10 times the analyte concentration. In these instances, the FMS recovery was reported and flagged as above 10 times the sample concentration.

For PFOA, PFBS, PFHS, and PFOS, when the field matrix spike level was not appropriate as compared to the endogenous sample concentration, the surrogate recovery standards were used to assess method accuracy. All field matrix spike and surrogate recovery standard recoveries met acceptance criteria except where noted below.

PP01: The average [$^{13}\text{C}_4$]-PFOS surrogate recovery was 133%. Since no other QC element was analyzed for PFHS, the data uncertainty was expanded to $\pm 33\%$ for PFHS.

P21B: The average [$^{13}\text{C}_4$]-PFOS surrogate recovery was 139%. Since no other QC element was analyzed for PFOS, the data uncertainty was expanded to $\pm 39\%$ for PFOS.

P263: The field matrix spike recovery was 62.0% for PFOSA. The data uncertainty for PFOSA was expanded to $\pm 38\%$ for this location.

Table 10. Location ID: D09

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-002	D09; Sample	182	NA	2490	NA
ISO17-14-04-002-DUP	D09; Sample Dup	180	NA	2440	NA
ISO17-14-04-002-FMS	D09; FMS	2010	90.1	4540	102
Average Concentration (ng/mL) ± %RPD		181 ng/mL ± 1.1%		2470 ng/mL ± 2.0%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-002	D09; Sample	1100	NA	17.3	NA
ISO17-14-04-002-DUP	D09; Sample Dup	1080	NA	16.6	NA
ISO17-14-04-002-FMS	D09; FMS	3030	95.6	40.8	95.4
Average Concentration (ng/mL) ± %RPD		1090 ng/mL ± 1.8%		17.0 ng/mL ± 4.1%	

NA = Not Applicable

Table 11. Location ID: P321

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-004	P321; Sample	2500	NA	639	NA
ISO17-14-04-004-DUP	P321; Sample Dup	2590	NA	649	NA
ISO17-14-04-004-FMS	P321; FMS	4750	87.5	3190	101
Average Concentration (ng/mL) ± %RPD		2550 ng/mL ± 3.5%		644 ng/mL ± 1.6%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-004	P321; Sample	6060	NA	0.190	NA
ISO17-14-04-004-DUP	P321; Sample Dup	6260	NA	0.174	NA
ISO17-14-04-004-FMS	P321; FMS	9150	NC	19.6	97.1 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		6160 ng/mL ± 3.2%		0.182 ng/mL ± 8.8%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) FMS concentration greater than 10 times the sample concentration.

Table 12. Location ID: Blokkersdijkvijver-standard

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-007	Blokkersdijkvijver-standard; Sample	1.32	NA	1.20	NA
ISO17-14-04-007-DUP	Blokkersdijkvijver-standard; Sample Dup	1.18	NA	1.02	NA
ISO17-14-04-007-FMS	Blokkersdijkvijver-standard; FMS	3.58	97.1	3.48	94.8
Average Concentration (ng/mL) ± %RPD		1.25 ng/mL ± 11%		1.11 ng/mL ± 16%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-007	Blokkersdijkvijver-standard; Sample	17.0	NA	0.0584	NA
ISO17-14-04-007-DUP	Blokkersdijkvijver-standard; Sample Dup	11.1	NA	0.0322	NA
ISO17-14-04-007-FMS	Blokkersdijkvijver-standard; FMS	13.2	NC	2.58	101 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		14.1 ng/mL ± 42%⁽²⁾		0.0453 ng/mL ± 58%⁽²⁾	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) FMS concentration greater than 10 times the sample concentration.

(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 13. Location ID: P115

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-013	P115; Sample	3.96	NA	1.93	NA
ISO17-14-04-013-DUP	P115; Sample Dup	3.84	NA	1.85	NA
ISO17-14-04-013-FMS	P115; FMS	6.42	105	4.36	98.8
Average Concentration (ng/mL) ± %RPD		3.90 ng/mL ± 3.1%		1.89 ng/mL ± 4.2%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-013	P115; Sample	1.05	NA	<0.0500	NA
ISO17-14-04-013-DUP	P115; Sample Dup	1.09	NA	<0.0500	NA
ISO17-14-04-013-FMS	P115; FMS	3.46	103	2.54	102 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		1.07 ng/mL ± 3.7%		<0.0500 ng/mL	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 14. Location ID: Effluent WWTP

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-015	Effluent WWTP; Sample	2.00	NA	2.66	NA
ISO17-14-04-015-DUP	Effluent WWTP; Sample Dup	1.96	NA	2.64	NA
ISO17-14-04-015-FMS	Effluent WWTP; FMS	4.40	101	5.08	97.2
Average Concentration (ng/mL) ± %RPD		1.98 ng/mL ± 2.0%		2.65 ng/mL ± 0.75%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-015	Effluent WWTP; Sample	3.64	NA	0.0708	NA
ISO17-14-04-015-DUP	Effluent WWTP; Sample Dup	3.84	NA	0.0558	NA
ISO17-14-04-015-FMS	Effluent WWTP; FMS	5.94	94.8	2.38	92.7 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		3.74 ng/mL ± 5.3%		0.0633 ng/mL ± 24%⁽²⁾	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 15. Location ID: PP04

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-018	PP04; Sample	718	NA	2830	NA	326	NA
ISO17-14-04-018-DUP	PP04; Sample Dup	714	NA	2840	NA	321	NA
ISO17-14-04-018-FMS	PP04; FMS	2570	91.3	2980	NC	2300	97.4
Average Concentration (ng/mL) ± %RPD		716 ng/mL ± 0.56%		2840 ng/mL ± 0.35%		324 ng/mL ± 1.5%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-018	PP04; Sample	6160	NA	56.2	NA
ISO17-14-04-018-DUP	PP04; Sample Dup	6100	NA	56.2	NA
ISO17-14-04-018-FMS	PP04; FMS	8480	NC	82.6	NC
Average Concentration (ng/mL) ± %RPD		6130 ng/mL ± 0.98%		56.2 ng/mL ± 0.0%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 16. Location ID: PP06

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-020	PP06; Sample	100	NA	13.2	NA	39.3	NA
ISO17-14-04-020-DUP	PP06; Sample Dup	99.7	NA	12.9	NA	38.5	NA
ISO17-14-04-020-FMS	PP06; FMS	189	89.2	100	87.0	133	94.1
Average Concentration (ng/mL) ± %RPD		99.9 ng/mL ± 0.30%		13.1 ng/mL ± 2.3%		38.9 ng/mL ± 2.1%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-020	PP06; Sample	988	NA	70.8	NA
ISO17-14-04-020-DUP	PP06; Sample Dup	1080	NA	70.6	NA
ISO17-14-04-020-FMS	PP06; FMS	1200	NC	145	74.3
Average Concentration (ng/mL) ± %RPD		1030 ng/mL ± 8.9%		70.7 ng/mL ± 0.28%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 17. Location ID: 13

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-026	13; Sample	66.8	NA	24.2	NA
ISO17-14-04-026-DUP	13; Sample Dup	59.5	NA	18.4	NA
ISO17-14-04-026-FMS	13; FMS	237	86.1	200	88.0
Average Concentration (ng/mL) ± %RPD		63.2 ng/mL ± 12%		21.3 ng/mL ± 27%⁽²⁾	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-026	13; Sample	200	NA	0.250	NA
ISO17-14-04-026-DUP	13; Sample Dup	196	NA	0.214	NA
ISO17-14-04-026-FMS	13; FMS	382	91.1	2.68	97.9 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		198 ng/mL ± 2.0%		0.232 ng/mL ± 16%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 18. Location ID: P263

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-031	P263; Sample	1300	NA	443	NA
ISO17-14-04-031-DUP	P263; Sample Dup	1320	NA	440	NA
ISO17-14-04-031-FMS	P263; FMS	2310	97.1	1470	99.9
Average Concentration (ng/mL) ± %RPD		1310 ng/mL ± 1.5%		442 ng/mL ± 0.68%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-031	P263; Sample	7530	NA	23.6	NA
ISO17-14-04-031-DUP	P263; Sample Dup	7380	NA	23.0	NA
ISO17-14-04-031-FMS	P263; FMS	10200	NC	38.8	62.0 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		7460 ng/mL ± 2.0%		23.3 ng/mL ± 2.6% ⁽²⁾	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) FMS recovery did not meet acceptance criteria of 100 ± 30%.

(2) The data uncertainty was expanded to ±38% for PFOSA.

Table 19. Location ID: Travel Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-032	Travel Blank	<0.0240	NA	<1.00	NA	<0.0250	NA
ISO17-14-04-032-FMS-LOW	Travel Blank FMS Low	2.32	96.7	2.46	98.4	2.40	96.0
ISO17-14-04-032-FMS-HIGH	Travel Blank FMS High	2370	94.0	22.5	113	2520	100

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-04-032	Travel Blank	<0.0464	NA	<0.0250	NA
ISO17-14-04-032-FMS-LOW	Travel Blank FMS Low	2.24	96.6	2.46	98.4
ISO17-14-04-032-FMS-HIGH	Travel Blank FMS High	2510	99.6	20.4	102

NA = Not Applicable

Table 20. Surrogate Recovery Standard Results (1)

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO17-14-04-001	BD24-3; Sample	99.3	107	NA
ISO17-14-04-001-DUP	BD24-3; Sample Dup	105	106	NA
ISO17-14-04-002	D09; Sample	104	106	NA
ISO17-14-04-002-DUP	D09; Sample Dup	102	106	NA
ISO17-14-04-002-FMS	D09; FMS	96.4	98.2	NA
ISO17-14-04-003	P121; Sample	101	105	NA
ISO17-14-04-003-DUP	P121; Sample Dup	103	102	NA
ISO17-14-04-004	P321; Sample	95.5	101	NA
ISO17-14-04-004-DUP	P321; Sample Dup	103	107	NA
ISO17-14-04-004-FMS	P321; FMS	95.8	100	NA
ISO17-14-04-005	3M vijver; Sample	111	112	NA
ISO17-14-04-005-DUP	3M vijver; Sample Dup	93.6	101	NA
ISO17-14-04-006	Blokkersdijkvijver-Noord; Sample	113	118	NA
ISO17-14-04-006-DUP	Blokkersdijkvijver-Noord; Sample Dup	103	103	NA
ISO17-14-04-007	Blokkersdijkvijver-standard; Sample	91.4 ⁽²⁾	87.5 ⁽²⁾	NA
ISO17-14-04-007-DUP	Blokkersdijkvijver-standard; Sample Dup	87.4 ⁽²⁾	84.8 ⁽²⁾	NA
ISO17-14-04-007-FMS	Blokkersdijkvijver-standard; FMS	88.0 ⁽²⁾	87.7 ⁽²⁾	NA
ISO17-14-04-008	L21; Sample	84.6 ⁽²⁾	85.4 ⁽²⁾	NA
ISO17-14-04-008-DUP	L21; Sample Dup	87.4 ⁽²⁾	81.3 ⁽²⁾	NA
ISO17-14-04-009	L22; Sample	82.0 ⁽²⁾	85.9 ⁽²⁾	NA
ISO17-14-04-009-DUP	L22; Sample Dup	84.6 ⁽²⁾	89.2 ⁽²⁾	NA
ISO17-14-04-010	L31; Sample	102	108	NA
ISO17-14-04-010-DUP	L31; Sample Dup	107	111	NA
ISO17-14-04-011	L4; Sample	114	118	NA
ISO17-14-04-011-DUP	L4; Sample Dup	101	104	NA
ISO17-14-04-012	P114bis; Sample	89.0 ⁽²⁾	86.1 ⁽²⁾	NA
ISO17-14-04-012-DUP	P114bis; Sample Dup	93.2 ⁽²⁾	86.3 ⁽²⁾	NA
ISO17-14-04-013	P115; Sample	105	106	NA
ISO17-14-04-013-DUP	P115; Sample Dup	100	105	NA
ISO17-14-04-013-FMS	P115; FMS	106	114	NA
ISO17-14-04-014	P116; Sample	103	103	NA
ISO17-14-04-014-DUP	P116; Sample Dup	109	114	NA
ISO17-14-04-015	Effluent WWTP; Sample	89.6 ⁽²⁾	82.5 ⁽²⁾	NA
ISO17-14-04-015-DUP	Effluent WWTP; Sample Dup	81.6 ⁽²⁾	93.4 ⁽²⁾	NA
ISO17-14-04-015-FMS	Effluent WWTP; FMS	89.0 ⁽²⁾	85.7 ⁽²⁾	NA
ISO17-14-04-016	PP01; Sample	101	103	133 ⁽³⁾
ISO17-14-04-016-DUP	PP01; Sample Dup	103	106	133 ⁽³⁾
ISO17-14-04-017	PP02; Sample	88.4	88.7	110
ISO17-14-04-017-DUP	PP02; Sample Dup	106	102	107

NA = Not Applicable

- (1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.
- (2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.
- (3) Surrogate recovery standard did not meet acceptance criteria of 100 ± 30%.

Table 20 continued. Surrogate Recovery Standard Results ⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO17-14-04-018	PP04; Sample	97.1	93.4	NA
ISO17-14-04-018-DUP	PP04; Sample Dup	106	104	NA
ISO17-14-04-018-FMS	PP04; FMS	98.6	99.5	NA
ISO17-14-04-019	PP05; Sample	103	102	NA
ISO17-14-04-019-DUP	PP05; Sample Dup	88.5	93.2	NA
ISO17-14-04-020	PP06; Sample	102	106	NA
ISO17-14-04-020-DUP	PP06; Sample Dup	91.5	98.0	NA
ISO17-14-04-020-FMS	PP06; FMS	108	111	NA
ISO17-14-04-021	PP07; Sample	111	114	NA
ISO17-14-04-021-DUP	PP07; Sample Dup	111	114	NA
ISO17-14-04-022	PP08; Sample	98.9	100	NA
ISO17-14-04-022-DUP	PP08; Sample Dup	108	106	NA
ISO17-14-04-023	PP09; Sample	101	102	NA
ISO17-14-04-023-DUP	PP09; Sample Dup	103	107	NA
ISO17-14-04-024	PP10; Sample	105	108	NA
ISO17-14-04-024-DUP	PP10; Sample Dup	96.8	98.6	NA
ISO17-14-04-025	12; Sample	99.7	104	NA
ISO17-14-04-025-DUP	12; Sample Dup	108	115	NA
ISO17-14-04-026	13; Sample	101	103	NA
ISO17-14-04-026-DUP	13; Sample Dup	107	111	NA
ISO17-14-04-026-FMS	13; FMS	98.2	102	NA
ISO17-14-04-027	5; Sample	87.2 ⁽²⁾	110	89.6 ⁽²⁾
ISO17-14-04-027-DUP	5; Sample Dup	89.2 ⁽²⁾	113	90.5 ⁽²⁾
ISO17-14-04-028	Bemalingsstation; Sample	111	113	NA
ISO17-14-04-028-DUP	Bemalingsstation; Sample Dup	101	105	120
ISO17-14-04-029	Collector put; Sample	100	107	121
ISO17-14-04-029-DUP	Collector put; Sample Dup	105	106	NA
ISO17-14-04-030	P21B; Sample	111	114	137 ⁽³⁾
ISO17-14-04-030-DUP	P21B; Sample Dup	88.8	89.2	140 ⁽³⁾
ISO17-14-04-031	P263; Sample	98.3	104	NA
ISO17-14-04-031-DUP	P263; Sample Dup	101	101	NA
ISO17-14-04-031-FMS	P263; FMS	101	105	NA
ISO17-14-04-032	Travel Blank; Sample	91.4 ⁽²⁾	81.5 ⁽²⁾	NA
ISO17-14-04-032-FMS-LOW	Travel Blank; FMS Low	91.8 ⁽²⁾	84.4 ⁽²⁾	NA
ISO17-14-04-032-FMS-HIGH	Travel Blank; FMS High	111	111	NA

NA = Not Applicable

- (1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.
- (2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.
- (3) Surrogate recovery standard did not meet acceptance criteria of 100 ± 30%.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 10-20 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M EHS Laboratory standard operating procedures.

7 Attachments

Chain of Custody Form

8 Signatures


Chelsie J. Grochow, 3M Report Author

Digitally signed by Chelsie J. Grochow
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Susan T. Wolf, 3M Principal Analytical Investigator

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William K. Reagen, Ph.D., 3M EHS Laboratory Technical Director

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DN: c=US, st=MN, l=St. Paul, ou=Laboratory Director, ou=3M Environmental Laboratory - authenticated by LRA, [REDACTED] o=3M, cn=William K. Reagen
Reason: I am approving this document
Date: 2018.01.04 12:04:57 -06'00'

Quality Assurance Representative

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3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
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3M Center, Bldg 260-5N-17
St. Paul, MN 55144

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Fax: [REDACTED]

Project: ISO17-14-04

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)

Completion Date:

Department: 832202 Site Source: 01WJWT10

Project Lead: Susan T. Wolf

Project Number:

Phone Number: [REDACTED]

Date Created: 3/27/2017

Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs; October 2017

*Gw = groundwater
sw = surface water*

Comments:

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO17-14-04-001	BD24-3; Sample	7/11/17	GW	/
ISO17-14-04-001-DUP	BD24-3; Sample Dup	7/11/17	GW	/
ISO17-14-04-002	D09; Sample	6/11/17	GW	/
ISO17-14-04-002-DUP	D09; Sample Dup	6/11/17	GW	/
ISO17-14-04-002-FMS	D09; FMS	6/11/17	GW	/
ISO17-14-04-003	P121; Sample	7/11/17	GW	/
ISO17-14-04-003-DUP	P121; Sample Dup	7/11/17	GW	/
ISO17-14-04-004	P321; Sample	7/11/17	GW	/
ISO17-14-04-004-DUP	P321; Sample Dup	7/11/17	GW	/
ISO17-14-04-004-FMS	P321; FMS	7/11/17	GW	/
ISO17-14-04-005	3M vijver; Sample	7/11/17	SW	/
ISO17-14-04-005-DUP	3M vijver; Sample Dup	7/11/17	SW	/
ISO17-14-04-006	Blokkersdijkvijver - Noord; Sample	7/11/17	SW	/
ISO17-14-04-006-DUP	Blokkersdijkvijver - Noord; Sample Dup	7/11/17	SW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 15 Deg C Received on Ice Other:

Collected by (print):	Collector's signature:						
Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Tina	10/11		FEDEX	Karen L. /per e-mail	11/14/17	11:15 AM

* ACCEPTABLE
~ 21 °C

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3M EHS LABORATORY
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St. Paul, MN 55144

Project: ISO17-14-04 (cont.)

Phone: [REDACTED]
Alt. Phone: [REDACTED]
Fax: [REDACTED]

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 3/27/2017

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

GW = Groundwater
SW = surface water

Project Description: 3M Antwerp Water Sampling for PFCs; October 2017

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
✓ ISO17-14-04-007	Blokkersdijkvijver – standard; Sample	7/11/17	SW	/
✓ ISO17-14-04-007-DUP	Blokkersdijkvijver – standard; Sample Dup	7/11/17	SW	/
✓ ISO17-14-04-007-FMS	Blokkersdijkvijver – standard; FMS	7/11/17	SW	/
✓ ISO17-14-04-008	L21; Sample	7/11/17	GW	/
✓ ISO17-14-04-008-DUP	L21; Sample Dup	7/11/17	GW	/
✓ ISO17-14-04-009	L22; Sample	7/11/17	GW	/
✓ ISO17-14-04-009-DUP	L22; Sample Dup	7/11/17	GW	/
✓ ISO17-14-04-010	L31; Sample	7/11/17	GW	SW /
✓ ISO17-14-04-010-DUP	L31; Sample Dup	7/11/17	GW	SW /
✓ ISO17-14-04-011	L4; Sample	7/11/17	GW	/
✓ ISO17-14-04-011-DUP	L4; Sample Dup	7/11/17	GW	/
✓ ISO17-14-04-012	P114bis; Sample	7/11/17	GW	/
✓ ISO17-14-04-012-DUP	P114bis; Sample Dup	7/11/17	GW	/
✓ ISO17-14-04-013	P115; Sample	7/11/17	GW	/
✓ ISO17-14-04-013-DUP	P115; Sample Dup	7/11/17	GW	/
✓ ISO17-14-04-013-FMS	P115; FMS	7/11/17	GW	/
✓ ISO17-14-04-014	P116; Sample	7/11/17	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 15 Deg C Received on Ice Other:

Collected by (print): Tina Mandony (TMA) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	10/11		FEDEX	Karen H. Pace	11/4/17	11:15am

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Project: ISO17-14-04 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 3/27/2017

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

GW = groundwater

Project Description: 3M Antwerp Water Sampling for PFCs; October 2017

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO17-14-04-014-DUP	P116; Sample Dup	6/11/17	GW	/
ISO17-14-04-015	Effluent WWTP; Sample	6/11/17	GW	/
ISO17-14-04-015-DUP	Effluent WWTP; Sample Dup	6/11/17	GW	/
ISO17-14-04-015-FMS	Effluent WWTP; FMS	6/11/17	GW	/
ISO17-14-04-016	PP01; Sample	6/11/17	GW	/
ISO17-14-04-016-DUP	PP01; Sample Dup	6/11/17	GW	/
ISO17-14-04-017	PP02; Sample	6/11/17	GW	/
ISO17-14-04-017-DUP	PP02; Sample Dup	6/11/17	GW	/
ISO17-14-04-018	PP04; Sample	6/11/17	GW	/
ISO17-14-04-018-DUP	PP04; Sample Dup	6/11/17	GW	/
ISO17-14-04-018-FMS	PP04; FMS	6/11/17	GW	/
ISO17-14-04-019	PP05; Sample	6/11/17	GW	BLACK
ISO17-14-04-019-DUP	PP05; Sample Dup	6/11/17	GW	BLACK
ISO17-14-04-020	PP06; Sample	6/11/17	GW	/
ISO17-14-04-020-DUP	PP06; Sample Dup	6/11/17	GW	/
ISO17-14-04-020-FMS	PP06; FMS	6/11/17	GW	/
ISO17-14-04-021	PP07; Sample	6/11/17	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 15 Deg C Received on Ice Other:

Collected by (print): Nicole Cauberghe (GW) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	<u>Nicole</u>	<u>10/11</u>		<u>FEDEX</u>	<u>Karen Huf /PACF</u>	<u>11/14/17</u>	<u>11:15AM</u>

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Project: ISO17-14-04 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 3/27/2017

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

GW = groundwater
SW = surface water
WW = wastewater

Project Description: 3M Antwerp Water Sampling for PFCs; October 2017

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
✓ ISO17-14-04-021-DUP	PP07; Sample Dup	6/11/17	GW	/
✓ ISO17-14-04-022	PP08; Sample	6/11/17	GW	/
✓ ISO17-14-04-022-DUP	PP08; Sample Dup	6/11/17	GW	/
✓ ISO17-14-04-023	PP09; Sample	6/11/17	GW	/
✓ ISO17-14-04-023-DUP	PP09; Sample Dup	6/11/17	GW	/
✓ ISO17-14-04-024	PP10; Sample	6/11/17	GW	/
✓ ISO17-14-04-024-DUP	PP10; Sample Dup	6/11/17	GW	/
✓ ISO17-14-04-025	12; Sample	7/11/17	SW	/
✓ ISO17-14-04-025-DUP	12; Sample Dup	7/11/17	SW	/
✓ ISO17-14-04-026	13; Sample	7/11/17	SW	/
✓ ISO17-14-04-026-DUP	13; Sample Dup	7/11/17	SW	/
✓ ISO17-14-04-026-FMS	13; FMS	7/11/17	SW	/
✓ ISO17-14-04-027	5; Sample	7/11/17	SW	/
✓ ISO17-14-04-027-DUP	5; Sample Dup	7/11/17	SW	/
✓ ISO17-14-04-028	Bemalingsstation; Sample	7/11/17	SW	/
✓ ISO17-14-04-028-DUP	Bemalingsstation; Sample Dup	7/11/17	SW	/
✓ ISO17-14-04-029	Collector put; Sample	6/11/17	WW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 15 Deg C Received on Ice Other:

Collected by (print): Tine Mandoux (GWW) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	Tine	10/11		FEDEX	Karen Hegg / PAC	11/14/17	11:15AM

* ACCEPTABLE
~21°C

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Project: ISO17-14-04 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 3/27/2017

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

GW = groundwater
WW = waste water

Project Description: 3M Antwerp Water Sampling for PFCs; October 2017

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
✓ ISO17-14-04-029-DUP	Collector put; Sample Dup	6/11/17	WW	/
✓ ISO17-14-04-030	P21B; Sample	6/11/17	GW	BLACK
✓ ISO17-14-04-030-DUP	P21B; Sample Dup	6/11/17	GW	BLACK
✓ ISO17-14-04-031	P263; Sample	7/11/17	GW	/
✓ ISO17-14-04-031-DUP	P263; Sample Dup	7/11/17	GW	/
✓ ISO17-14-04-031-FMS	P263; FMS	7/11/17	GW	/
✓ ISO17-14-04-032	Travel Blank; Sample	/	/	*
✓ ISO17-14-04-032-FMS-HIGH	Travel Blank; FMS High	/	/	*
✓ ISO17-14-04-032-FMS-LOW	Travel Blank; FMS Low	/	/	*

* Travel blank samples have been prepared by the 3M EHS Laboratory with the bottle order.

) delivered separately from the sample bottles
J trip glasses arrived on 8/11 at 3M Zwijndrecht

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 15 Deg C Received on Ice Other:

Collected by (print): Nicole Maertensx (WW) Collector's signature: Nicole Maertens

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
9	<u>Nicole Maertens</u>	<u>10/11</u>		<u>FEDEX</u>	<u>Naomi Schepers</u>	<u>11/14/17</u>	<u>11:15 AM</u>

* ACCEPTABLE
~21°C

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3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC18-00131

3M Lab Request Number: E18-0025

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: March 2nd, 2018

Requester

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SGS Belgium NV

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Member of the SGS Group (Société Générale de Surveillance)

Registered office: Noorderlaan 87 B-2030 Antwerpen H.R. Antwerpen 141.810 BTW BE 404.882.750 Dexia 550-3560000-93
All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction - Summary.

At the request (Lab Request Number: E18-0025) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected between January 29th and February 1st, 2018 from 29 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOA ($C_6F_{13}^{13}CF_2^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOS ($C_7F_{15}^{13}CF_2SO_3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Geert De Smet. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles. Table 1 summarizes the sample results. All PFBS, PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
D09		3001	196	1362	19.0
D17		68.0	222	57.1	<10.5
D18		129	238	39.7	3.35
P119A		25.7	74.5	24.9	6.02
P119B		621	1052	83.4	<5.73
P121		<0.164	<0.306	0.782	<0.176
L21		0.175	<0.306	2.67	<0.176
L22		<0.164	<0.306	6.27	<0.176
L31		0.716	0.985	2.04	<0.176
P115		1.60	5.17	0.377	<0.176
Effluent WWTP		35.5	34.7	64.4	1.56
Bemalingsstation		10.9	17.5	29.9	<0.262
Collector Put		10.3	38.2	130	8.22
P27	715	13.6	128	562	68.7
P304	304	132	424	973	15.8
P305	97.6	152	120	378	11.4
P42	39.7	230	351	4239	13.8
M4		196	1345	5747	37.7
P118C		156	392	1797	18.6
P119C		469	1030	4748	49.3
P264		160	293	1197	32.0
P265B		14.4	36.2	492	24.6
P371		76.8	209	2393	34.4
P374		617	578	1563	16.5
B3-bis		2.20	8.38	79.9	<0.525
B7		0.985	1.85	43.4	<0.262
P378		196	190	749	<5.73
PA112		89.5	367	195	28.7

2. Methods – Analytical and Preparatory.**2.1. Sample collection.**

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
D09	100
D16	100
D17	100
D18	100
P119A	100
P119B	100
P121	100
L21	100
L22	100
L31	100
P115	100
Effluent WWTP	100
Bemalingsstation	100
Collector Put	100
P27	100
P304	100
P305	100
P42	100
M4	100
P118C	100
P119C	100
P264	100
P265B	100
P371	100
P374	100
B3-bis	100
B7	100
P378	100
PA112	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
D09	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
D17	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
D18	1.0	0.1	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
P119A	1.0	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P119B	1.0	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P121	1.0	0.05	Solution A	10.0	-
L21	1.0	0.05	Solution A	1.0	-
L22	1.0	0.05	Solution A	1.0	-
L31	1.0	0.05	Solution A	1.0	-
P115	1.0	0.05	Solution A	1.0	-
Effluent WWTP	1.0	0.05	Solution A	1.0	-
Bemalingsstation	1.0	0.05	Solution A	2.0	-
Collector Put	0.5	0.1	Solution A	10.0	-
P27	1.0	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P304	0.5	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P305	1.0	0.1	Solution A	10.0	1.0mL extract + 2.0mL MeOH (*)
P42	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
M4	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P118C	0.5	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P119C	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P264	1.0	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P265B	1.0	0.1	Solution A	10.0	1.0mL extract + 2.0mL MeOH (*)
P371	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P374	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
B3-bis	1.0	0.1	Solution A	5.0	-
B7	1.0	0.05	Solution A	2.0	-
P378	1.0	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
PA112	1.0	0.1	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
Field Trip Blank	1.0	0.1	Solution A	10.0	-

(*): MeOH:LCMS-water (60:40)
Solution A = 1000 ng/mL (nominal) ^{13}C -PFC Surrogate Standards

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetri C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
PFBS	2.08 - 2.50	298.93/79.82	40	28	45
		298.93/98.78	40	29	45
PFHS	2.45 - 3.05	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.80 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
¹³ C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of \pm 25% (\pm 30% for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.0790 to 0.15 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.162 to 18.3 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within \pm 25%, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by diluting 1 mL water with 10.0 mL MeOH:LCMS-water (60:40). Method blanks were prepared to evaluate the levels of background contamination in the overall dilution process (glassware, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carryover.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of LCMS-water, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCS)

One lab control spike was prepared and analyzed.

LCS was prepared by spiking known amounts of the analytes into 1.0 mL of LCMS-water to produce the desired concentration.

The spiked water sample was diluted with 10.0 mL MeOH:LCMS-water (60:40) and analyzed in the same manner as the samples.

The LCS produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 7 summarizes the LCS recovery results

Table 7. Lab Control Spike Results.

Dilution date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
18 Feb 2018	LCS1	80	97.6	99.5	88.6	103	103	93.2	78.8

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing PFBS, PFHS, PFOA, ^{13}C -PFOA, PFOS, FOSA and ^{13}C -PFOS (QC Lab Low and QC Lab High).

The QCs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 8 summarizes the QCs recovery results.

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
25 Jan 2018	QC Lab Low inj1	10	103	86.5	103	124	101	97.8	110
	QC Lab High inj1	100	85.9	87.0	107	94.0	100	92.6	88.1
	QC Lab Low inj2	10	120	104	111	126	116	109	124
	QC Lab High inj2	100	90.3	88.8	88	84.3	102	95.0	101

3.7. Equations.Recovery.

$$\text{Recovery (\%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

The following tables summarize the results for the sample locations.

Table 9. Sample Results PFBS, PFHS, PFOA, PFOS and FOSA

	PFBS ng/ml	PFHS ng/ml	PFOA ng/ml	PFOS ng/ml	FOSA ng/ml
D09		3001	196	1362	19.0
D17		68.0	222	57.1	<10.5
D18		129	238	39.7	3.35
P119A		25.7	74.5	24.9	6.02
P119B		621	1052	83.4	<5.73
P121		<0.164	<0.306	0.782	<0.176
L21		0.175	<0.306	2.67	<0.176
L22		<0.164	<0.306	6.27	<0.176
L31		0.716	0.985	2.04	<0.176
P115		1.60	5.17	0.377	<0.176
Effluent WWTP		35.5	34.7	64.4	1.56
Bemalingsstation		10.9	17.5	29.9	<0.262
Collector Put		10.3	38.2	130	8.22
P27	715	13.6	128	562	68.7
P304	304	132	424	973	15.8
P305	97.6	152	120	378	11.4
P42	39.7	230	351	4239	13.8
M4		196	1345	5747	37.7
P118C		156	392	1797	18.6
P119C		469	1030	4748	49.3
P264		160	293	1197	32.0
P265B		14.4	36.2	492	24.6
P371		76.8	209	2393	34.4
P374		617	578	1563	16.5
B3-bls		2.20	8.38	79.9	<0.525
B7		0.985	1.85	43.4	<0.262
P378		196	190	749	<5.73
PA112		89.5	367	195	28.7
Field Trip Blank	<0.877	<0.887	<1.66	<1.33	<0.955

Table 10. Sample Results ^{13}C -PFOA and ^{13}C -PFOS

	^{13}C -PFOA		^{13}C -PFOS	
	ng/ml	% Rec	ng/ml	% Rec
D09	92.8	80.9	120	97.5
D17	111	96.8	152	124
D18	96.5	84.0	145	118
P119A	88.3	76.9	157	128
P119B	95.9	83.5	150	122
P121	65.7	114	76.5	124
L21	60.3	105	75.3	122
L22	65.0	113	76.8	125
L31	73.0	127	77.8	126
P115	74.0	129	79.1	129
Effluent WWTP	68.8	120	76.7	125
Bemalingsstation	69.8	122	75.9	123
Collector Put	111	96.8	153	124
P27	97.9	85.2	146	119
P304	103	89.3	154	125
P305	124	108	157	127
P42	91.9	80.0	139	112
M4	95.7	83.4	89.2	72.4
P118C	96.7	84.2	103	84.0
P119C	98.0	85.4	96.4	78.2
P264	122	106	104	84.1
P265B	99.3	86.5	140	113
P371	87.5	76.3	118	95.8
P374	102	88.9	123	100
B3-bis	116	101	155	126
B7	55.4	96.5	77.9	127
P378	98.5	85.8	155	126
PA112	97.6	85.0	151	122
Field Trip Blank	101	87.6	133	108

5. Conclusion.

- The recovery of ¹³C-PFOA and ¹³C-PFOS fell within the method acceptance criterion of 70-130%.
- Lab control spike recoveries fell within the method acceptance criteria for all compounds, indicating that the analysis method is in control,
- QC Lab Standards recoveries fell within the method acceptance criteria for all compounds.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Tine Mandonx (ERM Belgium).

8. Signatures.

Geert De Smet,
Lab Operations Manager

Date March 2nd, 2018



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date March 2nd, 2018

I.A.C.
A Division of SGS Belgium NV

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The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.



Final Report

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

January 2018 Sampling

Laboratory Request Number: ISO18-14-01

Report Date – Date of Last Signature

Testing Laboratory

3M Environment, Health, and Safety
3M EHS Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Nicole Cauberghe
3M Belgium EHS Operations
3M Belgium; ZW018/0/33
Phone: [REDACTED]



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M EHS Laboratory

3M EHS Laboratory Manager: Brian T. Mader
3M Principal Analytical Investigator: Susan Wolf
Report Author: Scott Porcher

Analytical Report ISO18-14-01

Water Sample Analysis at 3M Antwerp, Belgium
January 2018 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M EHS Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. All samples were collected on January 22, 29-31 and February 1, 2018, and returned to the 3M EHS Laboratory on February 7, 2018, at ambient temperature. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS) (select locations only), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO18-14-01.

The 3M EHS Laboratory prepared sample containers for sixty-six sampling locations. Each sample set consisted of a field sample and field sample duplicate. Eighteen locations also included a target analyte field matrix spike. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with a mass-labeled internal standard mix and select sample bottles were fortified with surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS prior to being sent to the field for sample collection. During sample collection, sample location D16 was not sampled.

Samples were prepared and analyzed using method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Select internal standards were used to aid in the data quality objectives.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: 2nd Aquifer					
ISO18-14-01-001	BD24-3; Sample	54.7	40.0	553	13.1
ISO18-14-01-001-DUP	BD24-3; Sample Dup	54.2	40.4	545	12.1
		Average	54.5	40.2	549
		%RPD Sample/Sample Dup	0.92	1.0	1.5
ISO18-14-01-002	BD24-4; Sample	10.5	3.74	6.32	0.422
ISO18-14-01-002-DUP	BD24-4; Sample Dup	10.7	3.84	6.68	0.508
		Average	10.6⁽²⁾	3.79⁽²⁾	6.50⁽²⁾
		%RPD Sample/Sample Dup	1.9	2.6	5.5
ISO18-14-01-003	D09; Sample	286	5460	2020	23.6
ISO18-14-01-003-DUP	D09; Sample Dup	275	5320	1910	20.6
		Average	281	5390	1970
		%RPD Sample/Sample Dup	3.9	2.6	5.6
ISO18-14-01-004	D10; Sample	335	139	24.3	0.144
ISO18-14-01-004-DUP	D10; Sample Dup	350	143	25.2	0.154
		Average	343	141	24.8
		%RPD Sample/Sample Dup	4.4	2.8	3.6
ISO18-14-01-005	D11; Sample	418	155	1500	13.9
ISO18-14-01-005-DUP	D11; Sample Dup	418	154	1490	13.7
		Average	418	155	1500
		%RPD Sample/Sample Dup	0.0	0.65	0.67
ISO18-14-01-006	D14; Sample	2.16	1.16	0.404	0.0868
ISO18-14-01-006-DUP	D14; Sample Dup	2.04	1.14	0.382	0.0704
		Average	2.10⁽²⁾	1.15⁽²⁾	0.393⁽²⁾
		%RPD Sample/Sample Dup	5.7	1.7	21⁽³⁾
ISO18-14-01-008	D17; Sample	782	303	204	0.126
ISO18-14-01-008-DUP	D17; Sample Dup	785	304	199	0.125
		Average	784	304	202
		%RPD Sample/Sample Dup	0.38	0.33	2.5
ISO18-14-01-009	D18; Sample	300	181	57.2	3.54
ISO18-14-01-009-DUP	D18; Sample Dup	298	179	55.8	3.40
		Average	299	180	56.5
		%RPD Sample/Sample Dup	0.67	1.1	2.5
ISO18-14-01-010	D5; Sample	505	264	465	7.46
ISO18-14-01-010-DUP	D5; Sample Dup	485	251	434	7.02
		Average	495	258	450
		%RPD Sample/Sample Dup	4.0	5.0	6.9
					7.24
					6.1

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 13%, PFBS \pm 30%, PFHS \pm 14%, and PFOS \pm 13%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 18%, PFBS \pm 13%, PFHS \pm 10%, PFOS \pm 22%, and PFOSA \pm 13%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of $\leq 20\%$.

Table 1 continued. Sample Results Summary⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: 2nd Aquifer					
ISO18-14-01-011	ND7; Sample	345	62.8	19.5	32.6
ISO18-14-01-011-DUP	ND7; Sample Dup	334	60.9	19.3	32.0
		Average	340	61.9	19.4
		%RPD Sample/Sample Dup	3.2	3.1	1.0
ISO18-14-01-012	P118A; Sample	1100	745	4850	19.1
ISO18-14-01-012-DUP	P118A; Sample Dup	1090	742	4910	19.1
		Average	1100	744	4880
		%RPD Sample/Sample Dup	0.91	0.40	1.2
ISO18-14-01-013	P118B; Sample	2010	2370	12400	3.02
ISO18-14-01-013-DUP	P118B; Sample Dup	1990	2390	13200	3.12
		Average	2000	2380	12800
		%RPD Sample/Sample Dup	1.0	0.84	6.3
ISO18-14-01-014	P119A; Sample	125	37.7	52.9	3.70
ISO18-14-01-014-DUP	P119A; Sample Dup	128	37.6	53.6	3.80
		Average	127	37.7	53.3
		%RPD Sample/Sample Dup	2.4	0.27	1.3
ISO18-14-01-015	P119B; Sample	1580	949	146	0.558
ISO18-14-01-015-DUP	P119B; Sample Dup	1580	947	147	0.560
		Average	1580	948	147
		%RPD Sample/Sample Dup	0.0	0.21	0.68
ISO18-14-01-016	P121; Sample	0.282	0.0986	1.10	0.0672
ISO18-14-01-016-DUP	P121; Sample Dup	0.258	0.0956	1.02	0.0656
		Average	0.270 ⁽³⁾	0.0971 ⁽³⁾	1.06 ⁽³⁾
		%RPD Sample/Sample Dup	8.9	3.1	7.5
ISO18-14-01-017	P321; Sample	2720	711	7920	0.580
ISO18-14-01-017-DUP	P321; Sample Dup	2730	714	8270	0.636
		Average	2730	713	8100
		%RPD Sample/Sample Dup	0.37	0.42	4.3
Zone: Blokkersdijk Nature Reserve					
ISO18-14-01-018	3M vijver; Sample	1.38	0.738	3.40	<0.0248
ISO18-14-01-018-DUP	3M vijver; Sample Dup	1.19	0.721	3.13	<0.0248
		Average	1.29	0.730	3.27
		%RPD Sample/Sample Dup	15	2.3	8.3
ISO18-14-01-019	Blokkersdijkvijver - Noord; Sample	1.11	0.643	2.00	<0.0248
ISO18-14-01-019-DUP	Blokkersdijkvijver - Noord; Sample Dup	1.13	0.623	1.93	<0.0248
		Average	1.12	0.633	1.97
		%RPD Sample/Sample Dup	1.8	3.2	3.6

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 13%, PFBS ± 30%, PFHS ± 14%, and PFOS ± 13%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 18%, PFBS ± 13%, PFHS ± 10%, PFOS ± 22%, and PFOSA ± 13%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Blokkersdijk Nature Reserve					
ISO18-14-01-020	Blokkersdijkvijver - standard; Sample	1.01	0.599	0.710	<0.0248
ISO18-14-01-020-DUP	Blokkersdijkvijver - standard; Sample Dup	1.07	0.634	0.643	<0.0248
Average		1.04	0.617	0.677	<0.0248
%RPD Sample/Sample Dup		5.8	5.7	9.9	NA
ISO18-14-01-021	L21; Sample	0.222	0.103	3.36	0.0470
ISO18-14-01-021-DUP	L21; Sample Dup	0.212	0.105	3.50	0.0512
Average		0.217⁽²⁾	0.104⁽²⁾	3.43⁽²⁾	0.0491
%RPD Sample/Sample Dup		4.6	1.9	4.1	8.55
ISO18-14-01-022	L22; Sample	0.298	0.170	6.54	0.0658
ISO18-14-01-022-DUP	L22; Sample Dup	0.312	0.167	6.62	0.0644
Average		0.305⁽²⁾	0.169⁽²⁾	6.58⁽²⁾	0.0651
%RPD Sample/Sample Dup		4.6	1.8	1.2	2.2
ISO18-14-01-023	L31; Sample	0.525	0.829	3.18	<0.0248
ISO18-14-01-023-DUP	L31; Sample Dup	0.489	0.870	3.17	<0.0248
Average		0.507	0.850	3.18	<0.0248
%RPD Sample/Sample Dup		7.1	4.8	0.31	NA
ISO18-14-01-024	L4; Sample	2.45	1.90	24.9	5.70
ISO18-14-01-024-DUP	L4; Sample Dup	2.37	1.87	25.3	6.00
Average		2.41	1.89	25.1	5.85
%RPD Sample/Sample Dup		3.3	1.6	1.6	5.1
ISO18-14-01-025	P114bis; Sample	2.13	1.24	13.3	<0.0248
ISO18-14-01-025-DUP	P114bis; Sample Dup	2.14	1.31	13.3	0.0322
Average		2.14	1.28	13.3	0.0322
%RPD Sample/Sample Dup		0.47	5.5	0.0	NA
ISO18-14-01-026	P115; Sample	4.56	2.28	0.800	0.0682
ISO18-14-01-026-DUP	P115; Sample Dup	4.32	2.19	0.801	0.0664
Average		4.44	2.24	0.801	0.0673
%RPD Sample/Sample Dup		5.4	4.0	0.12	2.7
ISO18-14-01-027	P116; Sample	0.529	0.513	8.47	0.0496
ISO18-14-01-027-DUP	P116; Sample Dup	0.556	0.528	9.01	0.0510
Average		0.543	0.521	8.74	0.0503
%RPD Sample/Sample Dup		5.0	2.9	6.2	2.8
Zone: Effluent WWTP					
ISO18-14-01-028	Effluent WWTP; Sample	32.1	39.0	88.2	3.34
ISO18-14-01-028-DUP	Effluent WWTP; Sample Dup	30.3	36.8	79.5	3.14
Average		31.2	37.9	83.9	3.24
%RPD Sample/Sample Dup		5.8	5.8	10	6.2

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 13%, PFBS \pm 30%, PFHS \pm 14%, and PFOS \pm 13%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 18%, PFBS \pm 13%, PFHS \pm 10%, PFOS \pm 22%, and PFOSA \pm 13%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of $\leq 20\%$.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Extraction Wells P&T						
ISO18-14-01-029	PP01; Sample	60.9	79.4	7990	2330	36.4
ISO18-14-01-029-DUP	PP01; Sample Dup	60.2	78.3	8080	2290	36.2
Average		60.6	78.9	8040	2310	36.3
%RPD Sample/Sample Dup		1.2	1.4	1.1	1.7	0.55
ISO18-14-01-030	PP02; Sample	547	47.2	3920	23900	26.2
ISO18-14-01-030-DUP	PP02; Sample Dup	587	44.2	3930	25100	25.6
Average		567	45.7	3930	24500	25.9
%RPD Sample/Sample Dup		7.1	6.6	0.25	4.9	2.3
ISO18-14-01-031	PP04; Sample	1390	2770	553	6540	30.6
ISO18-14-01-031-DUP	PP04; Sample Dup	1380	2760	551	6730	30.0
Average		1390	2770	552	6640	30.3
%RPD Sample/Sample Dup		0.72	0.36	0.36	2.9	2.0
ISO18-14-01-032	PP05; Sample	1900	14800	4790	678	99.0
ISO18-14-01-032-DUP	PP05; Sample Dup	1930	15000	4870	628	98.4
Average		1920	14900	4830	653	98.7
%RPD Sample/Sample Dup		1.6	1.3	1.7	7.7	0.61
ISO18-14-01-033	PP06; Sample	189	15.9	86.7	1930	58.8
ISO18-14-01-033-DUP	PP06; Sample Dup	191	17.1	89.3	1900	57.6
Average		190	16.5	88.0	1920	58.2
%RPD Sample/Sample Dup		1.1	7.3	3.0	1.6	2.1
ISO18-14-01-034	PP07; Sample	299	143	99.9	2430	67.8
ISO18-14-01-034-DUP	PP07; Sample Dup	301	143	99.0	2440	69.6
Average		300	143	99.5	2440	68.7
%RPD Sample/Sample Dup		0.67	0.0	0.90	0.41	2.6
ISO18-14-01-035	PP08; Sample	569	24.1	304	5200	33.4
ISO18-14-01-035-DUP	PP08; Sample Dup	559	22.7	298	5160	34.6
Average		564	23.4	301	5180	34.0
%RPD Sample/Sample Dup		1.8	6.0	2.0	0.77	3.5
ISO18-14-01-036	PP09; Sample	709	27.9	425	2400	13.3
ISO18-14-01-036-DUP	PP09; Sample Dup	727	28.4	428	2450	12.2
Average		718	28.2	427	2430	12.8
%RPD Sample/Sample Dup		2.5	1.8	0.70	2.1	8.6
ISO18-14-01-037	PP10; Sample	546	37.6	301	4780	35.8
ISO18-14-01-037-DUP	PP10; Sample Dup	539	37.9	303	4790	32.2
Average		543	37.8	302	4790	34.0
%RPD Sample/Sample Dup		1.3	0.79	0.66	0.21	11

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 13%, PFBS \pm 30%, PFHS \pm 14%, and PFOS \pm 13%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 18%, PFBS \pm 13%, PFHS \pm 10%, PFOS \pm 22%, and PFOSA \pm 13%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Palingbeek & Tophatgracht					
ISO18-14-01-038	12; Sample	32.4	9.87	144	0.362
ISO18-14-01-038-DUP	12; Sample Dup	31.8	9.83	141	0.370
Average %RPD Sample/Sample Dup		32.1 1.9	9.85 0.41	143 2.1	0.366 2.2
ISO18-14-01-039	13; Sample	34.1	11.7	137	0.328
ISO18-14-01-039-DUP	13; Sample Dup	34.4	11.4	139	0.330
Average %RPD Sample/Sample Dup		34.3 0.88	11.6 2.6	138 1.4	0.329 0.61
ISO18-14-01-040	5; Sample	16.3	13.3	33.8	0.103
ISO18-14-01-040-DUP	5; Sample Dup	15.9	13.0	32.8	0.104
Average %RPD Sample/Sample Dup		16.1 2.5	13.2 2.3	33.3 3.0	0.104 0.97
ISO18-14-01-041	Bemalingsstation; Sample	16.3	13.1	34.5	0.0986
ISO18-14-01-041-DUP	Bemalingsstation; Sample Dup	19.1	15.9	36.2	0.190
Average %RPD Sample/Sample Dup		17.7 16	14.5 19	35.4 4.8	0.144 63 ⁽³⁾
Zone: Sewer					
ISO18-14-01-042	Collector put; Sample	75.0	25.1	231	13.7
ISO18-14-01-042-DUP	Collector put; Sample Dup	75.6	25.4	237	13.9
Average %RPD Sample/Sample Dup		75.3 0.80	25.3 1.2	234 2.6	13.8 1.4

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area – Building 16						
ISO18-14-01-043	P27; Sample	165	748	18.8	712	136
ISO18-14-01-043-DUP	P27; Sample Dup	167	747	18.3	704	126
Average %RPD Sample/Sample Dup		166 1.2	748 0.13	18.6 2.7	708 1.1	131 7.6
ISO18-14-01-044	P21B; Sample	5570	4610	6630	37900	3.00
ISO18-14-01-044-DUP	P21B; Sample Dup	4820	3930	5570	32000	2.72
Average %RPD Sample/Sample Dup		5200 14	4270 16	6100 17	35000 17	2.86 9.8
ISO18-14-01-045	P304; Sample	528	349	170	1330	19.1
ISO18-14-01-045-DUP	P304; Sample Dup	511	345	168	1280	17.3
Average %RPD Sample/Sample Dup		520 3.3	347 1.2	169 1.2	1310 3.8	18.2 9.9

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 13%, PFBS \pm 30%, PFHS \pm 14%, and PFOS \pm 13%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 18%, PFBS \pm 13%, PFHS \pm 10%, PFOS \pm 22%, and PFOSA \pm 13%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - Building 16						
ISO18-14-01-046	P305; Sample	142	106	191	432	19.0
ISO18-14-01-046-DUP	P305; Sample Dup	146	107	196	425	17.2
Average %RPD Sample/Sample Dup		144 2.8	107 0.94	194 2.6	429 1.6	18.1 9.9
ISO18-14-01-047	P42; Sample	398	36.1	312	6250	14.7
ISO18-14-01-047-DUP	P42; Sample Dup	401	36.6	317	6310	16.0
Average %RPD Sample/Sample Dup		400 0.75	36.4 1.4	315 1.6	6280 0.96	15.4 8.5
3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾	
Zone: Source Area - WWTP						
ISO18-14-01-048	L19; Sample	284	134	6710	77.2	
ISO18-14-01-048-DUP	L19; Sample Dup	277	129	6570	83.0	
Average %RPD Sample/Sample Dup		281 2.5	132 3.8	6640 2.1	80.1 7.2	
ISO18-14-01-049	M4; Sample	1770	273	8580	54.6	
ISO18-14-01-049-DUP	M4; Sample Dup	1800	281	9290	56.8	
Average %RPD Sample/Sample Dup		1790 1.7	277 2.9	8940 7.9	55.7 3.9	
ISO18-14-01-050	P118C; Sample	574	245	2740	49.6	
ISO18-14-01-050-DUP	P118C; Sample Dup	583	252	2790	48.0	
Average %RPD Sample/Sample Dup		579 1.6	249 2.8	2770 1.8	48.8 3.3	
ISO18-14-01-051	P119C; Sample	1040	651	6950	75.6	
ISO18-14-01-051-DUP	P119C; Sample Dup	1050	653	6710	64.4	
Average %RPD Sample/Sample Dup		1050 0.96	652 0.31	6830 3.5	70.0 16	
ISO18-14-01-052	P262; Sample	26.9	5.75	220	110	
ISO18-14-01-052-DUP	P262; Sample Dup	28.2	6.15	213	107	
Average %RPD Sample/Sample Dup		27.6 4.7	5.95 6.7	217 3.2	109 2.8	
ISO18-14-01-053	P263; Sample	1380	482	8680	9.78	
ISO18-14-01-053-DUP	P263; Sample Dup	1380	481	8580	10.5	
Average %RPD Sample/Sample Dup		1380 0.0	482 0.21	8630 1.2	10.1 7.1	

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 13%, PFBS \pm 30%, PFHS \pm 14%, and PFOS \pm 13%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 18%, PFBS \pm 13%, PFHS \pm 10%, PFOS \pm 22%, and PFOSA \pm 13%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of $\leq 20\%$.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - WWTP					
ISO18-14-01-054	P264; Sample	293	195	1520	49.6
ISO18-14-01-054-DUP	P264; Sample Dup	303	192	1470	51.6
Average		298	194	1500	50.6
%RPD Sample/Sample Dup		3.4	1.6	3.3	4.0
ISO18-14-01-055	P265B; Sample	74.1	27.6	707	59.0
ISO18-14-01-055-DUP	P265B; Sample Dup	72.1	25.9	698	61.2
Average		73.1	26.8	703	60.1
%RPD Sample/Sample Dup		2.7	6.4	1.3	3.7
ISO18-14-01-056	P340; Sample	381	202	2560	36.2
ISO18-14-01-056-DUP	P340; Sample Dup	390	202	2710	39.2
Average		386	202	2640	37.7
%RPD Sample/Sample Dup		2.3	0.0	5.7	8.0
ISO18-14-01-057	P343; Sample	839	726	7420	10.2
ISO18-14-01-057-DUP	P343; Sample Dup	831	743	7590	10.7
Average		835	735	7510	10.5
%RPD Sample/Sample Dup		0.96	2.3	2.3	4.8
ISO18-14-01-058	P371; Sample	207	99.4	3080	32.2
ISO18-14-01-058-DUP	P371; Sample Dup	208	96.3	3120	32.6
Average		208	97.9	3100	32.4
%RPD Sample/Sample Dup		0.48	3.2	1.3	1.2
ISO18-14-01-059	P374; Sample	693	863	1970	28.6
ISO18-14-01-059-DUP	P374; Sample Dup	708	867	1970	28.0
Average		701	865	1970	28.3
%RPD Sample/Sample Dup		2.1	0.46	0.0	2.1
Zone: Southern Site Boundary					
ISO18-14-01-060	B3-bis; Sample	10.9	3.30	108	0.240
ISO18-14-01-060-DUP	B3-bis; Sample Dup	11.3	3.24	111	0.282
Average		11.1	3.27	110	0.261
%RPD Sample/Sample Dup		3.6	1.8	2.7	16
ISO18-14-01-061	B7; Sample	1.63	1.15	56.4	0.248
ISO18-14-01-061-DUP	B7; Sample Dup	1.54	1.19	56.3	0.258
Average		1.59	1.17	56.4	0.253
%RPD Sample/Sample Dup		5.7	3.4	0.18	4.0
ISO18-14-01-062	P372; Sample	72.1	54.7	1360	37.8
ISO18-14-01-062-DUP	P372; Sample Dup	69.0	54.9	1330	37.6
Average		70.6	54.8	1350	37.7
%RPD Sample/Sample Dup		4.4	0.36	2.2	0.53

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 13%, PFBS \pm 30%, PFHS \pm 14%, and PFOS \pm 13%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 18%, PFBS \pm 13%, PFHS \pm 10%, PFOS \pm 22%, and PFOSA \pm 13%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFBS	PFHS	PFOSA ⁽²⁾
Zone: Southern Site Boundary					
ISO18-14-01-063	P378; Sample	242	275	1030	1.72
ISO18-14-01-063-DUP	P378; Sample Dup	240	275	1050	1.68
		Average	241	275	1040
		%RPD Sample/Sample Dup	0.83	0.0	1.70
ISO18-14-01-064	PA109A; Sample	114	114	223	1.53
ISO18-14-01-064-DUP	PA109A; Sample Dup	114	114	222	1.46
		Average	114	114	223
		%RPD Sample/Sample Dup	0.0	0.0	1.50
ISO18-14-01-065	PA111A; Sample	752	996	2040	9.34
ISO18-14-01-065-DUP	PA111A; Sample Dup	753	1010	2050	8.88
		Average	753	1000	2050
		%RPD Sample/Sample Dup	0.13	1.4	9.11
ISO18-14-01-066	PA112; Sample	509	122	285	46.6
ISO18-14-01-066-DUP	PA112; Sample Dup	502	119	273	47.0
		Average	506	121	279
		%RPD Sample/Sample Dup	1.4	2.5	46.8
ISO18-14-01-067	Travel Blank	<0.0240	<0.0250	<0.0232	<0.0248

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
ISO18-14-01-067	Travel Blank	<0.0240 ⁽²⁾	<0.0250 ⁽²⁾	<0.0250 ⁽²⁾	<0.0232 ⁽²⁾	<0.0248 ⁽²⁾

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 13%, PFBS ± 30%, PFHS ± 14%, and PFOS ± 13%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 18%, PFBS ± 13%, PFHS ± 10%, PFOS ± 22%, and PFOSA ± 13%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis".

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected on January 22, 29-31 and February 1, 2018, in Nalgene™ (high-density polyethylene) bottles prepared at the 3M EHS Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. All sample locations were spiked with a mass-labeled internal standard mix at a nominal concentration of 1 ng/mL and select samples were fortified with surrogate recovery standards (SRSs) at a nominal concentration of 0.1 ng/mL prior to sample collection. Samples were received on February 7, 2018.

2.3 Sample Preparation

Select samples were prepared for PFOA, PFHS, PFOS and PFBS (select locations only) using a direct injection by methanol dilution method. Samples that required a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples that required a 1:100 dilution were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol. Samples that required a 1:500 dilution were prepared by diluting 0.02 mL of a well-mixed sample with 10.0 mL of methanol. Diluted samples and LCSs were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

Sample locations BD24-4, D14, P121, L21, L22 and Travel Blank sample and FMS Low were analyzed for PFOA, PFHS, and PFOS by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples along with the method blanks.

All samples were prepared for PFOSA by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The laboratory control samples and calibration standards were then diluted with methanol in the same manner.

2.4 Analysis

All samples and quality control samples were analyzed for the target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). SRSs were added and included in the analysis of these samples. Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.2. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

The following analytical runs were used to report the results herein:

2/15/18 (ETS Kirk) External Standard Calibration Analysis:

- All sample locations were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported **except** for the following: BD24-3, D14, P121, L21, L22, and Travel Blank sample and FMS Low (PFOA, PFHS, PFOS) and P118B (PFOS).

2/20/18 (ETS Kirk) External Standard Calibration Analysis:

- All sample locations were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported.

2/22/18 (ETS Rita) Internal Standard Calibration Analysis:

- All sample locations were analyzed for PFOSA. All sample results were reported **except** for the following: 3M vijver sample.

2/27/18 (ETS Rita) Internal Standard Calibration Analysis:

- All sample locations were analyzed for PFOSA. All sample results were reported.

3/14/17 (ETS Kirk) Internal Standard Calibration Analysis:

- All sample locations were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported.

3/19/18 (ETS Kirk) External Standard Calibration Analysis:

- Sample location P118B was analyzed for PFOS. All sample results were reported.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk	ETS Rita
Liquid Chromatograph	Agilent 1260	Agilent 1100
Analysis Method	ETS-8-044.3	ETS-8-044.3
Analysis Date	2/15/18, 2/20/18, 3/14/18, 3/19/18	2/22/18, 2/27/18
Guard column	Prism RP (2.1 mm X 50 mm), 5 μ	Prism RP (2.1 mm X 50 mm), 5 μ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ
Injection Volume	2 or 5 μ L	5 μ L
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 6500+
Ion Source	Turbo Spray	Turbo Spray
Polarity	Negative	Negative
Software	Analyst 1.6.3	Analyst 1.6.3

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis				
Step Number	Total Time (min)	Flow Rate (μ L/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0.00	300	90.0	10.0
1	0.50	300	90.0	10.0
2	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard	Mass Transition Q1/Q3
PFOA	413/369	[¹³ C ₈]-PFOA	421/376
	413/219		
	413/169		
PFBS	299/99	NA	NA
	299/80		
PFHS	399/99	[¹³ C ₃]-PFHS	402/80
	399/80		
PFOS	499/99	[¹³ C ₈]-PFOS	507/80
	499/80		
	499/130		
PFOSA	498/78	[¹³ C ₈]-PFOSA	506/78
[¹³ C ₄]-PFOA	417/372	[¹³ C ₈]-PFOA	421/376
[¹³ C ₄]-PFOS	503/80	[¹³ C ₈]-PFOS	507/80

NA = Not Applicable; An internal standard was not used for the analysis of PFBS.

The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

3 Data Analysis

3.1 Calibration

2/15/18, 2/20/18 and 3/19/18 Analysis (External Standard Calibration): Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL to 25 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

3/14/18 Analysis (Internal Standard Calibration): Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of twelve calibration standards ranging from 0.0125 ng/mL to 25 ng/mL (nominal) were analyzed. The calibration standards also contained surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

2/22/18 and 2/27/18 Analysis of PFOSA (Internal Standard Calibration): Samples were analyzed for PFOSA against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into laboratory reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 1.0 ng/mL. Calibration standards ranging from 0.025 ng/mL to 200 ng/mL (nominal) were analyzed. Prior to analysis, the calibration standards were diluted 2x by removing a 0.4 mL aliquot and diluting it with 0.4 mL of methanol. A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of $100\pm 25\%$ ($100\pm 30\%$ for the lowest curve point) were met for all analytes. The correlation coefficient (r) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.3 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the Table 6 below.

Table 6. LOQ

Analyte	LOQ, ng/mL ⁽¹⁾ 2/15/18 Analysis	LOQ, ng/mL ⁽¹⁾ 2/20/18 Analysis	LOQ, ng/mL ⁽²⁾ 2/22/18 Analysis	LOQ, ng/mL ⁽²⁾ 2/27/18 Analysis	LOQ, ng/mL ⁽²⁾ 3/14/18 Analysis	LOQ, ng/mL ⁽¹⁾ 3/19/18 Analysis
PFOA	0.0479	0.0479	NA	NA	0.0240	NA
PFBS	0.0500	0.0500	NA	NA	0.0250	NA
PFHS	0.0500	0.0500	NA	NA	0.0250	NA
PFOS	0.0464	0.0464	NA	NA	0.0232	0.0185
PFOSA	NA	NA	0.0248	0.0248	NA	NA

NA = Not Applicable

(1) A dilution factor was not applied to the LOQ.

(2) A dilution factor of 2 was applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of $100\pm 25\%$.

3.5 Blanks

Two types of blanks were prepared and analyzed with the samples: method/solvent blanks and field/trip blanks. Each blank result was reviewed and used to evaluate method performance. The method blanks were used to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. LCSs were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. Surrogates [¹³C₄]-PFOA and [¹³C₄]-PFOS were added post dilution when analyzed by external standard. The results in **Table 7** for these LCSs are reported with the dilution factor applied.

Method ETS-8-044.3 states that the average recovery of LCSs at each spiking level must be within 80%-120% with a RSD ≤20%. All LCS samples met criteria with the following exception:

- 2/20/18: All low level LCSs for PFBS were outside the method acceptance criteria of 100±20%, yielding an average recovery of 130%. A deviation is provided with the raw data.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report. The following calculations were used to generate data in **Table 7**.

$$\text{LCS Percent Recovery} = \frac{\text{Calculated Concentration}}{\text{Spike Concentration}} * 100\%$$

$$\text{LCS% RSD} = \frac{\text{standard deviation LCS replicates}}{\text{average LCS recovery}} * 100\%$$

Table 7. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 2/15/18	PFOA (Linear + Branched)			PFBS			
	Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-180215-1	99.8	109	109	109	100	117	117
LCS-180215-2	99.8	108	108	108	100	113	113
LCS-180215-3	99.8	111	111	111	100	116	116
Average ± %RSD	110% ± 1.4%			115% ± 1.8%			
LCS-180215-4	4990	5220	105	5000	5570	111	
LCS-180215-5	4990	5120	103	5000	5520	110	
LCS-180215-6	4990	5170	104	5000	5470	109	
Average ± %RSD	104% ± 0.97%			110% ± 0.91%			
LCS-180215-7	34900	34900	100	35000	38800	111	
LCS-180215-8	34900	34300	98.3	35000	38300	109	
LCS-180215-9	34900	35400	101	35000	39000	111	
Average ± %RSD	99.9% ± 1.6%			111% ± 0.93%			

NA = Not Applicable

(1) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 2/15/18	PFHS			PFOS (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-180215-1	100	120	120	99.8	99.4	99.6
LCS-180215-2	100	109	109	99.8	95.4	95.6
LCS-180215-3	100	117	117	99.8	97.9	98.1
Average ± %RSD	115% ± 4.9%			97.8% ± 2.1%		
LCS-180215-4	5010	5670	113	4990	4980	99.8
LCS-180215-5	5010	5520	110	4990	4930	98.8
LCS-180215-6	5010	5420	108	4990	4850	97.2
Average ± %RSD	111% ± 2.3%			98.6% ± 1.3%		
LCS-180215-7	35100	39900	114	34900	36800	105
LCS-180215-8	35100	39800	113	34900	36100	103
LCS-180215-9	35100	39500	113	34900	36900	106
Average ± %RSD	113% ± 0.52%			105% ± 1.2%		

ETS-8-044.3 External Calibration Analyzed 2/15/18	[¹³ C ₄]-PFOA			[¹³ C ₄]-PFOS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-180215-1	0.199	0.217	109	0.190	0.210	110
LCS-180215-2	0.199	0.205	103	0.190	0.198	104
LCS-180215-3	0.199	0.218	110	0.190	0.205	108
Average ± %RSD	107% ± 3.5%			107% ± 2.8%		
LCS-180215-4	1.99	2.29	115	1.90	2.16	114
LCS-180215-5	1.99	2.23	112	1.90	2.11	111
LCS-180215-6	1.99	2.24	113	1.90	2.08	109
Average ± %RSD	113% ± 1.3%			111% ± 2.3%		

NA = Not Applicable

(1) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 2/20/18	PFOA (Linear + Branched)			PFBS		
	Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)
LCS-180220-1	99.8	113	113	100	129	129
LCS-180220-2	99.8	106	106	100	133	133
LCS-180220-3	99.8	104	104	100	127	127
Average ± %RSD		108% ± 4.4%			130% ⁽¹⁾ ± 2.4%	
LCS-180220-4	4990	5320	107	5000	5720	114
LCS-180220-5	4990	5020	101	5000	5470	109
LCS-180220-6	4990	5170	104	5000	5420	108
Average ± %RSD		104% ± 2.9%			111% ± 2.9%	
LCS-180220-7	34900	34400	98.6	35000	38800	111
LCS-180220-8	34900	34400	98.6	35000	38200	109
LCS-180220-9	34900	33900	97.1	35000	38600	110
Average ± %RSD		98.1% ± 0.84%			110% ± 0.79%	

ETS-8-044.3 External Calibration Analyzed 2/20/18	PFHS			PFOS (Linear + Branched)		
	Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)
LCS-180220-1	100	123	123	99.8	102	102
LCS-180220-2	100	107	107	99.8	89.4	89.6
LCS-180220-3	100	117	117	99.8	98.9	99.1
Average ± %RSD		116% ± 7.0%			97.0% ± 6.8%	
LCS-180220-4	5010	5720	114	4990	5020	101
LCS-180220-5	5010	5520	110	4990	4820	96.6
LCS-180220-6	5010	5420	108	4990	4770	95.6
Average ± %RSD		111% ± 2.8%			97.6% ± 2.7%	
LCS-180220-7	35100	39100	111	34900	36500	105
LCS-180220-8	35100	39200	112	34900	36600	105
LCS-180220-9	35100	39300	112	34900	36200	104
Average ± %RSD		112% ± 0.26%			104% ± 0.57%	

NA = Not Applicable

(1) LCSs did not meet acceptance criteria of $100 \pm 20\%$.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 2/20/18	^{[13C₄]-PFOA}			^{[13C₄]-PFOS}		
	Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)
LCS-180220-1	0.199	0.215	108	0.190	0.215	113
LCS-180220-2	0.199	0.223	112	0.190	0.208	109
LCS-180220-3	0.199	0.213	107	0.190	0.208	109
Average ± %RSD	109% ± 2.4%			110% ± 2.1%		
LCS-180220-4	1.99	2.41	121	1.90	2.34	123
LCS-180220-5	1.99	2.31	116	1.90	2.21	116
LCS-180220-6	1.99	2.33	117	1.90	2.23	118
Average ± %RSD	118% ± 2.2%			119% ± 3.0%		

ETS-8-044.3 Internal Calibration Analyzed 2/22/18	^{PFOA}		
	Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)
LCS-180222-1	0.199	0.187	94.0
LCS-180222-2	0.199	0.178	89.4
LCS-180222-3	0.199	0.181	91.0
Average ± %RSD	91.5% ± 2.5%		
LCS-180222-4	19.9	18.9	95.0
LCS-180222-5	19.9	19.3	97.0
LCS-180222-6	19.9	18.4	92.5
Average ± %RSD	94.8% ± 2.4%		
LCS-180222-7	139	140	101
LCS-180222-8	139	135	97.1
LCS-180222-9	139	134	96.4
Average ± %RSD	98.1% ± 2.4%		

NA = Not Applicable

(1) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 2/27/18	PFOSA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-180227-1	0.199	0.179	89.9
LCS-180227-2	0.199	0.170	85.4
LCS-180227-3	0.199	0.169	84.9
Average ± %RSD	86.8% ± 3.2%		
LCS-180227-4	19.9	19.0	95.5
LCS-180227-5	19.9	18.1	91.0
LCS-180227-6	19.9	18.1	91.0
Average ± %RSD	92.5% ± 2.8%		
LCS-180227-7	139	135	97.1
LCS-180227-8	139	131	94.2
LCS-180227-9	139	130	93.5
Average ± %RSD	95.0% ± 2.0%		

ETS-8-044.3 Internal Calibration Analyzed 3/14/18	PFOA (Linear + Branched)			PFBS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-180314-1	0.190	0.187	98.4	0.198	0.194	98.0
LCS-180314-2	0.190	0.198	104	0.198	0.191	96.5
LCS-180314-3	0.190	0.194	102	0.198	0.191	96.5
Average ± %RSD	102% ± 2.9%			97.0% ± 0.90%		
LCS-180314-4	1.90	1.96	103	1.98	1.95	98.5
LCS-180314-5	1.90	1.90	100	1.98	1.87	94.4
LCS-180314-6	1.90	1.81	95.3	1.98	1.86	93.9
Average ± %RSD	99.5% ± 4.0%			95.6% ± 2.6%		
LCS-180314-7	33.2	31.8	95.8	34.7	32.8	94.5
LCS-180314-8	33.2	32.4	97.6	34.7	33.6	96.8
LCS-180314-9	33.2	32.8	98.8	34.7	33.8	97.4
Average ± %RSD	97.4% ± 1.6%			96.3% ± 1.6%		

NA = Not Applicable

(1) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 3/14/18	PFHS			PFOS (Linear + Branched)		
	Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)
LCS-180314-1	0.198	0.197	99.5	0.184	0.179	97.3
LCS-180314-2	0.198	0.188	94.9	0.184	0.174	94.6
LCS-180314-3	0.198	0.191	96.5	0.184	0.179	97.3
Average ± %RSD	97.0% ± 2.4%			96.4% ± 1.6%		
LCS-180314-4	1.98	1.95	98.5	1.84	1.87	102
LCS-180314-5	1.98	1.89	95.5	1.84	1.77	96.2
LCS-180314-6	1.98	1.85	93.4	1.84	1.76	95.7
Average ± %RSD	95.8% ± 2.7%			97.8% ± 3.4%		
LCS-180314-7	34.7	32.8	94.5	32.2	31.0	96.3
LCS-180314-8	34.7	32.6	93.9	32.2	31.2	96.9
LCS-180314-9	34.7	33.8	97.4	32.2	31.6	98.1
Average ± %RSD	95.3% ± 1.9%			97.1% ± 0.98%		

ETS-8-044.3 Internal Calibration Analyzed 3/14/18	[¹³ C ₄]-PFOA			[¹³ C ₄]-PFOS		
	Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)
LCS-180314-1	0.198	0.204	103	0.189	0.192	102
LCS-180314-2	0.198	0.212	107	0.189	0.191	101
LCS-180314-3	0.198	0.190	96.0	0.189	0.179	94.7
Average ± %RSD	102% ± 5.5%			99.1% ± 3.9%		
LCS-180314-4	1.98	2.08	105	1.89	1.88	99.5
LCS-180314-5	1.98	2.02	102	1.89	1.85	97.9
LCS-180314-6	1.98	2.00	101	1.89	1.81	95.8
Average ± %RSD	103% ± 2.0%			97.7% ± 1.9%		

NA = Not Applicable

(1) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 3/19/18	PFOS (Linear + Branched)			[¹³ C ₄]PFOS		
	Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)
LCS-180319-1	99.8	94.9	95.1	0.190	0.198	104
LCS-180319-2	99.8	88.4	88.6	0.190	0.181	95.2
LCS-180319-3	99.8	79.3	79.5	0.190	0.189	99.2
Average ± %RSD		87.7% ± 9.0%			99.5% ± 4.4%	
LCS-180319-4	4990	4890	98.0	1.90	2.01	106
LCS-180319-5	4990	4920	98.6	1.90	1.95	102
LCS-180319-6	4990	5120	103	1.90	1.99	105
Average ± %RSD		99.7% ± 2.5%			104% ± 2.0%	
LCS-180319-7	34900	38600	111			
LCS-180319-8	34900	36500	105			
LCS-180319-9	34900	35800	103			
Average ± %RSD		106% ± 3.9%				

NA = Not Applicable

(1) LCSs did not meet acceptance criteria of 100 ± 20%.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.4. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%. When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in Table 8 below.

- The data uncertainty for PFBS using external calibration was calculated at 24% following ETS-12-012.4; however, the data uncertainty was expanded to ±30% based on the PFBS recovery of the low-level LCSs from the analysis on 2/20/18.

Table 8. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	6.39	±13%
PFBS	External	NA	±30%
PFHS	External	6.99	±14%
PFOS	External	6.26	±13%
PFOA	Internal	8.84	±18%
PFBS	Internal	6.44	±13%
PFHS	Internal	5.15	±10%
PFOS	Internal	11.0	±22%
PFOSA	Internal	6.60	±12%

NA = Not Applicable

3.8 Field Matrix Spikes (FMS)

Target analyte field matrix spike samples were collected for select sampling points to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions at 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOA and PFOS. The standards used for the preparation of the field matrix spiking solutions greater than 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, select sample bottles contained stable isotope surrogate recovery spikes of [¹³C₄]-PFOA and [¹³C₄]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles prior to sample collection. The [¹³C₄]-PFOA was selected to represent the C8 perfluorocarboxylic acid. The [¹³C₄]-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

The following calculation was used to generate data in section 4 of the report.

$$FMS\ Recovery = \frac{Sample\ Concentration\ of\ FMS - Average\ Concentration\ (Field\ Sample\ & Field\ Sample\ Dup)}{Spike\ Concentration} \times 100\%$$

Table 9. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
D14, Blokkersdijkvijver – Noord	FMS	1.92	2.00	2.00	1.85	2.00
L21, P114bis	FMS	4.79	5.00	5.00	4.64	5.00
P118B	FMS	8.59	8.97	8.97	8.31	8.97
PP01	FMS	50.0	50.0	50.0	50.0	50.0
ND7, P118C, P265B, P372	FMS	100	100	100	100	100
Bemalingsstation	FMS	51.9	52.0	52.0	51.9	2.00
PA109A	FMS	54.8	55.0	55.0	54.6	5.00
D5, PP05, P304, P42, P343, P374	FMS	520	20.0	520	520	20.0
Trip Blank	Low	1.92	2.00	2.00	1.85	2.00
	High	520	20.0	520	520	20.0

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of $\pm 30\%$, demonstrate that the method is appropriate for the given matrix.

The method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. At times, the spike level exceeded the recommended upper limit of 10 times the analyte concentration. In these instances, the FMS recovery was reported and flagged as above 10 times the sample concentration.

For PFOA, PFBS, PFHS, and PFOS, when the field matrix spike level was not appropriate as compared to the endogenous sample concentration, the surrogate recovery standards were used to assess method accuracy. All field matrix spike and surrogate recovery standard recoveries met acceptance criteria with the following exception:

ISO18-14-01-064-FMS; The recovery for [¹³C₄]-PFOS was 135%. Since the average recovery for [¹³C₄]-PFOS for the sample set was 125%, which met method acceptance criteria, no adjustment was made to the analytical uncertainty.

Expired spiking solutions were inadvertently used during the bottle preparation and during sample preparation on 2/12/18 and 2/20/18. The spiking solutions were prepared from solutions that contained expired acetonitrile. The acetonitrile had expired on 1/12/18. Expired spiking solutions were used on 2/12/18 and 2/20/18 to prepare laboratory control samples with recoveries of the LCSs from both preparation batches meeting method acceptance criteria, except for PFBS from 2/20/18 sample prep. The FMS recovery for all analytes met method acceptance criteria. The compliant LCSs (except for PFBS) and study FMSs demonstrate that the spiking solutions, while expired at the time of use, had not degraded. The analytical data uncertainty has been expanded for samples analyzed by external standard calibration for PFBS. A standard operating procedure deviation is included with the raw data.

Table 10. Location ID: D14

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-006	D14; Sample	2.16	NA	1.16	NA
ISO18-14-01-006-DUP	D14; Sample Dup	2.04	NA	1.14	NA
ISO18-14-01-006-FMS	D14; FMS	4.02	100	2.94	89.5
Average Concentration (ng/mL) ± %RPD		2.10 ng/mL ± 5.7%		1.15 ng/mL ± 1.7%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-006	D14; Sample	0.404	NA	0.0868	NA
ISO18-14-01-006-DUP	D14; Sample Dup	0.382	NA	0.0704	NA
ISO18-14-01-006-FMS	D14; FMS	2.08	91.2	1.82	87.1 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		0.393 ng/mL ± 5.6%		0.0786 ng/mL ± 21% ⁽²⁾	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 11. Location ID: D5

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-010	D5; Sample	505	NA	264	NA
ISO18-14-01-010-DUP	D5; Sample Dup	485	NA	251	NA
ISO18-14-01-010-FMS	D5; FMS	1060	109	842	112
Average Concentration (ng/mL) ± %RPD		495 ng/mL ± 4.0%		258 ng/mL ± 5.0%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-010	D5; Sample	465	NA	7.46	NA
ISO18-14-01-010-DUP	D5; Sample Dup	434	NA	7.02	NA
ISO18-14-01-010-FMS	D5; FMS	971	NC	25.6	91.8
Average Concentration (ng/mL) ± %RPD		450 ng/mL ± 6.9%		7.24 ng/mL ± 6.1%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 12. Location ID: ND7

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-011	ND7; Sample	345	NA	62.8	NA
ISO18-14-01-011-DUP	ND7; Sample Dup	334	NA	60.9	NA
ISO18-14-01-011-FMS	ND7; FMS	444	NC	165	103
Average Concentration (ng/mL) ± %RPD		340 ng/mL ± 3.2%		61.9 ng/mL ± 3.1%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-011	ND7; Sample	19.5	NA	32.6	NA
ISO18-14-01-011-DUP	ND7; Sample Dup	19.3	NA	32.0	NA
ISO18-14-01-011-FMS	ND7; FMS	115	95.6	120	87.7
Average Concentration (ng/mL) ± %RPD		19.4 ng/mL ± 1.0%		32.3 ng/mL ± 1.9%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 13. Location ID: P118B

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-013	P118B; Sample	2010	NA	2370	NA
ISO18-14-01-013-DUP	P118B; Sample Dup	1990	NA	2390	NA
ISO18-14-01-013-FMS	P118B; FMS	2000	NC	2370	NC
Average Concentration (ng/mL) ± %RPD		2000 ng/mL ± 1.0%		2380 ng/mL ± 0.84%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-013	P118B; Sample	12400	NA	3.02	NA
ISO18-14-01-013-DUP	P118B; Sample Dup	13200	NA	3.12	NA
ISO18-14-01-013-FMS	P118B; FMS	12600	NC	10.7	85.1
Average Concentration (ng/mL) ± %RPD		12800 ng/mL ± 6.3%		3.07 ng/mL ± 3.3%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 14. Location ID: Blokkersdijkvijver Noord

3M LIMS ID	Sample Description	PFOA		PFHS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-019	Blokkersdijkvijver Noord; Sample	1.11	NA	0.643	NA
ISO18-14-01-019-DUP	Blokkersdijkvijver Noord; Sample Dup	1.13	NA	0.623	NA
ISO18-14-01-019-FMS	Blokkersdijkvijver Noord; FMS	3.06	101	2.70	103
Average Concentration (ng/mL) ± %RPD		1.12 ng/mL ± 1.8%		0.633 ng/mL ± 3.2%	

3M LIMS ID	Sample Description	PFOS		PFOSA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-019	Blokkersdijkvijver Noord; Sample	2.00	NA	<0.0248	NA
ISO18-14-01-019-DUP	Blokkersdijkvijver Noord; Sample Dup	1.93	NA	<0.0248	NA
ISO18-14-01-019-FMS	Blokkersdijkvijver Noord; FMS	3.84	101	1.74	87.0 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		1.97 ng/mL ± 3.6%		<0.0248 ng/mL	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 15. Location ID: L21

3M LIMS ID	Sample Description	PFOA		PFHS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-021	L21; Sample	0.222	NA	0.103	NA
ISO18-14-01-021-DUP	L21; Sample Dup	0.212	NA	0.105	NA
ISO18-14-01-021-FMS	L21; FMS	4.76	94.8 ⁽¹⁾	4.58	89.5 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		0.217 ng/mL ± 4.6%		0.104 ng/mL ± 1.9%	

3M LIMS ID	Sample Description	PFOS		PFOSA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-021	L21; Sample	3.88	NA	0.0470	NA
ISO18-14-01-021-DUP	L21; Sample Dup	3.75	NA	0.0512	NA
ISO18-14-01-021-FMS	L21; FMS	8.47	100	4.26	84.2 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		3.82 ng/mL ± 3.4%		0.0491 ng/mL ± 8.6%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 16. Location ID: P114bis

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-025	P114bis; Sample	2.13	NA	1.24	NA
ISO18-14-01-025-DUP	P114bis; Sample Dup	2.14	NA	1.31	NA
ISO18-14-01-025-FMS	P114bis; FMS	7.15	105	6.31	101
Average Concentration (ng/mL) ± %RPD		2.14 ng/mL ± 0.47%		1.28 ng/mL ± 5.5%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-025	P114bis; Sample	13.3	NA	<0.0248	NA
ISO18-14-01-025-DUP	P114bis; Sample Dup	13.3	NA	<0.0248	NA
ISO18-14-01-025-FMS	P114bis; FMS	18.0	NC	4.24	84.8 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		13.3 ng/mL ± 0.0%		<0.0248 ng/mL	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) FMS concentration greater than 10 times the sample concentration.

Table 17. Location ID: PP01

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-029	PP01; Sample	60.9	NA	79.4	NA	7990	NA
ISO18-14-01-029-DUP	PP01; Sample Dup	60.2	NA	78.3	NA	8080	NA
ISO18-14-01-029-FMS	PP01; FMS	114	107	130	102	8150	NC
Average Concentration (ng/mL) ± %RPD		60.6 ng/mL ± 1.2%		78.9 ng/mL ± 1.4%		8040 ng/mL ± 1.1%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-029	PP01; Sample	2330	NA	36.4	NA
ISO18-14-01-029-DUP	PP01; Sample Dup	2290	NA	36.2	NA
ISO18-14-01-029-FMS	PP01; FMS	2390	NC	78.4	84.2
Average Concentration (ng/mL) ± %RPD		2310 ng/mL ± 1.7%		36.3 ng/mL ± 0.55%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 18. Location ID: PP05

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-032	PP05; Sample	1900	NA	14800	NA	4790	NA
ISO18-14-01-032-DUP	PP05; Sample Dup	1930	NA	15000	NA	4870	NA
ISO18-14-01-032-FMS	PP05; FMS	2430	NC	14700	NC	5370	NC
Average Concentration (ng/mL) ± %RPD		1920 ng/mL ± 1.6%		14900 ng/mL ± 1.3%		4830 ng/mL ± 1.7%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-032	PP05; Sample	678	NA	99.0	NA
ISO18-14-01-032-DUP	PP05; Sample Dup	628	NA	98.4	NA
ISO18-14-01-032-FMS	PP05; FMS	1240	113	115	NC
Average Concentration (ng/mL) ± %RPD		653 ng/mL ± 7.7%		98.7 ng/mL ± 0.61%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 19. Location ID: Bemalingsstation

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-041	Bemalingsstation; Sample	16.3	NA	13.1	NA
ISO18-14-01-041-DUP	Bemalingsstation; Sample Dup	19.1	NA	15.9	NA
ISO18-14-01-041-FMS	Bemalingsstation; FMS	63.0	87.3	61.4	90.2
Average Concentration (ng/mL) ± %RPD		17.7 ng/mL ± 16%		14.5 ng/mL ± 19%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-041	Bemalingsstation; Sample	34.5	NA	0.0986	NA
ISO18-14-01-041-DUP	Bemalingsstation; Sample Dup	36.2	NA	0.1900	NA
ISO18-14-01-041-FMS	Bemalingsstation; FMS	74.5	75.4	1.86	85.8
Average Concentration (ng/mL) ± %RPD		35.4 ng/mL ± 4.8%		0.144 ng/mL ± 63%⁽¹⁾	

NA = Not Applicable

(1) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 20. Location ID: P304

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-045	P304; Sample	528	NA	349	NA	170	NA
ISO18-14-01-045-DUP	P304; Sample Dup	511	NA	345	NA	168	NA
ISO18-14-01-045-FMS	P304; FMS	1070	106	369	NC	746	111
Average Concentration (ng/mL) ± %RPD		520 ng/mL ± 3.3%		347 ng/mL ± 1.2%		169 ng/mL ± 1.2%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-045	P304; Sample	1330	NA	19.1	NA
ISO18-14-01-045-DUP	P304; Sample Dup	1280	NA	17.3	NA
ISO18-14-01-045-FMS	P304; FMS	1840	NC	35.4	86.0
Average Concentration (ng/mL) ± %RPD		1310 ng/mL ± 3.8%		18.2 ng/mL ± 9.9%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 21. Location ID: P42

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-047	P42; Sample	398	NA	36.1	NA	312	NA
ISO18-14-01-047-DUP	P42; Sample Dup	401	NA	36.6	NA	317	NA
ISO18-14-01-047-FMS	P42; FMS	839	84.5	53.8	87.3	783	90.1
Average Concentration (ng/mL) ± %RPD		400 ng/mL ± 0.75%		36.4 ng/mL ± 1.4%		315 ng/mL ± 1.6%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-047	P42; Sample	6250	NA	14.7	NA
ISO18-14-01-047-DUP	P42; Sample Dup	6310	NA	16.0	NA
ISO18-14-01-047-FMS	P42; FMS	6710	NC	35.2	99.3
Average Concentration (ng/mL) ± %RPD		6280 ng/mL ± 0.96%		15.4 ng/mL ± 8.5%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 22. Location ID: P118C

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-050	P118C; Sample	574	NA	245	NA
ISO18-14-01-050-DUP	P118C; Sample Dup	583	NA	252	NA
ISO18-14-01-050-FMS	P118C; FMS	676	NC	357	NC
Average Concentration (ng/mL) ± %RPD		579 ng/mL ± 1.6%		249 ng/mL ± 2.8%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-050	P118C; Sample	2740	NA	49.6	NA
ISO18-14-01-050-DUP	P118C; Sample Dup	2790	NA	48.0	NA
ISO18-14-01-050-FMS	P118C; FMS	2810	NC	143	94.2
Average Concentration (ng/mL) ± %RPD		2770 ng/mL ± 1.8%		48.8 ng/mL ± 3.3%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 23. Location ID: P265B

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-055	P265B; Sample	74.1	NA	27.6	NA
ISO18-14-01-055-DUP	P265B; Sample Dup	72.1	NA	25.9	NA
ISO18-14-01-055-FMS	P265B; FMS	173	99.9	130	103
Average Concentration (ng/mL) ± %RPD		73.1 ng/mL ± 2.7%		26.8 ng/mL ± 6.4%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-055	P265B; Sample	707	NA	59.0	NA
ISO18-14-01-055-DUP	P265B; Sample Dup	698	NA	61.2	NA
ISO18-14-01-055-FMS	P265B; FMS	801	NC	156	95.9
Average Concentration (ng/mL) ± %RPD		703 ng/mL ± 1.3%		60.1 ng/mL ± 3.7%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 24. Location ID: P343

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-057	P343; Sample	839	NA	726	NA
ISO18-14-01-057-DUP	P343; Sample Dup	831	NA	743	NA
ISO18-14-01-057-FMS	P343; FMS	1350	99.0	1250	99.1
Average Concentration (ng/mL) ± %RPD		835 ng/mL ± 0.96%		735 ng/mL ± 2.3%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-057	P343; Sample	7420	NA	10.2	NA
ISO18-14-01-057-DUP	P343; Sample Dup	7590	NA	10.7	NA
ISO18-14-01-057-FMS	P343; FMS	8000	NC	28.6	90.8
Average Concentration (ng/mL) ± %RPD		7510 ng/mL ± 2.3%		10.5 ng/mL ± 4.8%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 25. Location ID: P374

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-059	P374; Sample	693	NA	863	NA
ISO18-14-01-059-DUP	P374; Sample Dup	708	NA	867	NA
ISO18-14-01-059-FMS	P374; FMS	1260	108	1420	107
Average Concentration (ng/mL) ± %RPD		701 ng/mL ± 2.1%		865 ng/mL ± 0.46%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-059	P374; Sample	1970	NA	28.6	NA
ISO18-14-01-059-DUP	P374; Sample Dup	1970	NA	28.0	NA
ISO18-14-01-059-FMS	P374; FMS	2510	NC	47.2	94.5
Average Concentration (ng/mL) ± %RPD		1970 ng/mL ± 0.0%		28.3 ng/mL ± 2.1%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 26. Location ID: P372

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-062	P372; Sample	72.1	NA	54.7	NA
ISO18-14-01-062-DUP	P372; Sample Dup	69.0	NA	54.9	NA
ISO18-14-01-062-FMS	P372; FMS	167	96.5	157	102
Average Concentration (ng/mL) ± %RPD		70.6 ng/mL ± 4.4%		54.8 ng/mL ± 0.36%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-062	P372; Sample	1360	NA	37.8	NA
ISO18-14-01-062-DUP	P372; Sample Dup	1330	NA	37.6	NA
ISO18-14-01-062-FMS	P372; FMS	1430	NC	132	94.3
Average Concentration (ng/mL) ± %RPD		1350 ng/mL ± 2.2%		37.7 ng/mL ± 0.53%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 27. Location ID: PA109A

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-064	PA109A; Sample	114	NA	114	NA
ISO18-14-01-064-DUP	PA109A; Sample Dup	114	NA	114	NA
ISO18-14-01-064-FMS	PA109A; FMS	176	NC	182	124
Average Concentration (ng/mL) ± %RPD		114 ng/mL ± 0.0%		114 ng/mL ± 0.0%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-064	PA109A; Sample	223	NA	1.53	NA
ISO18-14-01-064-DUP	PA109A; Sample Dup	222	NA	1.46	NA
ISO18-14-01-064-FMS	PA109A; FMS	291	NC	5.86	87.3
Average Concentration (ng/mL) ± %RPD		223 ng/mL ± 0.45%		1.50 ng/mL ± 4.7%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 28. Location ID: Travel Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-067	Travel Blank	<0.0240	NA	<0.0250	NA	<0.0250	NA
ISO18-14-01-067-FMS-LOW	Travel Blank FMS Low	1.80	93.8	1.60	80.0	1.79	89.5
ISO18-14-01-067-FMS-HIGH	Travel Blank FMS High	536	103	22.2	111	550	106

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-01-067	Travel Blank	<0.0232	NA	<0.0248	NA
ISO18-14-01-067-FMS-LOW	Travel Blank FMS Low	1.73	93.5	1.85	92.5
ISO18-14-01-067-FMS-HIGH	Travel Blank FMS High	503	96.7	19.4	97.0

NA = Not Applicable

Table 29. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO18-14-01-001	BD24-3; Sample	119	121	NA
ISO18-14-01-001-DUP	BD24-3; Sample Duplicate	117	117	NA
ISO18-14-01-002	BD24-4; Sample ⁽²⁾⁽³⁾	96.0	96.5	NA
ISO18-14-01-002-DUP	BD24-4; Sample Duplicate ⁽²⁾⁽³⁾	98.4	93.2	NA
ISO18-14-01-003	D09; Sample	114	113	NA
ISO18-14-01-003-DUP	D09; Sample Duplicate	114	116	NA
ISO18-14-01-004	D10; Sample	112	114	NA
ISO18-14-01-004-DUP	D10; Sample Duplicate	113	116	NA
ISO18-14-01-005	D11; Sample	109	113	NA
ISO18-14-01-005-DUP	D11; Sample Duplicate	111	114	NA
ISO18-14-01-006	D14; Sample ⁽²⁾⁽³⁾	96.4	94.5	NA
ISO18-14-01-006-DUP	D14; Sample Duplicate ⁽²⁾⁽³⁾	93.4	91.7	NA
ISO18-14-01-006-FMS	D14; FMS ⁽²⁾⁽³⁾	94.2	94.0	NA
ISO18-14-01-008	D17; Sample	112	116	NA
ISO18-14-01-008-DUP	D17; Sample Duplicate	115	118	NA
ISO18-14-01-009	D18; Sample	115	117	NA
ISO18-14-01-009-DUP	D18; Sample Duplicate	117	118	NA
ISO18-14-01-010	D5; Sample	113	114	NA
ISO18-14-01-010-DUP	D5; Sample Duplicate	113	114	NA
ISO18-14-01-010-FMS	D5; FMS	117	119	NA
ISO18-14-01-011	ND7; Sample	118	117	NA
ISO18-14-01-011-DUP	ND7; Sample Duplicate	112	116	NA
ISO18-14-01-011-FMS	ND7; FMS	115	119	NA
ISO18-14-01-012	P118A; Sample	115	114	NA
ISO18-14-01-012-DUP	P118A; Sample Duplicate	116	114	NA
ISO18-14-01-013	P118B; Sample	115	113	103
ISO18-14-01-013-DUP	P118B; Sample Duplicate	114	114	104
ISO18-14-01-013-FMS	P118B; FMS	117	111	100
ISO18-14-01-014	P119A; Sample	115	114	NA
ISO18-14-01-014-DUP	P119A; Sample Duplicate	116	116	NA
ISO18-14-01-015	P119B; Sample	124	125	NA
ISO18-14-01-015-DUP	P119B; Sample Duplicate	120	120	NA
ISO18-14-01-016	P121; Sample ⁽²⁾⁽³⁾	83.8	91.3	NA
ISO18-14-01-016-DUP	P121; Sample Duplicate ⁽²⁾⁽³⁾	98.0	94.5	NA
ISO18-14-01-017	P321; Sample	118	116	NA
ISO18-14-01-017-DUP	P321; Sample Duplicate	117	118	NA
ISO18-14-01-018	3M vijver; Sample ⁽³⁾	118	117	NA
ISO18-14-01-018-DUP	3M vijver; Sample Duplicate ⁽³⁾	114	115	NA
ISO18-14-01-019	Blokkersdijkvijver Noord; Sample ⁽³⁾	119	119	NA

NA = Not Applicable

- (1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.
- (2) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were not added to the samples during sample preparation.
- (3) The surrogate recovery standards were added to the sample bottle prior to the sampling event.
- (4) Surrogate recovery standard did not meet acceptance criteria of 100 ± 30%; however, the average recovery for the sample set did meet acceptance criteria. No adjustment was made to the analytical uncertainty.

Table 29 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO18-14-01-019-DUP	Blokkersdijkvijver Noord; Sample Dup ⁽³⁾	110	110	NA
ISO18-14-01-019-FMS	Blokkersdijkvijver Noord; FMS ⁽³⁾	111	114	NA
ISO18-14-01-020	Blokkersdijkvijver standard; Sample ⁽³⁾	110	117	NA
ISO18-14-01-020-DUP	Blokkersdijkvijver standard; Sample Dup ⁽³⁾	114	115	NA
ISO18-14-01-021	L21; Sample ⁽²⁾⁽³⁾	96.6	92.6	NA
ISO18-14-01-021-DUP	L21; Sample Duplicate ⁽²⁾⁽³⁾	92.0	95.3	NA
ISO18-14-01-021-FMS	L21; FMS ⁽²⁾⁽³⁾	93.2	88.8	NA
ISO18-14-01-022	L22; Sample ⁽²⁾⁽³⁾	94.1	92.4	NA
ISO18-14-01-022-DUP	L22; Sample Duplicate ⁽²⁾⁽³⁾	90.4	91.9	NA
ISO18-14-01-023	L31; Sample ⁽³⁾	119	121	NA
ISO18-14-01-023-DUP	L31; Sample Duplicate ⁽³⁾	119	119	NA
ISO18-14-01-024	L4; Sample ⁽³⁾	113	117	NA
ISO18-14-01-024-DUP	L4; Sample Duplicate ⁽³⁾	113	117	NA
ISO18-14-01-025	P114bis; Sample ⁽³⁾	112	115	NA
ISO18-14-01-025-DUP	P114bis; Sample Duplicate ⁽³⁾	114	115	NA
ISO18-14-01-025-FMS	P114bis; FMS ⁽³⁾	116	119	NA
ISO18-14-01-026	P115; Sample ⁽³⁾	114	118	NA
ISO18-14-01-026-DUP	P115; Sample Duplicate ⁽³⁾	111	114	NA
ISO18-14-01-027	P116; Sample ⁽³⁾	117	118	NA
ISO18-14-01-027-DUP	P116; Sample Duplicate ⁽³⁾	111	117	NA
ISO18-14-01-028	Effluent WWTP; Sample ⁽³⁾	109	112	NA
ISO18-14-01-028-DUP	Effluent WWTP; Sample Duplicate ⁽³⁾	109	108	NA
ISO18-14-01-029	PP01; Sample	121	123	NA
ISO18-14-01-029-DUP	PP01; Sample Duplicate	116	116	NA
ISO18-14-01-029-FMS	PP01; FMS	113	115	NA
ISO18-14-01-030	PP02; Sample	118	114	NA
ISO18-14-01-030-DUP	PP02; Sample Duplicate	115	115	NA
ISO18-14-01-031	PP04; Sample	112	116	NA
ISO18-14-01-031-DUP	PP04; Sample Duplicate	112	115	NA
ISO18-14-01-032	PP05; Sample	113	118	NA
ISO18-14-01-032-DUP	PP05; Sample Duplicate	111	118	NA
ISO18-14-01-032-FMS	PP05; FMS	111	111	NA
ISO18-14-01-033	PP06; Sample	112	114	NA
ISO18-14-01-033-DUP	PP06; Sample Duplicate	115	117	NA
ISO18-14-01-034	PP07; Sample	122	128	NA
ISO18-14-01-034-DUP	PP07; Sample Duplicate	118	123	NA
ISO18-14-01-035	PP08; Sample	115	118	NA
ISO18-14-01-035-DUP	PP08; Sample Duplicate	117	119	NA
ISO18-14-01-036	PP09; Sample	114	116	NA
ISO18-14-01-036-DUP	PP09; Sample Duplicate	117	120	NA

NA = Not Applicable

- (1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.
- (2) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were not added to the samples during sample preparation.
- (3) The surrogate recovery standards were added to the sample bottle prior to the sampling event.
- (4) Surrogate recovery standard did not meet acceptance criteria of 100 ± 30%; however, the average recovery for the sample set did meet acceptance criteria. No adjustment was made to the analytical uncertainty.

Table 29 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO18-14-01-037	PP10; Sample	114	116	NA
ISO18-14-01-037-DUP	PP10; Sample Duplicate	112	116	NA
ISO18-14-01-038	12; Sample	120	120	NA
ISO18-14-01-038-DUP	12; Sample Duplicate	108	116	NA
ISO18-14-01-039	13; Sample	123	125	NA
ISO18-14-01-039-DUP	13; Sample Duplicate	118	123	NA
ISO18-14-01-040	5; Sample ⁽³⁾	114	122	NA
ISO18-14-01-040-DUP	5; Sample Duplicate ⁽³⁾	111	120	NA
ISO18-14-01-041	Bemalingsstation; Sample ⁽³⁾	115	122	NA
ISO18-14-01-041-DUP	Bemalingsstation; Sample Dup ⁽³⁾	115	120	NA
ISO18-14-01-041-FMS	Bemalingsstation; FMS ⁽³⁾	116	120	NA
ISO18-14-01-042	Collector put; Sample ⁽³⁾	115	121	NA
ISO18-14-01-042-DUP	Collector put; Sample Duplicate ⁽³⁾	117	118	NA
ISO18-14-01-043	P27; Sample	122	126	NA
ISO18-14-01-043-DUP	P27; Sample Duplicate	118	123	NA
ISO18-14-01-044	P21B; Sample	115	118	NA
ISO18-14-01-044-DUP	P21B; Sample Duplicate	115	119	NA
ISO18-14-01-045	P304; Sample	120	126	NA
ISO18-14-01-045-DUP	P304; Sample Duplicate	115	120	NA
ISO18-14-01-045-FMS	P304; FMS	118	121	NA
ISO18-14-01-046	P305; Sample	120	121	NA
ISO18-14-01-046-DUP	P305; Sample Duplicate	113	118	NA
ISO18-14-01-047	P42; Sample	118	116	NA
ISO18-14-01-047-DUP	P42; Sample Duplicate	117	119	NA
ISO18-14-01-047-FMS	P42; FMS	118	120	NA
ISO18-14-01-048	L19; Sample	113	117	NA
ISO18-14-01-048-DUP	L19; Sample Duplicate	109	113	NA
ISO18-14-01-049	M4; Sample	118	122	NA
ISO18-14-01-049-DUP	M4; Sample Duplicate	114	120	NA
ISO18-14-01-050	P118C; Sample	122	128	NA
ISO18-14-01-050-DUP	P118C; Sample Duplicate	116	123	NA
ISO18-14-01-050-FMS	P118C; FMS	116	123	NA
ISO18-14-01-051	P119C; Sample	122	125	NA
ISO18-14-01-051-DUP	P119C; Sample Duplicate	117	120	NA
ISO18-14-01-052	P262; Sample	118	125	NA
ISO18-14-01-052-DUP	P262; Sample Duplicate	113	119	NA
ISO18-14-01-053	P263; Sample	121	123	NA
ISO18-14-01-053-DUP	P263; Sample Duplicate	115	122	NA
ISO18-14-01-054	P264; Sample	116	124	NA
ISO18-14-01-054-DUP	P264; Sample Duplicate	120	124	NA

NA = Not Applicable

- (1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.
- (2) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were not added to the samples during sample preparation.
- (3) The surrogate recovery standards were added to the sample bottle prior to the sampling event.
- (4) Surrogate recovery standard did not meet acceptance criteria of 100 ± 30%; however, the average recovery for the sample set did meet acceptance criteria. No adjustment was made to the analytical uncertainty.

Table 29 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO18-14-01-055	P265B; Sample	120	126	NA
ISO18-14-01-055-DUP	P265B; Sample Duplicate	115	120	NA
ISO18-14-01-055-FMS	P265B; FMS	120	124	NA
ISO18-14-01-056	P340; Sample	117	124	NA
ISO18-14-01-056-DUP	P340; Sample Duplicate	120	119	NA
ISO18-14-01-057	P343; Sample	121	123	NA
ISO18-14-01-057-DUP	P343; Sample Duplicate	113	118	NA
ISO18-14-01-057-FMS	P343; FMS	119	125	NA
ISO18-14-01-058	P371; Sample	115	125	NA
ISO18-14-01-058-DUP	P371; Sample Duplicate	117	121	NA
ISO18-14-01-059	P374; Sample	113	119	NA
ISO18-14-01-059-DUP	P374; Sample Duplicate	118	123	NA
ISO18-14-01-059-FMS	P374; FMS	115	120	NA
ISO18-14-01-060	B3-bis; Sample ⁽³⁾⁽³⁾	118	127	NA
ISO18-14-01-060-DUP	B3-bis; Sample Duplicate	113	117	NA
ISO18-14-01-061	B7; Sample	116	126	NA
ISO18-14-01-061-DUP	B7; Sample Duplicate	116	127	NA
ISO18-14-01-062	P372; Sample	121	124	NA
ISO18-14-01-062-DUP	P372; Sample Duplicate	116	123	NA
ISO18-14-01-062-FMS	P372; FMS	116	124	NA
ISO18-14-01-063	P378; Sample	112	121	NA
ISO18-14-01-063-DUP	P378; Sample Duplicate	115	125	NA
ISO18-14-01-064	PA109A; Sample	116	123	NA
ISO18-14-01-064-DUP	PA109A; Sample Duplicate	116	118	NA
ISO18-14-01-064-FMS	PA109A; FMS	117	135 ⁽⁴⁾	NA
ISO18-14-01-065	PA111A; Sample	115	125	NA
ISO18-14-01-065-DUP	PA111A; Sample Duplicate	113	119	NA
ISO18-14-01-066	PA112; Sample	119	126	NA
ISO18-14-01-066-DUP	PA112; Sample Duplicate	107	116	NA
ISO18-14-01-067	Travel Blank ⁽²⁾⁽³⁾	101	94.7	NA
ISO18-14-01-067-FMS-LOW	Travel Blank FMS Low ⁽²⁾⁽³⁾	99.4	95.7	NA
ISO18-14-01-067-FMS-HIGH	Travel Blank FMS High	110	114	NA

NA = Not Applicable

- (1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.
- (2) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were not added to the samples during sample preparation.
- (3) The surrogate recovery standards were added to the sample bottle prior to the sampling event.
- (4) Surrogate recovery standard did not meet acceptance criteria of 100 ± 30%; however, the average recovery for the sample set did meet acceptance criteria. No adjustment was made to the analytical uncertainty.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 10-29 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M EHS Laboratory standard operating procedures.

7 Attachments

Chain of Custody Form

8 Signatures


Digitally signed by Scott T. Porcher
DN: c=US, st=MN, l=St. Paul, ou=EHS Laboratory,
o=3M, cn=Scott T. Porcher
Reason: I am the author of this document
Date: 2018.04.13 14:02:40 -05'00'

Scott Porcher, 3M Report Author


Susan T. Wolf
c=US, st=MN, l=St. Paul, ou=EHS Laboratory,
o=3M, cn=Susan T. Wolf
I have reviewed this document
2018.04.13 11:46:11 -05'00'

Susan T. Wolf, 3M Principal Analytical Investigator


Digitally signed by Brian T. Mader
DN: c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory -
authenticated by LRA, o=3M, cn=Brian T. Mader
Reason: I have reviewed this document
Date: 2018.04.19 12:42:23 -05'00'

Brian T. Mader, Ph.D., 3M EHS Laboratory Manager

The 3M EHS Laboratory's Quality Assurance Unit has audited the data and report for this project.


Digitally signed by Kent R. Lindstrom
DN: c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory - authenticated
by LRA, o=3M, cn=Kent R. Lindstrom
Reason: I agreed to the terms defined by the placement of my signature on this
document
Date: 2018.04.16 09:09:49 -05'00'

Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M EHS Laboratory.

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 3M Center, Bldg 260-5N-17
 St. Paul, MN 55144

Phone: [REDACTED]
 Alt. Ph: [REDACTED]
 Fax: (651) 733-1111

Project: ISO18-14-01

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
 Department: 832202 Site Source: 01WJWT10
 Project Number:
 Date Created: 1/11/2018

Project Description: 3M Antwerp Water Sampling for PFCs; January 2018

Comments:

Completion Date:
 Project Lead: Susan T. Wolf
 Phone Number:
 Email Address: [REDACTED]

Gw = groundwater

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO18-14-01-001	BD24-3; Sample	301118	GW	/
ISO18-14-01-001-DUP	BD24-3; Sample Duplicate	301118	GW	/
ISO18-14-01-002	BD24-4; Sample	301118	GW	/
ISO18-14-01-002-DUP	BD24-4; Sample Duplicate	301118	GW	/
ISO18-14-01-003	D09; Sample	291118	GW	/
ISO18-14-01-003-DUP	D09; Sample Duplicate	291118	GW	/
ISO18-14-01-004	D10; Sample	301118	GW	/
ISO18-14-01-004-DUP	D10; Sample Duplicate	301118	GW	/
ISO18-14-01-005	D11; Sample	291118	GW	/
ISO18-14-01-005-DUP	D11; Sample Duplicate	291118	GW	/
ISO18-14-01-006	D14; Sample	291118	GW	/
ISO18-14-01-006-DUP	D14; Sample Duplicate	291118	GW	/
ISO18-14-01-006-FMS	D14; FMS	291118	GW	/
ISO18-14-01-007	D16; Sample	/	/	NO SAMPLE

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: RT ^① Deg C Received on Ice Other:

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Tun	5/21/18		FEDEX	JOSEPH TUNN	2-7-18	1400

① SENDER WROTE "3" IN THIS FIELD, BUT SAMPLES WERE RECEIVED AT RT.
 JT 2-7-18

Page 1 of 10
 Page 39 of 48

3M EHS LABORATORY
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St. Paul, MN 55144

Project: ISO18-14-01 (cont.)

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Fax: ([REDACTED]

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/11/2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs; January 2018

GW = groundwater

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO18-14-01-007-DUP	D16; Sample Duplicate	/	/	NO SAMPLE
ISO18-14-01-008	D17; Sample	29/11/18	GW	/
ISO18-14-01-008-DUP	D17; Sample Duplicate	29/11/18	GW	/
ISO18-14-01-009	D18; Sample	29/11/18	GW	BLACK
ISO18-14-01-009-DUP	D18; Sample Duplicate	29/11/18	GW	BLACK
ISO18-14-01-010	D5; Sample	29/11/18	GW	/
ISO18-14-01-010-DUP	D5; Sample Duplicate	29/11/18	GW	/
ISO18-14-01-010-FMS	D5; FMS	29/11/18	GW	/
ISO18-14-01-011	ND7; Sample	30/11/18	GW	/
ISO18-14-01-011-DUP	ND7; Sample Duplicate	30/11/18	GW	/
ISO18-14-01-011-FMS	ND7; FMS	30/11/18	GW	/
ISO18-14-01-012	P118A; Sample	1/12/18	GW	/
ISO18-14-01-012-DUP	P118A; Sample Duplicate	1/12/18	GW	/
ISO18-14-01-013	P118B; Sample	1/12/18	GW	/
ISO18-14-01-013-DUP	P118B; Sample Duplicate	1/12/18	GW	/
ISO18-14-01-013-FMS	P118B; FMS	1/12/18	GW	/
ISO18-14-01-014	P119A; Sample	30/11/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: RT 30 Deg C Received on Ice Other:

Collected by (print): Tine Mandenx (TMA) Collector's signature: J. T. M.

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	5/12/18		FEDEX	JOSEPH TILMAN	2-7-18	14:00

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Project: ISO18-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/11/2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

GW = groundwater
SW = surface water

Project Description: 3M Antwerp Water Sampling for PFCs; January 2018

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
-------------------------	---------------------------	--------------------------	---------------	----------------

ISO18-14-01-014-DUP	P119A; Sample Duplicate	30/11/18	GW	/
ISO18-14-01-015	P119B; Sample	30/11/18	GW	/
ISO18-14-01-015-DUP	P119B; Sample Duplicate	30/11/18	GW	/
ISO18-14-01-016	P121; Sample	30/11/18	GW	/
ISO18-14-01-016-DUP	P121; Sample Duplicate	30/11/18	GW	/
ISO18-14-01-017	P321; Sample	1/12/18	GW	/
ISO18-14-01-017-DUP	P321; Sample Duplicate	1/12/18	GW	/
ISO18-14-01-018	3M vijver; Sample	30/11/18	SW	/
ISO18-14-01-018-DUP	3M vijver; Sample Duplicate	30/11/18	SW	/
ISO18-14-01-019	Blokkersdijkvijver – Noord; Sample	30/11/18	SW	/
ISO18-14-01-019-DUP	Blokkersdijkvijver – Noord; Sample Dup	30/11/18	SW	/
ISO18-14-01-019-FMS	Blokkersdijkvijver – Noord; FMS	30/11/18	SW	/
ISO18-14-01-020	Blokkersdijkvijver – standard; Sample	30/11/18	SW	/
ISO18-14-01-020-DUP	Blokkersdijkvijver – standard; Sample Dup	30/11/18	SW	/
ISO18-14-01-021	L21; Sample	30/11/18	GW	/
ISO18-14-01-021-DUP	L21; Sample Duplicate	30/11/18	GW	/
ISO18-14-01-021-FMS	L21; FMS	30/11/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: RT 30 Deg C Received on Ice Other:

Collected by (print): Nicole Mandenx (GMG) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	Nicole Mandenx	5/12/18		FEDEX	JOSEPH TILMAN	2-7-18	1400

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Project: ISO18-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/11/2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

*WW = surface water
GW = groundwater*

Project Description: 3M Antwerp Water Sampling for PFCs; January 2018

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
------------------	--------------------	-------------------	--------	---------

ISO18-14-01-022	L22; Sample	3/11/18	GW	/
ISO18-14-01-022-DUP	L22; Sample Duplicate	3/11/18	GW	/
ISO18-14-01-023	L31; Sample	3/11/18	GW	/
ISO18-14-01-023-DUP	L31; Sample Duplicate	3/11/18	GW	/
ISO18-14-01-024	L4; Sample	3/11/18	GW	/
ISO18-14-01-024-DUP	L4; Sample Duplicate	3/11/18	GW	/
ISO18-14-01-025	P114bis; Sample	3/11/18	GW	/
ISO18-14-01-025-DUP	P114bis; Sample Duplicate	3/11/18	GW	/
ISO18-14-01-025-FMS	P114bis; FMS	3/11/18	GW	/
ISO18-14-01-026	P115; Sample	3/11/18	GW	/
ISO18-14-01-026-DUP	P115; Sample Duplicate	3/11/18	GW	/
ISO18-14-01-027	P116; Sample	3/11/18	GW	/
ISO18-14-01-027-DUP	P116; Sample Duplicate	3/11/18	GW	/
ISO18-14-01-028	Effluent WWTP; Sample	3/11/18	WW	/
ISO18-14-01-028-DUP	Effluent WWTP; Sample Duplicate	3/11/18	WW	/
ISO18-14-01-029	PP01; Sample	3/11/18	GW	/
ISO18-14-01-029-DUP	PP01; Sample Duplicate	3/11/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: RT 30 Deg C Received on Ice Other:

Collected by (print): Tina Mandony (TMA) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	3/11/18		FEDEX	JOSPEH TILLMAN	2-7-18	1400

3M EHS LABORATORY
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Project: ISO18-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/11/2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs; January 2018

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO18-14-01-029-FMS	PP01; FMS	301118	GW	/
ISO18-14-01-030	PP02; Sample	301118	GW	/
ISO18-14-01-030-DUP	PP02; Sample Duplicate	301118	GW	/
ISO18-14-01-031	PP04; Sample	301118	GW	/
ISO18-14-01-031-DUP	PP04; Sample Duplicate	301118	GW	/
ISO18-14-01-032	PP05; Sample	301118	GW	/
ISO18-14-01-032-DUP	PP05; Sample Duplicate	301118	GW	/
ISO18-14-01-032-FMS	PP05; FMS	301118	GW	/
ISO18-14-01-033	PP06; Sample	301118	GW	/
ISO18-14-01-033-DUP	PP06; Sample Duplicate	301118	GW	/
ISO18-14-01-034	PP07; Sample	301118	GW	/
ISO18-14-01-034-DUP	PP07; Sample Duplicate	301118	GW	/
ISO18-14-01-035	PP08; Sample	301118	GW	/
ISO18-14-01-035-DUP	PP08; Sample Duplicate	301118	GW	/
ISO18-14-01-036	PP09; Sample	301118	GW	/
ISO18-14-01-036-DUP	PP09; Sample Duplicate	301118	GW	/
ISO18-14-01-037	PP10; Sample	301118	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: RT 30 Deg C Received on Ice Other:

Collected by (print): Nicole Mandony (CWA) Collector's signature:

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	1ma	5/21/18		FEDEX	JOSEPH TULLMAN	2-7-18	1400

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
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St. Paul, MN 55144

Project: ISO18-14-01 (cont.)

Phone: [REDACTED]
Alt. Ph: [REDACTED]
Fax: (651) 733-2000

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number: [REDACTED]
Date Created: 1/11/2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs; January 2018

GW = groundwater
SW = surface water
WW = waste water

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO18-14-01-037-DUP	PP10; Sample Duplicate	301-11-18	GW	/
ISO18-14-01-038	12; Sample	221-11-18	SW	/
ISO18-14-01-038-DUP	12; Sample Duplicate	221-11-18	SW	/
ISO18-14-01-039	13; Sample	221-11-18	SW	/
ISO18-14-01-039-DUP	13; Sample Duplicate	221-11-18	SW	/
ISO18-14-01-040	5; Sample	221-11-18	SW	/
ISO18-14-01-040-DUP	5; Sample Duplicate	221-11-18	SW	/
ISO18-14-01-041	Bemalingsstation; Sample	221-11-18	SW	/
ISO18-14-01-041-DUP	Bemalingsstation; Sample Dup	221-11-18	SW	/
ISO18-14-01-041-FMS	Bemalingsstation; FMS	221-11-18	SW	/
ISO18-14-01-042	Collector put; Sample	301-11-18	WW	/
ISO18-14-01-042-DUP	Collector put; Sample Duplicate	301-11-18	WW	/
ISO18-14-01-043	P27; Sample	301-11-18	GW	/
ISO18-14-01-043-DUP	P27; Sample Duplicate	301-11-18	GW	/
ISO18-14-01-044	P21B; Sample	291-11-18	GW	BLACK
ISO18-14-01-044-DUP	P21B; Sample Duplicate	291-11-18	GW	BLACK
ISO18-14-01-045	P304; Sample	291-11-18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 75 30 Deg C Received on Ice Other:

Collected by (print): Jine Mandenya (TMA) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	5/21/18		FEDEX	JOSEPH TIUMAN	2-7-18	1400

3M EHS LABORATORY
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Phone: [REDACTED]
Alt. Ph: [REDACTED]

Fax: [REDACTED]

Gw = groundwater

Project: ISO18-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/11/2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs; January 2018

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
-------------------------	---------------------------	--------------------------	---------------	----------------

ISO18-14-01-045-DUP	P304; Sample Duplicate	29/11/18	GW	/
ISO18-14-01-045-FMS	P304; FMS	29/11/18	GW	/
ISO18-14-01-046	P305; Sample	29/11/18	GW	/
ISO18-14-01-046-DUP	P305; Sample Duplicate	29/11/18	GW	/
ISO18-14-01-047	P42; Sample	29/11/18	GW	/
ISO18-14-01-047-DUP	P42; Sample Duplicate	29/11/18	GW	/
ISO18-14-01-047-FMS	P42; FMS	29/11/18	GW	/
ISO18-14-01-048	L19; Sample	30/11/18	GW	/
ISO18-14-01-048-DUP	L19; Sample Duplicate	30/11/18	GW	/
ISO18-14-01-049	M4; Sample	1/12/18	GW	/
ISO18-14-01-049-DUP	M4; Sample Duplicate	1/12/18	GW	/
ISO18-14-01-050	P118C; Sample	1/12/18	GW	/
ISO18-14-01-050-DUP	P118C; Sample Duplicate	1/12/18	GW	/
ISO18-14-01-050-FMS	P118C; FMS	1/12/18	GW	/
ISO18-14-01-051	P119C; Sample	30/11/18	GW	/
ISO18-14-01-051-DUP	P119C; Sample Duplicate	30/11/18	GW	/
ISO18-14-01-052	P262; Sample	1/12/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: RT 30 Deg C Received on Ice Other:

Collected by (print): Tine Mandenx (TMA) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	5/12/18		FEDEX	JOSEPH TULLMAN	2-7-18	1400

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Project: ISO18-14-01 (cont.)

Phone: [REDACTED]
Alt. Ph: [REDACTED]
Fax: (651) 733-2000

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/11/2018

Completion Date: *6w = groundwater*
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs; January 2018

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO18-14-01-052-DUP	P262; Sample Duplicate	1/21/18	GW	/
ISO18-14-01-053	P263; Sample	30/11/18	GW	/
ISO18-14-01-053-DUP	P263; Sample Duplicate	30/11/18	GW	/
ISO18-14-01-054	P264; Sample	1/21/18	GW	/
ISO18-14-01-054-DUP	P264; Sample Duplicate	1/21/18	GW	/
ISO18-14-01-055	P265B; Sample	1/21/18	GW	/
ISO18-14-01-055-DUP	P265B; Sample Duplicate	1/21/18	GW	/
ISO18-14-01-055-FMS	P265B; FMS	1/21/18	GW	/
ISO18-14-01-056	P340; Sample	30/11/18	GW	/
ISO18-14-01-056-DUP	P340; Sample Duplicate	30/11/18	GW	/
ISO18-14-01-057	P343; Sample	30/11/18	GW	/
ISO18-14-01-057-DUP	P343; Sample Duplicate	30/11/18	GW	/
ISO18-14-01-057-FMS	P343; FMS	30/11/18	GW	/
ISO18-14-01-058	P371; Sample	30/11/18	GW	/
ISO18-14-01-058-DUP	P371; Sample Duplicate	30/11/18	GW	/
ISO18-14-01-059	P374; Sample	30/11/18	GW	/
ISO18-14-01-059-DUP	P374; Sample Duplicate	30/11/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: *RT 30* Deg C Received on Ice Other:

Collected by (print): *Tina Mandane (TMA)* Collector's signature: *[Signature]*

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	5/21/18		FEDEX	JOSUA TUMAN	2-7-18	1400

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [REDACTED]
Alt. Ph: [REDACTED]
Fax: [REDACTED]

Project: ISO18-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/11/2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

GW = groundwater

Project Description: 3M Antwerp Water Sampling for PFCs; January 2018

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO18-14-01-059-FMS	P374; FMS	30/11/18	GW	/
ISO18-14-01-060	B3-bis; Sample	31/11/18	GW	/
ISO18-14-01-060-DUP	B3-bis; Sample Duplicate	31/11/18	GW	/
ISO18-14-01-061	B7; Sample	31/11/18	GW	/
ISO18-14-01-061-DUP	B7; Sample Duplicate	31/11/18	GW	/
ISO18-14-01-062	P372; Sample	30/11/18	GW	/
ISO18-14-01-062-DUP	P372; Sample Duplicate	30/11/18	GW	/
ISO18-14-01-062-FMS	P372; FMS	30/11/18	GW	/
ISO18-14-01-063	P378; Sample	31/11/18	GW	/
ISO18-14-01-063-DUP	P378; Sample Duplicate	31/11/18	GW	/
ISO18-14-01-064	PA109A; Sample	31/11/18	GW	/
ISO18-14-01-064-DUP	PA109A; Sample Duplicate	31/11/18	GW	/
ISO18-14-01-064-FMS	PA109A; FMS	31/11/18	GW	/
ISO18-14-01-065	PA111A; Sample	31/11/18	GW	/
ISO18-14-01-065-DUP	PA111A; Sample Duplicate	31/11/18	GW	/
ISO18-14-01-066	PA112; Sample	31/11/18	GW	/
ISO18-14-01-066-DUP	PA112; Sample Duplicate	31/11/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: RT 30 Deg C Received on Ice Other:

Collected by (print): Nicole Mandenx (LWA)

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	5/12/18		FEDEX	Julette Tillman	2-7-18	1400

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
 3M EHS Laboratory
 3M Center, Bldg 260-5N-17
 St. Paul, MN 55144

Project: ISO18-14-01 (cont.)

Phone: [REDACTED]
 Alt. Pho: [REDACTED]
 Fax: (651) [REDACTED]

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
 Department: 832202 Site Source: 01WJWT10
 Project Number:
 Date Created: 1/11/2018

Completion Date:
 Project Lead: Susan T. Wolf
 Phone Number: [REDACTED]
 Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs; January 2018

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO18-14-01-067	Travel Blank	/	/	*
ISO18-14-01-067-FMS-HIGH	Travel Blank FMS High	/	/	*
ISO18-14-01-067-FMS-LOW	Travel Blank FMS Low	/	/	*

* Travel Blank samples prepared by the 3M EHS Laboratory with the bottle order.

Sample Condition Upon Receipt: Acceptable All items accounted for
 Temperature: RT 30 Deg C Received on Ice Other:

Collected by (print):	Collector's signature:						
Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
3	JMA	5/2/18		FEDEX	JOSEPH TILMAN	2-7-18	1400



3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC18-01460-001

3M Lab Request Number: E18-0234

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: May 28th, 2018

Requester

Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000

SGS Belgium NV

Institute for Applied Chromatography Haven 407 Polderdijkweg 16 B-2030 Antwerpen
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Member of the SGS Group (Société Générale de Surveillance)

Registered office: Noorderlaan 87 B-2030 Antwerpen H.R. Antwerpen 141.810 BTW BE 404.882.750 Dexia 550-3580000-93
All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction - Summary.

At the request (Lab Request Number: E18-0025) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected between May 3rd and May 4th, 2018 from 11 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOA ($C_6F_{13}^{13}CF_2^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOS ($C_7F_{15}^{13}CF_2SO_3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Sven Herremans. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles. Table 1 summarizes the sample results. All PFBS, PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
P321		572	2177	3509	<0.787
P114bis		1.22	3.02	11.0	<0.145
Effluent WWTP		1.16	2.29	7.54	0.767
12		11.0	50.3	161	0.895
5		14.8	21.0	30.5	0.240
Bemalingsstation		11.7	17.0	25.6	0.228
Collector Put		21.6	74.3	152	8.29
PP02	<12.3	2427	575	13860	26.9
PP05	3172	1227	466	74.4	23.6
PP07	197	93.3	314	2558	31.6
PP09	12.3	258	509	2553	12.8

2. Methods – Analytical and Preparatory.**2.1. Sample collection.**

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
P321	100
P114bis	100
Effluent WWTP	100
12	100
5	100
Bemalingsstation	100
Collector Put	100
PP02	100
PP05	100
PP07	100
PP09	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefloreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
P321	1.0	0.1	Solution A	10.0	-
P114bis	1.0	0.05	Solution A	1.0	-
Effluent WWTP	1.0	0.1	Solution A	5.0	-
12	1.0	0.1	Solution A	10.0	-
5	1.0	0.1	Solution A	2.0	-
Bemalingsstation	1.0	0.1	Solution A	2.0	-
Collector Put	1.0	0.1	Solution A	5.0	-
PP02	1.0	0.2	Solution A	10.0	0.50mL extract + 10.0mL MeOH (*)
PP05	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP07	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP09	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)

(*): MeOH:LCMS-water (60:40)
 Solution A = 1000 ng/mL (nominal) ^{13}C -PFC Surrogate Standards

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ^{13}C -PFOA, PFOS, FOSA and ^{13}C -PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetrie C18 50 x 2.1mm, 3.5 μm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7 μm
Injection volume	5 μL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate ($\mu\text{L}/\text{min}$)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
		298.93/79.82	40	28	45
PFBS	2.08 - 2.50	298.93/98.78	40	29	45
		398.86/79.83	40	38	51
PFHS	2.45 - 3.05	398.86/98.79	40	35	51
		412.9/218.98	40	11	14
PFOA	2.80 - 3.65	412.9/368.87	40	11	14
		498.78/79.77	50	48	56
PFOS	2.92 - 4.20	498.78/98.73	50	39	56
		497.8 /77.82	125	34	44
¹³ C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of $\pm 25\%$ ($\pm 30\%$ for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.0524 to 0.0963 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.107 to 22.6 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within $\pm 25\%$, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by diluting 1 mL water with 10.0 mL MeOH:LCMS-water (60:40). Method blanks were prepared to evaluate the levels of background contamination in the overall dilution process (glassware, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carryover.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of LCMS-water, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCS)

One lab control spike was prepared and analyzed.

LCS was prepared by spiking known amounts of the analytes into 1.0 mL of LCMS-water to produce the desired concentration.

The spiked water sample was diluted with 10.0 mL MeOH:LCMS-water (60:40) and analyzed in the same manner as the samples.

The LCS produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 7 summarizes the LCS recovery results

Table 7. Lab Control Spike Results.

Dilution date	Description	Nominal Spike Level (ng/mL)	Percent Recovery					
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA
15 May 2018	LCS1	80	77.3	83.4	83.6	92.1	90.7	83.1
								85.2

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing PFBS, PFHS, PFOA, ^{13}C -PFOA, PFOS, FOSA and ^{13}C -PFOS (QC Lab Low and QC Lab High).

The QCs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 8 summarizes the QCs recovery results.

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery					
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA
25 Jan 2018	QC Lab Low inj1	10	104	89.1	113	124	102	97.8
	QC Lab High inj1	100	80.3	82.4	94.8	91.9	96.6	89.3
	QC Lab Low inj2	10	97.6	94.2	103	93.2	101	88.1
	QC Lab High inj2	100	80.7	77.3	94.0	83.3	92.9	86.5
								101
								81.1

3.7. Equations.Recovery.

$$\text{Recovery (\%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

The following tables summarize the results for the sample locations.

Table 9. Sample Results PFBS, PFHS, PFOA, PFOS and FOSA

	PFBS	PFHS	PFOA	PFOS	FOSA
	ng/ml	ng/ml	ng/ml	ng/ml	ng/ml
P321		572	2177	3509	<0.787
P114bis		1.22	3.02	11.0	<0.145
Effluent WWTP		1.16	2.29	7.54	0.767
12		11.0	50.3	161	0.895
5		14.8	21.0	30.5	0.240
Bemalingsstation		11.7	17.0	25.6	0.228
Collector Put		21.6	74.3	152	8.29
PP02	<12.3	2427	575	13860	26.9
PP05	3172	1227	466	74.4	23.6
PP07	197	93.3	314	2558	31.6
PP09	12.3	258	509	2553	12.8
Field Trip Blank	<0.582	<1.069	<0.608	<0.806	<0.787

Table 10. Sample Results ¹³C-PFOA and ¹³C-PFOS

	¹³ C-PFOA		¹³ C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
P321	98.9	86.1	87.8	71.3
P114bis	65.6	114	43.3	70.2
Effluent WWTP	107	93.5	118	95.4
12	108	94.8	117	95.1
5	114	99.3	112	90.9
Bemalingsstation	113	98.3	113	91.6
Collector Put	111	96.3	113	91.6
PP02	218	94.9	188	76.2
PP05	92.1	80.3	119	97.0
PP07	102	88.6	105	85.1
PP09	100	87.5	107	86.9
Field Trip Blank	118	103	118	95.6

5. Conclusion.

- The recovery of ¹³C-PFOA and ¹³C-PFOS fell within the method acceptance criterion of 70-130%.
- Lab control spike recoveries fell within the method acceptance criteria for all compounds, indicating that the analysis method is in control,
- QC Lab Standards recoveries fell within the method acceptance criteria for all compounds.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Tine Mandonx (ERM Belgium).

8. Signatures.

Sven Herremans,
Lab Operations Manager

Date May 28th, 2018

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date May 28th, 2018

I.A.C.
A Division of SGS Belgium NV

Reports are established on behalf of and for the account of the principal, who expressly accepts that these reports purely represent the situation at a given time and that they must always be presented and/or mentioned in their totality and in their particular context. SGS Belgium N.V., issuer of the reports, cannot be held liable for errors or modification of results during electronic or fax transmission. Only the originally signed report is binding.

The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.

Final Report

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

May 2018 Sampling

Laboratory Request Number: ISO18-14-02

Report Date – Date of Last Signature

Testing Laboratory

3M Environment, Health, and Safety
3M EHS Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Nicole Cauberghe
3M Belgium EHS Operations
3M Belgium; ZW018/0/33
Phone: [REDACTED]



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M EHS Laboratory

3M EHS Laboratory Manager: Brian T. Mader
3M Principal Analytical Investigator: Susan Wolf
Report Author: Chelsie Grochow

Analytical Report ISO18-14-02

Water Sample Analysis at 3M Antwerp, Belgium
May 2018 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M EHS Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. All samples were collected on May 3-4, 2018, and returned to the 3M EHS Laboratory on May 11, 2018, at ambient temperature. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS) (select locations only), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO18-14-02.

The 3M EHS Laboratory prepared sample containers for sixty-six sampling locations. Each sample set consisted of a field sample and field sample duplicate. Six locations also included a target analyte field matrix spike. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with a mass-labeled internal standard mix and select sample bottles were fortified with surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS prior to being sent to the field for sample collection. During sample collection, sample location "Blokkersdijkvijver Noord" was not sampled.

Samples were prepared and analyzed using method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Select internal standards were used to aid in the data quality objectives.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA
Zone: 2nd Aquifer					
ISO18-14-02-001	D16; Sample	387	133	2470	68.4
ISO18-14-02-001-DUP	D16; Sample Dup	384	134	2490	61.8
Average %RPD Sample/Sample Dup		386 ⁽²⁾ 0.78	134 ⁽²⁾ 0.75	2480 ⁽²⁾ 0.81	65.1 10
ISO18-14-02-002	P121; Sample	0.478	0.202	2.38	0.137
ISO18-14-02-002-DUP	P121; Sample Dup	0.496	0.202	2.42	0.126
Average %RPD Sample/Sample Dup		0.487 3.7	0.202 0.0	2.40 1.7	0.132 8.4
Zone: Blokkersdijk Nature Reserve					
ISO18-14-02-003	3M vijver; Sample	1.02	0.564	4.32	0.0378
ISO18-14-02-003-DUP	3M vijver; Sample Dup	1.06	0.558	4.42	0.0442
Average %RPD Sample/Sample Dup		1.04 3.8	0.561 1.1	4.37 2.3	0.0410 16
ISO18-14-02-005	Blokkersdijkvijver - standard; Sample	0.726	0.430	0.736	0.0258
ISO18-14-02-005-DUP	Blokkersdijkvijver - standard; Sample Dup	0.732	0.446	0.748	<0.0250
Average %RPD Sample/Sample Dup		0.729 0.82	0.438 3.7	0.742 1.6	0.0258 NA
ISO18-14-02-006	P321; Sample	2440	639	5300	0.374
ISO18-14-02-006-DUP	P321; Sample Dup	2450	657	5320	0.350
Average %RPD Sample/Sample Dup		2450 ⁽²⁾ 0.41	648 ⁽²⁾ 2.8	5310 ⁽²⁾ 0.38	0.362 6.6
ISO18-14-02-007	L21; Sample	0.218	0.113	4.40	0.105
ISO18-14-02-007-DUP	L21; Sample Dup	0.212	0.120	4.66	0.109
Average %RPD Sample/Sample Dup		0.215 2.8	0.117 6.0	4.53 5.7	0.107 3.7
ISO18-14-02-008	L22; Sample	0.270	0.185	4.44	0.0668
ISO18-14-02-008-DUP	L22; Sample Dup	0.252	0.173	3.96	0.0652
Average %RPD Sample/Sample Dup		0.261 6.9	0.179 6.7	4.20 11	0.0660 2.4
ISO18-14-02-009	L31; Sample	0.386	0.838	3.52	0.0768
ISO18-14-02-009-DUP	L31; Sample Dup	0.370	0.808	3.50	0.0696
Average %RPD Sample/Sample Dup		0.378 4.2	0.823 3.6	3.51 0.57	0.0732 9.8
ISO18-14-02-010	L4; Sample	2.16	1.70	18.9	3.70
ISO18-14-02-010-DUP	L4; Sample Dup	2.14	1.82	21.2	4.06
Average %RPD Sample/Sample Dup		2.15 0.93	1.76 6.8	20.1 11	3.88 9.3

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using internal standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 11%, PFBS \pm 13%, PFHS \pm 15%, PFOS \pm 12%, and PFOSA \pm 10%.
- (2) Sample set reported using external standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 8.8%, PFBS \pm 7.0%, PFHS \pm 7.8%, and PFOS \pm 17%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) Data uncertainty has been expanded to \pm 44% for PFOSA for location P21B based on field matrix spike recovery.

Table 1 continued. Sample Results Summary⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA
Zone: Blokkersdijk Nature Reserve					
ISO18-14-02-011	P114bis; Sample	2.12	1.25	13.7	0.0734
ISO18-14-02-011-DUP	P114bis; Sample Dup	2.16	1.26	14.0	0.0768
		Average	2.14	1.26	13.9
		%RPD Sample/Sample Dup	1.9	0.80	0.0751
				2.2	4.5
ISO18-14-02-012	P115; Sample	4.36	2.20	1.01	<0.100
ISO18-14-02-012-DUP	P115; Sample Dup	4.30	2.26	1.25	<0.100
		Average	4.33	2.23	1.13
		%RPD Sample/Sample Dup	1.4	2.7	<0.100
				21 ⁽³⁾	NA
ISO18-14-02-013	P116; Sample	0.760	0.544	9.10	0.128
ISO18-14-02-013-DUP	P116; Sample Dup	0.766	0.556	9.58	0.131
		Average	0.763	0.550	9.34
		%RPD Sample/Sample Dup	0.79	2.2	0.130
				5.1	2.3
Zone: Effluent WWTP					
ISO18-14-02-014	Effluent WWTP; Sample	2.10	1.44	10.4	0.820
ISO18-14-02-014-DUP	Effluent WWTP; Sample Dup	2.18	1.57	11.2	0.968
		Average	2.14	1.51	10.8
		%RPD Sample/Sample Dup	3.7	8.6	0.894
				7.4	17
Zone: Palingbeek & Tophatgracht					
ISO18-14-02-024	12; Sample	52.6	16.3	208	0.680
ISO18-14-02-024-DUP	12; Sample Dup	51.6	16.6	202	0.684
		Average	52.1	16.5	205 ⁽²⁾
		%RPD Sample/Sample Dup	1.9	1.8	0.682
				2.9	0.59
ISO18-14-02-025	13; Sample	50.8	17.6	172	0.562
ISO18-14-02-025-DUP	13; Sample Dup	54.6	18.5	185	0.620
		Average	52.7	18.1	179 ⁽²⁾
		%RPD Sample/Sample Dup	7.2	5.0	0.591
				7.3	9.8
ISO18-14-02-026	5; Sample	20.6	18.8	42.4	0.183
ISO18-14-02-026-DUP	5; Sample Dup	22.0	19.3	43.6	0.179
		Average	21.3	19.1	43.0
		%RPD Sample/Sample Dup	6.6	2.6	0.181
				2.8	2.2
ISO18-14-02-027	Bemalingsstation; Sample	18.0	15.9	39.4	0.166
ISO18-14-02-027-DUP	Bemalingsstation; Sample Dup	18.5	16.5	39.4	0.172
		Average	18.3	16.2	39.4
		%RPD Sample/Sample Dup	2.7	3.7	0.169
				0.0	3.6
Zone: Sewer					
ISO18-14-02-028	Collector put; Sample	77.8	26.4	210	21.8
ISO18-14-02-028-DUP	Collector put; Sample Dup	72.6	25.8	193	19.7
		Average	75.2	26.1	202 ⁽²⁾
		%RPD Sample/Sample Dup	6.9	2.3	20.8
				8.4	10

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using internal standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 11%, PFBS ± 13%, PFHS ± 15%, PFOS ± 12%, and PFOSA ± 10%.
- (2) Sample set reported using external standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 8.8%, PFBS ± 7.0%, PFHS ± 7.8%, and PFOS ± 17%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
- (4) Data uncertainty has been expanded to ±44% for PFOSA for location P21B based on field matrix spike recovery.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
Zone: Extraction Wells P&T						
ISO18-14-02-015	PP01; Sample	58.3	95.7	7760	2110	28.4
ISO18-14-02-015-DUP	PP01; Sample Dup	57.4	93.4	7670	2090	28.2
Average		57.9 ⁽²⁾	94.6 ⁽²⁾	7720 ⁽²⁾	2100 ⁽²⁾	28.3
%RPD Sample/Sample Dup		1.6	2.4	1.2	0.95	0.71
ISO18-14-02-016	PP02; Sample	547	32.9	2830	16900	31.0
ISO18-14-02-016-DUP	PP02; Sample Dup	567	32.2	2830	17200	32.2
Average		557 ⁽²⁾	32.6 ⁽²⁾	2830 ⁽²⁾	17100 ⁽²⁾	31.6
%RPD Sample/Sample Dup		3.6	2.2	0.0	1.8	3.8
ISO18-14-02-017	PP04; Sample	648	1990	286	5550	60.2
ISO18-14-02-017-DUP	PP04; Sample Dup	641	2080	284	5620	60.6
Average		645 ⁽²⁾	2040 ⁽²⁾	285 ⁽²⁾	5590 ⁽²⁾	60.4
%RPD Sample/Sample Dup		1.1	4.4	0.70	1.3	0.66
ISO18-14-02-018	PP05; Sample	653	4790	2140	241	73.4
ISO18-14-02-018-DUP	PP05; Sample Dup	723	5000	2580	216	78.8
Average		688 ⁽²⁾	4900 ⁽²⁾	2360 ⁽²⁾	229 ⁽²⁾	76.1
%RPD Sample/Sample Dup		10	4.3	19	11	7.1
ISO18-14-02-019	PP06; Sample	292	22.5	73.7	1630	216
ISO18-14-02-019-DUP	PP06; Sample Dup	295	23.2	75.2	1650	236
Average		294 ⁽²⁾	22.9 ⁽²⁾	74.5 ⁽²⁾	1640 ⁽²⁾	226
%RPD Sample/Sample Dup		1.0	3.1	2.0	1.2	8.8
ISO18-14-02-020	PP07; Sample	324	215	108	2900	102
ISO18-14-02-020-DUP	PP07; Sample Dup	322	215	108	2930	105
Average		323 ⁽²⁾	215 ⁽²⁾	108 ⁽²⁾	2920 ⁽²⁾	104
%RPD Sample/Sample Dup		0.62	0.0	0.0	1.0	2.9
ISO18-14-02-021	PP08; Sample	536	42.2	260	5350	52.2
ISO18-14-02-021-DUP	PP08; Sample Dup	556	44.7	271	5690	51.0
Average		546 ⁽²⁾	43.5 ⁽²⁾	266 ⁽²⁾	5520 ⁽²⁾	51.6
%RPD Sample/Sample Dup		3.7	5.8	4.1	6.2	2.3
ISO18-14-02-022	PP09; Sample	467	17.7	316	3100	20.6
ISO18-14-02-022-DUP	PP09; Sample Dup	482	18.3	322	3140	21.2
Average		475 ⁽²⁾	18.0 ⁽²⁾	319 ⁽²⁾	3120 ⁽²⁾	20.9
%RPD Sample/Sample Dup		3.2	3.3	1.9	1.3	2.9
ISO18-14-02-023	PP10; Sample	558	15.7	204	3480	22.8
ISO18-14-02-023-DUP	PP10; Sample Dup	551	16.3	205	3330	21.8
Average		555 ⁽²⁾	16.0 ⁽²⁾	205 ⁽²⁾	3410 ⁽²⁾	22.3
%RPD Sample/Sample Dup		1.3	3.8	0.49	4.4	4.5

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using internal standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 11%, PFBS \pm 13%, PFHS \pm 15%, PFOS \pm 12%, and PFOSA \pm 10%.
- (2) Sample set reported using external standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 8.8%, PFBS \pm 7.0%, PFHS \pm 7.8%, and PFOS \pm 17%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) Data uncertainty has been expanded to \pm 44% for PFOSA for location P21B based on field matrix spike recovery.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
Zone: Source Area – Building 16						
ISO18-14-02-029	P21B; Sample	9840	7730	11700	62000	7.38
ISO18-14-02-029-DUP	P21B; Sample Dup	10500	7980	12000	62400	6.76
Average %RPD Sample/Sample Dup		10200 ⁽²⁾ 6.5	7860 ⁽²⁾ 3.2	11900 ⁽²⁾ 2.5	62200 ⁽²⁾ 0.64	7.07 ⁽⁴⁾ 8.8
ISO18-14-02-030	Travel Blank	<0.0240	<0.100	<0.0250	<0.0464	<0.0250

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using internal standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 11%, PFBS ± 13%, PFHS ± 15%, PFOS ± 12%, and PFOSA ± 10%.
- (2) Sample set reported using external standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 8.8%, PFBS ± 7.0%, PFHS ± 7.8%, and PFOS ± 17%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
- (4) Data uncertainty has been expanded to ±44% for PFOSA for location P21B based on field matrix spike recovery.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis".

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluoroctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluoroctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluoroctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected on May 3-4, 2018, in Nalgene™ (high-density polyethylene) bottles prepared at the 3M EHS Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. All sample locations were spiked with a mass-labeled internal standard mix at a nominal concentration of 1 ng/mL and select samples were fortified with surrogate recovery standards (SRSs) at a nominal concentration of 0.1 ng/mL prior to sample collection. Samples were received on May 11, 2018.

2.3 Sample Preparation

Sample locations pre-spiked with surrogate recovery standards to be analyzed for PFOA, PFHS, and PFOS, and all samples to be analyzed for PFOSA were prepared by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples along with the method blanks.

Samples that required further dilution were prepared for PFOA, PFHS, PFOS and PFBS (select locations only) using a direct injection by methanol dilution method. Samples that required a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples that required a 1:100 dilution were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol. Samples that required a 1:500 dilution were prepared by diluting 0.02 mL of a well-mixed sample with 10.0 mL of methanol. Samples that required a 1:1000 dilution were prepared by first diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol, followed by a second dilution prepared by diluting 1 mL of the diluted sample into 9 mL of methanol. Diluted samples and LCCs were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

2.4 Analysis

All samples and quality control samples were analyzed for the target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). SRSs were added and included in the analysis of these samples. Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.2. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

The following analytical runs were used to report the results herein:

6/4/18 (ETS Kirk) Internal Standard Calibration Analysis:

- All sample locations pre-spiked with surrogate recovery standards were analyzed for PFOA, PFHS, PFOS, and PFOSA with the Travel Blank and Travel Blank FMS Low being analyzed for PFBS. All sample results were reported **except** for the following: L21 and P115 (PFOSA) and 12, 13, and Collector Put (PFOS).

6/5/18 (ETS Rita) Internal Standard Calibration Analysis:

- All remaining sample locations were analyzed for PFOSA. All sample results were reported **except** for PP06.

6/8/18 (ETS Kirk) External Standard Calibration Analysis:

- All remaining sample locations were analyzed for PFOA, PFHS, and PFOS with select locations analyzed for PFBS. In addition, sample locations 12, 13, and Collector Put were re-analyzed for PFOS. All sample results were reported **except** for the following: PP05 and P21B (PFOS).

6/12/18 (ETS Kirk) External Standard Calibration Analysis:

- Sample locations PP05 and P21B were analyzed for PFOS. All sample results were reported.

6/13/18 (ETS Rita) Internal Standard Calibration Analysis:

- Sample locations L21, P115, PP06, and P21B were re-analyzed for PFOSA. All sample results were reported **except** for P21B.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk	ETS Rita
Liquid Chromatograph	Agilent 1260	Agilent 1260
Analysis Method	ETS-8-044.3	ETS-8-044.3
Analysis Date	6/4/18, 6/8/18, 6/12/18	6/5/18, 6/13/18
Guard column	Prism RP (2.1 mm X 50 mm), 5 μ	Prism RP (2.1 mm X 50 mm), 5 μ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ
Injection Volume	2 or 5 μ L	2 or 4 μ L
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 6500+
Ion Source	Turbo Spray	Turbo Spray
Polarity	Negative	Negative
Software	Analyst 1.6.3	Analyst 1.6.3

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis				
Step Number	Total Time (min)	Flow Rate (μ L/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0.00	300	90.0	10.0
1	0.50	300	90.0	10.0
2	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard ⁽¹⁾	Mass Transition Q1/Q3
PFOA	413/369	[¹³ C ₈]-PFOA	421/376
	413/219		
	413/169		
PFBS	299/99	[¹⁸ O ₂]-PFBS	303/84
	299/80		
PFHS	399/99	[¹³ C ₃]-PFHS	402/80
	399/80		
PFOS	499/99	[¹³ C ₈]-PFOS	507/80
	499/80		
	499/130		
PFOSA	498/78	[¹³ C ₈]-PFOSA	506/78
[¹³ C ₄]-PFOA	417/372	[¹³ C ₈]-PFOA	421/376
[¹³ C ₄]-PFOS	503/80	[¹³ C ₈]-PFOS	507/80

The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

(1) Internal standard was not used for the external calibration analyses on 6/8/18 and 6/12/18.

3 Data Analysis

3.1 Calibration

6/4/18 and 6/5/18 Analysis (Internal Standard Calibration): Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of fourteen calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed. The calibration standards also contained surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

6/8/18 and 6/12/18 Analysis (External Standard Calibration): Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed on 6/8/18 and from 0.05 ng/mL to 100 ng/mL (nominal) on 6/12/18. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL or 0.05 ng/mL to 25 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

6/13/18 Analysis of PFOSA (Internal Standard Calibration): Samples were analyzed for PFOSA against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into laboratory reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 1.0 ng/mL. Calibration standards ranging from 0.025 ng/mL to 300 ng/mL (nominal) were analyzed. Prior to analysis, the calibration standards were diluted 2x by removing a 0.4 mL aliquot and diluting it with 0.4 mL of methanol. A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of $100 \pm 25\%$ ($100 \pm 30\%$ for the lowest curve point) were met for all analytes. The correlation coefficient (r) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.3 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the **Table 6** below.

Table 6. LOQ

Analyte	LOQ, ng/mL ⁽¹⁾ 6/4/18 Analysis	LOQ, ng/mL ⁽¹⁾ 6/5/18 Analysis	LOQ, ng/mL ⁽²⁾ 6/8/18 Analysis	LOQ, ng/mL ⁽²⁾ 6/12/18 Analysis	LOQ, ng/mL ⁽¹⁾ 6/13/18 Analysis
PFOA	0.0240	NA	0.0192	NA	NA
PFBS	0.100	NA	0.0200	NA	NA
PFHS	0.0250	NA	0.0200	NA	NA
PFOS	0.0464	NA	0.0464	0.0464	NA
PFOSA	0.0250	0.100	NA	NA	0.100

NA = Not Applicable

(1) A dilution factor of 2 was applied to the LOQ.

(2) A dilution factor was not applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of 100%±25%.

3.5 Blanks

Two types of blanks were prepared and analyzed with the samples: method/solvent blanks and field/trip blanks. Each blank result was reviewed and used to evaluate method performance. The method blanks were used to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. LCSs were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. Surrogates [¹³C₄]-PFOA and [¹³C₄]-PFOS were added post dilution when analyzed by external standard.

Method ETS-8-044.3 states that the average recovery of LCSs at each spiking level must be within 80%-120% with a RSD ≤20%. All LCS samples met criteria with the following exception:

- 6/4/18: High level LCSs (140 ng/mL) for PFBS and PFOSA were spiked above the resulting ULOQ. The Low and Mid-level LCSs were appropriate for the sample concentrations and the data were reported.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report. The following calculations were used to generate data in **Table 7**.

$$\text{LCS Percent Recovery} = \frac{\text{Calculated Concentration}}{\text{Spike Concentration}} * 100\%$$

$$\text{LCS% RSD} = \frac{\text{standard deviation LCS replicates}}{\text{average LCS recovery}} * 100\%$$

Table 7. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 6/4/18	PFOA (Linear + Branched)			PFBS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-180604-1	0.190	0.202	106	0.198	0.210	106
LCS-180604-2	0.190	0.210	111	0.198	0.210	106
LCS-180604-3	0.190	0.204	107	0.198	0.212	107
Average \pm %RSD	$108\% \pm 2.4\%$			$106\% \pm 0.54\%$		
LCS-180604-4	19.0	18.7	98.4	19.8	19.5	98.3
LCS-180604-5	19.0	18.2	95.9	19.8	19.2	97.1
LCS-180604-6	19.0	18.8	98.8	19.8	18.9	95.7
Average \pm %RSD	$97.7\% \pm 1.6\%$			$97.0\% \pm 1.3\%$		
LCS-180604-7	133	126	94.5	139	>ULOQ	NA
LCS-180604-8	133	122	91.5	139	>ULOQ	NA
LCS-180604-9	133	122	91.9	139	>ULOQ	NA
Average \pm %RSD	$92.6\% \pm 1.8\%$			NA ⁽¹⁾		

ETS-8-044.3 Internal Calibration Analyzed 6/4/18	PFHS			PFOS (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-180604-1	0.198	0.208	105	0.184	0.206	112
LCS-180604-2	0.198	0.204	103	0.184	0.189	103
LCS-180604-3	0.198	0.210	106	0.184	0.192	104
Average \pm %RSD	$105\% \pm 1.5\%$			$106\% \pm 4.6\%$		
LCS-180604-4	19.8	19.7	99.4	18.4	18.3	99.7
LCS-180604-5	19.8	19.4	98.2	18.4	18.5	101
LCS-180604-6	19.8	19.2	96.9	18.4	18.4	100
Average \pm %RSD	$98.2\% \pm 1.3\%$			$100\% \pm 0.68\%$		
LCS-180604-7	139	118	85.2	129	117	91.1
LCS-180604-8	139	116	83.8	129	110	85.0
LCS-180604-9	139	110	79.2	129	109	84.7
Average \pm %RSD	$82.7\% \pm 3.8\%$			$86.9\% \pm 4.2\%$		

NA = Not Applicable

ULOQ = Upper Limit of Quantitation

(1) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

PFOSA			
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-180604-1	0.198	0.214	109
LCS-180604-2	0.198	0.202	102
LCS-180604-3	0.198	0.206	104
Average ± %RSD	105% ± 3.4%		
LCS-180604-4	19.8	19.7	99.3
LCS-180604-5	19.8	19.6	98.8
LCS-180604-6	19.8	19.2	97.0
Average ± %RSD	98.4% ± 1.2%		
LCS-180604-7	139	>ULOQ	NA
LCS-180604-8	139	>ULOQ	NA
LCS-180604-9	139	>ULOQ	NA
Average ± %RSD	NA⁽¹⁾		

[¹³ C ₄]-PFOA				[¹³ C ₄]-PFOS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-180604-1	0.198	0.208	105	0.189	0.185	97.6
LCS-180604-2	0.198	0.194	98.1	0.189	0.190	100
LCS-180604-3	0.198	0.208	105	0.189	0.195	103
Average ± %RSD	103% ± 3.9%			100% ± 2.7%		
LCS-180604-4	1.98	2.10	106	1.89	1.90	100
LCS-180604-5	1.98	2.02	102	1.89	1.92	102
LCS-180604-6	1.98	2.12	107	1.89	1.94	103
Average ± %RSD	105% ± 2.5%			102% ± 1.5%		

NA = Not Applicable

ULOQ = Upper Limit of Quantitation

(1) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 6/5/18		PFOSA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	
LCS-180604-1	0.198	0.232	117	
LCS-180604-2	0.198	0.226	114	
LCS-180604-3	0.198	0.226	114	
Average ± %RSD	115% ± 1.5%			
LCS-180604-4	19.8	19.1	96.6	
LCS-180604-5	19.8	18.9	95.5	
LCS-180604-6	19.8	18.9	95.3	
Average ± %RSD	95.8% ± 0.73%			
LCS-180604-7	139	129	92.6	
LCS-180604-8	139	129	92.8	
LCS-180604-9	139	130	93.3	
Average ± %RSD	92.9% ± 0.39%			

ETS-8-044.3 External Calibration Analyzed 6/8/18		PFOA (Linear + Branched)			PFBS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	
LCS-180608-1	100	86.3	86.5	100	92.4	92.6	
LCS-180608-2	100	98.4	98.7	100	101	101	
LCS-180608-3	100	89.9	90.1	100	98.4	98.5	
Average ± %RSD	91.8% ± 6.8%			97.4% ± 4.4%			
LCS-180608-4	5000	4760	95.3	5000	5170	103	
LCS-180608-5	5000	4840	96.8	5000	5120	102	
LCS-180608-6	5000	4790	95.7	5000	4980	100	
Average ± %RSD	95.9% ± 0.81%			102% ± 1.7%			
LCS-180608-7	35000	33400	95.4	35000	35700	102	
LCS-180608-8	35000	32600	93.0	35000	35600	102	
LCS-180608-9	35000	31700	90.5	35000	34700	99.0	
Average ± %RSD	93.0% ± 2.6%			101% ± 1.7%			

NA = Not Applicable

ULOQ = Upper Limit of Quantitation

(1) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 6/8/18		PFHS			PFOS (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	
LCS-180608-1	100	91.4	91.4	100.0	84.8	84.8	
LCS-180608-2	100	98.4	98.6	100.0	91.9	92.1	
LCS-180608-3	100	95.9	95.9	100.0	92.9	92.9	
Average ± %RSD	95.3% ± 3.8%			89.9% ± 5.0%			
LCS-180608-4	5000	5070	101	5000	4870	97.4	
LCS-180608-5	5000	5120	102	5000	4820	96.4	
LCS-180608-6	5000	4960	99.3	5000	4770	95.4	
Average ± %RSD	101% ± 1.4%			96.4% ± 1.0%			
LCS-180608-7	35000	36000	103	35000	36000	103	
LCS-180608-8	35000	35800	102	35000	35100	100	
LCS-180608-9	35000	35400	101	35000	34500	98.4	
Average ± %RSD	102% ± 0.98%			100% ± 2.3%			

ETS-8-044.3 External Calibration Analyzed 6/8/18		[¹³ C ₄]-PFOA			[¹³ C ₄]-PFOS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	
LCS-180608-1	0.199	0.219	110	0.190	0.211	111	
LCS-180608-2	0.199	0.228	115	0.190	0.220	116	
LCS-180608-3	0.199	0.220	110	0.190	0.216	113	
Average ± %RSD	112% ± 2.6%			113% ± 2.2%			
LCS-180608-4	1.99	2.26	114	1.90	2.18	115	
LCS-180608-5	1.99	2.31	116	1.90	2.19	115	
LCS-180608-6	1.99	2.26	114	1.90	2.18	115	
Average ± %RSD	115% ± 1.0%			115% ± 0.0%			

NA = Not Applicable

ULOQ = Upper Limit of Quantitation

(1) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 6/12/18	PFOS (Linear + Branched)			[¹³ C ₄]-PFOS		
	Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)
LCS-180612-1	200	164	81.9	0.191	0.206	108
LCS-180612-2	200	177	88.6	0.191	0.209	110
LCS-180612-3	200	166	83.2	0.191	0.208	109
Average ± %RSD	84.6% ± 4.2%			109% ± 0.92%		
LCS-180612-4	20000	19900	99.5	1.91	2.18	114
LCS-180612-5	20000	19800	99.2	1.91	2.23	117
LCS-180612-6	20000	19400	97.2	1.91	2.18	114
Average ± %RSD	98.6% ± 1.3%			115% ± 1.5%		
LCS-180612-7	70000	71700	102			
LCS-180612-8	70000	69700	99.5			
LCS-180612-9	70000	70000	100			
Average ± %RSD	101% ± 1.3%					

ETS-8-044.3 Internal Calibration Analyzed 6/13/18	PFOSA			
	Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-180613-1	0.199	0.206	103	
LCS-180613-2	0.199	0.208	105	
LCS-180613-3	0.199	0.202	102	
Average ± %RSD	103% ± 1.5%			
LCS-180613-4	19.9	19.9	100	
LCS-180613-5	19.9	19.4	97.3	
LCS-180613-6	19.9	19.8	99.5	
Average ± %RSD	98.9% ± 1.5%			
LCS-180613-7	199	200	100	
LCS-180613-8	199	200	101	
LCS-180613-9	199	204	102	
Average ± %RSD	101% ± 0.99%			

NA = Not Applicable

ULOQ = Upper Limit of Quantitation

(1) LCSs spiked outside the calibration range.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.4. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in **Table 8** below.

Table 8. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	4.41	±8.8%
PFBS	External	3.52	±7.0%
PFHS	External	3.88	±7.8%
PFOS	External	8.26	±17%
PFOA	Internal	5.29	±11%
PFBS	Internal	6.38	±13%
PFHS	Internal	7.64	±15%
PFOS	Internal	5.92	±12%
PFOSA	Internal	5.24	±10%

3.8 Field Matrix Spikes (FMS)

Target analyte field matrix spike samples were collected for select sampling points to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions at 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOA and PFOS. The standards used for the preparation of the field matrix spiking solutions greater than 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, select sample bottles contained stable isotope surrogate recovery spikes of [¹³C₄]-PFOA and [¹³C₄]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles prior to sample collection. The [¹³C₄]-PFOA was selected to represent the C8 perfluorocarboxylic acid. The [¹³C₄]-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

The following calculation was used to generate data in section 4 of the report.

$$FMS\ Recovery = \frac{Sample\ Concentration\ of\ FMS - Average\ Concentration\ (Field\ Sample\ & Field\ Sample\ Dup)}{Spike\ Concentration} \times 100\%$$

Table 9. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
L22	FMS	0.958	1.00	1.00	0.927	1.00
P21B	FMS	4.79	5.00	5.00	4.64	5.00
5	FMS	21.0	21.0	21.0	20.9	1.00
PP08, PP09	FMS	525	25.0	525	525	25.0
D16	FMS	500	100	500	500	100
Trip Blank	Low	0.958	1.00	1.00	0.927	1.00
	High	525	25.0	525	525	25.0

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of $\pm 30\%$, demonstrate that the method is appropriate for the given matrix.

The method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. At times, the spike level exceeded the recommended upper limit of 10 times the analyte concentration. In these instances, the FMS recovery was reported and flagged as above 10 times the sample concentration.

For PFOA, PFBS, PFHS, and PFOS, when the field matrix spike level was not appropriate as compared to the endogenous sample concentration, the surrogate recovery standards were used to assess method accuracy. All field matrix spike and surrogate recovery standard recoveries met acceptance criteria with the following exception:

P21B: The field matrix spike recovery was 56.2% for PFOSA. The sample set was re-prepared and analyzed on 6/13/18 to confirm the spike recovery. As there was no improvement in the recovery from the re-analysis, the initial sample results are reported. The data uncertainty for PFOSA was expanded to $\pm 44\%$ for this location.

Table 10. Location ID: D16

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-02-001	D16; Sample	387	NA	133	NA
ISO18-14-02-001-DUP	D16; Sample Duplicate	384	NA	134	NA
ISO18-14-02-001-FMS	D16; Sample FMS	807	84.3	594	92.1
Average Concentration (ng/mL) ± %RPD		386 ng/mL ± 0.78%		134 ng/mL ± 0.75%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-02-001	D16; Sample	2470	NA	68.4	NA
ISO18-14-02-001-DUP	D16; Sample Duplicate	2490	NA	61.8	NA
ISO18-14-02-001-FMS	D16; Sample FMS	2940	NC	156	90.9
Average Concentration (ng/mL) ± %RPD		2480 ng/mL ± 0.81%		65.1 ng/mL ± 10%	

NA = Not Applicable

Table 11. Location ID: L22

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-02-008	L22; Sample	0.270	NA	0.185	NA
ISO18-14-02-008-DUP	L22; Sample Duplicate	0.252	NA	0.173	NA
ISO18-14-02-008-FMS	L22; Sample FMS	1.26	104	1.22	104
Average Concentration (ng/mL) ± %RPD		0.261 ng/mL ± 6.9%		0.179 ng/mL ± 6.7%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-02-008	L22; Sample	4.44	NA	0.0668	NA
ISO18-14-02-008-DUP	L22; Sample Duplicate	3.96	NA	0.0652	NA
ISO18-14-02-008-FMS	L22; Sample FMS	5.34	NC	1.09	102 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		4.20 ng/mL ± 11%		0.0660 ng/mL ± 2.4%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) FMS concentration greater than 10 times the sample concentration.

Table 12. Location ID: PP08

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-02-021	PP08; Sample	536	NA	42.2	NA	260	NA
ISO18-14-02-021-DUP	PP08; Sample Duplicate	556	NA	44.7	NA	271	NA
ISO18-14-02-021-FMS	PP08; Sample FMS	1050	96.0	71.7	113	796	101
Average Concentration (ng/mL) ± %RPD		546 ng/mL ± 3.7%		43.5 ng/mL ± 5.8%		266 ng/mL ± 4.1%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-02-021	PP08; Sample	5350	NA	52.2	NA
ISO18-14-02-021-DUP	PP08; Sample Duplicate	5690	NA	51.0	NA
ISO18-14-02-021-FMS	PP08; Sample FMS	5840	NC	65.4	NC
Average Concentration (ng/mL) ± %RPD		5520 ng/mL ± 6.2%		51.6 ng/mL ± 2.3%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 13. Location ID: PP09

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-02-022	PP09; Sample	467	NA	17.7	NA	316	NA
ISO18-14-02-022-DUP	PP09; Sample Duplicate	482	NA	18.3	NA	322	NA
ISO18-14-02-022-FMS	PP09; Sample FMS	973	95.0	43.6	102	850	101
Average Concentration (ng/mL) ± %RPD		475 ng/mL ± 3.2%		18.0 ng/mL ± 3.3%		319 ng/mL ± 1.9%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-02-022	PP09; Sample	3100	NA	20.6	NA
ISO18-14-02-022-DUP	PP09; Sample Duplicate	3140	NA	21.2	NA
ISO18-14-02-022-FMS	PP09; Sample FMS	3610	NC	41.6	82.8
Average Concentration (ng/mL) ± %RPD		3120 ng/mL ± 1.3%		20.9 ng/mL ± 2.9%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 14. Location ID: 5

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-02-026	5; Sample	20.6	NA	18.8	NA
ISO18-14-02-026-DUP	5; Sample Duplicate	22.0	NA	19.3	NA
ISO18-14-02-026-FMS	5; Sample FMS	41.4	95.7	40.0	99.8
Average Concentration (ng/mL) ± %RPD		21.3 ng/mL ± 6.6%		19.1 ng/mL ± 2.6%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-02-026	5; Sample	42.4	NA	0.183	NA
ISO18-14-02-026-DUP	5; Sample Duplicate	43.6	NA	0.179	NA
ISO18-14-02-026-FMS	5; Sample FMS	61.4	NC	1.17	98.9
Average Concentration (ng/mL) ± %RPD		43.0 ng/mL ± 2.8%		0.181 ng/mL ± 2.2%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 15. Location ID: P21B

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-02-022	PP09; Sample	9840	NA	7730	NA	11700	NA
ISO18-14-02-022-DUP	PP09; Sample Duplicate	10500	NA	7980	NA	12000	NA
ISO18-14-02-022-FMS	PP09; Sample FMS	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾	NA ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		10200 ng/mL ± 6.5%		7860 ng/mL ± 3.2%		11900 ng/mL ± 2.5%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-02-029	P21B; Sample	62000	NA	7.38	NA
ISO18-14-02-029-DUP	P21B; Sample Duplicate	62400	NA	6.76	NA
ISO18-14-02-029-FMS	P21B; Sample FMS	NA ⁽¹⁾	NA ⁽¹⁾	9.88	56.2 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		62200 ng/mL ± 0.64%		7.07 ng/mL ± 8.8% ⁽²⁾	

NA = Not Applicable

(1) FMS not analyzed for analyte due to inappropriate spike level.

(2) FMS recovery does not meet acceptance criteria of 100 ± 30%.

(3) Data uncertainty expanded to ±44%.

Table 16. Location ID: Travel Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-02-030	Travel Blank	<0.0240	NA	<0.100	NA	<0.0250	NA
ISO18-14-02-030-FMS-LOW	Travel Blank FMS Low	0.958	100	0.926	92.6	1.05	105
ISO18-14-02-030-FMS-HIGH	Travel Blank FMS High	515	98.1	26.8	107	544	104

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-02-030	Travel Blank	<0.0464	NA	<0.0250	NA
ISO18-14-02-030-FMS-LOW	Travel Blank FMS Low	0.998	108	1.01	101
ISO18-14-02-030-FMS-HIGH	Travel Blank FMS High	509	97.0	25.6	102

NA = Not Applicable

Table 17. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO18-14-02-001	D16; Sample	113 ⁽²⁾	116 ⁽²⁾	NA
ISO18-14-02-001-DUP	D16; Sample Duplicate	115 ⁽²⁾	120 ⁽²⁾	NA
ISO18-14-02-001-FMS	D16; Sample FMS	116 ⁽²⁾	115 ⁽²⁾	NA
ISO18-14-02-002	P121; Sample	101	94.2	NA
ISO18-14-02-002-DUP	P121; Sample Duplicate	97.0	98.6	NA
ISO18-14-02-003	3M vijver; Sample	104	98.6	NA
ISO18-14-02-003-DUP	3M vijver; Sample Duplicate	104	98.0	NA
ISO18-14-02-005	Blokkersdijkvijver standaard; Sample	106	99.7	NA
ISO18-14-02-005-DUP	Blokkersdijkvijver standaard; Sample Duplicate	107	103	NA
ISO18-14-02-006	P321; Sample	104 ⁽²⁾	110 ⁽²⁾	NA
ISO18-14-02-006-DUP	P321; Sample Duplicate	111 ⁽²⁾	115 ⁽²⁾	NA
ISO18-14-02-007	L21; Sample	96.6	97.6	NA
ISO18-14-02-007-DUP	L21; Sample Duplicate	100	94.9	NA
ISO18-14-02-008	L22; Sample	91.4	94.9	NA
ISO18-14-02-008-DUP	L22; Sample Duplicate	99.8	95.9	NA
ISO18-14-02-008-FMS	L22; Sample FMS	105	98.0	NA
ISO18-14-02-009	L31; Sample	99.4	98.8	NA
ISO18-14-02-009-DUP	L31; Sample Duplicate	108	97.8	NA
ISO18-14-02-010	L4; Sample	98.4	99.1	NA
ISO18-14-02-010-DUP	L4; Sample Duplicate	94.0	95.9	NA
ISO18-14-02-011	P114bis; Sample	91.2	90.9	NA
ISO18-14-02-011-DUP	P114bis; Sample Duplicate	102	106	NA
ISO18-14-02-012	P115; Sample	107	94.5	NA
ISO18-14-02-012-DUP	P115; Sample Duplicate	109	98.6	NA
ISO18-14-02-013	P116; Sample	89.0	98.8	NA
ISO18-14-02-013-DUP	P116; Sample Duplicate	100	92.6	NA
ISO18-14-02-014	Effluent WWTP; Sample	101	101	NA
ISO18-14-02-014-DUP	Effluent WWTP; Sample Duplicate	109	95.5	NA
ISO18-14-02-015	PP01; Sample	107 ⁽²⁾	110 ⁽²⁾	NA
ISO18-14-02-015-DUP	PP01; Sample Duplicate	112 ⁽²⁾	114 ⁽²⁾	NA
ISO18-14-02-016	PP02; Sample	117 ⁽²⁾	118 ⁽²⁾	NA
ISO18-14-02-016-DUP	PP02; Sample Duplicate	114 ⁽²⁾	114 ⁽²⁾	NA
ISO18-14-02-017	PP04; Sample	118 ⁽²⁾	118 ⁽²⁾	NA
ISO18-14-02-017-DUP	PP04; Sample Duplicate	118 ⁽²⁾	118 ⁽²⁾	NA
ISO18-14-02-018	PP05; Sample	115 ⁽²⁾	119 ⁽²⁾	117 ⁽²⁾
ISO18-14-02-018-DUP	PP05; Sample Duplicate	109 ⁽²⁾	114 ⁽²⁾	116 ⁽²⁾
ISO18-14-02-019	PP06; Sample	119 ⁽²⁾	119 ⁽²⁾	NA
ISO18-14-02-019-DUP	PP06; Sample Duplicate	120 ⁽²⁾	119 ⁽²⁾	NA
ISO18-14-02-020	PP07; Sample	118 ⁽²⁾	119 ⁽²⁾	NA
ISO18-14-02-020-DUP	PP07; Sample Duplicate	117 ⁽²⁾	118 ⁽²⁾	NA

NA = Not Applicable

(1) The surrogate recovery standards were added to the sample bottle prior to the sampling event unless noted otherwise.

(2) The surrogate recovery standards were added to the samples during sample preparation.

Table 17 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO18-14-02-021	PP08; Sample	116 ⁽²⁾	117 ⁽²⁾	NA
ISO18-14-02-021-DUP	PP08; Sample Duplicate	115 ⁽²⁾	119 ⁽²⁾	NA
ISO18-14-02-021-FMS	PP08; Sample FMS	118 ⁽²⁾	118 ⁽²⁾	NA
ISO18-14-02-022	PP09; Sample	117 ⁽²⁾	118 ⁽²⁾	NA
ISO18-14-02-022-DUP	PP09; Sample Duplicate	114 ⁽²⁾	112 ⁽²⁾	NA
ISO18-14-02-022-FMS	PP09; Sample FMS	109 ⁽²⁾	112 ⁽²⁾	NA
ISO18-14-02-023	PP10; Sample	114 ⁽²⁾	115 ⁽²⁾	NA
ISO18-14-02-023-DUP	PP10; Sample Duplicate	110 ⁽²⁾	117 ⁽²⁾	NA
ISO18-14-02-024	12; Sample	98.2	97.8	117 ⁽²⁾
ISO18-14-02-024-DUP	12; Sample Duplicate	96.2	94.0	114 ⁽²⁾
ISO18-14-02-025	13; Sample	97.4	97.6	122 ⁽²⁾
ISO18-14-02-025-DUP	13; Sample Duplicate	101	98.2	123 ⁽²⁾
ISO18-14-02-026	5; Sample	96.0	98.6	NA
ISO18-14-02-026-DUP	5; Sample Duplicate	110	97.0	NA
ISO18-14-02-026-FMS	5; Sample FMS	96.2	94.0	NA
ISO18-14-02-027	Bemalingstation; Sample	99.8	96.1	NA
ISO18-14-02-027-DUP	Bemalingstation; Sample Duplicate	99.8	100	NA
ISO18-14-02-028	Collector put; Sample	98.4	94.9	128 ⁽²⁾
ISO18-14-02-028-DUP	Collector put; Sample Duplicate	97.2	87.7	118 ⁽²⁾
ISO18-14-02-029	P21B; Sample	111 ⁽²⁾	111 ⁽²⁾	113 ⁽²⁾
ISO18-14-02-029-DUP	P21B; Sample Duplicate	112 ⁽²⁾	116 ⁽²⁾	114 ⁽²⁾
ISO18-14-02-030	Travel Blank	95.6	93.0	NA
ISO18-14-02-030-FMS-LOW	Travel Blank FMS Low	90.8	99.9	NA
ISO18-14-02-030-FMS-HIGH	Travel Blank FMS High	117	120	NA

NA = Not Applicable

(1) The surrogate recovery standards were added to the sample bottle prior to the sampling event unless noted otherwise.

(2) The surrogate recovery standards were added to the samples during sample preparation.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 10-17 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M EHS Laboratory standard operating procedures.

7 Attachments

Chain of Custody Form

8 Signatures

Digitally signed by Chelsie J. Grochow
DN: c=US, st=MN, l=St. Paul, ou=EHS Laboratory,
o=3M, cn=Chelsie J. Grochow
Reason: I am the author of this document
Date: 2018.07.05 09:25:07 -05'00'

Chelsie Grochow, 3M Report Author

Susan T. Wolf
c=US, st=MN, l=St. Paul, ou=EHS Laboratory,
o=3M, cn=Susan T. Wolf
I have reviewed this document
2018.07.02 13:15:58 -05'00'

Susan T. Wolf, 3M Principal Analytical Investigator

Digitally signed by Brian T. Mader
DN: c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory -
authenticated by LRA, o=3M, cn=Brian T. Mader
Reason: I have reviewed this document
Date: 2018.07.02 15:36:33 -05'00'

Brian T. Mader, Ph.D., 3M EHS Laboratory Manager

The 3M EHS Laboratory's Quality Assurance Unit has audited the data and report for this project.

Digitally signed by Daryl K. Peterson
DN: c=US, st=MN, l=St. Paul, ou=EHS Laboratory,
o=3M, cn=Daryl K. Peterson
Reason: I have reviewed this document
Date: 2018.07.03 07:02:18 -05'00'

Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M EHS Laboratory.

3M EHS LABORATORY
Chain-of-Custody

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St. Paul, MN 55144

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Fax: (651) 733-1111

Project: ISO18-14-02

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)

Department: 832202 Site Source: 01WJWT10

Project Number:

Date Created: 4/9/2018

Project Description: 3M Antwerp Water Sampling for PFCs; April 2018

Comments:

Completion Date:
Project Lead: Susan T. Wolf
Phone Number:
Email Address: [REDACTED]

GW = ground water

SW = surface water

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO18-14-02-001	D16; Sample 11	3/15/18	GW	/
ISO18-14-02-001-DUP	D16; Sample Duplicate 11	3/15/18	GW	/
ISO18-14-02-001-FMS	D16; Sample FMS 11	3/15/18	GW	/
ISO18-14-02-002	P121; Sample 11	4/15/18	GW	/
ISO18-14-02-002-DUP	P121; Sample Duplicate 11	4/15/18	GW	/
ISO18-14-02-003	3M vijver; Sample 11	3/15/18	SW	/
ISO18-14-02-003-DUP	3M vijver; Sample Duplicate 11	3/15/18	SW	/
ISO18-14-02-004	Blokkersdijkvijver Noord; Sample 11	6/18/18	SW	NO SAMPLE
ISO18-14-02-004-DUP	Blokkersdijkvijver Noord; Sample Duplicate 11	7/18/18	SW	NO SAMPLE
ISO18-14-02-005	Blokkersdijkvijver standaard; Sample 11	3/15/18	SW	/
ISO18-14-02-005-DUP	Blokkersdijkvijver standaard; Sample Duplicate 11	3/15/18	SW	/
ISO18-14-02-006	P321; Sample 11	3/15/18	GW	/
ISO18-14-02-006-DUP	P321; Sample Duplicate 11	3/15/18	GW	/
ISO18-14-02-007	L21; Sample 11	4/15/18	GW	/

Sample Condition Upon Receipt: (1) Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print):	Collector's signature:						
Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	J. M. Mandel	8/15/18	/	FEDEX	JOSEPH TILLMAN	5-11-18	1300

(1) "ACCEPTABLE" AND "TEMPERATURE" WERE MISTAKENLY FILLED OUT BY COLLECTOR.
ALL SAMPLES ARE ACCEPTABLE, AND ARRIVED AT ROOM TEMPERATURE
JT 5-11-18

Page 1 of 5

Page 26 of 30

3M EHS LABORATORY
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St. Paul, MN 55144

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Project: ISO18-14-02 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 4/9/2018

Project Description: 3M Antwerp Water Sampling for PFCs; April 2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

GW = groundwater
WW = waste water

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO18-14-02-007-DUP	L21; Sample Duplicate //	4/15/18	GW	/
ISO18-14-02-008	L22; Sample //	4/15/18	GW	/
ISO18-14-02-008-DUP	L22; Sample Duplicate //	4/15/18	GW	/
ISO18-14-02-008-FMS	L22; Sample FMS //	4/15/18	GW	/
ISO18-14-02-009	L31; Sample //	4/15/18	GW	/
ISO18-14-02-009-DUP	L31; Sample Duplicate //	4/15/18	GW	/
ISO18-14-02-010	L4; Sample //	4/15/18	GW	/
ISO18-14-02-010-DUP	L4; Sample Duplicate //	4/15/18	GW	/
ISO18-14-02-011	P114bis; Sample //	4/15/18	GW	/
ISO18-14-02-011-DUP	P114bis; Sample Duplicate //	4/15/18	GW	/
ISO18-14-02-012	P115; Sample //	4/15/18	GW	/
ISO18-14-02-012-DUP	P115; Sample Duplicate //	4/15/18	GW	/
ISO18-14-02-013	P116; Sample //	4/15/18	GW	/
ISO18-14-02-013-DUP	P116; Sample Duplicate //	4/15/18	GW	/
ISO18-14-02-014	Effluent WWTP; Sample //	3/15/18	WW	/
ISO18-14-02-014-DUP	Effluent WWTP; Sample Duplicate //	3/15/18	WW	/
ISO18-14-02-015	PP01; Sample //	3/15/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Tine Mandson (CMA) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TINA	8/15/18	/	FEDEX	JOSEPH TILLMAN	5-11-18	1300

3M EHS LABORATORY
Chain-of-Custody

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Project: ISO18-14-02 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
 Department: 832202 Site Source: 01WJWT10

Project Number:

Date Created: 4/9/2018

Project Description: 3M Antwerp Water Sampling for PFCs; April 2018

Completion Date:
 Project Lead: Susan T. Wolf
 Phone Number:
 Email Address: [REDACTED]

Gw = groundwater

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO18-14-02-015-DUP	PP01; Sample Duplicate //	3/5/18	GW	/
ISO18-14-02-016	PP02; Sample //	3/5/18	GW	/
ISO18-14-02-016-DUP	PP02; Sample Duplicate //	3/5/18	GW	/
ISO18-14-02-017	PP04; Sample //	3/5/18	GW	/
ISO18-14-02-017-DUP	PP04; Sample Duplicate //	3/5/18	GW	/
ISO18-14-02-018	PP05; Sample //	3/5/18	GW	BLACK
ISO18-14-02-018-DUP	PP05; Sample Duplicate //	3/5/18	GW	BLACK
ISO18-14-02-019	PP06; Sample //	3/5/18	GW	/
ISO18-14-02-019-DUP	PP06; Sample Duplicate //	3/5/18	GW	/
ISO18-14-02-020	PP07; Sample //	3/5/18	GW	/
ISO18-14-02-020-DUP	PP07; Sample Duplicate //	3/5/18	GW	/
ISO18-14-02-021	PP08; Sample //	3/5/18	GW	/
ISO18-14-02-021-DUP	PP08; Sample Duplicate //	3/5/18	GW	/
ISO18-14-02-021-FMS	PP08; Sample FMS //	3/5/18	GW	/
ISO18-14-02-022	PP09; Sample //	3/5/18	GW	/
ISO18-14-02-022-DUP	PP09; Sample Duplicate //	3/5/18	GW	/
ISO18-14-02-022-FMS	PP09; Sample FMS //	3/5/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 17 Deg C Received on Ice Other:

Collected by (print): Tina Mandana (TMA)

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	5/8/18	/	FEDEX	JOSEPH TILMAN	5-11-18	1300

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St. Paul, MN 55144

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Alt. Ph: [REDACTED]
Fax: [REDACTED]

Project: ISO18-14-02 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 4/9/2018

Project Description: 3M Antwerp Water Sampling for PFCs; April 2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

GW = ground water
SW = surface water
WW = waste water

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO18-14-02-023	PP10; Sample 11	3/15/18	GW	/
ISO18-14-02-023-DUP	PP10; Sample Duplicate 11	3/15/18	GW	/
ISO18-14-02-024	12; Sample 11	3/15/18	SW	/
ISO18-14-02-024-DUP	12; Sample Duplicate 11	3/15/18	SW	/
ISO18-14-02-025	13; Sample 11	3/15/18	SW	/
ISO18-14-02-025-DUP	13; Sample Duplicate 11	3/15/18	SW	/
ISO18-14-02-026	5; Sample 11	3/15/18	SW	/
ISO18-14-02-026-DUP	5; Sample Duplicate 11	3/15/18	SW	/
ISO18-14-02-026-FMS	5; Sample FMS 11	3/15/18	SW	/
ISO18-14-02-027	Bemalingstation; Sample 11	3/15/18	SW	/
ISO18-14-02-027-DUP	Bemalingstation; Sample Duplicate 11	3/15/18	GW	/
ISO18-14-02-028	Collector put; Sample 11	3/15/18	WW	/
ISO18-14-02-028-DUP	Collector put; Sample Duplicate 11	3/15/18	WW	/
ISO18-14-02-029	P21B; Sample 11	3/15/18	GW	BLACK
ISO18-14-02-029-DUP	P21B; Sample Duplicate 11	3/15/18	GW	BLACK
ISO18-14-02-029-FMS	P21B; Sample FMS 11	3/15/18	GW	BLACK
ISO18-14-02-030	Travel Blank 1	CJG 4/11/18	/	*

* Travel Blank samples prepared by the 3M EHS Laboratory with the bottle order.

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print):	Tine Mandonx (TMA)	Collector's signature:					
Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	5/18/18	/	FEDEX	JOSEPH TILMAN	5-11-18	1300

3M EHS LABORATORY
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Project: ISO18-14-02 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 4/9/2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number:
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs; April 2018

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO18-14-02-030-FMS-HIGH	Travel Blank FMS High	CJG 4/11/18	/	*
ISO18-14-02-030-FMS-LOW	Travel Blank FMS Low	CJG 4/11/18	/	*

* Travel Blank samples prepared by the 3M EHS Laboratory with the bottle order.

Sample Condition Upon Receipt: ① Acceptable All items accounted for
Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Tina Mandony (TMA) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
2	TMA	5/18/18	/	FEDEX	JOSEPH TILLMAN	5-11-18	1300



3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC18-02979_002

3M Lab Request Number: E18-0387

Analysis of Total Petroleum Hydrocarbons in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: August 14th, 2018

Requester

Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000

SGS Belgium NV Institute for Applied Chromatography Haven 407 Polderdijkweg 16 B-2030 Antwerpen
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Registered office: Noorderlaan 87 B-2030 Antwerpen H.R. Antwerpen 141.810 BTW BE 404.882.750 Citibank BE87 5701 3412 5594
All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction.

At the request (Lab Request Number: E18-0387) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected between July, 2018 from 2 locations and analyzed for:

- Total Petroleum Hydrocarbons (TPH)

2. Methods – Analytical and Preparatory.**2.1. Sample collection.**

The sample was collected in a 1L glass container containing HCl as a preservative. The bottle was sent by SGS Belgium NV, Division IAC Laboratory.

2.2. Analysis of mineral oil.

The samples was analyzed according to CMA/3/R.1 and WAC/IV/B/025, the official Dutch method.

3. Results.

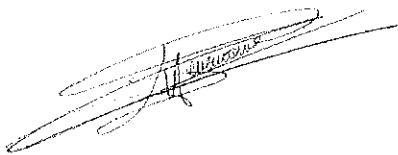
Table 1 summarizes the sample results.

Table 1. Mineral oil results.

Sample identification	Concentration (in µg/L)				
	Fraction C-10 -C-12	Fraction C-12 -C-20	Fraction C-20 -C-30	Fraction C-30 -C-40	Mineral oil
P18	81	680	450	<25	1200
P28	240	880	290	<25	1400

4. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

5. Signatures.

Sven Herremans,
Technical Manager

Date August 14th, 2017



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date August 14th, 2017

I.A.C.
A Division of SGS Belgium NV

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The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.



3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC18-02979_001

3M Lab Request Number: E18-0387

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: August 14th, 2018

Requester

Jim Kotsmith
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SGS Belgium NV

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All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction - Summary.

At the request (Lab Request Number: E18-0387) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected in July, 2018 from 30 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOA ($C_6F_{13}^{13}CF_2^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOS ($C_7F_{15}^{13}CF_2SO_3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Geert De Smet. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles.

Table 1 summarizes the sample results. All PFBS, PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
Effluent WWTp	<0.360	<0.244	0.549	<0.366	
Bemalingsstation	49.1	70.4	75.0	<0.732	
Collector Put	19.2	65.6	176	8.31	
BD24-3	24.3	28.1	855	<4.0	
BD24-4	4.75	20.4	4.22	<0.366	
D10	159	512	41.6	<2.66	
D11	56.0	129	706	33.0	
D14	1.57	2.52	0.918	<0.366	
D2	<0.360	0.317	6.76	<0.366	
D5	131	422	338	5.04	
ND7	59.3	158	21.1	21.2	
P118A	1444	1486	93.8	2.13	
P118B	2672	2394	10000	<7.99	
K3	238	9936	44.9	3767	<14.7
P21B	6586	8051	9477	39698	<28.2
P56	7.18	11.7	26.3	178	<28.2
L19	86.3	231	3171	62.1	
P262	668	1567	4678	36.1	
P263	356	1401	6389	17.7	
P340	202	499	2155	26.5	
P341	185	699	4237	48.8	
P343	274	539	4639	<14.7	
P379	15.7	56.2	591	31.9	
P380	33.4	97.9	679	46.3	
P381	124	540	936	4.70	
P382	106	484	686	34.0	
P372	32.5	53.1	1096	19.7	
PA109A	33.8	43.8	119	0.999	
PA111A	676	725	1521	5.73	

2. Methods – Analytical and Preparatory.**2.1. Sample collection.**

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
Effluent WWTp	100
Bemalingsstation	100
Collector Put	100
BD24-3	100
BD24-4	100
D10	100
D11	100
D14	100
D2	100
D5	100
ND7	100
P118A	100
P118B	100
K3	100
P21B	100
P56	100
L19	100
P262	100
P263	100
P340	100
P341	100
P343	100
P379	100
P380	100
P381	100
P382	100
P372	100
PA109A	100
PA111A	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
Effluent WWTp	1.0	0.05	Solution A	2.0	-
Bemalingsstation	1.0	0.1	Solution A	5.0	-
Collector Put	1.0	0.1	Solution A	10.0	-
BD24-3	1.0	0.1	Solution A	10.0	1.0 mL extract + 2.0 mL MeOH (*)
BD24-4	1.0	0.05	Solution A	2.0	-
D10	1.0	0.1	Solution A	10.0	1.0 mL extract + 1.0 mL MeOH (*)
D11	1.0	0.1	Solution A	10.0	1.0 mL extract + 1.0 mL MeOH (*)
D14	1.0	0.05	Solution A	2.0	-
D2	1.0	0.05	Solution A	2.0	-
D5	1.0	0.1	Solution A	10.0	1.0 mL extract + 1.0 mL MeOH (*)
ND7	1.0	0.1	Solution A	5.0	-
P118A	1.0	0.1	Solution A	10.0	1.0 mL extract + 5.0 mL MeOH (*)
P118B	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
K3	1.0	0.2	Solution A	10.0	0.5 mL extract + 10.0 mL MeOH (*)
P21B	1.0	0.2	Solution A	10.0	0.5 mL extract + 10.0 mL MeOH (*)
P56	1.0	0.1	Solution A	10.0	-
L19	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
P262	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
P263	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
P340	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
P341	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
P343	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
P379	1.0	0.1	Solution A	10.0	1.0 mL extract + 2.0 mL MeOH (*)
P380	1.0	0.1	Solution A	10.0	1.0 mL extract + 2.0 mL MeOH (*)
P381	1.0	0.1	Solution A	10.0	1.0 mL extract + 2.0 mL MeOH (*)
P382	1.0	0.1	Solution A	10.0	1.0 mL extract + 2.0 mL MeOH (*)
P372	1.0	0.1	Solution A	10.0	-
PA109A	1.0	0.1	Solution A	10.0	1.0 mL extract + 5.0 mL MeOH (*)
PA111A	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)

(*): MeOH:LCMS-water (60:40)
Solution A = 1000 ng/mL (nominal) ¹³C-PFC Surrogate Standards

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetrie C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C-PFOA	2.91 - 3.80	416.8 / 371.8	60	11	14
PFBS	2.08 - 2.50	298.93 / 79.82	40	28	45
		298.93 / 98.78	40	29	45
PFHS	2.45 - 3.05	398.86 / 79.83	40	38	51
		398.86 / 98.79	40	35	51
PFOA	2.80 - 3.65	412.9 / 218.98	40	11	14
		412.9 / 368.87	40	11	14
PFOS	2.92 - 4.20	498.78 / 79.77	50	48	56
		498.78 / 98.73	50	39	56
FOSA	4.00 - 5.60	497.8 / 77.82	125	34	44
¹³ C-PFOS	3.40 - 4.20	503.0 / 80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of $\pm 25\%$ ($\pm 30\%$ for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.0780 to 0.12 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.244 to 28.2 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within $\pm 25\%$, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by diluting 1 mL water with 10.0 mL MeOH:LCMS-water (60:40). Method blanks were prepared to evaluate the levels of background contamination in the overall dilution process (glassware, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carryover.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of LCMS-water, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCS)

One lab control spike was prepared and analyzed.

LCS was prepared by spiking known amounts of the analytes into 1.0 mL of LCMS-water to produce the desired concentration.

The spiked water sample was diluted with 10.0 mL MeOH:LCMS-water (60:40) and analyzed in the same manner as the samples.

The LCS produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 7 summarizes the LCS recovery results

Table 7. Lab Control Spike Results.

Dilution date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
2 August 2018	LCS1	80	81.8	82.9	94.4	94.7	95.1	81.9	93.1

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing PFBS, PFHS, PFOA, ^{13}C -PFOA, PFOS, FOSA and ^{13}C -PFOS (QC Lab Low and QC Lab High).

The QCs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 8 summarizes the QCs recovery results.

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
25 Jan 2018	QC Lab Low inj1	10	102	87.13	111	124	103	93.3	120
	QC Lab High inj1	100	79.4	75.8	95.7	97.6	95.9	84.0	88.5
	QC Lab Low inj2	10	99.8	90.1	116	114	105	99.9	105
	QC Lab High inj2	100	83.1	81.5	104	106	105	91.9	73.1

3.7. Equations.Recovery.

$$\text{Recovery (\%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

The following tables summarize the results for the sample locations.

Table 9. Sample Results PFBS, PFHS, PFOA, PFOS and FOSA

	PFBS ng/ml	PFHS ng/ml	PFOA ng/ml	PFOS ng/ml	FOSA ng/ml
Effluent WWTP		<0.360	<0.244	0.549	<0.366
Bemalingsstation		49.1	70.4	75.0	<0.732
Collector Put		19.2	65.6	176	8.31
BD24-3		24.3	28.1	855	<4.0
BD24-4		4.75	20.4	4.22	<0.366
D10		159	512	41.6	<2.66
D11		56.0	129	706	33.0
D14		1.57	2.52	0.918	<0.366
D2		<0.360	0.317	6.76	<0.366
D5		131	422	338	5.04
ND7		59.3	158	21.1	21.2
P118A		1444	1486	93.8	2.13
P118B		2672	2394	10000	<7.99
K3	238	9936	44.9	3767	<14.7
P21B	6586	8051	9477	39698	<28.2
P56	7.18	11.7	26.3	178	<28.2
L19		86.3	231	3171	62.1
P262		668	1567	4678	36.1
P263		356	1401	6389	17.7
P340		202	499	2155	26.5
P341		185	699	4237	48.8
P343		274	539	4639	<14.7
P379		15.7	56.2	591	31.9
P380		33.4	97.9	679	46.3
P381		124	540	936	4.70
P382		106	484	686	34.0
P372		32.5	53.1	1096	19.7
PA109A		33.8	43.8	119	0.999
PA111A		676	725	1521	5.73
Field Trip Blank	<0.866	<1.31	<0.888	<1.29	<1.33

Table 10. Sample Results ^{13}C -PFOA and ^{13}C -PFOS

	^{13}C -PFOA		^{13}C -PFOS	
	ng/ml	% Rec	ng/ml	% Rec
Effluent WWTP	70.1	122	57.4	93.3
Bemalingsstation	129	113	114	92.4
Collector Put	132	115	119	96.6
BD24-3	133	116	117	95.3
BD24-4	73.4	128	51.8	84.2
D10	124	108	116	94.5
D11	137	120	117	94.9
D14	73.0	127	52.3	84.9
D2	71.6	125	53.4	86.8
D5	129	112	118	96.0
ND7	120	104	115	93.3
P118A	126	110	108	87.7
P118B	138	120	94.7	77.0
K3	246	107	228	92.7
P21B	238	104	194	78.9
P56	138	120	114	92.7
L19	131	114	103	83.5
P262	135	117	96.1	78.0
P263	124	108	104	84.6
P340	131	114	104	84.2
P341	119	104	94.9	77.0
P343	132	115	100	81.5
P379	133	127	113	91.8
P380	131	125	111	90.2
P381	124	119	117	94.8
P382	131	125	113	92.1
P372	132	126	110	89.4
PA109A	132	115	117	95.0
PA111A	135	117	112	91.3
Field Trip Blank	134	117	133	108

5. Conclusion.

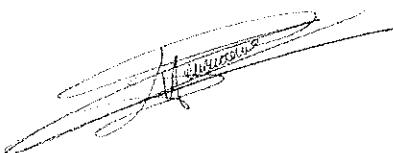
- The recovery of ¹³C-PFOA and ¹³C-PFOS fell within the method acceptance criterion of 70-130%.
- Lab control spike recoveries fell within the method acceptance criteria for all compounds, indicating that the analysis method is in control,
- QC Lab Standards recoveries fell within the method acceptance criteria for all compounds.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Elien Kemme (ERM Belgium).

8. Signatures.

Sven Herremans,
Technical Manager

Date August 14th, 2018



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date August 14th, 2018

I.A.C.
A Division of SGS Belgium NV

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The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.

Final Report

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

July 2018 Sampling

Laboratory Request Number: ISO18-14-03

Report Date – Date of Last Signature

Testing Laboratory

3M Environment, Health, and Safety
3M EHS Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Nicole Cauberghe
3M Belgium EHS Operations
3M Belgium; ZW018/0/33
Phone: [REDACTED]



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M EHS Laboratory

3M EHS Laboratory Manager: Brian T. Mader
3M Principal Analytical Investigator: Susan Wolf
Report Author: Chelsie Grochow

Analytical Report ISO18-14-03

Water Sample Analysis at 3M Antwerp, Belgium
July 2018 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M EHS Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. All samples were collected July 9-12, 2018 and returned to the 3M EHS Laboratory on July 16, 2018, at ambient temperature. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS) (select locations only), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO18-14-03.

The 3M EHS Laboratory prepared sample containers for seventy-four sampling locations. Each sample set consisted of a field sample and field sample duplicate. Eighteen locations also included a target analyte field matrix spike. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with a mass-labeled internal standard mix and select sample bottles were fortified with surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS prior to being sent to the field for sample collection. During sample collection, sample locations B3-bis and B7 were not sampled.

Samples were prepared and analyzed using method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Select internal standards were used to aid in the data quality objectives.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: 2nd Aquifer					
ISO18-14-03-001	BD24-3; Sample	34.3	34.5	1060	3.38
ISO18-14-03-001-DUP	BD24-3; Sample Dup	36.3	37.1	990	4.84
		Average	35.3	35.8	1030
		%RPD Sample/Sample Dup	5.7	7.3	4.11
ISO18-14-03-002	BD24-4; Sample	14.9	5.10	4.32	0.127
ISO18-14-03-002-DUP	BD24-4; Sample Dup	15.6	5.34	4.72	0.150
		Average	15.3 ⁽²⁾	5.22 ⁽²⁾	4.52 ⁽²⁾
		%RPD Sample/Sample Dup	4.6	4.6	0.139
ISO18-14-03-003	D09; Sample	300	3770	1410	15.0
ISO18-14-03-003-DUP	D09; Sample Dup	320	3810	1400	16.0
		Average	310	3790	1410
		%RPD Sample/Sample Dup	6.5	1.1	15.5
ISO18-14-03-004	D10; Sample	559	231	59.0	<0.250
ISO18-14-03-004-DUP	D10; Sample Dup	539	226	58.5	<0.250
		Average	549	229	58.8
		%RPD Sample/Sample Dup	3.6	2.2	<0.250
ISO18-14-03-005	D11; Sample	135	87.3	838	61.4
ISO18-14-03-005-DUP	D11; Sample Dup	126	79.8	793	60.8
		Average	131	83.6	816
		%RPD Sample/Sample Dup	6.9	9.0	61.1
ISO18-14-03-006	D14; Sample	2.02	1.63	1.13	0.0880
ISO18-14-03-006-DUP	D14; Sample Dup	1.83	1.56	1.03	0.0818
		Average	1.93 ⁽²⁾	1.60 ⁽²⁾	1.08 ⁽²⁾
		%RPD Sample/Sample Dup	9.9	4.4	0.0849
ISO18-14-03-007	D16; Sample	678	297	3940	15.8
ISO18-14-03-007-DUP	D16; Sample Dup	644	286	3900	16.2
		Average	661	292	3920
		%RPD Sample/Sample Dup	5.1	3.8	16.0
ISO18-14-03-008	D17; Sample	495	169	280	1.61
ISO18-14-03-008-DUP	D17; Sample Dup	485	172	266	1.38
		Average	490	171	273
		%RPD Sample/Sample Dup	2.0	1.8	1.50
ISO18-14-03-009	D18; Sample	225	153	53.9	2.92
ISO18-14-03-009-DUP	D18; Sample Dup	215	151	50.4	2.80
		Average	220	152	52.2
		%RPD Sample/Sample Dup	4.5	1.3	2.86
					4.2

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 15%, PFBS \pm 12%, PFHS \pm 13%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 14%, PFBS \pm 11%, PFHS \pm 13%, PFOS \pm 11%, and PFOSA \pm 20%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) The analytical data uncertainty has been expanded for location P119C for PFOSA to \pm 31% based on field matrix spike recovery.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: 2nd Aquifer					
ISO18-14-03-010	D2; Sample	0.344	0.176	7.84	0.394
ISO18-14-03-010-DUP	D2; Sample Dup	0.388	0.159	8.14	0.454
		Average %RPD Sample/Sample Dup	0.366 ⁽²⁾ 12	0.168 ⁽²⁾ 10	7.99 ⁽²⁾ 3.8
ISO18-14-03-011	D5; Sample	434	177	436	8.90
ISO18-14-03-011-DUP	D5; Sample Dup	431	170	416	8.64
		Average %RPD Sample/Sample Dup	433 0.69	174 4.0	426 4.7
ISO18-14-03-012	ND7; Sample	177	86.5	26.6	39.4
ISO18-14-03-012-DUP	ND7; Sample Dup	174	82.9	26.8	36.0
		Average %RPD Sample/Sample Dup	176 1.7	84.7 4.3	26.7 0.75
ISO18-14-03-013	P118A; Sample	1390	2040	114	1.31
ISO18-14-03-013-DUP	P118A; Sample Dup	1440	2040	113	1.16
		Average %RPD Sample/Sample Dup	1420 3.5	2040 0.0	114 0.88
ISO18-14-03-014	P118B; Sample	2110	3420	12000	8.00
ISO18-14-03-014-DUP	P118B; Sample Dup	2090	3370	11900	7.20
		Average %RPD Sample/Sample Dup	2100 0.95	3400 1.5	12000 0.84
ISO18-14-03-015	P119A; Sample	63.6	19.0	19.9	2.18
ISO18-14-03-015-DUP	P119A; Sample Dup	60.7	18.1	19.8	2.20
		Average %RPD Sample/Sample Dup	62.2 4.7	18.6 4.9	19.9 0.50
ISO18-14-03-016	P119B; Sample	874	425	177	2.10
ISO18-14-03-016-DUP	P119B; Sample Dup	879	423	175	2.06
		Average %RPD Sample/Sample Dup	877 0.57	424 0.47	176 1.1
ISO18-14-03-017	P121; Sample	0.302	0.118	0.982	0.0814
ISO18-14-03-017-DUP	P121; Sample Dup	0.318	0.173	1.03	0.0774
		Average %RPD Sample/Sample Dup	0.310 ⁽²⁾ 5.2	0.146 ⁽²⁾ 38 ⁽³⁾	1.01 ⁽²⁾ 4.8
ISO18-14-03-018	P321; Sample	2330	658	4710	1.89
ISO18-14-03-018-DUP	P321; Sample Dup	2400	683	4930	2.08
		Average %RPD Sample/Sample Dup	2370 3.0	671 3.7	4820 4.6

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 15%, PFBS \pm 12%, PFHS \pm 13%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 14%, PFBS \pm 11%, PFHS \pm 13%, PFOS \pm 11%, and PFOSA \pm 20%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) The analytical data uncertainty has been expanded for location P119C for PFOSA to \pm 31% based on field matrix spike recovery.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Blokkersdijk Nature Reserve					
ISO18-14-03-019	3M vijver; Sample	1.77	0.784	12.7	0.0856
ISO18-14-03-019-DUP	3M vijver; Sample Dup	1.70	0.766	12.4	0.0808
Average %RPD Sample/Sample Dup		1.74 ⁽²⁾	0.775 ⁽²⁾	12.6 ⁽²⁾	0.0832
%RPD Sample/Sample Dup		4.0	2.3	2.4	5.8
ISO18-14-03-020	Blokkersdijkvijver Noord; Sample	0.934	0.568	0.766	<0.250
ISO18-14-03-020-DUP	Blokkersdijkvijver Noord; Sample Dup	0.818	0.504	0.702	<0.250
Average %RPD Sample/Sample Dup		0.876 ⁽²⁾	0.536 ⁽²⁾	0.734 ⁽²⁾	<0.250
%RPD Sample/Sample Dup		13	12	8.7	NA
ISO18-14-03-021	Blokkersdijkvijver standard; Sample	1.28	0.698	5.82	0.168
ISO18-14-03-021-DUP	Blokkersdijkvijver standard; Sample Dup	1.34	0.718	5.92	0.169
Average %RPD Sample/Sample Dup		1.31 ⁽²⁾	0.708 ⁽²⁾	5.87 ⁽²⁾	0.169
%RPD Sample/Sample Dup		4.6	2.8	1.7	0.59
ISO18-14-03-022	L21; Sample	0.288	0.121	4.92	0.118
ISO18-14-03-022-DUP	L21; Sample Dup	0.276	0.157	5.10	0.0972
Average %RPD Sample/Sample Dup		0.282 ⁽²⁾	0.139 ⁽²⁾	5.01 ⁽²⁾	0.108
%RPD Sample/Sample Dup		4.3	26 ⁽³⁾	3.6	19
ISO18-14-03-023	L22; Sample	0.184	0.148	2.86	0.0262
ISO18-14-03-023-DUP	L22; Sample Dup	0.204	0.170	2.76	0.0258
Average %RPD Sample/Sample Dup		0.194 ⁽²⁾	0.159 ⁽²⁾	2.81 ⁽²⁾	0.0260
%RPD Sample/Sample Dup		10	14	3.6	1.5
ISO18-14-03-024	L31; Sample	0.648	0.602	11.1	0.338
ISO18-14-03-024-DUP	L31; Sample Dup	0.640	0.590	10.5	0.428
Average %RPD Sample/Sample Dup		0.644 ⁽²⁾	0.596 ⁽²⁾	10.8 ⁽²⁾	0.383
%RPD Sample/Sample Dup		1.2	2.0	5.6	23 ⁽³⁾
ISO18-14-03-025	L4; Sample	2.28	2.08	29.0	0.684
ISO18-14-03-025-DUP	L4; Sample Dup	2.10	1.94	27.4	0.724
Average %RPD Sample/Sample Dup		2.19 ⁽²⁾	2.01 ⁽²⁾	28.2 ⁽²⁾	0.704
%RPD Sample/Sample Dup		8.2	7.0	5.7	5.7
ISO18-14-03-026	P114bis; Sample	1.35	1.43	12.0	0.0550
ISO18-14-03-026-DUP	P114bis; Sample Dup	1.30	1.38	11.8	0.0440
Average %RPD Sample/Sample Dup		1.33 ⁽²⁾	1.41 ⁽²⁾	11.9 ⁽²⁾	0.0495
%RPD Sample/Sample Dup		3.8	3.6	1.7	22 ⁽³⁾
ISO18-14-03-027	P115; Sample	3.20	1.74	0.568	<0.0250
ISO18-14-03-027-DUP	P115; Sample Dup	3.28	1.76	0.654	<0.0250
Average %RPD Sample/Sample Dup		3.24 ⁽²⁾	1.75 ⁽²⁾	0.611 ⁽²⁾	<0.0250
%RPD Sample/Sample Dup		2.5	1.1	14	NA

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 15%, PFBS \pm 12%, PFHS \pm 13%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 14%, PFBS \pm 11%, PFHS \pm 13%, PFOS \pm 11%, and PFOSA \pm 20%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) The analytical data uncertainty has been expanded for location P119C for PFOSA to \pm 31% based on field matrix spike recovery.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFBS	PFHS	PFOSA ⁽²⁾
Zone: Blokkersdijk Nature Reserve					
ISO18-14-03-028	P116; Sample	0.960	0.528	8.00	0.105
ISO18-14-03-028-DUP	P116; Sample Dup	0.942	0.532	7.70	0.105
		Average	0.951 ⁽²⁾	0.530 ⁽²⁾	7.85 ⁽²⁾
		%RPD Sample/Sample Dup	1.9	0.75	3.8
Zone: Effluent WWTP					
ISO18-14-03-029	Effluent WWTP; Sample	0.0940	0.109	0.704	0.0606
ISO18-14-03-029-DUP	Effluent WWTP; Sample Dup	0.0626	0.0736	0.640	0.0612
		Average	0.0783 ⁽²⁾	0.0913 ⁽²⁾	0.672 ⁽²⁾
		%RPD Sample/Sample Dup	40 ⁽³⁾	39 ⁽³⁾	9.5

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
ISO18-14-03-030	PP01; Sample	50.3	58.1	8320	1800	60.8
ISO18-14-03-030-DUP	PP01; Sample Dup	53.4	60.2	8280	1770	51.6
		Average	51.9	59.2	8300	1790
		%RPD Sample/Sample Dup	6.0	3.6	0.48	1.7
ISO18-14-03-031	PP02; Sample	653	52.7	3630	20000	29.4
ISO18-14-03-031-DUP	PP02; Sample Dup	628	53.7	3630	19100	26.6
		Average	641	53.2	3630	19600
		%RPD Sample/Sample Dup	3.9	1.9	0.0	4.6
ISO18-14-03-032	PP04; Sample	427	1480	234	5640	66.4
ISO18-14-03-032-DUP	PP04; Sample Dup	419	1420	219	5490	63.4
		Average	423	1450	227	5570
		%RPD Sample/Sample Dup	1.9	4.1	6.6	2.7
ISO18-14-03-033	PP05; Sample	587	5850	2600	176	17.2
ISO18-14-03-033-DUP	PP05; Sample Dup	602	6070	2650	158	17.2
		Average	595	5960	2630	167
		%RPD Sample/Sample Dup	2.5	3.7	1.9	11
ISO18-14-03-034	PP06; Sample	268	18.5	76.8	1280	58.6
ISO18-14-03-034-DUP	PP06; Sample Dup	270	19.1	77.3	1300	61.0
		Average	269	18.8	77.1	1290
		%RPD Sample/Sample Dup	0.74	3.2	0.65	1.6
ISO18-14-03-035	PP07; Sample	308	223	136	2670	72.0
ISO18-14-03-035-DUP	PP07; Sample Dup	288	192	111	2610	76.4
		Average	298	208	124	2640
		%RPD Sample/Sample Dup	6.7	15	20	2.3
						5.9

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 15%, PFBS \pm 12%, PFHS \pm 13%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 14%, PFBS \pm 11%, PFHS \pm 13%, PFOS \pm 11%, and PFOSA \pm 20%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) The analytical data uncertainty has been expanded for location P119C for PFOSA to \pm 31% based on field matrix spike recovery.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
ISO18-14-03-036	PP08; Sample	510	90.7	654	5480	64.8
ISO18-14-03-036-DUP	PP08; Sample Dup	494	87.2	648	5340	65.0
	Average	502	89.0	651	5410	64.9
	%RPD Sample/Sample Dup	3.2	3.9	0.92	2.6	0.31
ISO18-14-03-037	PP09; Sample	662	23.6	341	2740	10.4
ISO18-14-03-037-DUP	PP09; Sample Dup	654	23.7	331	2630	9.68
	Average	658	23.7	336	2690	10.0
	%RPD Sample/Sample Dup	1.2	0.42	3.0	4.1	7.2
ISO18-14-03-038	PP10; Sample	542	19.4	210	3380	15.9
ISO18-14-03-038-DUP	PP10; Sample Dup	536	18.8	213	3340	16.4
	Average	539	19.1	212	3360	16.2
	%RPD Sample/Sample Dup	1.1	3.1	1.4	1.2	3.1

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Palingbeek & Tophatgracht					
ISO18-14-03-039	12; Sample	119	34.8	446	0.492
ISO18-14-03-039-DUP	12; Sample Dup	122	34.4	452	0.818
	Average	121	34.6	449	0.655
	%RPD Sample/Sample Dup	2.5	1.2	1.3	50 ⁽³⁾
ISO18-14-03-040	13; Sample	109	36.8	367	0.494
ISO18-14-03-040-DUP	13; Sample Dup	106	36.3	371	0.306
	Average	108	36.6	369	0.400
	%RPD Sample/Sample Dup	2.8	1.4	1.1	47 ⁽³⁾
ISO18-14-03-041	5; Sample	89.0	81.8	126	0.284
ISO18-14-03-041-DUP	5; Sample Dup	89.6	81.8	123	0.286
	Average	89.3 ⁽²⁾	81.8 ⁽²⁾	125 ⁽²⁾	0.285
	%RPD Sample/Sample Dup	0.67	0.0	2.4	0.70
ISO18-14-03-042	Bemalingsstation; Sample	69.2	61.6	93.8	0.224
ISO18-14-03-042-DUP	Bemalingsstation; Sample Dup	65.6	62.4	95.6	0.228
	Average	67.4 ⁽²⁾	62.0 ⁽²⁾	94.7 ⁽²⁾	0.226
	%RPD Sample/Sample Dup	5.3	1.3	1.9	1.8
Zone: Sewer					
ISO18-14-03-043	Collector put; Sample	59.0	22.6	204	14.1
ISO18-14-03-043-DUP	Collector put; Sample Dup	61.6	22.6	202	14.6
	Average	60.3 ⁽²⁾	22.6 ⁽²⁾	203	14.4
	%RPD Sample/Sample Dup	4.3	0.0	0.99	3.5

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 15%, PFBS \pm 12%, PFHS \pm 13%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 14%, PFBS \pm 11%, PFHS \pm 13%, PFOS \pm 11%, and PFOSA \pm 20%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) The analytical data uncertainty has been expanded for location P119C for PFOSA to \pm 31% based on field matrix spike recovery.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - Building 16						
ISO18-14-03-044	K3; Sample	79.8	297	17000	4800	13.1
ISO18-14-03-044-DUP	K3; Sample Dup	76.3	290	17100	4860	14.5
Average		78.1	294	17100	4830	13.8
%RPD Sample/Sample Dup		4.5	2.4	0.59	1.2	10
ISO18-14-03-045	P27; Sample	231	198	87.2	1780	84.4
ISO18-14-03-045-DUP	P27; Sample Dup	231	192	83.6	2020	108
Average		231	195	85.4	1900	96.2
%RPD Sample/Sample Dup		0.0	3.1	4.2	13	25 ⁽³⁾
ISO18-14-03-046	P21B; Sample	10100	7830	12100	63000	6.34
ISO18-14-03-046-DUP	P21B; Sample Dup	9840	7830	11800	62300	6.50
Average		9970	7830	12000	62700	6.42
%RPD Sample/Sample Dup		2.6	0.0	2.5	1.1	2.5
ISO18-14-03-047	P304; Sample	404	248	132	1070	19.0
ISO18-14-03-047-DUP	P304; Sample Dup	421	252	128	1110	20.6
Average		413	250	130	1090	19.8
%RPD Sample/Sample Dup		4.1	1.6	3.1	3.7	8.1
ISO18-14-03-048	P305; Sample	177	149	245	486	18.8
ISO18-14-03-048-DUP	P305; Sample Dup	176	161	257	483	18.8
Average		177	155	251	485	18.8
%RPD Sample/Sample Dup		0.57	7.7	4.8	0.62	0.0
ISO18-14-03-049	P42; Sample	328	40.8	308	5560	18.3
ISO18-14-03-049-DUP	P42; Sample Dup	320	38.3	307	5410	16.9
Average		324	39.6	308	5490	17.6
%RPD Sample/Sample Dup		2.5	6.3	0.33	2.7	8.0
ISO18-14-03-050	P56; Sample	27.4	8.50	16.1	198	34.0
ISO18-14-03-050-DUP	P56; Sample Dup	21.0	6.68	12.2	197	31.8
Average		24.2 ⁽²⁾	7.59 ⁽²⁾	14.2 ⁽²⁾	198	32.9
%RPD Sample/Sample Dup		26 ⁽³⁾	24 ⁽³⁾	28 ⁽³⁾	0.51	6.7

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - WWTP					
ISO18-14-03-051	L19; Sample	225	114	3470	94.4
ISO18-14-03-051-DUP	L19; Sample Dup	224	115	3460	92.8
Average		225	115	3470	93.6
%RPD Sample/Sample Dup		0.45	0.87	0.29	1.7

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 15%, PFBS \pm 12%, PFHS \pm 13%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 14%, PFBS \pm 11%, PFHS \pm 13%, PFOS \pm 11%, and PFOSA \pm 20%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) The analytical data uncertainty has been expanded for location P119C for PFOSA to \pm 31% based on field matrix spike recovery.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - WWTP					
ISO18-14-03-052	M4; Sample	588	148	9790	118
ISO18-14-03-052-DUP	M4; Sample Dup	564	142	8790	119
		Average	145	9290	119
		%RPD Sample/Sample Dup	4.2	11	0.84
ISO18-14-03-053	P118C; Sample	216	86.4	2950	52.2
ISO18-14-03-053-DUP	P118C; Sample Dup	212	87.9	3010	50.4
		Average	87.2	2980	51.3
		%RPD Sample/Sample Dup	1.9	2.0	3.5
ISO18-14-03-054	P119C; Sample	233	107	3990	71.0
ISO18-14-03-054-DUP	P119C; Sample Dup	240	106	3950	66.6
		Average	107	3970	68.8 ⁽⁴⁾
		%RPD Sample/Sample Dup	3.0	1.0	6.4
ISO18-14-03-055	P262; Sample	1470	826	5840	65.4
ISO18-14-03-055-DUP	P262; Sample Dup	1460	847	5810	63.6
		Average	837	5830	64.5
		%RPD Sample/Sample Dup	0.68	2.5	0.52
ISO18-14-03-056	P263; Sample	1410	463	9340	22.4
ISO18-14-03-056-DUP	P263; Sample Dup	1440	471	9090	22.0
		Average	467	9220	22.2
		%RPD Sample/Sample Dup	2.1	1.7	2.7
ISO18-14-03-057	P264; Sample	263	154	1460	74.0
ISO18-14-03-057-DUP	P264; Sample Dup	255	151	1510	76.4
		Average	153	1490	75.2
		%RPD Sample/Sample Dup	3.1	2.0	3.4
ISO18-14-03-058	P265C; Sample	97.3	42.3	829	97.2
ISO18-14-03-058-DUP	P265C; Sample Dup	93.6	40.3	823	95.4
		Average	41.3	826	96.3
		%RPD Sample/Sample Dup	3.9	4.8	0.73
ISO18-14-03-059	P340; Sample	493	267	2520	54.0
ISO18-14-03-059-DUP	P340; Sample Dup	471	261	2440	50.6
		Average	264	2480	52.3
		%RPD Sample/Sample Dup	4.6	3.2	6.5
ISO18-14-03-060	P341; Sample	643	223	5180	83.2
ISO18-14-03-060-DUP	P341; Sample Dup	659	223	5290	85.8
		Average	223	5240	84.5
		%RPD Sample/Sample Dup	2.5	0.0	2.1
					3.1

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 15%, PFBS \pm 12%, PFHS \pm 13%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 14%, PFBS \pm 11%, PFHS \pm 13%, PFOS \pm 11%, and PFOSA \pm 20%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) The analytical data uncertainty has been expanded for location P119C for PFOSA to \pm 31% based on field matrix spike recovery.

Table 1 continued. Sample Results Summary⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - WWTP					
ISO18-14-03-061	P343; Sample	506	366	5520	12.9
ISO18-14-03-061-DUP	P343; Sample Dup	526	366	5960	14.6
		Average	516	366	5740
		%RPD Sample/Sample Dup	3.9	0.0	13.8
				7.7	12
ISO18-14-03-062	P371; Sample	474	188	6610	23.8
ISO18-14-03-062-DUP	P371; Sample Dup	478	190	6750	23.4
		Average	476	189	6680
		%RPD Sample/Sample Dup	0.84	1.1	23.6
				2.1	1.7
ISO18-14-03-063	P374; Sample	160	193	2490	13.2
ISO18-14-03-063-DUP	P374; Sample Dup	159	184	2480	12.2
		Average	160	189	2490
		%RPD Sample/Sample Dup	0.63	4.8	12.7
				0.40	7.9
ISO18-14-03-064	P379; Sample	59.4	19.2	675	61.6
ISO18-14-03-064-DUP	P379; Sample Dup	59.6	19.3	657	59.6
		Average	59.5	19.3	666
		%RPD Sample/Sample Dup	0.34	0.52	60.6
				2.7	3.3
ISO18-14-03-065	P380; Sample	102	40.4	827	97.4
ISO18-14-03-065-DUP	P380; Sample Dup	102	42.4	812	97.2
		Average	102	41.4	820
		%RPD Sample/Sample Dup	0.0	4.8	97.3
				1.8	0.21
ISO18-14-03-066	P381; Sample	529	165	1160	9.26
ISO18-14-03-066-DUP	P381; Sample Dup	541	161	1230	9.76
		Average	535	163	1200
		%RPD Sample/Sample Dup	2.2	2.5	9.51
				5.9	5.3
ISO18-14-03-067	P382; Sample	470	140	776	75.4
ISO18-14-03-067-DUP	P382; Sample Dup	469	142	785	71.4
		Average	470	141	781
		%RPD Sample/Sample Dup	0.21	1.4	73.4
				1.2	5.4
Zone: Southern Site Boundary					
ISO18-14-03-070	P372; Sample	51.9	42.3	1260	51.0
ISO18-14-03-070-DUP	P372; Sample Dup	51.6	41.9	1210	50.8
		Average	51.8	42.1	1240
		%RPD Sample/Sample Dup	0.58	0.95	50.9
				4.0	0.39
ISO18-14-03-071	P378; Sample	205	236	732	1.48
ISO18-14-03-071-DUP	P378; Sample Dup	209	240	722	1.45
		Average	207	238	727
		%RPD Sample/Sample Dup	1.9	1.7	1.47
				1.4	2.0

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 15%, PFBS \pm 12%, PFHS \pm 13%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 14%, PFBS \pm 11%, PFHS \pm 13%, PFOS \pm 11%, and PFOSA \pm 20%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) The analytical data uncertainty has been expanded for location P119C for PFOSA to \pm 31% based on field matrix spike recovery.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFBS	PFHS	PFOSA ⁽²⁾
Zone: Southern Site Boundary					
ISO18-14-03-072	PA109A; Sample	38.4	41.0	157	2.04
ISO18-14-03-072-DUP	PA109A; Sample Dup	42.0	43.8	157	2.66
Average %RPD Sample/Sample Dup		40.2 ⁽²⁾ 9.0	42.4 ⁽²⁾ 6.6	157 ⁽²⁾ 0.0	2.35 26 ⁽³⁾
ISO18-14-03-073	PA111A; Sample	740	993	2040	10.1
ISO18-14-03-073-DUP	PA111A; Sample Dup	743	1000	2100	10.2
Average %RPD Sample/Sample Dup		742 0.40	997 0.70	2070 2.9	10.2 0.99
ISO18-14-03-074	PA112; Sample	134	32.1	346	98.6
ISO18-14-03-074-DUP	PA112; Sample Dup	135	31.4	353	98.2
Average %RPD Sample/Sample Dup		135 0.74	31.8 2.2	350 2.0	98.4 0.41
Travel Blank					
3M LIMS ID	Sample Description	PFOA	PFBS	PFHS	PFOS
ISO18-14-03-067	Travel Blank	<0.0480 ⁽²⁾	<0.0500 ⁽²⁾	<0.0250 ⁽²⁾	<0.0928 ⁽²⁾
PFOSA					
<0.0250 ⁽²⁾					

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 15%, PFBS ± 12%, PFHS ± 13%, and PFOS ± 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 14%, PFBS ± 11%, PFHS ± 13%, PFOS ± 11%, and PFOSA ± 20%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
- (4) The analytical data uncertainty has been expanded for location P119C for PFOSA to ±31% based on field matrix spike recovery.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis".

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluoroctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected on July 9-12, 2018, in Nalgene™ (high-density polyethylene) bottles prepared at the 3M EHS Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. All sample locations were spiked with a mass-labeled internal standard mix at a nominal concentration of 1 ng/mL.

and select samples were fortified with surrogate recovery standards (SRSs) at a nominal concentration of 0.1 ng/mL prior to sample collection. Samples were received on July 16, 2018.

2.3 Sample Preparation

Sample locations pre-spiked with internal standard and surrogates were analyzed for all analytes (PFBS for select locations) by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples along with the method blanks.

Samples requiring dilutions were prepared for PFOA, PFHS, PFOS and PFBS (select locations only) using a direct injection by methanol dilution method. Samples that required a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples that required a 1:100 dilution were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol. Samples that required a 1:500 dilution were prepared by diluting 0.02 mL of a well-mixed sample with 10.0 mL of methanol. Samples that required a 1:1000 dilution were prepared by first diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol, followed by a second dilution prepared by diluting 1 mL of the diluted sample into 9 mL of methanol. Diluted samples and LCSs were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

Select samples were prepared for PFOSA by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The laboratory control samples and calibration standards were then diluted with methanol in the same manner.

2.4 Analysis

All samples and quality control samples were analyzed for the target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). SRSs were added and included in the analysis of these samples. Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.2. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

The following analytical runs were used to report the results herein:

8/1/18 (ETS Kirk) Internal Standard Calibration Analysis:

- Sample locations pre-spiked with surrogates were analyzed for PFOA, PFHS, PFOS, and PFOSA with select locations being analyzed for PFBS. All sample results were reported **except** for the following sample locations: 12 (PFOA, PFHS, PFOS, PFOSA), P119A and 13 (PFOA, PFHS, PFOS), Blokkersdijkvijver Noord (PFOSA), and Collector put and P56 (PFOS).

8/7/18 (ETS Tesla) Internal Standard Calibration Analysis:

- All sample locations were analyzed for PFOSA. All sample results were reported **except** for the re-analysis of sample location 13.

8/8/18 (ETS Kirk) External Standard Calibration Analysis:

- All sample locations were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported **except** for the following sample location: PP05 (all analytes).

8/15/18 (ETS Kirk) External Standard Calibration Analysis:

- All sample locations were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported **except** for the following sample location: P21B, M4, and P263 (PFOS).

8/21/18 (ETS Kirk) External Standard Calibration Analysis:

- Sample locations P21B, M4, and P263 were analyzed for PFOS. All sample results were reported.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk	ETS Tesla
Liquid Chromatograph	Agilent 1260	Agilent 1260
Analysis Method	ETS-8-044.3	ETS-8-044.3
Analysis Date	8/1/18, 8/8/18, 8/15/18, 8/21/18	8/7/18
Guard column	Prism RP (2.1 mm X 50 mm), 5 μ	Prism RP (2.1 mm X 50 mm), 5 μ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ
Injection Volume	2 μ L	2 μ L
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 6500+
Ion Source	Turbo Spray	Turbo Spray
Polarity	Negative	Negative
Software	Analyst 1.6.3	Analyst 1.6.3

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis				
Step Number	Total Time (min)	Flow Rate (μ L/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0.00	300	90.0	10.0
1	0.50	300	90.0	10.0
2	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard	Mass Transition Q1/Q3
PFOA	413/369	$[^{13}\text{C}_8]\text{-PFOA}$	421/376
	413/219		
	413/169		
PFBS	299/99	$[^{18}\text{O}_2]\text{-PFBS}$	303/84
	299/80		
PFHS	399/99	$[^{13}\text{C}_3]\text{-PFHS}$	402/80
	399/80		
PFOS	499/99	$[^{13}\text{C}_8]\text{-PFOS}$	507/80
	499/80		
	499/130		
PFOSA	498/78	$[^{13}\text{C}_8]\text{-PFOSA}$	506/78
$[^{13}\text{C}_3]\text{-PFOA}$	417/372	$[^{13}\text{C}_8]\text{-PFOA}$	421/376
$[^{13}\text{C}_3]\text{-PFOS}$	503/80	$[^{13}\text{C}_8]\text{-PFOS}$	507/80

The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

3 Data Analysis

3.1 Calibration

8/1/18 Analysis (Internal Standard Calibration): Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of fourteen calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed. The calibration standards also contained surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

8/8/18, 8/15/18, and 8/21/18 Analysis (External Standard Calibration): Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL or 0.05 ng/mL to 100 ng/mL (nominal) were analyzed. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL or 0.05 ng/mL to 25 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

8/7/18 Analysis of PFOSA (Internal Standard Calibration): Samples were analyzed for PFOSA against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into laboratory reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 1.0 ng/mL. Calibration standards ranging from 0.025 ng/mL

to 200 ng/mL (nominal) were analyzed. Prior to analysis, the calibration standards were diluted 2x by removing a 0.4 mL aliquot and diluting it with 0.4 mL of methanol. A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of $100\pm 25\%$ ($100\pm 30\%$ for the lowest curve point) were met for all analytes. The correlation coefficient (r) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes with the exception of PFOS analyzed on 8/15/18 with an area count RSD of 7.0%.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.3 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the **Table 6** below.

Table 6. LOQ

Analyte	LOQ, ng/mL ⁽²⁾ 8/1/18 Analysis	LOQ, ng/mL ⁽²⁾ 8/7/18 Analysis	LOQ, ng/mL ⁽¹⁾ 8/8/18 Analysis	LOQ, ng/mL ⁽¹⁾ 8/15/18 Analysis	LOQ, ng/mL ⁽¹⁾ 8/21/18 Analysis
PFOA	0.0480	NA	0.0958	0.0479	NA
PFBS	0.0500	NA	0.100	0.0200	NA
PFHS	0.0250	NA	0.100	0.0200	NA
PFOS	0.0928	NA	0.0464	0.0464	0.0464
PFOSA	0.0250	0.250	NA	NA	NA

NA = Not Applicable

(1) A dilution factor was not applied to the LOQ.

(2) A dilution factor of 2 was applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of $100\pm 25\%$.

3.5 Blanks

Two types of blanks were prepared and analyzed with the samples: method/solvent blanks and field/trip blanks. Each blank result was reviewed and used to evaluate method performance. The method blanks were used to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. Target analyte LCSs analyzed on 8/1/18 were prepared at nominal concentrations of 0.2 ng/mL, 20 ng/mL, and 140 ng/mL. Target analyte LCSs analyzed on 8/7/18 were prepared at nominal concentrations of 0.2 ng/mL, 20 ng/mL, and 200 ng/mL. Target analyte LCSs analyzed on 8/8/18 were prepared at nominal concentrations of 100 ng/mL, 5000 ng/mL, and 35000 ng/mL. Target analyte LCSs analyzed on 8/15/18 were prepared at nominal concentrations of 100 ng/mL, 5000 ng/mL, and 35000 ng/mL. Target analyte LCSs analyzed on 8/21/18 were prepared at nominal concentrations of 200 ng/mL, 20000 ng/mL, and 70000 ng/mL. LCSs were

prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. Surrogates [¹³C₄]-PFOA and [¹³C₄]-PFOS were added post dilution when analyzed by external standard.

Method ETS-8-044.3 states that the average recovery of LCSs at each spiking level must be within 80%-120% with a RSD ≤20%. All LCS samples met criteria with the following exceptions:

- 8/1/18: High-level LCSs for PFOSA were spiked above the resulting ULOQ (100 ng/mL).
- 8/7/18: Low-level LCSs for PFOSA were spiked below the results LLOQ (0.250 ng/mL).

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report. Individual LCS results are included in the raw data and available upon request.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.4. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in Table 7 below.

Table 7. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	7.66	±15%
PFBS	External	5.88	±12%
PFHS	External	6.33	±13%
PFOS	External	7.38	±15%
PFOA	Internal	6.89	±14%
PFBS	Internal	5.36	±11%
PFHS	Internal	6.28	±13%
PFOS	Internal	5.61	±11%
PFOSA	Internal	10.1	±20%

3.8 Field Matrix Spikes (FMS)

Target analyte field matrix spike samples were collected for select sampling points to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions at 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOA and PFOS. The standards used for the preparation of the field matrix spiking solutions greater than 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, select sample bottles contained stable isotope surrogate recovery spikes of [¹³C₄]-PFOA and [¹³C₄]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles prior to sample collection. The [¹³C₄]-PFOA was selected to represent the C8 perfluorocarboxylic acid. The [¹³C₄]-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

The following calculation was used to generate data in section 4 of the report.

$$FMS\ Recovery = \frac{Sample\ Concentration\ of\ FMS - Average\ Concentration\ (Field\ Sample\ & Field\ Sample\ Dup)}{Spike\ Concentration} \times 100\%$$

Table 8. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
L31	FMS	1.92	2.00	2.00	1.85	2.00
D2, P116	FMS	10.0	10.0	10.0	10.0	10.0
Collector put, P56	FMS	50.0	50.0	50.0	50.0	50.0
12, B3-bis	FMS	51.9	52.0	52.0	51.9	2.00
D10, D17, P119A, P378	FMS	255	255	255	255	5.00
PP07, K3, P265C, P381	FMS	1050	1050	1050	1050	50.0
P119C, P340 ⁽¹⁾	FMS	942	942	942	942	44.8
PP02	FMS	1050	50.0	1050	1050	50.0
Trip Blank	Low	1.92	2.00	2.00	1.85	2.00
	High	1050	50.0	1050	1050	50.0

(1) Sample container for the FMS sample was overfilled by 10%. The FMS true values were adjusted accordingly.

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of $\pm 30\%$, demonstrate that the method is appropriate for the given matrix.

The method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. At times, the spike level exceeded the recommended upper limit of 10 times the analyte concentration. In these instances, the FMS recovery was reported and flagged as above 10 times the sample concentration.

For PFOA, PFBS, PFHS, and PFOS, when the field matrix spike level was not appropriate as compared to the endogenous sample concentration, the surrogate recovery standards were used to assess method accuracy. All field matrix spike and surrogate recovery standard recoveries met acceptance criteria with the following exception:

P119C: The FMS recovery was 69.2% for PFOSA. The analytical data uncertainty was expanded to $\pm 31\%$ for PFOSA based on the FMS %bias.

Table 9. Location ID: D10

3M LIMS ID	Sample Description	PFOA		PFHS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-004	D10; Sample	559	NA	231	NA
ISO18-14-03-004-DUP	D10; Sample Dup	539	NA	226	NA
ISO18-14-03-004-FMS	D10; FMS	745	NC	493	104
Average Concentration (ng/mL) ± %RPD		549 ng/mL ± 3.6%		229 ng/mL ± 2.2%	

3M LIMS ID	Sample Description	PFOS		PFOSA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-004	D10; Sample	59.0	NA	<0.250	NA
ISO18-14-03-004-DUP	D10; Sample Dup	58.5	NA	<0.250	NA
ISO18-14-03-004-FMS	D10; FMS	291	91.2	4.80	96.0 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		58.8 ng/mL ± 0.85%		<0.250 ng/mL	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration

(1) FMS concentration greater than 10 times the sample concentration.

Table 10. Location ID: D17

3M LIMS ID	Sample Description	PFOA		PFHS	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-008	D17; Sample	495	NA	169	NA
ISO18-14-03-008-DUP	D17; Sample Dup	485	NA	172	NA
ISO18-14-03-008-FMS	D17; FMS	704	84.0	418	97.1
Average Concentration (ng/mL) ± %RPD		490 ng/ml ± 2.0%		171 ng/mL ± 1.8%	

3M LIMS ID	Sample Description	PFOS		PFOSA	
		Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-008	D17; Sample	280	NA	1.61	NA
ISO18-14-03-008-DUP	D17; Sample Dup	266	NA	1.38	NA
ISO18-14-03-008-FMS	D17; FMS	476	79.7	7.10	112
Average Concentration (ng/mL) ± %RPD		273 ng/mL ± 5.1%		1.50 ng/mL ± 15%	

NA = Not Applicable

Table 7. Location ID: D2

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-010	D2; Sample	0.344	NA	0.176	NA
ISO18-14-03-010-DUP	D2; Sample Dup	0.388	NA	0.159	NA
ISO18-14-03-010-FMS	D2; FMS	9.54	91.7 ⁽¹⁾	9.54	93.7 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		0.366 ng/mL ± 12%		0.168 ng/mL ± 10%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-010	D2; Sample	7.84	NA	0.394	NA
ISO18-14-03-010-DUP	D2; Sample Dup	8.14	NA	0.454	NA
ISO18-14-03-010-FMS	D2; FMS	16.6	86.1	10.5	101 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		7.99 ng/mL ± 3.8%		0.424 ng/mL ± 14%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 8. Location ID: P119A

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-015	P119A; Sample	63.6	NA	19.0	NA
ISO18-14-03-015-DUP	P119A; Sample Dup	60.7	NA	18.1	NA
ISO18-14-03-015-FMS	P119A; FMS	298	92.6	272	99.4 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		62.2 ng/mL ± 4.7%		18.6 ng/mL ± 4.9%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-015	P119A; Sample	19.9	NA	2.18	NA
ISO18-14-03-015-DUP	P119A; Sample Dup	19.8	NA	2.20	NA
ISO18-14-03-015-FMS	P119A; FMS	248	89.6 ⁽¹⁾	7.86	113
Average Concentration (ng/mL) ± %RPD		19.9 ng/mL ± 0.50%		2.19 ng/mL ± 0.91%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 9. Location ID: L31

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-024	L31; Sample	0.648	NA	0.602	NA
ISO18-14-03-024-DUP	L31; Sample Duplicate	0.640	NA	0.590	NA
ISO18-14-03-024-FMS	L31; FMS	2.44	93.6	2.50	95.2
Average Concentration (ng/mL) ± %RPD		0.644 ng/mL ± 1.2%		0.596 ng/mL ± 2.0%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-024	L31; Sample	11.1	NA	0.338	NA
ISO18-14-03-024-DUP	L31; Sample Duplicate	10.5	NA	0.428	NA
ISO18-14-03-024-FMS	L31; FMS	13.4	NC	2.2	91.9
Average Concentration (ng/mL) ± %RPD		10.8 ng/mL ± 5.6%		0.383 ng/mL ± 23% (1)	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 10. Location ID: P116

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-028	P116; Sample	0.960	NA	0.528	NA
ISO18-14-03-028-DUP	P116; Sample Dup	0.942	NA	0.532	NA
ISO18-14-03-028-FMS	P116; FMS	10.2	92.5 (1)	10.0	94.7 (1)
Average Concentration (ng/mL) ± %RPD		0.951 ng/mL ± 1.9%		0.530 ng/mL ± 0.75%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-028	P116; Sample	8.00	NA	0.105	NA
ISO18-14-03-028-DUP	P116; Sample Dup	7.70	NA	0.105	NA
ISO18-14-03-028-FMS	P116; FMS	16.6	87.5	10.2	101 (1)
Average Concentration (ng/mL) ± %RPD		7.85 ng/mL ± 3.8%		0.105 ng/mL ± 0.0%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 15. Location ID: PP02

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-031	PP02; Sample	653	NA	52.7	NA	3630	NA
ISO18-14-03-031-DUP	PP02; Sample Dup	628	NA	53.7	NA	3630	NA
ISO18-14-03-031-FMS	PP02; FMS	1610	92.3	104	102	4770	NC
Average Concentration (ng/mL) ± %RPD		641 ng/mL ± 3.9%		53.2 ng/mL ± 1.9%		3630 ng/mL ± 0.0%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-031	PP02; Sample	20000	NA	29.4	NA
ISO18-14-03-031-DUP	PP02; Sample Dup	19100	NA	26.6	NA
ISO18-14-03-031-FMS	PP02; FMS	21100	NC	78.8	102
Average Concentration (ng/mL) ± %RPD		19600 ng/mL ± 4.6%		28.0 ng/mL ± 10%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 16. Location ID: PP07

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-035	PP07; Sample	308	NA	223	NA	136	NA
ISO18-14-03-035-DUP	PP07; Sample Dup	288	NA	192	NA	111	NA
ISO18-14-03-035-FMS	PP07; FMS	1260	91.6	1190	93.6	1110	94.0
Average Concentration (ng/mL) ± %RPD		298 ng/mL ± 6.7%		208 ng/mL ± 15%		124 ng/mL ± 20%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-035	PP07; Sample	2670	NA	72.0	NA
ISO18-14-03-035-DUP	PP07; Sample Dup	2610	NA	76.4	NA
ISO18-14-03-035-FMS	PP07; FMS	3640	NC	122	95.6
Average Concentration (ng/mL) ± %RPD		2640 ng/mL ± 2.3%		74.2 ng/mL ± 5.9%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 17. Location ID: 12

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-039	12; Sample	119	NA	34.8	NA
ISO18-14-03-039-DUP	12; Sample Dup	122	NA	34.4	NA
ISO18-14-03-039-FMS	12; FMS	169	NC	83.6	94.2
Average Concentration (ng/mL) ± %RPD		121 ng/mL ± 2.5%		34.6 ng/mL ± 1.2%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-039	12; Sample	446	NA	0.492	NA
ISO18-14-03-039-DUP	12; Sample Dup	452	NA	0.818	NA
ISO18-14-03-039-FMS	12; FMS	484	NC	2.66	100
Average Concentration (ng/mL) ± %RPD		449 ng/mL ± 1.3%		0.655 ng/mL ± 50%⁽¹⁾	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 18. Location ID: Collector put

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-043	Collector put; Sample	59.0	NA	22.6	NA
ISO18-14-03-043-DUP	Collector put; Sample Dup	61.6	NA	22.6	NA
ISO18-14-03-043-FMS	Collector put; FMS	104	87.4	69.4	93.6
Average Concentration (ng/mL) ± %RPD		60.3 ng/mL ± 4.3%		22.6 ng/mL ± 0.0%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-043	Collector put; Sample	204	NA	14.1	NA
ISO18-14-03-043-DUP	Collector put; Sample Dup	202	NA	14.6	NA
ISO18-14-03-043-FMS	Collector put; FMS	NA ⁽¹⁾	NA ⁽¹⁾	68.2	108
Average Concentration (ng/mL) ± %RPD		203 ng/mL ± 0.99%		14.4 ng/mL ± 3.5%	

NA = Not Applicable

(1) The FMS was not re-analyzed for PFOS since the spike level was not appropriate based on the endogenous sample concentration.

Table 19. Location ID: K3

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-044	K3; Sample	79.8	NA	297	NA	17000	NA
ISO18-14-03-044-DUP	K3; Sample Dup	76.3	NA	290	NA	17100	NA
ISO18-14-03-044-FMS	K3; FMS	1030	90.7 ⁽¹⁾	1320	97.8	17900	NC
Average Concentration (ng/mL) ± %RPD		78.1 ng/mL ± 4.5%		294 ng/mL ± 2.4%		17100 ng/mL ± 0.59%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-044	K3; Sample	4800	NA	13.1	NA
ISO18-14-03-044-DUP	K3; Sample Dup	4860	NA	14.5	NA
ISO18-14-03-044-FMS	K3; FMS	5870	NC	60.4	93.2
Average Concentration (ng/mL) ± %RPD		4830 ng/mL ± 1.2%		13.8 ng/mL ± 10%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) FMS concentration greater than 10 times the sample concentration.

Table 20. Location ID: P56

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-050	P56; Sample	27.4	NA	8.50	NA	16.1	NA
ISO18-14-03-050-DUP	P56; Sample Dup	21.0	NA	6.68	NA	12.2	NA
ISO18-14-03-050-FMS	P56; FMS	66.2	84.0	55.4	95.6	56.6	84.9
Average Concentration (ng/mL) ± %RPD		24.2 ng/mL ± 26%⁽¹⁾		7.59 ng/mL ± 24%⁽¹⁾		14.2 ng/mL ± 28%⁽¹⁾	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-050	P56; Sample	198	NA	34.0	NA
ISO18-14-03-050-DUP	P56; Sample Dup	197	NA	31.8	NA
ISO18-14-03-050-FMS	P56; FMS	NA ⁽²⁾	NA ⁽²⁾	77.2	88.6
Average Concentration (ng/mL) ± %RPD		198 ng/mL ± 0.51%		32.9 ng/mL ± 6.7%	

NA = Not Applicable

(1) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(2) FMS not re-analyzed for PFOS since the spike level was not appropriate based on the endogenous sample concentration.

Table 21. Location ID: P119C

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-054	P119C; Sample	233	NA	107	NA
ISO18-14-03-054-DUP	P119C; Sample Dup	240	NA	106	NA
ISO18-14-03-054-FMS	P119C; FMS	1220	104	1110	107
Average Concentration (ng/mL) ± %RPD		237 ng/mL ± 3.0%		107 ng/mL ± 0.94%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-054	P119C; Sample	3990	NA	71.0	NA
ISO18-14-03-054-DUP	P119C; Sample Dup	3950	NA	66.6	NA
ISO18-14-03-054-FMS	P119C; FMS	5030	NC	99.8	69.2 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		3970 ng/mL ± 1.0%		68.8 ng/mL ± 6.4% ⁽²⁾	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) FMS did not meet acceptance criteria of $100 \pm 30\%$.

(2) Analytical data uncertainty has been expanded to $\pm 31\%$.

Table 22. Location ID: P265C

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-058	P265C; Sample	97.3	NA	42.3	NA
ISO18-14-03-058-DUP	P265C; Sample Dup	93.6	NA	40.3	NA
ISO18-14-03-058-FMS	P265C; FMS	1090	94.7 ⁽¹⁾	1100	101 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		95.5 ng/mL ± 3.9%		41.3 ng/mL ± 4.8%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-058	P265C; Sample	829	NA	97.2	NA
ISO18-14-03-058-DUP	P265C; Sample Dup	823	NA	95.4	NA
ISO18-14-03-058-FMS	P265C; FMS	1800	92.8	138	83.4
Average Concentration (ng/mL) ± %RPD		826 ng/mL ± 0.73%		96.3 ng/mL ± 1.9%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 23. Location ID: P340

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-059	P340; Sample	493	NA	267	NA
ISO18-14-03-059-DUP	P340; Sample Dup	471	NA	261	NA
ISO18-14-03-059-FMS	P340; FMS	1440	102	1260	106
Average Concentration (ng/mL) ± %RPD		482 ng/mL ± 4.6%		264 ng/mL ± 2.3%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-059	P340; Sample	2520	NA	54.0	NA
ISO18-14-03-059-DUP	P340; Sample Dup	2440	NA	50.6	NA
ISO18-14-03-059-FMS	P340; FMS	3320	NC	90.2	84.6
Average Concentration (ng/mL) ± %RPD		2480 ng/mL ± 3.2%		52.3 ng/mL ± 6.5%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 24. Location ID: P381

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-066	P381; Sample	529	NA	165	NA
ISO18-14-03-066-DUP	P381; Sample Dup	541	NA	161	NA
ISO18-14-03-066-FMS	P381; FMS	1600	101	1330	111
Average Concentration (ng/mL) ± %RPD		535 ng/mL ± 2.2%		163 ng/mL ± 2.5%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-066	P381; Sample	1160	NA	9.26	NA
ISO18-14-03-066-DUP	P381; Sample Dup	1230	NA	9.76	NA
ISO18-14-03-066-FMS	P381; FMS	2380	113	56.2	93.4
Average Concentration (ng/mL) ± %RPD		1200 ng/mL ± 5.9%		9.51 ng/mL ± 5.3%	

NA = Not Applicable

Table 25. Location ID: P378

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-071	P378; Sample	205	NA	236	NA
ISO18-14-03-071-DUP	P378; Sample Dup	209	NA	240	NA
ISO18-14-03-071-FMS	P378; FMS	426	85.9	477	93.7
Average Concentration (ng/mL) ± %RPD		207 ng/mL ± 1.9%		238 ng/mL ± 1.7%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-071	P378; Sample	732	NA	1.48	NA
ISO18-14-03-071-DUP	P378; Sample Dup	722	NA	1.45	NA
ISO18-14-03-071-FMS	P378; FMS	926	NC	6.50	101
Average Concentration (ng/mL) ± %RPD		727 ng/mL ± 1.4%		1.47 ng/mL ± 2.0%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 26. Location ID: Travel Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-075	Travel Blank	<0.0480	NA	<0.0500	NA	<0.0250	NA
ISO18-14-03-075-FMS-LOW	Travel Blank FMS Low	1.78	92.7	1.92	96.0	1.80	90.0
ISO18-14-03-075-FMS-HIGH	Travel Blank FMS High	1020	97.1	51.1	102	1130	108

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-03-075	Travel Blank	<0.0928	NA	<0.0250	NA
ISO18-14-03-075-FMS-LOW	Travel Blank FMS Low	1.74	94.1	1.88	94.0
ISO18-14-03-075-FMS-HIGH	Travel Blank FMS High	1050	100	47.6	95.2

NA = Not Applicable

Table 27. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)	
		[¹³ C ₄]PFOA	[¹³ C ₄]PFOS
ISO18-14-03-001	BD24-3; Sample	100	105
ISO18-14-03-001-DUP	BD24-3; Sample Duplicate	104	112
ISO18-14-03-002	BD24-4; Sample	86.2 ⁽²⁾	84.2 ⁽²⁾
ISO18-14-03-002-DUP	BD24-4; Sample Duplicate	82.6 ⁽²⁾	94.5 ⁽²⁾
ISO18-14-03-003	D09; Sample	107	113
ISO18-14-03-003-DUP	D09; Sample Duplicate	105	111
ISO18-14-03-004	D10; Sample	101	108
ISO18-14-03-004-DUP	D10; Sample Duplicate	97.7	109
ISO18-14-03-004-FMS	D10; FMS	98.6	108
ISO18-14-03-005	D11; Sample	102	112
ISO18-14-03-005-DUP	D11; Sample Duplicate	107	114
ISO18-14-03-006	D14; Sample	88.6 ⁽²⁾	93.6 ⁽²⁾
ISO18-14-03-006-DUP	D14; Sample Duplicate	86.7 ⁽²⁾	89.1 ⁽²⁾
ISO18-14-03-007	D16; Sample	108	113
ISO18-14-03-007-DUP	D16; Sample Duplicate	105	115
ISO18-14-03-008	D17; Sample	106	113
ISO18-14-03-008-DUP	D17; Sample Duplicate	109	117
ISO18-14-03-008-FMS	D17; FMS	110	118
ISO18-14-03-009	D18; Sample	108	114
ISO18-14-03-009-DUP	D18; Sample Duplicate	109	114
ISO18-14-03-010	D2; Sample	98.4 ⁽²⁾	91.1 ⁽²⁾
ISO18-14-03-010-DUP	D2; Sample Duplicate	87.4 ⁽²⁾	95.9 ⁽²⁾
ISO18-14-03-010-FMS	D2; FMS	89.0 ⁽²⁾	89.0 ⁽²⁾
ISO18-14-03-011	D5; Sample	110	117
ISO18-14-03-011-DUP	D5; Sample Duplicate	108	115
ISO18-14-03-012	ND7; Sample	102	113
ISO18-14-03-012-DUP	ND7; Sample Duplicate	102	111
ISO18-14-03-013	P118A; Sample	108	116
ISO18-14-03-013-DUP	P118A; Sample Duplicate	106	117
ISO18-14-03-014	P118B; Sample	107	108
ISO18-14-03-014-DUP	P118B; Sample Duplicate	103	110
ISO18-14-03-015	P119A; Sample	102	109
ISO18-14-03-015-DUP	P119A; Sample Duplicate	96.7	109
ISO18-14-03-015-FMS	P119A; FMS	104	111
ISO18-14-03-016	P119B; Sample	111	120
ISO18-14-03-016-DUP	P119B; Sample Duplicate	108	118
ISO18-14-03-017	P121; Sample	87.2 ⁽²⁾	90.1 ⁽²⁾
ISO18-14-03-017-DUP	P121; Sample Duplicate	91.8 ⁽²⁾	83.8 ⁽²⁾
ISO18-14-03-018	P321; Sample	103	111
ISO18-14-03-018-DUP	P321; Sample Duplicate	107	111
ISO18-14-03-019	3M vijver; Sample	90.2 ⁽²⁾	94.5 ⁽²⁾
ISO18-14-03-019-DUP	3M vijver; Sample Duplicate	85.0 ⁽²⁾	81.0 ⁽²⁾

NA = Not Applicable

- (1) The surrogate recovery standards [¹³C₄]PFOA and [¹³C₄]PFOS were added to the samples during sample preparation unless noted otherwise.
- (2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 27 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)	
		[¹³ C ₄]PFOA	[¹³ C ₄]PFOS
ISO18-14-03-020	Blokkersdijkvijver Noord; Sample	95.0 ⁽²⁾	88.2 ⁽²⁾
ISO18-14-03-020-DUP	Blokkersdijkvijver Noord; Sample Dup	93.4 ⁽²⁾	90.3 ⁽²⁾
ISO18-14-03-021	Blokkersdijkvijver standard; Sample	97.0 ⁽²⁾	90.9 ⁽²⁾
ISO18-14-03-021-DUP	Blokkersdijkvijver standard; Sample Dup	94.4 ⁽²⁾	95.5 ⁽²⁾
ISO18-14-03-022	L21; Sample	93.0 ⁽²⁾	96.8 ⁽²⁾
ISO18-14-03-022-DUP	L21; Sample Duplicate	95.4 ⁽²⁾	91.5 ⁽²⁾
ISO18-14-03-023	L22; Sample	93.6 ⁽²⁾	86.7 ⁽²⁾
ISO18-14-03-023-DUP	L22; Sample Duplicate	96.0 ⁽²⁾	92.8 ⁽²⁾
ISO18-14-03-024	L31; Sample	89.8 ⁽²⁾	90.9 ⁽²⁾
ISO18-14-03-024-DUP	L31; Sample Duplicate	107 ⁽²⁾	87.7 ⁽²⁾
ISO18-14-03-024-FMS	L31; FMS	104 ⁽²⁾	95.3 ⁽²⁾
ISO18-14-03-025	L4; Sample	101 ⁽²⁾	91.5 ⁽²⁾
ISO18-14-03-025-DUP	L4; Sample Duplicate	91.2 ⁽²⁾	88.9 ⁽²⁾
ISO18-14-03-026	P114bis; Sample	92.8 ⁽²⁾	89.8 ⁽²⁾
ISO18-14-03-026-DUP	P114bis; Sample Duplicate	91.0 ⁽²⁾	92.6 ⁽²⁾
ISO18-14-03-027	P115; Sample	86.4 ⁽²⁾	92.4 ⁽²⁾
ISO18-14-03-027-DUP	P115; Sample Duplicate	91.0 ⁽²⁾	87.7 ⁽²⁾
ISO18-14-03-028	P116; Sample	92.2 ⁽²⁾	99.3 ⁽²⁾
ISO18-14-03-028-DUP	P116; Sample Duplicate	91.6 ⁽²⁾	89.4 ⁽²⁾
ISO18-14-03-028-FMS	P116; FMS	90.4 ⁽²⁾	89.8 ⁽²⁾
ISO18-14-03-029	Effluent WWTP; Sample	87.0 ⁽²⁾	85.9 ⁽²⁾
ISO18-14-03-029-DUP	Effluent WWTP; Sample Duplicate	92.6 ⁽²⁾	91.9 ⁽²⁾
ISO18-14-03-030	PP01; Sample	109	114
ISO18-14-03-030-DUP	PP01; Sample Duplicate	115	120
ISO18-14-03-031	PP02; Sample	108	109
ISO18-14-03-031-DUP	PP02; Sample Duplicate	103	110
ISO18-14-03-031-FMS	PP02; FMS	108	112
ISO18-14-03-032	PP04; Sample	112	113
ISO18-14-03-032-DUP	PP04; Sample Duplicate	110	119
ISO18-14-03-033	PP05; Sample	105	113
ISO18-14-03-033-DUP	PP05; Sample Duplicate	114	114
ISO18-14-03-034	PP06; Sample	112	118
ISO18-14-03-034-DUP	PP06; Sample Duplicate	109	120
ISO18-14-03-035	PP07; Sample	103	112
ISO18-14-03-035-DUP	PP07; Sample Duplicate	106	118
ISO18-14-03-035-FMS	PP07; FMS	112	116
ISO18-14-03-036	PP08; Sample	110	116
ISO18-14-03-036-DUP	PP08; Sample Duplicate	91.9	102
ISO18-14-03-037	PP09; Sample	101	106
ISO18-14-03-037-DUP	PP09; Sample Duplicate	109	116
ISO18-14-03-038	PP10; Sample	109	114
ISO18-14-03-038-DUP	PP10; Sample Duplicate	112	114

NA = Not Applicable

- (1) The surrogate recovery standards [¹³C₄]PFOA and [¹³C₄]PFOS were added to the samples during sample preparation unless noted otherwise.
- (2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 27 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]PFOA	[¹³ C ₄]PFOS	[¹³ C ₄]PFOS Re-analysis
ISO18-14-03-039	12; Sample	105	112	NA
ISO18-14-03-039-DUP	12; Sample Duplicate	107	111	NA
ISO18-14-03-039-FMS	12; FMS	106	111	NA
ISO18-14-03-040	13; Sample	109	116	NA
ISO18-14-03-040-DUP	13; Sample Duplicate	108	114	NA
ISO18-14-03-041	5; Sample	96.0 ⁽²⁾	90.9 ⁽²⁾	NA
ISO18-14-03-041-DUP	5; Sample Duplicate	91.6 ⁽²⁾	93.8 ⁽²⁾	NA
ISO18-14-03-042	Bemalingsstation; Sample	93.2 ⁽²⁾	102 ⁽²⁾	NA
ISO18-14-03-042-DUP	Bemalingsstation; Sample Duplicate	99.6 ⁽²⁾	91.7 ⁽²⁾	NA
ISO18-14-03-043	Collector put; Sample	92.8 ⁽²⁾	111	NA
ISO18-14-03-043-DUP	Collector put; Sample Duplicate	91.8 ⁽²⁾	114	NA
ISO18-14-03-043-FMS	Collector put; FMS	96.0 ⁽²⁾	NA	NA
ISO18-14-03-044	K3; Sample	109	114	NA
ISO18-14-03-044-DUP	K3; Sample Duplicate	105	114	NA
ISO18-14-03-044-FMS	K3; FMS	111	116	NA
ISO18-14-03-045	P27; Sample	110	112	NA
ISO18-14-03-045-DUP	P27; Sample Duplicate	102	110	NA
ISO18-14-03-046	P21B; Sample	106	104	111
ISO18-14-03-046-DUP	P21B; Sample Duplicate	109	112	111
ISO18-14-03-047	P304; Sample	110	114	NA
ISO18-14-03-047-DUP	P304; Sample Duplicate	111	113	NA
ISO18-14-03-048	P305; Sample	112	118	NA
ISO18-14-03-048-DUP	P305; Sample Duplicate	112	118	NA
ISO18-14-03-049	P42; Sample	115	117	NA
ISO18-14-03-049-DUP	P42; Sample Duplicate	103	107	NA
ISO18-14-03-050	P56; Sample	85.0 ⁽²⁾	114	NA
ISO18-14-03-050-DUP	P56; Sample Duplicate	87.2 ⁽²⁾	115	NA
ISO18-14-03-050-FMS	P56; FMS	89.8 ⁽²⁾	NA	NA
ISO18-14-03-051	L19; Sample	117	117	NA
ISO18-14-03-051-DUP	L19; Sample Duplicate	111	116	NA
ISO18-14-03-052	M4; Sample	111	113	109
ISO18-14-03-052-DUP	M4; Sample Duplicate	114	116	110
ISO18-14-03-053	P118C; Sample	117	115	NA
ISO18-14-03-053-DUP	P118C; Sample Duplicate	113	118	NA
ISO18-14-03-054	P119C; Sample	114	121	NA
ISO18-14-03-054-DUP	P119C; Sample Duplicate	112	119	NA
ISO18-14-03-054-FMS	P119C; FMS	115	118	NA
ISO18-14-03-055	P262; Sample	116	121	NA
ISO18-14-03-055-DUP	P262; Sample Duplicate	114	121	NA
ISO18-14-03-056	P263; Sample	115	119	114
ISO18-14-03-056-DUP	P263; Sample Duplicate	111	115	115

NA = Not Applicable

- (1) The surrogate recovery standards [¹³C₄]PFOA and [¹³C₄]PFOS were added to the samples during sample preparation unless noted otherwise.
- (2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 27 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]PFOA	[¹³ C ₄]PFOS	[¹³ C ₄]PFOS Re-analysis
ISO18-14-03-057	P264; Sample	114	120	NA
ISO18-14-03-057-DUP	P264; Sample Duplicate	111	119	NA
ISO18-14-03-058	P265C; Sample	110	119	NA
ISO18-14-03-058-DUP	P265C; Sample Duplicate	106	112	NA
ISO18-14-03-058-FMS	P265C; FMS	116	120	NA
ISO18-14-03-059	P340; Sample	111	113	NA
ISO18-14-03-059-DUP	P340; Sample Duplicate	108	118	NA
ISO18-14-03-059-FMS	P340; FMS	115	118	NA
ISO18-14-03-060	P341; Sample	113	116	NA
ISO18-14-03-060-DUP	P341; Sample Duplicate	111	119	NA
ISO18-14-03-061	P343; Sample	109	118	NA
ISO18-14-03-061-DUP	P343; Sample Duplicate	111	115	NA
ISO18-14-03-062	P371; Sample	116	123	NA
ISO18-14-03-062-DUP	P371; Sample Duplicate	113	121	NA
ISO18-14-03-063	P374; Sample	109	117	NA
ISO18-14-03-063-DUP	P374; Sample Duplicate	112	118	NA
ISO18-14-03-064	P379; Sample	115	124	NA
ISO18-14-03-064-DUP	P379; Sample Duplicate	116	127	NA
ISO18-14-03-065	P380; Sample	115	124	NA
ISO18-14-03-065-DUP	P380; Sample Duplicate	118	128	NA
ISO18-14-03-066	P381; Sample	115	120	NA
ISO18-14-03-066-DUP	P381; Sample Duplicate	116	125	NA
ISO18-14-03-066-FMS	P381; FMS	112	119	NA
ISO18-14-03-067	P382; Sample	118	119	NA
ISO18-14-03-067-DUP	P382; Sample Duplicate	115	121	NA
ISO18-14-03-070	P372; Sample	111	118	NA
ISO18-14-03-070-DUP	P372; Sample Duplicate	112	118	NA
ISO18-14-03-071	P378; Sample	124	130	NA
ISO18-14-03-071-DUP	P378; Sample Duplicate	109	115	NA
ISO18-14-03-071-FMS	P378; FMS	105	114	NA
ISO18-14-03-072	PA109A; Sample	102 ⁽²⁾	85.4 ⁽²⁾	NA
ISO18-14-03-072-DUP	PA109A; Sample Duplicate	102 ⁽²⁾	88.4 ⁽²⁾	NA
ISO18-14-03-073	PA111A; Sample	110	119	NA
ISO18-14-03-073-DUP	PA111A; Sample Duplicate	119	125	NA
ISO18-14-03-074	PA112; Sample	117	126	NA
ISO18-14-03-074-DUP	PA112; Sample Duplicate	118	126	NA
ISO18-14-03-075	Travel Blank	82.0 ⁽²⁾	94.2 ⁽²⁾	NA
ISO18-14-03-075-FMS-LOW	Travel Blank FMS Low	95.6 ⁽²⁾	94.2 ⁽²⁾	NA
ISO18-14-03-075-FMS-HIGH	Travel Blank FMS High	118	125	NA

NA = Not Applicable

(1) The surrogate recovery standards [¹³C₄]PFOA and [¹³C₄]PFOS were added to the samples during sample preparation unless noted otherwise.

(2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 9-27 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M EHS Laboratory standard operating procedures.

7 Attachments

Chain of Custody Form

8 Signatures


Chelsie J. Grochow, 3M Report Author
Digitally signed by Chelsie J. Grochow
DN: c=US, st=MN, l=St. Paul, ou=EHS Laboratory, o=3M, cn=Chelsie J. Grochow
Reason: I am the author of this document
Date: 2018.09.17 15:26:37 -05'00'


Susan T. Wolf, 3M Principal Analytical Investigator
Digitally signed by Susan T. Wolf
DN: c=US, st=MN, l=St. Paul, ou=EHS Laboratory, o=3M, cn=Susan T. Wolf
Reason: I have reviewed this document
Date: 2018.09.13 21:48:13 -05'00'


Brian T. Mader, Ph.D., 3M EHS Laboratory Manager
Digitally signed by Brian T. Mader
DN: c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory - authenticated by LRA, o=3M, cn=Brian T. Mader
Reason: I have reviewed this document
Date: 2018.09.14 16:13:22 -05'00'

The 3M EHS Laboratory's Quality Assurance Unit has audited the data and report for this project.


Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M EHS Laboratory.

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
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St. Paul, MN 55144

Phone: [REDACTED]
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Fax: [REDACTED]

Project: ISO18-14-03

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 6/13/2018
Project Description: 3M Antwerp Water Sampling for PFCs; July 2018
Comments:

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

GW = Groundwater

3M Sample Number	Sample Description	D/M/Y		Comment
		Date/Time Sampled	Matrix	
ISO18-14-03-001	BD24-3; Sample ✓ ✓	19/7/18	GW	/
ISO18-14-03-001-DUP	BD24-3; Sample Duplicate ✓ ✓	10/7/18	GW	/
ISO18-14-03-002	BD24-4; Sample ✓ ✓	10/7/18	GW	/
ISO18-14-03-002-DUP	BD24-4; Sample Duplicate ✓ ✓	10/07/18	GW	/
ISO18-14-03-003	D09; Sample ✓ ✓	11/07/18	GW	/
ISO18-14-03-003-DUP	D09; Sample Duplicate ✓ ✓	11/07/18	GW	/
ISO18-14-03-004	D10; Sample ✓ ✓	10/07/18	GW	/
ISO18-14-03-004-DUP	D10; Sample Duplicate ✓ ✓	10/07/18	GW	/
ISO18-14-03-004-FMS	D10; FMS ✓ ✓	10/07/18	GW	/
ISO18-14-03-005	D11; Sample ✓ ✓	12/07/18	GW	/
ISO18-14-03-005-DUP	D11; Sample Duplicate ✓ ✓	12/07/18	GW	/
ISO18-14-03-006	D14; Sample ✓ ✓	11/07/18	GW	/
ISO18-14-03-006-DUP	D14; Sample Duplicate ✓ ✓	11/07/18	GW	/
ISO18-14-03-007	D16; Sample ✓ ✓	11/07/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 40 Deg C Received on Ice Other:

Collected by (print): Elin Kemme (EKE) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	EKE	13/7/18	/	FEDEX	[Signature]	18/07/2018	12:00pm

Page 1 of 11

3M EHS LABORATORY
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Project: ISO18-14-03 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 6/13/2018

Project Description: 3M Antwerp Water Sampling for PFCs; July 2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

D/M/Y GW = Groundwater
Date/Timed Sampled Matrix Comment

3M Sample Number	Sample Description	Date/Timed Sampled	Matrix	Comment
ISO18-14-03-007-DUP .	D16; Sample Duplicate ✓✓	11/07/18	GW	/
ISO18-14-03-008	D17; Sample ✓✓	11/07/18	GW	/
ISO18-14-03-008-DUP	D17; Sample Duplicate ✓✓	11/07/18	GW	/
ISO18-14-03-008-FMS	D17; FMS ✓✓	11/07/18	GW	/
ISO18-14-03-009	D18; Sample ✓✓	11/07/18	GW	/
ISO18-14-03-009-DUP	D18; Sample Duplicate ✓✓	11/07/18	GW	/
ISO18-14-03-010	D2; Sample ✓✓	10/07/18	GW	/
ISO18-14-03-010-DUP	D2; Sample Duplicate ✓✓	10/07/18	GW	/
ISO18-14-03-010-FMS	D2; FMS ✓✓	10/07/18	GW	/
ISO18-14-03-011	D5; Sample ✓✓	10/07/18	GW	/
ISO18-14-03-011-DUP	D5; Sample Duplicate ✓✓	10/07/18	GW	/
ISO18-14-03-012	ND7; Sample ✓✓	11/07/18	GW	/
ISO18-14-03-012-DUP	ND7; Sample Duplicate ✓✓	11/07/18	GW	/
ISO18-14-03-013	P118A; Sample ✓✓	11/07/18	GW	/
ISO18-14-03-013-DUP	P118A; Sample Duplicate ✓✓	11/07/18	GW	/
ISO18-14-03-014	P118B; Sample ✓✓	11/07/18	GW	/
ISO18-14-03-014-DUP	P118B; Sample Duplicate ✓✓	11/07/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Elein Kemme (EKE)

Collector's signature: S. W. S.

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
147	EKE	13/7/18	/	FEDEX	S. W. S.	16 Jul 2018	12:00pm

3M EHS LABORATORY
Chain-of-Custody

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Fax: [REDACTED]

Project: ISO18-14-03 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 6/13/2018

Project Description: 3M Antwerp Water Sampling for PFCs; July 2018

Completion Date: 11/7/18
Project Lead: Susan T. Wolf SW = Surface Water
Phone Number: [REDACTED]
Email Address: [REDACTED]

GW = Groundwater

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO18-14-03-015	P119A; Sample ✓ ✓	11/7/18	GW	/
ISO18-14-03-015-DUP	P119A; Sample Duplicate ✓ ✓	11/7/18	GW	/
ISO18-14-03-015-PMS	P119A; FMS ✓✓	11/7/18	GW	/
ISO18-14-03-016	P119B; Sample ✓ ✓	11/7/18	GW	/
ISO18-14-03-016-DUP	P119B; Sample Duplicate ✓ ✓	11/7/18	GW	/
ISO18-14-03-017	P121; Sample ✓ ✓	10/7/18	GW	/
ISO18-14-03-017-DUP	P121; Sample Duplicate ✓ ✓	10/7/18	GW	/
ISO18-14-03-018	P321; Sample ✓ ✓	9/7/18	GW	/
ISO18-14-03-018-DUP	P321; Sample Duplicate ✓ ✓	9/7/18	GW	/
ISO18-14-03-019	3M vijver; Sample ✓ ✓	9-12/9/18 10/7/18	SW	/
ISO18-14-03-019-DUP	3M vijver; Sample Duplicate ✓ ✓	10/7/18	SW	/
ISO18-14-03-020	Blokkersdijkvijver Noord; Sample ✓ ✓	10/7/18	SW	/
ISO18-14-03-020-DUP	Blokkersdijkvijver Noord; Sample Dup ✓ ✓	10/7/18	SW	/
ISO18-14-03-021	Blokkersdijkvijver standard; Sample ✓ ✓	10/7/18	SW	/
ISO18-14-03-021-DUP	Blokkersdijkvijver standard; Sample Dup ✓ ✓	10/7/18	SW	/
ISO18-14-03-022	L21; Sample ✓ ✓	10/7/18	GW	/
ISO18-14-03-022-DUP	L21; Sample Duplicate ✓ ✓	10/7/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Elien Kamme

Collector's signature:

Item #	Relinquished by:	Date DD/MM/YY	Time	Shipped Via	Received by:	Date DD/MM/YY	Time
17	EKE	13/07/18	/	FEDEX	Susan T. Wolf	16Jul 2018	12:00pm

3M EHS LABORATORY
Chain-of-Custody

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Project: ISO18-14-03 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 6/13/2018

Completion Date:

Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

GW = Ground water
WW = Wastewater

Project Description: 3M Antwerp Water Sampling for PFCs; July 2018

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO18-14-03-023	L22; Sample ✓✓	10/7/18	GW	/
ISO18-14-03-023-DUP	L22; Sample Duplicate ✓✓	10/7/18	GW	/
ISO18-14-03-024	L31; Sample ✓✓	10/7/18	GW	/
ISO18-14-03-024-DUP	L31; Sample Duplicate ✓✓	10/7/18	GW	/
ISO18-14-03-024-FMS	L31; FMS ✓✓	10/7/18	GW	/
ISO18-14-03-025	L4; Sample ✓✓	10/7/18	GW	/
ISO18-14-03-025-DUP	L4; Sample Duplicate ✓✓	10/7/18	GW	/
ISO18-14-03-026	P114bis; Sample ✓✓	10/7/18 10/7/18	GW	/
ISO18-14-03-026-DUP	P114bis; Sample Duplicate ✓✓	10/7/18	GW	/
ISO18-14-03-027	P115; Sample ✓✓	10/7/18	GW	/
ISO18-14-03-027-DUP	P115; Sample Duplicate ✓✓	10/7/18	GW	/
ISO18-14-03-028	P116; Sample ✓✓	10/7/18	GW	/
ISO18-14-03-028-DUP	P116; Sample Duplicate ✓✓	10/7/18	GW	/
ISO18-14-03-028-FMS	P116; FMS ✓✓	10/7/18	GW	/
ISO18-14-03-029	Effluent WWTP; Sample ✓✓	10/7/18	WW	/
ISO18-14-03-029-DUP	Effluent WWTP, Sample Duplicate ✓✓	10/7/18	WW	/
ISO18-14-03-030	PP01; Sample ✓✓	10/7/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Elain Kimmie (EKE) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	EKE	10/7/18	/	FEDEX	Porter D. St.	10/31/2018	12:00pm

3M EHS LABORATORY
Chain-of-Custody

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Fax: ([REDACTED]

Project: ISO18-14-03 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 6/13/2018

Completion Date:

Project Lead: Susan T. Wolf
Phone Number: [REDACTED] GW=Ground Water
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs; July 2018

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO18-14-03-030-DUP	PP01; Sample Duplicate ✓ ✓	10/17/18	GW	/
ISO18-14-03-031	PP02; Sample ✓ ✓	10/17/18	GW	/
ISO18-14-03-031-DUP	PP02; Sample Duplicate ✓ ✓	10/17/18	GW	/
ISO18-14-03-031-FMS	PP02; FMS ✓ ✓	10/17/18	GW	/
ISO18-14-03-032	PP04; Sample ✓ ✓	10/17/18	GW	/
ISO18-14-03-032-DUP	PP04; Sample Duplicate ✓ ✓	10/17/18	GW	/
ISO18-14-03-033	PP05; Sample ✓ ✓	10/17/18	GW	/
ISO18-14-03-033-DUP	PP05; Sample Duplicate ✓ ✓	9-18-17/18 2017/18	GW	/
ISO18-14-03-034	PP06; Sample ✓ ✓	10/17/18	GW	/
ISO18-14-03-034-DUP	PP06; Sample Duplicate ✓ ✓	10/17/18	GW	/
ISO18-14-03-035	PP07; Sample ✓ ✓	10/17/18	GW	/
ISO18-14-03-035-DUP	PP07; Sample Duplicate ✓ ✓	10/17/18	GW	/
ISO18-14-03-035-FMS	PP07; FMS ✓ ✓	10/17/18	GW	/
ISO18-14-03-036	PP08; Sample ✓ ✓	10/17/18	GW	/
ISO18-14-03-036-DUP	PP08; Sample Duplicate ✓ ✓	10/17/18	GW	/
ISO18-14-03-037	PP09; Sample ✓ ✓	10/17/18	GW	/
ISO18-14-03-037-DUP	PP09; Sample Duplicate ✓ ✓	10/17/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Elien Kremme (EKE) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	EKE	10/17/18	/	FEDEX	Guburu. Sh	10/17/2018	12:00pm

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Project: ISO18-14-03 (cont.)

Phone: [REDACTED]
Alt. Phone: [REDACTED]
Fax: (651) 777-2100

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 6/13/2018

Completion Date:

GW = Groundwater
SW = Surface Water
WW = Waste Water

Project Description: 3M Antwerp Water Sampling for PFCs; July 2018

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
		D/M/Y		
ISO18-14-03-038	PP10, Sample ✓ ✓	10/7/18	GW	/
ISO18-14-03-038-DUP	PP10, Sample Duplicate ✓ ✓	10/7/18	GW	/
ISO18-14-03-039	12; Sample ✓ ✓	10/7/18	SW	/
ISO18-14-03-039-DUP	12; Sample Duplicate ✓ ✓	10/7/18	SW	/
ISO18-14-03-039-FMS	12; FMS ✓ ✓	9/7/18	SW	/
ISO18-14-03-040	13; Sample ✓ ✓	9/7/18	SW	/
ISO18-14-03-040-DUP	13; Sample Duplicate ✓ ✓	9/7/18	GW	/
ISO18-14-03-041	5; Sample ✓ ✓	9/7/18	SW	/
ISO18-14-03-041-DUP	5; Sample Duplicate ✓ ✓	9/7/18	SW	/
ISO18-14-03-042	Bemalingsstation; Sample ✓ ✓	9/7/18	SW	/
ISO18-14-03-042-DUP	Bemalingsstation; Sample Duplicate ✓ ✓	9/7/18	SW	/
ISO18-14-03-043	Collector put; Sample ✓ ✓	10/7/18	WW	/
ISO18-14-03-043-DUP	Collector put; Sample Duplicate ✓ ✓	10/7/18	WW	/
ISO18-14-03-043-FMS	Collector put; FMS ✓ ✓	10/7/18	WW	/
ISO18-14-03-044	K3; Sample ✓ ✓	11/7/18	GW	/
ISO18-14-03-044-DUP	K3; Sample Duplicate ✓ ✓	11/7/18	GW	/
ISO18-14-03-044-FMS	K3; FMS ✓ ✓	11/7/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print):

Collector's signature:

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	EKE	13/7/18	/	FEDEX	Susan T. Wolf	16Jul2018	12:00pm

3M EHS LABORATORY
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Project: ISO18-14-03 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)

Completion Date:

Department: 832202 Site Source: 01WJWT10

Project Lead: Susan T. Wolf

Project Number:

Phone Number: [REDACTED]

Date Created: 6/13/2018

Email Address: [REDACTED]

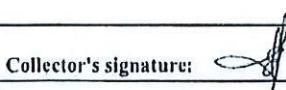
GW=Ground Water

Project Description: 3M Antwerp Water Sampling for PFCs; July 2018

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO18-14-03-045	P27; Sample ✓ ✓	11/7/18	GW	/
ISO18-14-03-045-DUP	P27; Sample Duplicate ✓ ✓	11/7/18	GW	/
ISO18-14-03-046	P21B; Sample ✓ ✓	12/07/18	GW	/
ISO18-14-03-046-DUP	P21B; Sample Duplicate ✓ ✓	12/07/18	GW	/
ISO18-14-03-047	P304; Sample ✓ ✓	11/7/18	GW	/
ISO18-14-03-047-DUP	P304; Sample Duplicate ✓ ✓	11/7/18	GW	/
ISO18-14-03-048	P305; Sample ✓ ✓	12/7/18	GW	/
ISO18-14-03-048-DUP	P305; Sample Duplicate ✓ ✓	12/7/18	GW	/
ISO18-14-03-049	P42; Sample ✓ ✓	12/7/18	GW	/
ISO18-14-03-049-DUP	P42; Sample Duplicate ✓ ✓	11/07/18	GW	/
ISO18-14-03-050	P56; Sample ✓ ✓	11/07/18	GW	/
ISO18-14-03-050-DUP	P56; Sample Duplicate ✓ ✓	11/07/18	GW	/
ISO18-14-03-050-FMS	P56; FMS ✓ ✓	11/07/18	GW	/
ISO18-14-03-051	L19; Sample ✓ ✓	11/07/18	GW	/
ISO18-14-03-051-DUP	L19; Sample Duplicate ✓ ✓	11/07/18	GW	/
ISO18-14-03-052	M4; Sample ✓ ✓	09/07/18	GW	/
ISO18-14-03-052-DUP	M4; Sample Duplicate ✓ ✓	09/07/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 16 Deg C Received on Ice Other:

Collected by (print): Elien Kemme (EKE) Collector's signature: 

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	Elien Kemme	13/7/18	/	FEDEX	Sahni, S.	16/07/2018	12:04pm

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [REDACTED]
Alt. Ph: [REDACTED]
Fax: [REDACTED]

Project: ISO18-14-03 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)

Department: 832202 Site Source: 01WJWT10

Project Number:

Date Created: 6/13/2018

Project Description: 3M Antwerp Water Sampling for PFCs; July 2018

Completion Date:

Project Lead: Susan T. Wolf GW = Ground Water

Phone Number: [REDACTED]

Email Address: [REDACTED]

3M Sample Number

Sample Description

Date/Time Sampled Matrix Comment

D/M/Y

ISO18-14-03-053	P118C; Sample ✓ ✓	11/07/18	GW	/
ISO18-14-03-053-DUP	P118C; Sample Duplicate ✓ ✓	11/07/18	GW	/
ISO18-14-03-054	P119C; Sample ✓ ✓	11/07/18	GW	/
ISO18-14-03-054-DUP	P119C; Sample Duplicate ✓ ✓	11/07/18	GW	/
ISO18-14-03-054-FMS	P119C; FMS ✓ ✓	11/07/18	GW	/
ISO18-14-03-055	P262; Sample ✓ ✓	12/07/18	GW	/
ISO18-14-03-055-DUP	P262; Sample Duplicate ✓ ✓	12/07/18	GW	/
ISO18-14-03-056	P263; Sample ✓ ✓	12/07/18	GW	/
ISO18-14-03-056-DUP	P263; Sample Duplicate ✓ ✓	12/07/18	GW	/
ISO18-14-03-057	P264; Sample ✓ ✓	11/17/18	GW	/
ISO18-14-03-057-DUP	P264; Sample Duplicate ✓ ✓	11/17/18	GW	/
ISO18-14-03-058	P265C; Sample ✓ ✓	11/17/18	GW	/
ISO18-14-03-058-DUP	P265C; Sample Duplicate ✓ ✓	11/17/18	GW	/
ISO18-14-03-058-FMS	P265C; FMS ✓ ✓	11/17/18	GW	/
ISO18-14-03-059	P340; Sample ✓ ✓	09/07/18	GW	/
ISO18-14-03-059-DUP	P340; Sample Duplicate ✓ ✓	09/07/18	GW	/
ISO18-14-03-059-FMS	P340; FMS ✓ ✓	09/07/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 40 Deg C Received on Ice Other:

Collected by (print): Elein Kromme (EKE) Collector's signature: [Signature]

Item #	Relinquished by:	Date D/M/Y	Time	Shipped Via	Received by:	Date D/M/Y	Time
17	EKE	13/7/18	/	FEDEX	Susan T. Wolf	16/7/2018	12:09pm

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
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St. Paul, MN 55144

Phone: [REDACTED]
Alt. Pho: [REDACTED]
Fax: (651) 777-4222

Project: ISO18-14-03 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)

Completion Date:

GW = Ground Water

Department: 832202 Site Source: 01WJWT10

Project Lead: Susan T. Wolf

Project Number:

Phone Number:

Date Created: 6/13/2018

Email Address:

Project Description: 3M Antwerp Water Sampling for PFCs; July 2018

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO18-14-03-060	P341; Sample ✓ ✓	09/07/18	GW	/
ISO18-14-03-060-DUP	P341; Sample Duplicate ✓ ✓	09/07/18	GW	/
ISO18-14-03-061	P343; Sample ✓ ✓	12/07/18	GW	/
ISO18-14-03-061-DUP	P343; Sample Duplicate ✓ ✓	12/07/18	GW	/
ISO18-14-03-062	P371; Sample ✓ ✓	12/07/18	GW	/
ISO18-14-03-062-DUP	P371; Sample Duplicate ✓ ✓	12/07/18	GW	/
ISO18-14-03-063	P374; Sample ✓ ✓	12/07/18	GW	/
ISO18-14-03-063-DUP	P374; Sample Duplicate ✓ ✓	12/07/18	GW	/
ISO18-14-03-064	P379; Sample ✓ ✓	12/07/18	GW	/
ISO18-14-03-064-DUP	P379; Sample Duplicate ✓ ✓	12/07/18	GW	/
ISO18-14-03-065	P380; Sample ✓ ✓	12/07/18	GW	/
ISO18-14-03-065-DUP	P380; Sample Duplicate ✓ ✓	12/07/18	GW	/
ISO18-14-03-066	P381; Sample ✓ ✓	12/07/18	GW	/
ISO18-14-03-066-DUP	P381; Sample Duplicate ✓ ✓	12/07/18	GW	/
ISO18-14-03-066-FMS	P381; FMS ✓ ✓	12/07/18	GW	/
ISO18-14-03-067	P382; Sample ✓ ✓	11/07/18	GW	/
ISO18-14-03-067-DUP	P382; Sample Duplicate ✓ ✓	11/07/18	GW	/

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Elien Kemme (EKE) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	EKE	13/7/18	/	FEDEX	<u>S. M. W. S.</u>	16 Jul 2018	10:00pm

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Project: ISO18-14-03 (cont.)

Phone [REDACTED]
Alt. Ph [REDACTED]
Fax: [REDACTED]

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 6/13/2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs; July 2018

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO18-14-03-068	B3-bis; Sample ✓ ✓		/	NO SAMPLE
ISO18-14-03-068-DUP	B3-bis; Sample Duplicate ✓ ✓		/	NO SAMPLE
ISO18-14-03-068-FMS	B3-bis; FMS ✓ ✓		/	NO SAMPLE
ISO18-14-03-069	B7; Sample ✓ ✓		/	NO SAMPLE
ISO18-14-03-069-DUP	B7; Sample Duplicate ✓ ✓		/	NO SAMPLE
ISO18-14-03-070	P372; Sample ✓ ✓	12/7/18	GW	/
ISO18-14-03-070-DUP	P372; Sample Duplicate ✓ ✓	12/7/18	GW	/
ISO18-14-03-071	P378; Sample ✓ ✓	9-12/7/18 11/7/18	GW	/
ISO18-14-03-071-DUP	P378; Sample Duplicate ✓ ✓	11/7/18	GW	/
ISO18-14-03-071-FMS	P378; FMS ✓ ✓	11/7/18	GW	/
ISO18-14-03-072	PA109A; Sample ✓ ✓	11/07/18	GW	/
ISO18-14-03-072-DUP	PA109A; Sample Duplicate ✓ ✓	11/07/18	GW	/
ISO18-14-03-073	PA111A; Sample ✓ ✓	11/07/18	GW	/
ISO18-14-03-073-DUP	PA111A; Sample Duplicate ✓ ✓	11/07/18	GW	/
ISO18-14-03-074	PA112; Sample ✓ ✓	11/7/18	GW	/
ISO18-14-03-074-DUP	PA112; Sample Duplicate ✓ ✓	11/7/18	GW	/
ISO18-14-03-075	Travel Blank ✓	6/18/18 C.J.G	/	*

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Item #	Collected by (print):	Collector's signature:	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	EKE			13/7/18	/	FEDEX	R.W.G.	16/7/2018	12:00pm

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
 3M EHS Laboratory
 3M Center, Bldg 280-6N-17
 St. Paul, MN 55144

Phone: [REDACTED]
 Alt. Ph: [REDACTED]
 Fax: [REDACTED]

Project: ISO18-14-03 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
 Department: 832202 Site Source: 01WJWT10
 Project Number:
 Date Created: 6/13/2018

Completion Date:
 Project Lead: Susan T. Wolf
 Phone Number: [REDACTED]
 Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PPCs; July 2018

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO18-14-03-075-FMS-HIGH	Travel Blank FMS High	✓	(6)18/18 C3G	*
ISO18-14-03-075-FMS-LOW	Travel Blank FMS Low	✓	(6)18/18 C3G	*

* Travel blank samples prepared by the 3M EHS Laboratory with the bottle order.

Sample Condition Upon Receipt: Acceptable All items accounted for
 Temperature: 10 Deg C Received on Ice Other:

Collected by (print): <u>Elien Kemme</u>		Collector's signature: <u>[Signature]</u>					
Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
2	EKE	13/7/18	/	FEDEX	<u>Julia QD</u>	16.1.2018	12:00pm

Page 11 of 11



3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC18-05733-002

3M Lab Request Number: E18-0748

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: November 19th, 2018

Requester

Jim Kotsmith
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Member of the SGS Group (Société Générale de Surveillance)

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3412 5594. All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction - Summary.

At the request (Lab Request Number: E18-0748) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, one water sample was collected in October 2018 from 1 location and analyzed for the following perfluorinated compounds:

- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$) (and its ^{13}C -labeled analogues)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$) (and its ^{13}C -labeled analogues)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$) (and its ^{13}C -labeled analogues)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$) (and its ^{13}C -labeled analogue)
- ^{13}C -labeled analogue of PFUdA ($C_4F_{21}^{13}C_6F_2^{13}COO^-$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared one sample container for each sampling location under the direction of Sven Herremans. Each empty container was marked with a "fill to here" line and was fortified with a surrogate recovery spike and an internal standard spike, prior to being sent to the field for sample collection.

Table 1 summarizes the sample results with their uncertainty. All PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$ unless noted otherwise. See Section 4 of the report for additional information on the method QC used to assess method uncertainty.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)			
	PFHS	PFOA	PFOS	FOSA
3M vijver	0.578	1.83	4.30	0.030 (a)

(a): The recovery of FOSA for 3M vijver is $\pm 70\%$.

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

The sample was collected in a polyethylene bottle prepared at the SGS Belgium NV, Division IAC Laboratory. Based on the concentrations as reported in previous reports, the bottle was spiked, prior to sample collection, in the laboratory with a known volume of a surrogate recovery solution ($^{13}\text{C}_2\text{-PFHS}$, $^{13}\text{C}_4\text{-PFOA}$, $^{13}\text{C}_4\text{-PFOS}$, $^{13}\text{C}_7\text{-PFUdA}$) and internal standard solution ($^{13}\text{C}_3\text{-PFHS}$, $^{13}\text{C}_8\text{-PFOA}$, $^{13}\text{C}_8\text{-PFOS}$, $^{13}\text{C}_8\text{-FOSA}$). Table 2 below details the sample collected and spikes added to the bottle.

Table 2. Sample Collection and Spike Information.

Sample Identification	Nominal Final Volume Collected (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Final Nominal Spike Concentration (ng/mL)	
				$^{13}\text{C-PFC-SS}$	$^{13}\text{C-PFC-IS}$
3M vijver	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0

Solution A = 1000 ng/mL (nominal) $^{13}\text{C-PFC}$ Surrogate Recovery Standards

Solution B = 1500 ng/mL (nominal) $^{13}\text{C-PFC}$ Internal Standards

2.2. Extraction.

All samples, calibration standards, and associated quality control samples were extracted using a modified procedure of ECO/AV/IAC/064 “Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)”. Briefly, an amount of sample (see table 3) was loaded, onto a pre-conditioned Waters tC18 solid phase extraction (SPE) cartridge (Sep-Pak, 1g, 6cc) using a vacuum manifold. The loaded SPE cartridges were then eluted with 5 mL of methanol using vacuum.

Table 3. Sample amount used.

Sample	Amount of Sample used (mL)	Concentration Factor
3M vijver	40	8
Field Trip Blank	40	8

2.3. Determination of suspended solids in water.

No suspended solids

2.4. Analysis.

All solutions and extracts were analyzed for the PFCs (PFHS, PFOA, PFOS, FOSA) and the surrogate recovery standards ($^{13}\text{C}_2\text{-PFHS}$, $^{13}\text{C}_4\text{-PFOA}$, $^{13}\text{C}_4\text{-PFOS}$, $^{13}\text{C}_7\text{-PFUdA}$) and internal standards ($^{13}\text{C}_3\text{-PFHS}$, $^{13}\text{C}_8\text{-PFOA}$, $^{13}\text{C}_8\text{-PFOS}$, $^{13}\text{C}_8\text{-FOSA}$) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetrie C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
$^{13}\text{C}_3\text{-PFHS}$	2.00 - 3.00	402.0 /80.0	60	38	51
$^{13}\text{C}_2\text{-PFHS}$	2.30 - 3.30	403.0/84.0	60	38	51
$^{13}\text{C}_8\text{-PFOS}$	3.00 - 4.00	507.0/80.0	60	48	56
$^{13}\text{C}_4\text{-PFOA}$	2.30 - 3.50	417.0 /372.0	100	11	14
$^{13}\text{C}_8\text{-PFOA}$	2.30 - 3.50	421.0 /376.0	60	11	14
PFHS	2.00 - 3.00	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.30 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
		498.78/98.73	50	39	56
¹³ C ₄ -PFOS	3.00 - 4.00	503.0 /80.0	60	48	56
¹³ C ₇ PFUdA	4.30 - 5.60	570.0 /525.0	60	12	17
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
¹³ C ₈ FOSA	4.30 - 5.70	506.0/78.0	60	34	44

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Extracted Calibration Standard.

Extracted calibration standards were prepared by spiking known amounts of stock solutions containing PFHS, PFOA, PFOS, FOSA and ^{13}C -labeled analogues into 40 mL of HPLC water. Each spiked water standard was then extracted in the same manner as the collected samples. A total of 12 spiked standards ranging from 0.005 ng/mL to 100 ng/mL (nominal) were prepared. Each curve point contains the mixture of internal standards at a nominal concentration of 1 ng/mL. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. The calibration curve will be generated by taking the ratio of the standard peak area counts over the internal standard peak area counts to fit the data for each analyte. Each extracted calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of $\pm 25\%$ ($\pm 30\%$ for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The 0.025ng/mL (nominal) reporting limit is a practical quantitation limit (PQL) required by the requester and it is possible that the samples contain target analytes at quantifiable concentrations below the PQL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within $\pm 25\%$, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by loading 40 mL of HPLC water onto a Waters tC18 solid phase extraction (SPE) cartridge (SEP-Pak, 1g, 6cc) and eluting with 5 mL of methanol using the same extraction procedure as the samples. Method blanks were prepared to evaluate the levels of background contamination in the overall extraction process (glassware, SPE cartridges, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carry over.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of HPLC water, spiked with the surrogate recovery standards and internal standards, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCSs)

Low (0.125 ng/mL nominal concentration) and high (2.5 ng/mL nominal concentration) lab control spikes were prepared and analyzed in duplicate. LCSs were prepared by spiking known amounts of the analytes and surrogates into 40 mL of HPLC water to produce the desired concentration. The spiked water samples were extracted and analyzed in the same manner as the samples.

All LCSs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

All LCSs produced recoveries within the method acceptance criteria of $\pm 15\%$ RPD for precision, except for PFOS LCSH.

Table 8 summarize the LCS recovery results.

Table 7. Lab Control Spike Results.

Extraction date	Description	Nominal Spike Level (ng/mL)	Percent Recovery							
			¹³ C ₄ -PFOA	¹³ C ₄ -PFOS	¹³ C ₂ -PFHS	¹³ C ₇ -PFuDA	PFHS	PFOA	PFOS	FOSA
8 November 2018	LCSL01	0.125	95.6	102	99.8	82.9	90.1	96.3	95.5	90.4
	LCSL02	0.125	88.1	97.7	99.8	84.1	91.6	106	91.2	85.1
	Average		91.9	100	99.8	83.5	90.9	101	93.4	87.8
	%RPD		8.2	4.3	0.0	1.4	1.7	9.1	4.6	6.0
	LCSH 01	2.5	97.4	107	102	92.9	91.4	98.8	98.2	91.2
	LCSH 02	2.5	106	106	108	91.5	102	117	114	103
	Average		101	106	105	92.2	96.6	108	106	97.1
	%RPD		8.0	0.8	5.0	1.5	11	17 (a)	15	12

(a) The recovery of the RPD fell outside the method acceptance criterion of $\pm 15\%$.

3.6. Surrogates.

Surrogate recovery standards were added to all samples to evaluate overall method performance.

3.7. Internal Standards.

Internal standards were added to all samples to calculate the concentration of PFCs in the samples by using internal standard calibration.

3.8. Equations.

Recovery.

$$\text{Recovery (\%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

Tabel 8 and 9 summarizes the results for the sample locations.

Table 8. Sample Results PFHS and PFOA.

	PFHS ng/ml	$^{13}\text{C}_2\text{-PFHS}$ %Rec	PFOA ng/ml	$^{13}\text{C}_4\text{-PFOA}$ %Rec
Field Trip Blank	<0.025	96.0	<0.025	88.3
3M vijver	0.578	83.3	1.83	89.6

Table 9. Sample Results PFOS and FOSA.

	PFOS ng/ml	$^{13}\text{C}_4\text{-PFOS}$ %Rec	FOSA ng/ml	$^{13}\text{C}_7\text{-PFuDA}$ %Rec
Field Trip Blank	<0.025	87.4	<0.025	75.2
3M vijver	4.30	80.3	0.030	30.7 (a)

(a): The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130%.

All PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$, except for FOSA for the following sample locations:

(a): The recovery of FOSA for 3M vijver is $\pm 70\%$.

5. Conclusion.

- The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130% for one sample location.
- Lab control spike recoveries fell within the method acceptance criteria of 25%.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Elien Kemme (ERM Belgium).

8. Signatures.

Sven Herremans,
Lab Operations Manager

Date November 19th, 2018



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date November 19th, 2018

I.A.C.
A Division of SGS Belgium NV

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The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.



3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC18-05733-001

3M Lab Request Number: E18-0748

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: November 19th, 2018

Requester

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1. Introduction - Summary.

At the request (Lab Request Number: E18-0748) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected in October 2018 from 11 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOA ($C_6F_{13}^{13}CF_2^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOS ($C_7F_{15}^{13}CF_2SO_3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Sven Herremans. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles.

Table 1 summarizes the sample results. All PFBS, PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$ unless noted otherwise. See section 4 of the report for additional information on the method QC used to assess method uncertainty.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
L4		2.01	3.59	12.0 (a)	<0.203
P116		0.689	1.33	3.27	<0.203
Effluent WWTP		14.3	16.2	8.56 (b)	<0.203
PP01	108	17285	48.8	4718	15.0
PP04	1228	193	525	3363	<28.7
PP06	12.0	38.7	126	549	<4.06
PP08	26.5	241	401	4000	<14.9
PP10	17.9	194	568	2581	3.33
13		22.1	66.3	113	<1.35
Bemalingsstation		26.6	28.8	14.2	<0.372
Collector Put		19.5	54.3	110	1.99

(a): The recovery of PFOS for L4 is $\pm 35\%$.

(b): The recovery of PFOS for Effluent WWTP is $\pm 35\%$.

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
L4	100
P116	100
Effluent WWTP	100
PP01	100
PP04	100
PP06	100
PP08	100
PP10	100
13	100
Bemalingsstation	100
Collector Put	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
L4	1.0	0.05	Solution A	1.0	-
P116	1.0	0.05	Solution A	1.0	-
Effluent WWTP	1.0	0.05	Solution A	1.0	-
PP01	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP04	1.0	0.2	Solution A	10.0	0.5mL extract + 10.0mL MeOH (*)
PP06	1.0	0.1	Solution A	10.0	1.0mL extract + 2.0mL MeOH (*)
PP08	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP10	1.0	0.1	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
13	1.0	0.1	Solution A	10.0	-
Bemalingsstation	1.0	0.05	Solution A	2.0	-
Collector Put	1.0	0.1	Solution A	10.0	-

(*): MeOH:LCMS-water (60:40)
 Solution A = 1000 ng/mL (nominal) ^{13}C -PFC Surrogate Standards

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ^{13}C -PFOA, PFOS, FOSA and ^{13}C -PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetre C18 50 x 2.1mm, 3.5 μm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7 μm
Injection volume	5 μL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate ($\mu\text{L}/\text{min}$)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5

7	6.50	400	95	5
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Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
PFBS	2.08 - 2.50	298.93/79.82	40	28	45
		298.93/98.78	40	29	45
PFHS	2.45 - 3.05	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.80 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
¹³ C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of \pm 25% (\pm 30% for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.099 to 0.121 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.203 to 28.5 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within \pm 25%, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by diluting 1 mL water with 10.0 mL MeOH:LCMS-water (60:40). Method blanks were prepared to evaluate the levels of background contamination in the overall dilution process (glassware, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carryover.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of LCMS-water, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCS)

One lab control spike was prepared and analyzed.

LCS was prepared by spiking known amounts of the analytes into 1.0 mL of LCMS-water to produce the desired concentration.

The spiked water sample was diluted with 10.0 mL MeOH:LCMS-water (60:40) and analyzed in the same manner as the samples.

The LCS produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 7 summarizes the LCS recovery results

Table 7. Lab Control Spike Results.

Dilution date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
08 November 2018	LCS1	80	84.7	87.2	86.3	90.5	99.4	90.9	91.4

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing PFBS, PFHS, PFOA, ^{13}C -PFOA, PFOS, FOSA and ^{13}C -PFOS (QC Lab Low and QC Lab High).

The QCs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 8 summarizes the QCs recovery results.

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
25 Jan 2018	QC Lab Low inj1	10	87.1	78.7	89.8	124	92.7	93.3	114
	QC Lab High inj1	100	89.8	80.5	99.8	103	99.3	92.8	79.1
	QC Lab Low inj2	10	90.1	83.5	88.3	90.8	95.8	95.2	108
	QC Lab High inj2	100	82.0	75.3	91.2	95.1	94.7	87.6	80.8

3.7. Equations.Recovery.

$$\text{Recovery (\%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

The following tables summarize the results for the sample locations.

Table 9. Sample Results PFBS, PFHS, PFOA, PFOS and FOSA

	PFBS	PFHS	PFOA	PFOS	FOSA
	ng/ml	ng/ml	ng/ml	ng/ml	ng/ml
L4		2.01	3.59	12.0	<0.203
P116		0.689	1.33	3.27	<0.203
Effluent WWTP		14.3	16.2	8.56	<0.203
PP01	108	17285	48.8	4718	15.0
PP04	1228	193	525	3363	<28.7
PP06	12.0	38.7	126	549	<4.06
PP08	26.5	241	401	4000	<14.9
PP10	17.9	194	568	2581	3.33
13		22.1	66.3	113	<1.35
Bemalingsstation		26.6	28.8	14.2	<0.372
Collector Put		19.5	54.3	110	1.99
Field Trip Blank	<1.17	<1.25	<1.34	<1.35	<1.10

Table 10. Sample Results ¹³C-PFOA and ¹³C-PFOS

	¹³ C-PFOA		¹³ C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
L4	70.6	123	41.0	66.6*
P116	69.3	121	44.2	71.8
Effluent WWTP	69.5	121	41.8	67.9*
PP01	118	103	129	105
PP04	252	110	245	99.6
PP06	111	96.9	135	110
PP08	104	90.5	127	103
PP10	134	117	109	88.5
13	109	95.4	137	111
Bemalingsstation	65.3	114	56.0	90.9
Collector Put	109	95.1	137	112
Field Trip Blank	105	91.8	145	118

* fell outside the method acceptance criterion of 70-130%

5. Conclusion.

- The recovery of ¹³C-PFOA and ¹³C-PFOS fell within the method acceptance criterion of 70-130%, except for two samples.
- Lab control spike recoveries fell within the method acceptance criteria for all compounds, indicating that the analysis method is in control,
- QC Lab Standards recoveries fell within the method acceptance criteria for all compounds.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Elien Kemme (ERM Belgium).

8. Signatures.

Sven Herremans,
Lab Operations Manager

Date November 19th, 2018



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date November 19th, 2018

I.A.C.
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The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.

Final Report

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

October 2018 Sampling

Laboratory Request Number: ISO18-14-04

Report Date – Date of Last Signature

Testing Laboratory

3M Environment, Health, Safety & Medical
3M EHS Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Nicole Cauberghe
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3M Belgium; ZW018/0/33
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The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M EHS Laboratory

3M EHS Laboratory Manager: Brian T. Mader
3M Principal Analytical Investigator: Susan Wolf
Report Author: Chelsie Grochow

Analytical Report ISO18-14-04

Water Sample Analysis at 3M Antwerp, Belgium
October 2018 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M EHS Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. All samples were collected on October 23-25, 2018, and returned to the 3M EHS Laboratory on October 29, 2018, at ambient temperature. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS) (select locations only), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO18-14-04.

The 3M EHS Laboratory prepared sample containers for thirty-seven sampling locations. Each sample set consisted of a field sample and field sample duplicate. Eleven locations also included a target analyte field matrix spike. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with a mass-labeled internal standard mix and select sample bottles were fortified with surrogate recovery standards [$^{13}\text{C}_4$]-PFOA and [$^{13}\text{C}_4$]-PFOS prior to being sent to the field for sample collection. During sample collection, several sample locations were not sampled including: PP01, K3, P56, P41, PB402, P219, P40, P300, P1065, P215, and P216.

Samples were prepared and analyzed using method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Select internal standards were used to aid in the data quality objectives.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary ⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA
Zone: Blokkersdijk Nature Reserve					
ISO18-14-04-001	3M vijver; Sample	1.65	0.698	4.82	0.0374
ISO18-14-04-001-DUP	3M vijver; Sample Dup	1.78	0.732	5.06	0.0348
		Average	1.72	0.715	4.94
		%RPD Sample/Sample Dup	7.6	4.8	0.0361
ISO18-14-04-002	Blokkersdijkvijver Noord; Sample	1.12	0.644	1.04	0.0352
ISO18-14-04-002-DUP	Blokkersdijkvijver Noord; Sample Dup	1.07	0.626	1.03	0.0318
		Average	1.10	0.635	1.04
		%RPD Sample/Sample Dup	4.6	2.8	0.0335
ISO18-14-04-003	Blokkersdijkvijver - standard; Sample	1.16	0.740	1.07	0.0538
ISO18-14-04-003-DUP	Blokkersdijkvijver - standard; Sample Dup	1.29	0.794	1.18	0.0640
		Average	1.23	0.767	1.13
		%RPD Sample/Sample Dup	11	7.0	0.0589
ISO18-14-04-004	P321; Sample	2960	750	5970	0.206
ISO18-14-04-004-DUP	P321; Sample Dup	2970	777	6060	0.200
		Average	2970⁽²⁾	764⁽²⁾	6020⁽²⁾
		%RPD Sample/Sample Dup	0.34	3.5	1.5
ISO18-14-04-005	L21; Sample	0.174	0.0916	6.66	0.0816
ISO18-14-04-005-DUP	L21; Sample Dup	0.175	0.0960	5.18	0.0616
		Average	0.175	0.0938	5.92
		%RPD Sample/Sample Dup	0.57	4.7	0.0716
				25⁽³⁾	28⁽³⁾
ISO18-14-04-006	L22; Sample	0.402	0.256	2.56	0.0652
ISO18-14-04-006-DUP	L22; Sample Dup	0.392	0.236	2.48	0.0528
		Average	0.397	0.246	2.52
		%RPD Sample/Sample Dup	2.5	8.1	0.0590
				3.2	21⁽³⁾
ISO18-14-04-007	L31; Sample	0.282	0.326	7.40	0.0746
ISO18-14-04-007-DUP	L31; Sample Dup	0.280	0.316	7.16	0.0686
		Average	0.281	0.321	7.28
		%RPD Sample/Sample Dup	0.71	3.1	0.0716
				3.3	8.4
ISO18-14-04-008	L4; Sample	2.38	2.04	28.4	0.206
ISO18-14-04-008-DUP	L4; Sample Dup	2.40	2.02	28.0	0.204
		Average	2.39	2.03	28.2
		%RPD Sample/Sample Dup	0.84	0.99	0.205
				1.4	0.98
ISO18-14-04-009	P114bis; Sample	3.24	1.44	9.60	0.0570
ISO18-14-04-009-DUP	P114bis; Sample Dup	3.22	1.45	9.86	0.0608
		Average	3.23	1.45	9.73
		%RPD Sample/Sample Dup	0.62	0.69	0.0589
				2.7	6.5

- (1) Unless noted otherwise, samples were reported using internal standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 18%, PFHS \pm 11%, PFOS \pm 13%, and PFOSA \pm 25%.
- (2) Sample set reported using external standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 18%, PFBS \pm 16%, PFHS \pm 14%, and PFOS \pm 22%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of $\leq 20\%$.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA
Zone: Blokkersdijk Nature Reserve					
ISO18-14-04-010	P115; Sample	4.08	2.18	1.45	0.0398
ISO18-14-04-010-DUP	P115; Sample Dup	4.20	2.18	1.43	0.0320
		Average	4.14	2.18	1.44
		%RPD Sample/Sample Dup	2.9	0.0	22 ⁽³⁾
ISO18-14-04-011	P116; Sample	0.922	0.474	7.94	0.0828
ISO18-14-04-011-DUP	P116; Sample Dup	1.08	0.576	9.88	0.0982
		Average	1.00	0.525	8.91
		%RPD Sample/Sample Dup	16	19	22 ⁽³⁾
Zone: Effluent WWTP					
ISO18-14-04-012	Effluent WWTP; Sample	13.6	14.4	28.8	0.466
ISO18-14-04-012-DUP	Effluent WWTP; Sample Dup	12.4	13.5	27.8	0.458
		Average	13.0	14.0	28.3
		%RPD Sample/Sample Dup	9.2	6.5	0.462
Zone: Palingbeek & Tophatgracht					
ISO18-14-04-022	12; Sample	110	30.2	364	0.262
ISO18-14-04-022-DUP	12; Sample Dup	108	29.6	346	0.268
		Average	109	29.9	355 ⁽²⁾
		%RPD Sample/Sample Dup	1.8	2.0	0.265
ISO18-14-04-023	13; Sample	97.0	34.6	284	0.256
ISO18-14-04-023-DUP	13; Sample Dup	84.6	33.0	277	0.252
		Average	90.8	33.8	281 ⁽²⁾
		%RPD Sample/Sample Dup	14	4.7	0.254
ISO18-14-04-024	5; Sample	36.0	37.2	53.4	0.137
ISO18-14-04-024-DUP	5; Sample Dup	36.2	35.6	51.4	0.138
		Average	36.1	36.4	52.4
		%RPD Sample/Sample Dup	0.55	4.4	0.138
ISO18-14-04-025	Bemalingsstation; Sample	38.0	38.2	53.0	0.131
ISO18-14-04-025-DUP	Bemalingsstation; Sample Dup	33.8	34.8	46.2	0.111
		Average	35.9	36.5	49.6
		%RPD Sample/Sample Dup	12	9.3	0.121
Zone: Sewer					
ISO18-14-04-026	Collector put; Sample	73.1	30.6	287	18.8
ISO18-14-04-026-DUP	Collector put; Sample Dup	70.6	28.9	281	19.3
		Average	71.9 ⁽²⁾	29.8 ⁽²⁾	284 ⁽²⁾
		%RPD Sample/Sample Dup	3.5	5.7	19.1
					2.1
					2.6

- (1) Unless noted otherwise, samples were reported using internal standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 18%, PFHS \pm 11%, PFOS \pm 13%, and PFOSA \pm 25%.
- (2) Sample set reported using external standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 18%, PFBS \pm 16%, PFHS \pm 14%, and PFOS \pm 22%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of $\leq 20\%$.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
Zone: Extraction Wells P&T						
ISO18-14-04-014	PP02; Sample	703	43.7	4810	22500	28.0
ISO18-14-04-014-DUP	PP02; Sample Dup	668	36.3	4650	21500	29.2
		Average	686 ⁽²⁾	40.0 ⁽²⁾	4730 ⁽²⁾	22000 ⁽²⁾
		%RPD Sample/Sample Dup	5.1	19	3.4	4.5
ISO18-14-04-015	PP04; Sample	668	1540	300	5170	56.4
ISO18-14-04-015-DUP	PP04; Sample Dup	678	1580	296	4760	61.0
		Average	673 ⁽²⁾	1560 ⁽²⁾	298 ⁽²⁾	4970 ⁽²⁾
		%RPD Sample/Sample Dup	1.5	2.6	1.3	8.3
ISO18-14-04-016	PP05; Sample	735	6510	3280	290	10.8
ISO18-14-04-016-DUP	PP05; Sample Dup	738	6660	3320	229	10.4
		Average	737 ⁽²⁾	6590 ⁽²⁾	3300 ⁽²⁾	260 ⁽²⁾
		%RPD Sample/Sample Dup	0.41	2.3	1.2	24 ⁽³⁾
ISO18-14-04-017	PP06; Sample	150	16.8	52.1	1040	53.2
ISO18-14-04-017-DUP	PP06; Sample Dup	154	16.9	53.1	1050	52.0
		Average	152 ⁽²⁾	16.9 ⁽²⁾	52.6 ⁽²⁾	1050 ⁽²⁾
		%RPD Sample/Sample Dup	2.6	0.59	1.9	0.96
ISO18-14-04-018	PP07; Sample	318	225	118	2170	70.4
ISO18-14-04-018-DUP	PP07; Sample Dup	311	226	116	2130	73.4
		Average	315 ⁽²⁾	226 ⁽²⁾	117 ⁽²⁾	2150 ⁽²⁾
		%RPD Sample/Sample Dup	2.2	0.44	1.7	1.9
ISO18-14-04-019	PP08; Sample	538	34.7	344	7060	53.8
ISO18-14-04-019-DUP	PP08; Sample Dup	530	35.0	346	6950	47.2
		Average	534 ⁽²⁾	34.9 ⁽²⁾	345 ⁽²⁾	7010 ⁽²⁾
		%RPD Sample/Sample Dup	1.5	0.86	0.58	1.6
ISO18-14-04-020	PP09; Sample	648	20.1	316	2520	13.3
ISO18-14-04-020-DUP	PP09; Sample Dup	606	18.2	290	2310	11.1
		Average	627 ⁽²⁾	19.2 ⁽²⁾	303 ⁽²⁾	2420 ⁽²⁾
		%RPD Sample/Sample Dup	6.7	9.9	8.6	8.7
ISO18-14-04-021	PP10; Sample	588	16.5	232	3900	16.0
ISO18-14-04-021-DUP	PP10; Sample Dup	548	15.5	217	3590	15.1
		Average	568 ⁽²⁾	16.0 ⁽²⁾	225 ⁽²⁾	3750 ⁽²⁾
		%RPD Sample/Sample Dup	7.0	6.3	6.7	8.3
Zone: Source Area – Building 16						
ISO18-14-04-028	P21B; Sample	9190	7230	11400	57400	5.06
ISO18-14-04-028-DUP	P21B; Sample Dup	8940	7030	11100	55600	5.00
		Average	9070 ⁽²⁾	7130 ⁽²⁾	11300 ⁽²⁾	56500 ⁽²⁾
		%RPD Sample/Sample Dup	2.8	2.8	2.7	3.2
Travel Blank						
ISO18-14-04-038	Travel Blank	<0.0240	<0.200 ⁽²⁾	<0.0250	<0.0464	<0.0250

- (1) Unless noted otherwise, samples were reported using internal standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 18%, PFHS \pm 11%, PFOS \pm 13%, and PFOSA \pm 25%.
- (2) Sample set reported using external standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 18%, PFBS \pm 16%, PFHS \pm 14%, and PFOS \pm 22%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis".

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluoroctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected on October 23-25, 2018, in Nalgene™ (high-density polyethylene) bottles prepared at the 3M EHS Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. All sample locations were spiked with a mass-labeled internal standard mix at a nominal concentration of 1 ng/mL and select samples were fortified with surrogate recovery standards (SRSs) at a nominal concentration of 0.1 ng/mL prior to sample collection. Samples were received on October 29, 2018.

2.3 Sample Preparation

Sample locations pre-spiked with surrogate recovery standards to be analyzed for PFOA, PFHS, and PFOS, and all samples to be analyzed for PFOSA were prepared by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples along with the method blanks.

Samples that required further dilution were prepared for PFOA, PFHS, PFOS and PFBS (select locations only) using a direct injection by methanol dilution method. Samples that required a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples that required a 1:100 dilution were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol. Samples that required a 1:500 dilution were prepared by diluting 0.02 mL of a well-mixed sample with 10.0 mL of methanol. Samples that required a 1:1000 dilution were prepared by first diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol, followed by a second dilution prepared by diluting 1 mL of the diluted sample into 9 mL of methanol. Diluted samples and LCSs were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

2.4 Analysis

All samples and quality control samples were analyzed for the target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). SRSs were added and included in the analysis of these samples. Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-

010.2. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

The following analytical runs were used to report the results herein:

11/8/18 (ETS Kirk) Internal Standard Calibration Analysis:

- All sample locations pre-spiked with surrogate recovery standards were analyzed for PFOA, PFHS, PFOS, and PFOSA. All sample results were reported **except** for the following: L21 (PFOA and PFHS), 12 and 13 (PFOS), and Collector put (PFOA, PFHS, and PFOS).

11/16/18 (ETS Rita) Internal Standard Calibration Analysis:

- All remaining sample locations were analyzed for PFOSA. All sample results were reported.

11/26/18 (ETS Kirk) External Standard Calibration Analysis:

- All remaining sample locations were analyzed for PFOA, PFHS, and PFOS with select locations analyzed for PFBS. In addition, sample locations 12 and 13 were re-analyzed for PFOS and Collector put was re-analyzed for PFOA, PFHS, and PFOS. All sample results were reported **except** for the following: PP05 and P21B (PFOS).

11/30/18 (ETS Kirk) Internal Standard Calibration Analysis:

- Sample location L21 was re-analyzed and reported for PFOA and PFHS.

12/4/18 (ETS Kirk) External Standard Calibration Analysis:

- Sample locations PP05 and P21B were re-analyzed and reported for PFOS.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk	ETS Rita
Liquid Chromatograph	Agilent 1260	Agilent 1260
Analysis Method	ETS-8-044.3	ETS-8-044.3
Analysis Date	11/8/18, 11/26/18, 11/30/18, 12/4/18	11/16/18
Guard column	Prism RP (2.1 mm X 50 mm), 5 μ	Prism RP (2.1 mm X 50 mm), 5 μ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ
Injection Volume	2 or 5 μ L	2 μ L
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 6500+
Ion Source	Turbo Spray	Turbo Spray
Polarity	Negative	Negative
Software	Analyst 1.6.3	Analyst 1.6.3

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis				
Step Number	Total Time (min)	Flow Rate (μ L/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0.00	300	90.0	10.0
1	0.50	300	90.0	10.0
2	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard ⁽¹⁾	Mass Transition Q1/Q3
PFOA	413/369	[¹³ C ₈]-PFOA	421/376
	413/219		
	413/169		
PFBS	299/99	NA	NA
	299/80		
PFHS	399/99	[¹³ C ₃]-PFHS	402/80
	399/80		
PFOS	499/99	[¹³ C ₈]-PFOS	507/80
	499/80		
	499/130		
PFOSA	498/78	[¹³ C ₈]-PFOSA	506/78
[¹³ C ₄]-PFOA	417/372	[¹³ C ₈]-PFOA	421/376
[¹³ C ₄]-PFOS	503/80	[¹³ C ₈]-PFOS	507/80

The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

(1) Internal standard was not used for the external calibration analyses on 11/26/18 or 12/4/18.

3 Data Analysis

3.1 Calibration

11/8/18, 11/16/18, and 11/30/18 Analysis (Internal Standard Calibration): Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of fourteen calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed. The calibration standards also contained surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

11/26/18 and 12/4/18 Analysis (External Standard Calibration): Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed on 11/26/18 and from 0.05 ng/mL to 100 ng/mL (nominal) on 12/4/18. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL or 0.05 ng/mL to 25 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±25% (100±30% for the lowest curve point) were met for all analytes. The correlation coefficient (*r*) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.3 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the **Table 6** below.

Table 6. LOQ

Analyte	LOQ, ng/mL ⁽¹⁾ 11/8/18 Analysis	LOQ, ng/mL ⁽¹⁾ 11/16/18 Analysis	LOQ, ng/mL ⁽²⁾ 11/26/18 Analysis	LOQ, ng/mL ⁽¹⁾ 11/30/18 Analysis	LOQ, ng/mL ⁽²⁾ 12/4/18 Analysis
PFOA	0.0240	NA	0.0479	0.0240	NA
PFBS	NA	NA	0.0200	NA	NA
PFHS	0.0250	NA	0.0200	0.0250	NA
PFOS	0.0464	NA	0.0927	NA	0.0464
PFOSA	0.0250	0.100	NA	NA	NA

NA = Not Applicable

(1) A dilution factor of 2 was applied to the LOQ.

(2) A dilution factor was not applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of $100\%\pm 25\%$.

3.5 Blanks

Two types of blanks were prepared and analyzed with the samples: method/solvent blanks and field/trip blanks. Each blank result was reviewed and used to evaluate method performance. The method blanks were used to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. LCSs analyzed on 11/8/18, 11/16/18, and 11/30/18 were prepared at 0.2, 20, and 140 ng/mL. LCSs analyzed on 11/26/18 were prepared at 100, 5,000, and 35,000 ng/mL. LCSs analyzed on 12/4/18 were prepared at 200, 20,000, and 70,000 ng/mL. LCSs were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. Surrogates [$^{13}\text{C}_4$]-PFOA and [$^{13}\text{C}_4$]-PFOS were added post dilution when analyzed by external standard.

Method ETS-8-044.3 states that the average recovery of LCSs at each spiking level must be within 80%-120% with a RSD $\leq 20\%$. All LCS samples met criteria with the following exception:

- 11/8/18: High level LCSs (140 ng/mL) for PFHS and PFOSA were spiked above the resulting ULOQ. The Low and Mid-level LCSs were appropriate for the sample concentrations and the data were reported.
- 11/16/18: Low level LCSs (0.2 ng/mL) initially analyzed on 11/8/18 were re-injected for PFOSA and had an average recovery of 125%.

- 11/30/18: High level LCSs (140 ng/mL) for PFOA and PFBS were spiked above the resulting ULOQ. The Low and Mid-level LCSs were appropriate for the sample concentrations and the data were reported.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report. Individual LCS results are included in the raw data and available upon request.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.4. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in **Table 7** below.

- The data uncertainty for PFOSA was calculated at 17% following ETS-12-012.4; however, the data uncertainty was increased to $\pm 25\%$ based on the PFOSA recovery of the low-level LCSs from the analysis on 11/16/18.

Table 7. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	9.17	$\pm 18\%$
PFBS	External	8.19	$\pm 16\%$
PFHS	External	7.21	$\pm 14\%$
PFOS	External	10.9	$\pm 22\%$
PFOA	Internal	9.20	$\pm 18\%$
PFHS	Internal	5.43	$\pm 11\%$
PFOS	Internal	6.59	$\pm 13\%$
PFOSA	Internal	NA	$\pm 25\%$

NA = Not Applicable

3.8 Field Matrix Spikes (FMS)

Target analyte field matrix spike samples were collected for select sampling points to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions at 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOA and PFOS. The standards used for the preparation of the field matrix spiking solutions greater than 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, select sample bottles contained stable isotope surrogate recovery spikes of [¹³C₄]-PFOA and [¹³C₄]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles prior to sample collection. The [¹³C₄]-PFOA was selected to represent the C8 perfluorocarboxylic acid. The [¹³C₄]-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

The following calculation was used to generate data in section 4 of the report.

$$FMS\ Recovery = \frac{Sample\ Concentration\ of\ FMS - Average\ Concentration\ (Field\ Sample\ & Field\ Sample\ Dup)}{Spike\ Concentration} \times 100\%$$

Table 8. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
3M vijver	FMS	1.92	2.00	2.00	1.85	2.00
L4	FMS	10.0	10.0	10.0	10.0	10.0
Effluent WWTP	FMS	51.9	52.0	52.0	51.9	2.00
13 ⁽¹⁾	FMS	43.8	43.9	43.9	43.8	1.69
Collector Put	FMS	110	110	110	109	10.0
P321	FMS	2050	2050	2050	2050	50.0
PP04 ⁽¹⁾	FMS	1840	1840	1840	1840	44.8
PP10	FMS	2050	50.0	2050	2050	50.0
Trip Blank	Low	1.92	2.00	2.00	1.85	2.00
	High	520	20.0	520	520	20.0

(1) The sample container for the FMS sample was overfilled by 10%. The FMS true values were adjusted accordingly.

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of $\pm 30\%$, demonstrate that the method is appropriate for the given matrix.

The method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. At times, the spike level exceeded the recommended upper limit of 10 times the analyte concentration. In these instances, the FMS recovery was reported and flagged as above 10 times the sample concentration.

For PFOA, PFBS, PFHS, and PFOS, when the field matrix spike level was not appropriate as compared to the endogenous sample concentration, the surrogate recovery standards were used to assess method accuracy. All field matrix spike and surrogate recovery standard recoveries met acceptance criteria.

Table 9. Location ID: 3M vijver

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-001	3M vijver; Sample	1.65	NA	0.698	NA
ISO18-14-04-001-DUP	3M vijver; Sample Duplicate	1.78	NA	0.732	NA
ISO18-14-04-001-FMS	3M vijver; Sample FMS	3.72	104	2.68	98.3
Average Concentration (ng/mL) \pm %RPD		1.72 ng/mL \pm 7.6%		0.715 ng/mL \pm 4.8%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-001	3M vijver; Sample	4.82	NA	0.0374	NA
ISO18-14-04-001-DUP	3M vijver; Sample Duplicate	5.06	NA	0.0348	NA
ISO18-14-04-001-FMS	3M vijver; Sample FMS	7.22	NC	1.99	97.7 ⁽¹⁾
Average Concentration (ng/mL) \pm %RPD		4.94 ng/mL \pm 4.9%		0.0361 ng/mL \pm 7.2%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) FMS concentration greater than 10 times the sample concentration.

Table 10. Location ID: P321

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-004	P321; Sample	2960	NA	750	NA
ISO18-14-04-004-DUP	P321; Sample Duplicate	2970	NA	777	NA
ISO18-14-04-004-FMS	P321; Sample FMS	4990	98.8	3100	114
Average Concentration (ng/mL) ± %RPD		2970 ng/mL ± 0.34%		764 ng/mL ± 3.5%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-004	P321; Sample	5970	NA	0.206	NA
ISO18-14-04-004-DUP	P321; Sample Duplicate	6060	NA	0.200	NA
ISO18-14-04-004-FMS	P321; Sample FMS	8760	NC	49.2	98.0 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		6020 ng/mL ± 1.5%		0.203 ng/mL ± 3.0%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) FMS concentration greater than 10 times the sample concentration.

Table 11. Location ID: L4

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-008	L4; Sample	2.38	NA	2.04	NA
ISO18-14-04-008-DUP	L4; Sample Duplicate	2.40	NA	2.02	NA
ISO18-14-04-008-FMS	L4; Sample FMS	12.1	97.1	11.9	98.7
Average Concentration (ng/mL) ± %RPD		2.39 ng/mL ± 0.84%		2.03 ng/mL ± 0.99%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-008	L4; Sample	28.4	NA	0.206	NA
ISO18-14-04-008-DUP	L4; Sample Duplicate	28.0	NA	0.204	NA
ISO18-14-04-008-FMS	L4; Sample FMS	37.8	NC	10.3	101 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		28.2 ng/mL ± 1.4%		0.205 ng/mL ± 0.98%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) FMS concentration greater than 10 times the sample concentration.

Table 12. Location ID: Effluent WWTP

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-012	Effluent WWTP; Sample	13.6	NA	14.4	NA
ISO18-14-04-012-DUP	Effluent WWTP; Sample Duplicate	12.4	NA	13.5	NA
ISO18-14-04-012-FMS	Effluent WWTP; Sample FMS	59.6	89.8	66.4	101
Average Concentration (ng/mL) ± %RPD		13.0 ng/mL ± 9.2%		14.0 ng/mL ± 6.5%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-012	Effluent WWTP; Sample	28.8	NA	0.466	NA
ISO18-14-04-012-DUP	Effluent WWTP; Sample Duplicate	27.8	NA	0.458	NA
ISO18-14-04-012-FMS	Effluent WWTP; Sample FMS	80.8	101	2.34	93.9
Average Concentration (ng/mL) ± %RPD		28.3 ng/mL ± 3.5%		0.462 ng/mL ± 1.7%	

NA = Not Applicable

Table 13. Location ID: PP04

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-015	PP04; Sample	668	NA	1540	NA	300	NA
ISO18-14-04-015-DUP	PP04; Sample Duplicate	678	NA	1580	NA	296	NA
ISO18-14-04-015-FMS	PP04; Sample FMS	2500	99.3	3520	107	2280	108
Average Concentration (ng/mL) ± %RPD		673 ng/mL ± 1.5%		1560 ng/mL ± 2.6%		298 ng/mL ± 1.3%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-015	PP04; Sample	5170	NA	56.4	NA
ISO18-14-04-015-DUP	PP04; Sample Duplicate	4760	NA	61.0	NA
ISO18-14-04-015-FMS	PP04; Sample FMS	6680	NC	94.0	78.8
Average Concentration (ng/mL) ± %RPD		4970 ng/mL ± 8.3%		58.7 ng/mL ± 7.8%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 14. Location ID: PP10

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-021	PP10; Sample	588	NA	16.5	NA	232	NA
ISO18-14-04-021-DUP	PP10; Sample Duplicate	548	NA	15.5	NA	217	NA
ISO18-14-04-021-FMS	PP10; Sample FMS	2550	96.7	78.3	125	2400	106
Average Concentration (ng/mL) ± %RPD		568 ng/mL ± 7.0%		16.0 ng/mL ± 6.3%		225 ng/mL ± 6.7%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-021	PP10; Sample	3900	NA	16.0	NA
ISO18-14-04-021-DUP	PP10; Sample Duplicate	3590	NA	15.1	NA
ISO18-14-04-021-FMS	PP10; Sample FMS	6160	118	64.8	98.5
Average Concentration (ng/mL) ± %RPD		3750 ng/mL ± 8.3%		15.6 ng/mL ± 5.8%	

NA = Not Applicable

Table 15. Location ID: 13

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-023	13; Sample	97.0	NA	34.6	NA
ISO18-14-04-023-DUP	13; Sample Duplicate	84.6	NA	33.0	NA
ISO18-14-04-023-FMS	13; Sample FMS	123	NC	82.8	112
Average Concentration (ng/mL) ± %RPD		90.8 ng/mL ± 14%		33.8 ng/mL ± 4.7%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-023	13; Sample	284	NA	0.256	NA
ISO18-14-04-023-DUP	13; Sample Duplicate	277	NA	0.252	NA
ISO18-14-04-023-FMS	13; Sample FMS	400	NC	1.89	96.8
Average Concentration (ng/mL) ± %RPD		281 ng/mL ± 2.5%		0.254 ng/mL ± 1.6%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 16. Location ID: Collector Put

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-026	Collector put; Sample	73.1	NA	30.6	NA
ISO18-14-04-026-DUP	Collector put; Sample Duplicate	70.6	NA	28.9	NA
ISO18-14-04-026-FMS	Collector put; Sample FMS	173	92.0	136	96.6
Average Concentration (ng/mL) ± %RPD		71.9 ng/mL ± 3.5%		29.8 ng/mL ± 5.7%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-026	Collector put; Sample	287	NA	18.8	NA
ISO18-14-04-026-DUP	Collector put; Sample Duplicate	281	NA	19.3	NA
ISO18-14-04-026-FMS	Collector put; Sample FMS	368	NC	28.8	97.5
Average Concentration (ng/mL) ± %RPD		284 ng/mL ± 2.1%		19.1 ng/mL ± 2.6%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 17. Location ID: Travel Blank

		PFOA		PFBS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-038	Travel Blank	<0.0240	NA	<0.200	NA
ISO18-14-04-038-FMS-LOW	Travel Blank FMS Low	1.79	93.2	2.06	103
ISO18-14-04-038-FMS-HIGH	Travel Blank FMS High	1990	97.1	53.6	107

		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO18-14-04-038	Travel Blank	<0.0250	NA	<0.0464	NA
ISO18-14-04-038-FMS-LOW	Travel Blank FMS Low	1.87	93.5	1.92	104
ISO18-14-04-038-FMS-HIGH	Travel Blank FMS High	2150	105	2060	100

		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery
ISO18-14-04-038	Travel Blank	<0.0250	NA
ISO18-14-04-038-FMS-LOW	Travel Blank FMS Low	1.92	96.0
ISO18-14-04-038-FMS-HIGH	Travel Blank FMS High	49.4	98.8

NA = Not Applicable

Table 18. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]PFOA	[¹³ C ₄]PFOS	[¹³ C ₄]PFOS Re-analysis
ISO18-14-04-001	3M vijver; Sample	85.0	88.0	NA
ISO18-14-04-001-DUP	3M vijver; Sample Duplicate	93.9	85.5	NA
ISO18-14-04-001-FMS	3M vijver; Sample FMS	93.2	83.8	NA
ISO18-14-04-002	Blokkersdijkvijver Noord; Sample	90.5	91.5	NA
ISO18-14-04-002-DUP	Blokkersdijkvijver Noord; Sample Duplicate	90.9	87.8	NA
ISO18-14-04-003	Blokkersdijkvijver standaard; Sample	76.4	83.8	NA
ISO18-14-04-003-DUP	Blokkersdijkvijver standaard; Sample Duplicate	93.8	84.4	NA
ISO18-14-04-004	P321; Sample	111 ⁽²⁾	109 ⁽²⁾	NA
ISO18-14-04-004-DUP	P321; Sample Duplicate	106 ⁽²⁾	112 ⁽²⁾	NA
ISO18-14-04-004-FMS	P321; Sample FMS	102 ⁽²⁾	104 ⁽²⁾	NA
ISO18-14-04-005	L21; Sample	98.5	89.4	84.4
ISO18-14-04-005-DUP	L21; Sample Duplicate	102	92.4	90.7
ISO18-14-04-006	L22; Sample	90.5	92.4	NA
ISO18-14-04-006-DUP	L22; Sample Duplicate	97.3	80.9	NA
ISO18-14-04-007	L31; Sample	96.9	92.3	NA
ISO18-14-04-007-DUP	L31; Sample Duplicate	93.2	85.9	NA
ISO18-14-04-008	L4; Sample	92.8	91.9	NA
ISO18-14-04-008-DUP	L4; Sample Duplicate	84.4	89.6	NA
ISO18-14-04-008-FMS	L4; Sample FMS	93.6	88.0	NA
ISO18-14-04-009	P114bis; Sample	103	94.7	NA
ISO18-14-04-009-DUP	P114bis; Sample Duplicate	88.0	90.1	NA
ISO18-14-04-010	P115; Sample	88.2	88.2	NA
ISO18-14-04-010-DUP	P115; Sample Duplicate	94.4	93.0	NA
ISO18-14-04-011	P116; Sample	94.5	88.2	NA
ISO18-14-04-011-DUP	P116; Sample Duplicate	85.2	93.6	NA
ISO18-14-04-012	Effluent WWTP; Sample	96.8	88.0	NA
ISO18-14-04-012-DUP	Effluent WWTP; Sample Duplicate	101	89.2	NA
ISO18-14-04-012-FMS	Effluent WWTP; Sample FMS	95.6	92.4	NA
ISO18-14-04-014	PP02; Sample	115 ⁽²⁾	110 ⁽²⁾	NA
ISO18-14-04-014-DUP	PP02; Sample Duplicate	112 ⁽²⁾	109 ⁽²⁾	NA
ISO18-14-04-015	PP04; Sample	120 ⁽²⁾	118 ⁽²⁾	NA
ISO18-14-04-015-DUP	PP04; Sample Duplicate	120 ⁽²⁾	122 ⁽²⁾	NA
ISO18-14-04-015-FMS	PP04; Sample FMS	116 ⁽²⁾	121 ⁽²⁾	NA
ISO18-14-04-016	PP05; Sample	112 ⁽²⁾	110 ⁽²⁾	113 ⁽²⁾
ISO18-14-04-016-DUP	PP05; Sample Duplicate	108 ⁽²⁾	106 ⁽²⁾	111 ⁽²⁾
ISO18-14-04-017	PP06; Sample	116 ⁽²⁾	116 ⁽²⁾	NA
ISO18-14-04-017-DUP	PP06; Sample Duplicate	113 ⁽²⁾	116 ⁽²⁾	NA
ISO18-14-04-018	PP07; Sample	112 ⁽²⁾	113 ⁽²⁾	NA
ISO18-14-04-018-DUP	PP07; Sample Duplicate	105 ⁽²⁾	107 ⁽²⁾	NA

NA = Not Applicable

(1) The surrogate recovery standards were added to the sample bottle prior to the sampling event unless noted otherwise.

(2) The surrogate recovery standards were added to the samples during sample preparation.

Table 18 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]PFOA	[¹³ C ₄]PFOS	[¹³ C ₄]PFOS Re-analysis
ISO18-14-04-019	PP08; Sample	117 ⁽²⁾	121 ⁽²⁾	NA
ISO18-14-04-019-DUP	PP08; Sample Duplicate	110 ⁽²⁾	110 ⁽²⁾	NA
ISO18-14-04-020	PP09; Sample	112 ⁽²⁾	114 ⁽²⁾	NA
ISO18-14-04-020-DUP	PP09; Sample Duplicate	103 ⁽²⁾	100 ⁽²⁾	NA
ISO18-14-04-021	PP10; Sample	104 ⁽²⁾	110 ⁽²⁾	NA
ISO18-14-04-021-DUP	PP10; Sample Duplicate	106 ⁽²⁾	104 ⁽²⁾	NA
ISO18-14-04-021-FMS	PP10; Sample FMS	111 ⁽²⁾	113 ⁽²⁾	NA
ISO18-14-04-022	12; Sample	100	91.6	114 ⁽²⁾
ISO18-14-04-022-DUP	12; Sample Duplicate	93.6	87.6	108 ⁽²⁾
ISO18-14-04-023	13; Sample	94.0	82.3	113 ⁽²⁾
ISO18-14-04-023-DUP	13; Sample Duplicate	95.5	88.2	118 ⁽²⁾
ISO18-14-04-023-FMS	13; Sample FMS	105	87.6	115 ⁽²⁾
ISO18-14-04-024	5; Sample	88.6	88.8	NA
ISO18-14-04-024-DUP	5; Sample Duplicate	90.8	91.7	NA
ISO18-14-04-025	Bemalingstation; Sample	88.2	90.7	NA
ISO18-14-04-025-DUP	Bemalingstation; Sample Duplicate	92.4	88.0	NA
ISO18-14-04-026	Collector put; Sample	117 ⁽²⁾	119 ⁽²⁾	NA
ISO18-14-04-026-DUP	Collector put; Sample Duplicate	108 ⁽²⁾	115 ⁽²⁾	NA
ISO18-14-04-026-FMS	Collector put; Sample FMS	114 ⁽²⁾	116 ⁽²⁾	NA
ISO18-14-04-028	P21B; Sample	115 ⁽²⁾	114 ⁽²⁾	111 ⁽²⁾
ISO18-14-04-028-DUP	P21B; Sample Duplicate	117 ⁽²⁾	115 ⁽²⁾	109 ⁽²⁾
ISO18-14-04-038	Travel Blank	87.8	90.1	NA
ISO18-14-04-038-FMS-LOW	Travel Blank FMS Low	86.8	92.4	NA
ISO18-14-04-038-FMS-HIGH	Travel Blank FMS High	111 ⁽²⁾	113 ⁽²⁾	NA

NA = Not Applicable

(1) The surrogate recovery standards were added to the sample bottle prior to the sampling event unless noted otherwise.

(2) The surrogate recovery standards were added to the samples during sample preparation.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. Analysis was completed using 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 9-18 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M EHS Laboratory standard operating procedures.

7 Attachments

Chain of Custody Form

8 Signatures

Chelsie Grochow, 3M Report Author

Susan T. Wolf, 3M Principal Analytical Investigator

Brian T. Mader, Ph.D., 3M EHS Laboratory Manager

The 3M EHS Laboratory's Quality Assurance Unit has audited the data and report for this project.

Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M EHS Laboratory.

3M EHS LABORATORY
Chain-of-Custody

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St. Paul, MN 55144

Phone: [REDACTED]
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Fax: [REDACTED]

Project: ISO18-14-04

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 10/3/2018

Project Description: 3M Antwerp Water Sampling for PFCs - October 2018

Comments:

Completion Date:

Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
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ISO18-14-04-001	3M vijver; Sample ✓ ✓	25/10/2018	WG	
ISO18-14-04-001-DUP	3M vijver; Sample Duplicate ✓ ✓	25/10/2018	WG	
ISO18-14-04-001-FMS	3M vijver; Sample FMS ✓ ✓	25/10/2018	WQ	
ISO18-14-04-002	Blokkersdijkvijver Noord; Sample ✓ ✓	25/10/2018	WG	
ISO18-14-04-002-DUP	Blokkersdijkvijver Noord; Sample Duplicate ✓ ✓	25/10/2018	WG	
ISO18-14-04-003	Blokkersdijkvijver standaard; Sample ✓ ✓	25/10/2018	WG	
ISO18-14-04-003-DUP	Blokkersdijkvijver standaard; Sample Duplicate ✓ ✓	25/10/2018	WG	
ISO18-14-04-004	P321; Sample ✓ ✓	24/10/18	WG	
ISO18-14-04-004-DUP	P321; Sample Duplicate ✓ ✓	24/10/18	WG	
ISO18-14-04-004-FMS	P321; Sample FMS ✓ ✓	24/10/18	WQ	
ISO18-14-04-005	L21; Sample ✓ ✓	23/10/18	WG	
ISO18-14-04-005-DUP	L21; Sample Duplicate ✓ ✓	23/10/18	WG	
ISO18-14-04-006	L22; Sample ✓ ✓	23/10/18	WG	
ISO18-14-04-006-DUP	L22; Sample Duplicate ✓ ✓	23/10/18	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 15 Deg C Received on Ice Other:

Collected by (print): Elein Kemme (EKE) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
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14	EKE	26/10/18	/	FEDEX	Joseph Tiernan / 3m	10-29-18	12:00 pm

3M EHS LABORATORY
Chain-of-Custody

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Project: ISO18-14-04 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 10/3/2018

Project Description: 3M Antwerp Water Sampling for PFCs - October 2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO18-14-04-007	L31; Sample ✓ ✓	23/10/18	WG	
ISO18-14-04-007-DUP	L31; Sample Duplicate ✓ ✓	24/10/18	WG	
ISO18-14-04-008	L4; Sample ✓ ✓	24/10/18	WG	
ISO18-14-04-008-DUP	L4; Sample Duplicate ✓ ✓	24/10/18	WG	
ISO18-14-04-008-FMS	L4; Sample FMS ✓ ✓	24/10/18	WQ	
ISO18-14-04-009	P114bis; Sample ✓ ✓	24/10/18	WG	
ISO18-14-04-009-DUP	P114bis; Sample Duplicate ✓ ✓	24/10/18	WG	
ISO18-14-04-010	P115; Sample ✓ ✓	24/10/18	WG	
ISO18-14-04-010-DUP	P115; Sample Duplicate ✓ ✓	24/10/18	WG	
ISO18-14-04-011	P116; Sample ✓ ✓	23/10/18	WG	
ISO18-14-04-011-DUP	P116; Sample Duplicate ✓ ✓	23/10/18	WG	
ISO18-14-04-012	Effluent WWTP; Sample ✓ ✓	25/10/18	WG	
ISO18-14-04-012-DUP	Effluent WWTP; Sample Duplicate ✓ ✓	25/10/18	WG	
ISO18-14-04-012-FMS	Effluent WWTP; Sample FMS ✓ ✓	25/10/18	WQ	
ISO18-14-04-013	PP01; Sample ✓ -	/	WG	OUT OF USE, NOT SAMPLED
ISO18-14-04-013-DUP	PP01; Sample Duplicate ✓ -	/	WG	OUT OF USE, NOT SAMPLED
ISO18-14-04-014	PP02; Sample ✓ ✓	25/10/18	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 15 Deg C Received on Ice Other:

Collected by (print): Eileen Kemme (EKE) Collector's signature: J

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	EKE	26/10/18	/	FEDEX	JOSEPH TILUNAN / 3m	10-29-18	12:00 pm

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
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Fax: [REDACTED]

Project: ISO18-14-04 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 10/3/2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number:
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs - October 2018

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO18-14-04-014-DUP	PP02; Sample Duplicate ✓ ✓	25/10/18	WG	
ISO18-14-04-015	PP04; Sample ✓ ✓	25/10/18	WG	
ISO18-14-04-015-DUP	PP04; Sample Duplicate ✓ ✓	25/10/18	WG	
ISO18-14-04-015-FMS	PP04; Sample FMS ✓ ✓	25/10/18	WQ	
ISO18-14-04-016	PP05; Sample ✓ ✓	25/10/18	WG	
ISO18-14-04-016-DUP	PP05; Sample Duplicate ✓ ✓	25/10/18	WG	
ISO18-14-04-017	PP06; Sample ✓ ✓	25/10/18	WG	
ISO18-14-04-017-DUP	PP06; Sample Duplicate ✓ ✓	25/10/18	WG	
ISO18-14-04-018	PP07; Sample ✓ ✓	25/10/18	WG	
ISO18-14-04-018-DUP	PP07; Sample Duplicate ✓ ✓	25/10/18	WG	
ISO18-14-04-019	PP08; Sample ✓ ✓	25/10/18	WG	
ISO18-14-04-019-DUP	PP08; Sample Duplicate ✓ ✓	25/10/18	WG	
ISO18-14-04-020	PP09; Sample ✓ ✓	25/10/18	WG	
ISO18-14-04-020-DUP	PP09; Sample Duplicate ✓ ✓	25/10/18	WG	
ISO18-14-04-021	PP10; Sample ✓ ✓	25/10/18	WG	
ISO18-14-04-021-DUP	PP10; Sample Duplicate ✓ ✓	25/10/18	WG	
ISO18-14-04-021-FMS	PP10; Sample FMS ✓ ✓	25/10/18	WQ	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 15 Deg C Received on Ice Other:

Collected by (print): Elein Hemme (EKE) Collector's signature:

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
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17	EKE	26/10/18	/	FEDEX	JOSEPH TILLMAN / 3M	10-29-18	1200 pm

3M EHS LABORATORY
Chain-of-Custody

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Alt. Ph: [REDACTED]
Fax: [REDACTED]

Project: ISO18-14-04 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 10/3/2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs - October 2018

SW = surface water
WW = waste water

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
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ISO18-14-04-022	12; Sample ✓ ✓	25/10/18	SW	
ISO18-14-04-022-DUP	12; Sample Duplicate ✓ ✓	25/10/18	SW	
ISO18-14-04-023	13; Sample ✓ ✓	25/10/18	SW	
ISO18-14-04-023-DUP	13; Sample Duplicate ✓ ✓	25/10/18	SW	
ISO18-14-04-023-FMS	13; Sample FMS ✓ ✓	25/10/18	WQ	
ISO18-14-04-024	5; Sample ✓ ✓	25/10/18	SW	
ISO18-14-04-024-DUP	5; Sample Duplicate ✓ ✓	25/10/18	SW	
ISO18-14-04-025	Bemalingstation, Sample ✓ ✓	25/10/18	SW	
ISO18-14-04-025-DUP	Bemalingstation, Sample Duplicate ✓ ✓	25/10/18	SW	
ISO18-14-04-026	Collector put, Sample ✓ ✓	25/10/18	WW	
ISO18-14-04-026-DUP	Collector put; Sample Duplicate ✓ ✓	25/10/18	WW	
ISO18-14-04-026-FMS	Collector put, Sample FMS ✓ ✓	25/10/18	WQ	
ISO18-14-04-027	K3; Sample ✓ -	/	WG	NOT SAMPLED
ISO18-14-04-027-DUP	K3; Sample Duplicate ✓ -	/	WG	NOT SAMPLED
ISO18-14-04-028	P21B; Sample ✓ ✓	24/10/18	WG	
ISO18-14-04-028-DUP	P21B; Sample Duplicate ✓ ✓	24/10/18	WG	
ISO18-14-04-029	P56; Sample ✓ -	/	WG	NOT SAMPLED

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 15 Deg C Received on Ice Other:

Collected by (print): Elien Kemme (EKE) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
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157	EKE	26/10/18	/	FEDEX	JOGJA TILLMAN /3m	10-29-18	1200 PM

3M EHS LABORATORY
Chain-of-Custody

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Project: ISO18-14-04 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 10/3/2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs - October 2018

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
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ISO18-14-04-029-DUP	P56; Sample Duplicate ✓	/	WG	NOT SAMPLED
ISO18-14-04-030	P41; Sample ✓	/		NOT SAMPLED
ISO18-14-04-030-DUP	P41; Sample Duplicate ✓	/		W
ISO18-14-04-031	PB402; Sample ✓	/		W
ISO18-14-04-031-DUP	PB402; Sample Duplicate ✓	/		W
ISO18-14-04-031-FMS	PB402; FMS ✓	/		W
ISO18-14-04-032	P219; Sample ✓	/		W
ISO18-14-04-032-DUP	P219; Sample Duplicate ✓	/		W
ISO18-14-04-033	P40; Sample ✓	/		W
ISO18-14-04-033-DUP	P40; Sample Duplicate ✓	/		W
ISO18-14-04-034	P300; Sample ✓	/		W
ISO18-14-04-034-DUP	P300; Sample Duplicate ✓	/		W
ISO18-14-04-034-FMS	P300; FMS ✓	/		W
ISO18-14-04-035	P1065; Sample ✓	/		W
ISO18-14-04-035-DUP	P1065; Sample Duplicate ✓	/		W
ISO18-14-04-036	P215; Sample ✓	/		W
ISO18-14-04-036-DUP	P215; Sample Duplicate ✓	/		W

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 15 Deg C Received on Ice Other:

Collected by (print): Elien Kemme (EKE) Collector's signature:

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
--------	------------------	------	------	-------------	--------------	------	------

17	EKE	26/10/18	/	FEDEX	JOSEPH TILLMAN/3M	10-29-18	1200pm

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [REDACTED]
Alt. Ph: [REDACTED]
Fax: [REDACTED]

Project: ISO18-14-04 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 10/3/2018

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs - October 2018

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
-------------------------	---------------------------	--------------------------	---------------	----------------

ISO18-14-04-036-FMS	P215; FMS ✓	/		NOT SAMPLED
ISO18-14-04-037	P216; Sample ✓	/		NOT SAMPLED
ISO18-14-04-037-DUP	P216; Sample Duplicate ✓	/		NOT SAMPLED
ISO18-14-04-038	Travel Blank ✓			*
ISO18-14-04-038-FMS-HIGH	Travel Blank FMS High ✓			*
ISO18-14-04-038-FMS-LOW	Travel Blank FMS Low ✓			*

* Travel Blank samples were prepared by the 3M EHS Laboratory with the bottle order.

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 15 Deg C Received on Ice Other:

Collected by (print): Elin Kemme (EKE) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
--------	------------------	------	------	-------------	--------------	------	------

<u>6</u>	<u>EKE</u>	<u>26/10/18</u>	<u>/</u>	<u>FEDEX</u>	<u>JOSEPH TILLMAN /3M</u>	<u>10-29-18</u>	<u>1200pm</u>



3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC19-00136

3M Lab Request Number: E19-0176

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: March 15th, 2019

Requester

Jim Kotsmith
3M Center, Bldg 224-5w-17
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SGS Belgium NV

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Member of the SGS Group (Société Générale de Surveillance)

Registered office: Noorderlaan 87 B-2030 Antwerpen H.R. Antwerpen 141.810 BTW BE 404.882.750 Dexia 550-3560000-93
All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction - Summary.

At the request (Lab Request Number: E19-0176) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected in January 2019 from 3 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOA ($C_6F_{13}^{13}CF_2^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOS ($C_7F_{15}^{13}CF_2SO_3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Sven Herremans. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles.

Table 1 summarizes the sample results. All PFBS, PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
Effluent WWTP	6.06	10.5	23.8	0.340	
Bemalingsstation	12.9	16.1	18.2	<0.209	
Collector Put	23.7	60.3	137	7.50	

2. Methods – Analytical and Preparatory.**2.1. Sample collection.**

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
Effluent WWTP	100
Bemalingsstation	100
Collector Put	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefloreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
Effluent WWTP	1.0	0.05	Solution A	1.0	-
Bemalingsstation	1.0	0.05	Solution A	1.0	-
Collector Put	1.0	0.05	Solution A	1.0	-

(*): MeOH:LCMS-water (60:40)
 Solution A = 1000 ng/mL (nominal) ¹³C-PFC Surrogate Standards

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetre C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
PFBS	2.08 - 2.50	298.93/79.82	40	28	45
		298.93/98.78	40	29	45
PFHS	2.45 - 3.05	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.80 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
¹³ C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of $\pm 25\%$ ($\pm 30\%$ for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.0880 to 0.122 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.18 to 0.25 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within $\pm 25\%$, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by diluting 1 mL water with 10.0 mL MeOH:LCMS-water (60:40). Method blanks were prepared to evaluate the levels of background contamination in the overall dilution process (glassware, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carryover.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of LCMS-water, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCS)

One lab control spike was prepared and analyzed.

LCS was prepared by spiking known amounts of the analytes into 1.0 mL of LCMS-water to produce the desired concentration.

The spiked water sample was diluted with 10.0 mL MeOH:LCMS-water (60:40) and analyzed in the same manner as the samples.

The LCS produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 7 summarizes the LCS recovery results

Table 7. Lab Control Spike Results.

Dilution date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
27 February 2019	LCS1	80	88.7	90.6	75.1	76.1	104	96.1	101

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing PFBS, PFHS, PFOA, ^{13}C -PFOA, PFOS, FOSA and ^{13}C -PFOS (QC Lab Low and QC Lab High).

The QCs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy, except for one sample (QC lab high inj2, ^{13}C -PFOA < 25%)

Table 8 summarizes the QCs recovery results.

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
25 Jan 2018	QC Lab Low inj1	10	100	92.4	103	124	107	102	107
	QC Lab High inj1	100	86.8	77.3	99.2	81.0	101	90.7	101
	QC Lab Low inj2	10	98.9	91.6	101	90.3	109	105	111
	QC Lab High inj2	100	91.2	81.7	103	68.9 (a)	108	95.7	107

(a) Outside the acceptance criteria

3.7. Equations.Recovery.

$$\text{Recovery (\%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

The following tables summarize the results for the sample locations.

Table 9. Sample Results PFBS, PFHS, PFOA, PFOS and FOSA

	PFBS		PFHS		PFOA	PFOS	FOSA
	ng/ml		ng/ml		ng/ml	ng/ml	ng/ml
Effluent WWTP			6.06		10.5	23.8	0.340
Bemalingsstation			12.9		16.1	18.2	<0.209
Collector Put			23.7		60.3	137	7.50
Field Trip Blank	<1.14		<1.25		<0.977	<1.35	<1.13

Table 10. Sample Results ¹³C-PFOA and ¹³C-PFOS

	¹³ C-PFOA		¹³ C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
Effluent WWTP	45.1	78.6	49.5	80.4
Bemalingsstation	43.5	75.9	50.8	82.5
Collector Put	41.0	71.4	47.0	76.4
Field Trip Blank	7.80	90.3	128	104

5. Conclusion.

- The recovery of ¹³C-PFOA and ¹³C-PFOS fell within the method acceptance criterion of 70-130%.
- Lab control spike recoveries fell within the method acceptance criteria for all compounds, indicating that the analysis method is in control,
- QC Lab Standards recoveries fell within the method acceptance criteria for all compounds, except for one sample (QC Lab high inj2, recovery of ¹³C-PFOA < 25%).

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

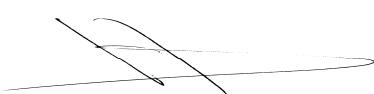
7. Addendum.

Addendum: e-mail from Elien Kemme (ERM Belgium).

8. Signatures.

Sven Herremans,
Technical Manager

Date March 15th, 2019



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date March 15th, 2019

I.A.C.
A Division of SGS Belgium NV

Reports are established on behalf of and for the account of the principal, who expressly accepts that these reports purely represent the situation at a given time and that they must always be presented and/or mentioned in their totality and in their particular context. SGS Belgium N.V., issuer of the reports, cannot be held liable for errors or modification of results during electronic or fax transmission. Only the originally signed report is binding.

The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.

Final Report

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

January 2019 Sampling

Laboratory Request Number: ISO19-14-01

Report Date – Date of Last Signature

Testing Laboratory

3M Environment, Health, Safety & Medical
3M EHS Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Charlotte Tack
3M Belgium
3M Belgium; ZW019/01/01
Phone: [REDACTED]



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M EHS Laboratory

3M EHS Laboratory Manager: Brian T. Mader
3M Principal Analytical Investigator: Susan Wolf
Report Author: Chelsie Grochow

Analytical Report ISO19-14-01

Water Sample Analysis at 3M Antwerp, Belgium
January 2019 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M EHS Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. All samples were collected January 30 – February 1, 2019, and returned to the 3M EHS Laboratory on February 6, 2019, at ambient temperature. The results in this report apply to the samples as received from ERM. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS) (select locations only), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO19-14-01.

The 3M EHS Laboratory prepared sample containers for sixty-three sampling locations. Each sample set consisted of a field sample and field sample duplicate. Fourteen locations also included a target analyte field matrix spike. Each empty container was marked with a “fill to here” line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with a mass-labeled internal standard mix and select sample bottles were fortified with surrogate recovery standards [$^{13}\text{C}_4$]-PFOA and [$^{13}\text{C}_4$]-PFOS prior to being sent to the field for sample collection. During sample collection, sample location P262 was not sampled and sample location P265C was changed to P265B as indicated on the chain of custody.

Samples were prepared and analyzed using method ETS-8-044.3 “Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis”. Select internal standards were used to aid in the data quality objectives.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 “General Requirements for the Competence of Testing and Calibration Laboratories”, in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary ⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: 2nd Aquifer					
ISO19-14-01-001	BD24-3; Sample	29.2	28.6	980	3.40
ISO19-14-01-001-DUP	BD24-3; Sample Dup	27.0	28.2	974	3.02
		Average	28.1	28.4	977
		%RPD Sample/Sample Dup	7.8	1.4	0.61
ISO19-14-01-002	BD24-4; Sample	21.6	8.54	3.10	0.157
ISO19-14-01-002-DUP	BD24-4; Sample Dup	22.0	8.60	3.14	0.147
		Average	21.8 ⁽²⁾	8.57 ⁽²⁾	3.12 ⁽²⁾
		%RPD Sample/Sample Dup	1.8	0.70	1.3
ISO19-14-01-003	D09; Sample	382	14200	3120	23.0
ISO19-14-01-003-DUP	D09; Sample Dup	385	13900	3230	22.4
		Average	384	14100	3180
		%RPD Sample/Sample Dup	0.78	2.1	3.5
ISO19-14-01-004	D10; Sample	420	177	42.7	0.0716
ISO19-14-01-004-DUP	D10; Sample Dup	436	176	43.2	0.0752
		Average	428	177	43.0
		%RPD Sample/Sample Dup	3.7	0.57	1.2
ISO19-14-01-005	D11; Sample	36.7	51.4	711	96.6
ISO19-14-01-005-DUP	D11; Sample Dup	34.4	49.2	713	96.4
		Average	35.6	50.3	712
		%RPD Sample/Sample Dup	6.5	4.4	0.28
ISO19-14-01-006	D14; Sample	2.02	1.61	1.78	0.102
ISO19-14-01-006-DUP	D14; Sample Dup	2.18	1.69	1.87	0.138
		Average	2.10 ⁽²⁾	1.65 ⁽²⁾	1.83 ⁽²⁾
		%RPD Sample/Sample Dup	7.6	4.8	4.9
ISO19-14-01-007	D16; Sample	188	64.0	1210	88.0
ISO19-14-01-007-DUP	D16; Sample Dup	189	63.4	1200	84.6
		Average	189	63.7	1210
		%RPD Sample/Sample Dup	0.53	0.94	0.83
ISO19-14-01-008	D17; Sample	747	236	232	0.113
ISO19-14-01-008-DUP	D17; Sample Dup	714	240	235	0.0902
		Average	731	238	234
		%RPD Sample/Sample Dup	4.5	1.7	1.3
ISO19-14-01-009	D18; Sample	286	205	72.1	1.96
ISO19-14-01-009-DUP	D18; Sample Dup	284	204	61.5	1.83
		Average	285	205	66.8
		%RPD Sample/Sample Dup	0.70	0.49	16
					6.9

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 12%, PFBS \pm 5.7%, PFHS \pm 10%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 16%, PFBS \pm 14%, PFHS \pm 30%, PFOS \pm 28%, and PFOSA \pm 17%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary ⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: 2nd Aquifer					
ISO19-14-01-010	D5; Sample	317	175	287	2.46
ISO19-14-01-010-DUP	D5; Sample Dup	304	172	284	2.42
		Average	311	174	286
		%RPD Sample/Sample Dup	4.2	1.7	1.1
ISO19-14-01-011	ND7; Sample	161	75.0	47.4	8.32
ISO19-14-01-011-DUP	ND7; Sample Dup	147	75.0	47.1	8.56
		Average	154	75.0	47.3
		%RPD Sample/Sample Dup	9.1	0.0	0.63
ISO19-14-01-012	P118A; Sample	1450	2350	141	1.03
ISO19-14-01-012-DUP	P118A; Sample Dup	1470	2350	140	1.07
		Average	1460	2350	141
		%RPD Sample/Sample Dup	1.4	0.0	0.71
ISO19-14-01-013	P118B; Sample	1980	2320	13900	7.26
ISO19-14-01-013-DUP	P118B; Sample Dup	1980	2360	14400	6.36
		Average	1980	2340	14200
		%RPD Sample/Sample Dup	0.0	1.7	3.5
ISO19-14-01-014	P119A; Sample	34.2	10.7	6.16	0.930
ISO19-14-01-014-DUP	P119A; Sample Dup	36.2	10.6	5.76	0.914
		Average	35.2	10.7	5.96
		%RPD Sample/Sample Dup	5.7	0.94	6.7
ISO19-14-01-015	P119B; Sample	1410	670	300	0.528
ISO19-14-01-015-DUP	P119B; Sample Dup	1430	673	305	0.528
		Average	1420	672	303
		%RPD Sample/Sample Dup	1.4	0.45	1.7
ISO19-14-01-016	P121; Sample	0.534	0.296	0.462	0.0666
ISO19-14-01-016-DUP	P121; Sample Dup	0.532	0.310	0.448	0.0810
		Average	0.533 ⁽²⁾	0.303 ⁽²⁾	0.455 ⁽²⁾
		%RPD Sample/Sample Dup	0.38	4.6	3.1
ISO19-14-01-017	3M vijver; Sample	0.716	0.380	1.93	0.0306
ISO19-14-01-017-DUP	3M vijver; Sample Dup	0.722	0.392	1.93	<0.0250
		Average	0.719 ⁽²⁾	0.386 ⁽²⁾	1.93 ⁽²⁾
		%RPD Sample/Sample Dup	0.83	3.1	0.0
ISO19-14-01-018	Blokkersdijkvijver Noord; Sample	0.872	0.484	4.98	0.202
ISO19-14-01-018-DUP	Blokkersdijkvijver Noord; Sample Dup	0.884	0.478	5.42	0.210
		Average	0.878 ⁽²⁾	0.481 ⁽²⁾	5.20 ⁽²⁾
		%RPD Sample/Sample Dup	1.4	1.2	8.5

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 12%, PFBS \pm 5.7%, PFHS \pm 10%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 16%, PFBS \pm 14%, PFHS \pm 30%, PFOS \pm 28%, and PFOSA \pm 17%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Blokkersdijk Nature Reserve					
ISO19-14-01-019	Blokkersdijkvijver standard; Sample	0.822	0.476	1.07	0.0378
ISO19-14-01-019-DUP	Blokkersdijkvijver standard; Sample Dup	0.796	0.464	1.03	0.0394
		Average %RPD Sample/Sample Dup	0.809 ⁽²⁾ 3.2	0.470 ⁽²⁾ 2.6	1.05 ⁽²⁾ 3.8
		0.0386 4.1			
ISO19-14-01-020	P321; Sample	2440	547	5670	0.262
ISO19-14-01-020-DUP	P321; Sample Dup	2480	552	6120	0.256
		Average %RPD Sample/Sample Dup	2460 1.6	550 0.91	5900 7.6
		0.259 2.3			
ISO19-14-01-021	L21; Sample	<0.192	0.133	4.20	0.370
ISO19-14-01-021-DUP	L21; Sample Dup	<0.192	0.131	4.16	0.348
		Average %RPD Sample/Sample Dup	<0.192 ⁽²⁾ NA	0.132 ⁽²⁾ 1.5	4.18 ⁽²⁾ 0.96
		0.359 6.1			
ISO19-14-01-022	L22; Sample	0.358	0.230	2.20	0.118
ISO19-14-01-022-DUP	L22; Sample Dup	0.332	0.238	2.36	0.0536
		Average %RPD Sample/Sample Dup	0.345 ⁽²⁾ 7.5	0.234 ⁽²⁾ 3.4	2.28 ⁽²⁾ 7.0
		0.0858 75 ⁽³⁾			
ISO19-14-01-023	L31; Sample	0.252	0.752	3.96	0.0540
ISO19-14-01-023-DUP	L31; Sample Dup	0.248	0.812	4.34	0.0500
		Average %RPD Sample/Sample Dup	0.250 ⁽²⁾ 1.6	0.782 ⁽²⁾ 7.7	4.15 ⁽²⁾ 9.2
		0.0520 7.7			
ISO19-14-01-024	L4; Sample	2.32	1.61	25.6	1.39
ISO19-14-01-024-DUP	L4; Sample Dup	2.52	1.90	28.0	1.65
		Average %RPD Sample/Sample Dup	2.42 ⁽²⁾ 8.3	1.76 ⁽²⁾ 17	26.8 ⁽²⁾ 9.0
		1.52 17			
ISO19-14-01-025	P114bis; Sample	3.46	1.47	4.24	0.0692
ISO19-14-01-025-DUP	P114bis; Sample Dup	3.30	1.41	3.60	0.0576
		Average %RPD Sample/Sample Dup	3.38 ⁽²⁾ 4.7	1.44 ⁽²⁾ 4.2	3.92 ⁽²⁾ 16
		0.0634 18			
ISO19-14-01-026	P115; Sample	2.88	1.75	2.16	<0.0250
ISO19-14-01-026-DUP	P115; Sample Dup	2.76	1.61	1.68	<0.0250
		Average %RPD Sample/Sample Dup	2.82 ⁽²⁾ 4.3	1.68 ⁽²⁾ 8.3	1.92 ⁽²⁾ 25 ⁽³⁾
		<0.0250 NA			
ISO19-14-01-027	P116; Sample	0.702	0.410	7.58	0.115
ISO19-14-01-027-DUP	P116; Sample Dup	0.720	0.480	8.38	0.152
		Average %RPD Sample/Sample Dup	0.711 ⁽²⁾ 2.5	0.445 ⁽²⁾ 16	7.98 ⁽²⁾ 10
		0.134 28 ⁽³⁾			

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 12%, PFBS \pm 5.7%, PFHS \pm 10%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 16%, PFBS \pm 14%, PFHS \pm 30%, PFOS \pm 28%, and PFOSA \pm 17%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFBS	PFHS	PFOSA ⁽²⁾
Zone: Effluent WWTP					
ISO19-14-01-028	Effluent WWTP; Sample	10.4	6.08	40.6	0.934
ISO19-14-01-028-DUP	Effluent WWTP; Sample Dup	11.4	6.44	47.6	1.15
Average		10.9⁽²⁾	6.26⁽²⁾	44.1⁽²⁾	1.04
%RPD Sample/Sample Dup		9.2	5.8	16	21⁽³⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
ISO19-14-01-029	PP01; Sample	164	95.1	116	1270	29.2
ISO19-14-01-029-DUP	PP01; Sample Dup	170	97.0	118	1260	25.8
Average		167	96.1	117	1270	27.5
%RPD Sample/Sample Dup		3.6	2.0	1.7	0.79	12
ISO19-14-01-030	PP02; Sample	487	29.7	3940	20800	26.6
ISO19-14-01-030-DUP	PP02; Sample Dup	517	30.5	4060	21700	27.6
Average		502	30.1	4000	21300	27.1
%RPD Sample/Sample Dup		6.0	2.7	3.0	4.2	3.7
ISO19-14-01-031	PP04; Sample	691	1270	289	5370	57.6
ISO19-14-01-031-DUP	PP04; Sample Dup	672	1270	292	5480	58.6
Average		682	1270	291	5430	58.1
%RPD Sample/Sample Dup		2.8	0.0	1.0	2.0	1.7
ISO19-14-01-032	PP05; Sample	469	3380	2390	417	38.8
ISO19-14-01-032-DUP	PP05; Sample Dup	483	3410	2400	437	39.8
Average		476	3400	2400	427	39.3
%RPD Sample/Sample Dup		2.9	0.88	0.42	4.7	2.5
ISO19-14-01-033	PP06; Sample	241	21.5	78.3	1730	64.4
ISO19-14-01-033-DUP	PP06; Sample Dup	232	21.5	77.7	1650	59.6
Average		237	21.5	78.0	1690	62.0
%RPD Sample/Sample Dup		3.8	0.0	0.77	4.7	7.7
ISO19-14-01-034	PP07; Sample	317	179	107	2080	58.0
ISO19-14-01-034-DUP	PP07; Sample Dup	289	175	105	2150	59.2
Average		303	177	106	2120	58.6
%RPD Sample/Sample Dup		9.2	2.3	1.9	3.3	2.0
ISO19-14-01-035	PP08; Sample	565	29.5	258	5880	38.8
ISO19-14-01-035-DUP	PP08; Sample Dup	572	29.9	266	5950	37.4
Average		569	29.7	262	5920	38.1
%RPD Sample/Sample Dup		1.2	1.3	3.1	1.2	3.7

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 12%, PFBS \pm 5.7%, PFHS \pm 10%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 16%, PFBS \pm 14%, PFHS \pm 30%, PFOS \pm 28%, and PFOSA \pm 17%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary⁽¹⁾

		Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
ISO19-14-01-036	PP09; Sample	580	18.3	303	2340	14.5
ISO19-14-01-036-DUP	PP09; Sample Dup	599	18.3	302	2370	14.2
		Average	590	18.3	303	2360
		%RPD Sample/Sample Dup	3.2	0.0	0.33	1.3
ISO19-14-01-037	PP10; Sample	537	16.1	204	3730	17.5
ISO19-14-01-037-DUP	PP10; Sample Dup	558	15.4	200	3600	17.2
		Average	548	15.8	202	3670
		%RPD Sample/Sample Dup	3.8	4.4	2.0	3.5
						1.7

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Palingbeek & Tophatgracht					
ISO19-14-01-038	12; Sample	43.4	14.4	179	0.344
ISO19-14-01-038-DUP	12; Sample Dup	45.0	14.2	166	0.298
		Average	44.2⁽²⁾	14.3⁽²⁾	173⁽²⁾
		%RPD Sample/Sample Dup	3.6	1.4	7.5
ISO19-14-01-039	13; Sample	44.0	15.9	154	0.324
ISO19-14-01-039-DUP	13; Sample Dup	43.6	15.3	156	0.330
		Average	43.8⁽²⁾	15.6⁽²⁾	155⁽²⁾
		%RPD Sample/Sample Dup	0.91	3.8	1.3
ISO19-14-01-040	5; Sample	17.1	14.6	32.8	0.206
ISO19-14-01-040-DUP	5; Sample Dup	16.3	14.5	33.0	0.222
		Average	16.7⁽²⁾	14.6⁽²⁾	32.9⁽²⁾
		%RPD Sample/Sample Dup	4.8	0.69	0.61
ISO19-14-01-041	Bemalingsstation; Sample	17.2	12.5	29.6	0.134
ISO19-14-01-041-DUP	Bemalingsstation; Sample Dup	16.1	12.2	30.2	0.168
		Average	16.7	12.4⁽²⁾	29.9⁽²⁾
		%RPD Sample/Sample Dup	6.6	2.4	2.0
					23⁽³⁾
Zone: Sewer					
ISO19-14-01-042	Collector put; Sample	50.8	23.2	212	18.4
ISO19-14-01-042-DUP	Collector put; Sample Dup	50.8	23.2	208	20.0
		Average	50.8⁽²⁾	23.2⁽²⁾	210
		%RPD Sample/Sample Dup	0.0	0.0	1.9
					8.3

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 12%, PFBS \pm 5.7%, PFHS \pm 10%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 16%, PFBS \pm 14%, PFHS \pm 30%, PFOS \pm 28%, and PFOSA \pm 17%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary⁽¹⁾

		Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - Building 16						
ISO19-14-01-043	P27; Sample	234	14900	42.5	1720	36.4
ISO19-14-01-043-DUP	P27; Sample Dup	248	15000	41.6	1700	39.2
		Average	241	15000	42.1	1710
		%RPD Sample/Sample Dup	5.8	0.67	2.1	1.2
ISO19-14-01-044	P21B; Sample	9640	7930	12200	66100	3.86
ISO19-14-01-044-DUP	P21B; Sample Dup	9440	7880	12100	64900	3.74
		Average	9540	7910	12200	65500
		%RPD Sample/Sample Dup	2.1	0.63	0.82	1.8
ISO19-14-01-045	P304; Sample	412	262	121	1100	14.6
ISO19-14-01-045-DUP	P304; Sample Dup	391	258	119	1060	14.9
		Average	402	260	120	1080
		%RPD Sample/Sample Dup	5.2	1.5	1.7	3.7
ISO19-14-01-046	P305; Sample	152	132	185	500	10.4
ISO19-14-01-046-DUP	P305; Sample Dup	137	132	183	473	9.64
		Average	145	132	184	487
		%RPD Sample/Sample Dup	10	0.0	1.1	5.5
ISO19-14-01-047	P42; Sample	276	31.9	267	3610	12.0
ISO19-14-01-047-DUP	P42; Sample Dup	275	32.1	268	3760	12.2
		Average	276	32.0	268	3690
		%RPD Sample/Sample Dup	0.36	0.63	0.37	4.1
						1.7

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area – WWTP					
ISO19-14-01-048	L19; Sample	253	176	3950	26.4
ISO19-14-01-048-DUP	L19; Sample Dup	261	175	3960	27.4
		Average	257	176	3960
		%RPD Sample/Sample Dup	3.1	0.57	0.25
ISO19-14-01-049	M4; Sample	196	79.8	2120	112
ISO19-14-01-049-DUP	M4; Sample Dup	192	77.8	2110	114
		Average	194	78.8	2120
		%RPD Sample/Sample Dup	2.1	2.5	0.47
ISO19-14-01-050	P118C; Sample	188	83.6	1760	53.2
ISO19-14-01-050-DUP	P118C; Sample Dup	190	84.5	1760	48.4
		Average	189	84.1	1760
		%RPD Sample/Sample Dup	1.1	1.1	0.0
					9.4

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 12%, PFBS \pm 5.7%, PFHS \pm 10%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 16%, PFBS \pm 14%, PFHS \pm 30%, PFOS \pm 28%, and PFOSA \pm 17%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - WWTP					
ISO19-14-01-051	P119C; Sample	1720	655	10500	132
ISO19-14-01-051-DUP	P119C; Sample Dup	1790	659	10500	128
		Average	657	10500	130
		%RPD Sample/Sample Dup	4.0	0.61	3.1
ISO19-14-01-053	P263; Sample	1210	393	9090	9.34
ISO19-14-01-053-DUP	P263; Sample Dup	1160	390	8940	8.84
		Average	392	9020	9.09
		%RPD Sample/Sample Dup	4.2	0.77	5.5
ISO19-14-01-054	P264; Sample	339	218	1490	69.4
ISO19-14-01-054-DUP	P264; Sample Dup	332	223	1500	67.2
		Average	221	1500	68.3
		%RPD Sample/Sample Dup	2.1	2.3	3.2
ISO19-14-01-055	P265B; Sample	128	44.8	581	81.0
ISO19-14-01-055-DUP	P265B; Sample Dup	121	43.5	582	79.8
		Average	44.2	582	80.4
		%RPD Sample/Sample Dup	5.6	2.9	0.17
ISO19-14-01-056	P340; Sample	497	260	2270	22.6
ISO19-14-01-056-DUP	P340; Sample Dup	519	265	2290	22.2
		Average	263	2280	22.4
		%RPD Sample/Sample Dup	4.3	1.9	0.88
ISO19-14-01-057	P341; Sample	795	247	5670	50.6
ISO19-14-01-057-DUP	P341; Sample Dup	795	248	5910	50.2
		Average	248	5790	50.4
		%RPD Sample/Sample Dup	0.0	0.40	4.1
ISO19-14-01-058	P343; Sample	657	461	6770	12.1
ISO19-14-01-058-DUP	P343; Sample Dup	617	452	6550	11.0
		Average	457	6660	11.6
		%RPD Sample/Sample Dup	6.3	2.0	3.3
ISO19-14-01-059	P371; Sample	589	488	1710	10.3
ISO19-14-01-059-DUP	P371; Sample Dup	621	489	1730	10.3
		Average	489	1720	10.3
		%RPD Sample/Sample Dup	5.3	0.20	1.2
ISO19-14-01-060	P374; Sample	197	252	1470	47.6
ISO19-14-01-060-DUP	P374; Sample Dup	206	255	1450	49.6
		Average	254	1460	48.6
		%RPD Sample/Sample Dup	4.5	1.2	1.4
					4.1

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 12%, PFBS \pm 5.7%, PFHS \pm 10%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 16%, PFBS \pm 14%, PFHS \pm 30%, PFOS \pm 28%, and PFOSA \pm 17%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - WWTP					
ISO19-14-01-061	P379; Sample	50.6	16.0	658	65.4
ISO19-14-01-061-DUP	P379; Sample Dup	46.2	15.5	630	68.4
		Average	48.4	15.8	644
		%RPD Sample/Sample Dup	9.1	3.2	66.9
ISO19-14-01-062	P380; Sample	553	495	2230	37.8
ISO19-14-01-062-DUP	P380; Sample Dup	552	494	2200	37.4
		Average	553	495	2220
		%RPD Sample/Sample Dup	0.18	0.20	37.6
				1.4	1.1
Zone: Southern Site Boundary					
ISO19-14-01-063	P372; Sample	53.4	46.9	1190	26.6
ISO19-14-01-063-DUP	P372; Sample Dup	50.8	46.8	1240	26.4
		Average	52.1	46.9	1220
		%RPD Sample/Sample Dup	5.0	0.21	26.5
				4.1	0.75

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
ISO19-14-01-064	Travel Blank	<0.192 ⁽²⁾	<0.200 ⁽²⁾	<0.100 ⁽²⁾	<0.185 ⁽²⁾	<0.0250 ⁽²⁾

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 12%, PFBS ± 5.7%, PFHS ± 10%, and PFOS ± 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 16%, PFBS ± 14%, PFHS ± 30%, PFOS ± 28%, and PFOSA ± 17%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis".

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected on January 30 – February 1, 2019, in Nalgene™ (high-density polyethylene) bottles prepared at the 3M EHS Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. All sample locations were spiked with a mass-labeled internal standard mix at a nominal concentration

of 1 ng/mL and select samples were fortified with surrogate recovery standards (SRSs) at a nominal concentration of 0.1 ng/mL prior to sample collection. Samples were received on February 6, 2019.

2.3 Sample Preparation

Sample locations pre-spiked with internal standard and surrogates were analyzed for all analytes (PFBS for select locations) by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples along with the method blanks.

Samples requiring dilutions were prepared for PFOA, PFHS, PFOS and PFBS (select locations only) using a direct injection by methanol dilution method. Samples that required a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples that required a 1:100 dilution were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol. Samples that required a 1:500 dilution were prepared by diluting 0.02 mL of a well-mixed sample with 10.0 mL of methanol. Samples that required a 1:1000 dilution were prepared by first diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol, followed by a second dilution prepared by diluting 1 mL of the diluted sample into 9 mL of methanol. Diluted samples and LSCs were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

2.4 Analysis

All samples and quality control samples were analyzed for the target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). SRSs were added and included in the analysis of these samples. Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.2. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

The following analytical runs were used to report the results herein:

2/21/19 (ETS Kirk) Internal Standard Calibration Analysis:

- Sample locations pre-spiked with surrogates were analyzed for PFOA, PFHS, PFOS, and PFOSA with select locations being analyzed for PFBS. All sample results were reported **except** for the following sample locations: L22, L31, and P115 (PFOSA), Bemalingstation (PFOA), and Collector put (PFOS).

2/25/19 (ETS DaVinci) Internal Standard Calibration Analysis:

- All sample locations not included in the first analysis were analyzed and reported for PFOSA.

2/26/19 (ETS Tesla) External Standard Calibration Analysis:

- All sample locations were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported **except** for the following sample location: D09 (PFHS) and PP05 (all analytes).

2/28/19 (ETS Tesla) External Standard Calibration Analysis:

- All sample locations were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS except for locations reanalyzed for specific analytes: D09 (PFHS), Bemalingstation (PFOA), and Collector put (PFOS). All sample results were reported **except** for the following sample locations: P119C (PFOS), P27 (PFBS), and P21B (PFOS).

3/5/19 (ETS Tesla) External Standard Calibration Analysis:

- Sample locations were reanalyzed and reported for select analytes: P119C (PFOS), P27 (PFBS), and P21B (PFOS).

3/6/19 (ETS Tesla) Internal Standard Calibration Analysis:

- Sample locations L22, L31, and P115 were reanalyzed and reported for PFOSA.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk	ETS DaVinci	ETS Tesla
Liquid Chromatograph	Agilent 1260	Agilent 1200	Agilent 1260
Analysis Method	ETS-8-044.3	ETS-8-044.3	ETS-8-044.3
Analysis Date	2/21/19	2/25/19	2/26/19, 2/28/19, 3/5/19, 3/6/19
Guard column	Prism RP (2.1 mm X 50 mm), 5 μ	Prism RP (2.1 mm X 50 mm), 5 μ	Prism RP (2.1 mm X 50 mm), 5 μ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ
Injection Volume	5 μ L	10 μ L	5 μ L
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 6500	AB Sciex Triple Quad 6500+
Ion Source	Turbo Spray	Turbo Spray	Turbo Spray
Polarity	Negative	Negative	Negative
Software	Analyst 1.6.3	Analyst 1.6.3	Analyst 1.6.3

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis				
Step Number	Total Time (min)	Flow Rate (μ L/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0.00	300	90.0	10.0
1	0.50	300	90.0	10.0
2	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard	Mass Transition Q1/Q3
PFOA	413/369	$[^{13}\text{C}_8]\text{-PFOA}$	421/376
	413/219		
	413/169		
PFBS	299/99	$[^{18}\text{O}_2]\text{-PFBS}$	303/84
	299/80		
PFHS	399/99	$[^{13}\text{C}_3]\text{-PFHS}$	402/80
	399/80		
PFOS	499/99	$[^{13}\text{C}_8]\text{-PFOS}$	507/80
	499/80		
	499/130		
PFOSA	498/78	$[^{13}\text{C}_8]\text{-PFOSA}$	506/78
$[^{13}\text{C}_4]\text{-PFOA}$	417/372	$[^{13}\text{C}_8]\text{-PFOA}$	421/376
$[^{13}\text{C}_4]\text{-PFOS}$	503/80	$[^{13}\text{C}_8]\text{-PFOS}$	507/80

The individual transitions were summed to produce a “total ion chromatogram” (TIC), which was used for quantitation.

3 Data Analysis

3.1 Calibration

2/21/19, 2/25/19, and 3/6/19 Analysis (Internal Standard Calibration): Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of fourteen calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed on 2/21/19 and 2/25/19. Nine calibration standards ranging from 0.0125 ng/mL to 5.0 ng/mL (nominal) were analyzed on 3/6/19. The calibration standards also contained surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

2/26/19, 2/28/19, and 3/5/19 Analysis (External Standard Calibration): Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL to 25 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±25% (100±30% for the lowest curve point) were met for all analytes. The correlation coefficient (*r*) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes for each analysis.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.3 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the **Table 6** below.

Table 6. LOQ

Analyte	LOQ, ng/mL ⁽²⁾ 2/21/19 Analysis	LOQ, ng/mL ⁽²⁾ 2/25/19 Analysis	LOQ, ng/mL ⁽¹⁾ 2/26/19 Analysis	LOQ, ng/mL ⁽¹⁾ 2/28/19 Analysis	LOQ, ng/mL ⁽¹⁾ 3/5/19 Analysis	LOQ, ng/mL ⁽²⁾ 3/6/19 Analysis
PFOA	0.192	NA	0.0192	0.0479	NA	NA
PFBS	0.200	NA	0.0200	0.0200	0.0200	NA
PFHS	0.100	NA	0.0200	0.0200	NA	NA
PFOS	0.185	NA	0.0464	0.0464	0.232	NA
PFOSA	0.0250	0.0250	NA	NA	NA	0.0250

NA = Not Applicable

(1) A dilution factor was not applied to the LOQ.

(2) A dilution factor of 2 was applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of $100\% \pm 25\%$.

3.5 Blanks

Two types of blanks were prepared and analyzed with the samples: method/solvent blanks and field/trip blanks. Each blank result was reviewed and used to evaluate method performance. The method blanks were used to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. Target analyte LCSs analyzed on 2/21/19 and 2/25/19 were prepared at nominal concentrations of 0.2 ng/mL, 20 ng/mL, and 140 ng/mL. Target analyte LCSs analyzed on 2/26/19 and 2/28/19 were prepared at nominal concentrations of 100 ng/mL, 5000 ng/mL, and 35000 ng/mL. Target analyte LCSs analyzed on 3/5/19 were prepared at nominal concentrations of 200 ng/mL, 20000 ng/mL, and 70000 ng/mL. Target analyte LCSs analyzed on 3/6/19 were prepared at nominal concentrations of 0.2 ng/mL, 2 ng/mL, and 7 ng/mL. LCSs were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. Surrogates [¹³C₄]-PFOA and [¹³C₄]-PFOS were added post dilution when analyzed by external standard.

Method ETS-8-044.3 states that the average recovery of LCSs at each spiking level must be within 80%-120% with an RSD $\leq 20\%$. All LCS samples met criteria with the following exceptions:

- 2/21/19: All high-level LCSs for PFOA, PFBS, and PFOSA and two high-level LCSs for PFHS were spiked above the resulting ULOQ. Two low-level LCSs for PFBS were spiked below the resulting LLOQ. The average recovery of the high-level LCSs was 70.5% for PFHS and 72.2% for PFOS.
- 3/5/19: Low-level LCSs for PFOS were spiked below the resulting LLOQ.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report. Individual LCS results are included in the raw data and available upon request.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.4. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in **Table 7** below.

- The data uncertainty for PFHS using internal calibration was calculated at $\pm 17\%$ following ETS-12-012.4; however, the data uncertainty was expanded to $\pm 30\%$ based on the PFHS recovery of the high-level LCSs from the analysis on 2/21/19.
- The data uncertainty for PFOS using internal calibration was calculated at $\pm 20\%$ following ETS-12-012.4; however, the data uncertainty was expanded to $\pm 28\%$ based on the PFOS recovery of the high-level LCSs from the analysis on 2/21/19.

Table 7. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	6.01	$\pm 12\%$
PFBS	External	2.84	$\pm 5.7\%$
PFHS	External	4.99	$\pm 10\%$
PFOS	External	7.30	$\pm 15\%$
PFOA	Internal	8.20	$\pm 16\%$
PFBS	Internal	7.20	$\pm 14\%$
PFHS	Internal	NA	$\pm 30\%$
PFOS	Internal	NA	$\pm 28\%$
PFOSA	Internal	8.48	$\pm 17\%$

NA = Not Applicable

3.8 Field Matrix Spikes (FMS)

Target analyte field matrix spike samples were collected for select sampling points to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions at 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOA and PFOS. The standards used for the preparation of the field matrix spiking solutions greater than 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, select sample bottles contained stable isotope surrogate recovery spikes of [¹³C₄]-PFOA and [¹³C₄]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles prior to sample collection. The [¹³C₄]-PFOA was selected to represent the C8 perfluorocarboxylic acid. The [¹³C₄]-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

The following calculation was used to generate data in section 4 of the report.

$$FMS\ Recovery = \frac{Sample\ Concentration\ of\ FMS - Average\ Concentration\ (Field\ Sample\ & Field\ Sample\ Dup)}{Spike\ Concentration} \times 100\%$$

Table 8. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
P121, P115	FMS	1.92	2.00	2.00	1.85	2.00
Blokkersdijkvijver - standard	FMS	10.0	10.0	10.0	10.0	10.0
BD24-3, Bemalingstation	FMS	110	110	110	109	10.0
P118A, P305	FMS	1010	1010	1010	1010	10.0
P27	FMS	2050	2050	2050	2050	50.0
PP01, PP06, L19, P264, P371	FMS	2050	50.0	2050	2050	50.0
D11 ⁽¹⁾	FMS	1810	44.2	1810	1810	44.2
Trip Blank	Low	1.92	2.00	2.00	1.85	2.00
	High	2050	50.0	2050	2050	50.0

(1) Sample container for the FMS sample was overfilled by 10%. The FMS true values were adjusted accordingly.

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of $\pm 30\%$, demonstrate that the method is appropriate for the given matrix.

The method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. At times, the spike level exceeded the recommended upper limit of 10 times the analyte concentration. In these instances, the FMS recovery was reported and flagged as above 10 times the sample concentration.

For PFOA, PFBS, PFHS, and PFOS, when the field matrix spike level was not appropriate as compared to the endogenous sample concentration, the surrogate recovery standards were used to assess method accuracy. All field matrix spike and surrogate recovery standard recoveries met acceptance criteria.

Table 9. Location ID: BD24-3

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-001	BD24-3; Sample	29.2	NA	28.6	NA
ISO19-14-01-001-DUP	BD24-3; Sample Dup	27.0	NA	28.2	NA
ISO19-14-01-001-FMS	BD24-3; FMS	151	112	145	106
Average Concentration (ng/mL) ± %RPD		28.1 ng/mL ± 7.8%		28.4 ng/mL ± 1.4%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-001	BD24-3; Sample	980	NA	3.40	NA
ISO19-14-01-001-DUP	BD24-3; Sample Dup	974	NA	3.02	NA
ISO19-14-01-001-FMS	BD24-3; FMS	1100	NC	11.9	86.9
Average Concentration (ng/mL) ± %RPD		977 ng/mL ± 0.61%		3.21 ng/mL ± 12%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration

Table 10. Location ID: D11

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-005	D11; Sample	36.7	NA	51.4	NA
ISO19-14-01-005-DUP	D11; Sample Dup	34.4	NA	49.2	NA
ISO19-14-01-005-FMS	D11; FMS	2080	113 ⁽¹⁾	2100	113 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		35.6 ng/mL ± 6.5%		50.3 ng/mL ± 4.4%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-005	D11; Sample	711	NA	96.6	NA
ISO19-14-01-005-DUP	D11; Sample Dup	713	NA	96.4	NA
ISO19-14-01-005-FMS	D11; FMS	2690	109	127	NC
Average Concentration (ng/mL) ± %RPD		712 ng/mL ± 0.28%		96.5 ng/mL ± 0.21%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration

(1) FMS concentration greater than 10 times the sample concentration.

Table 11. Location ID: P118A

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-012	P118A; Sample	1450	NA	2350	NA
ISO19-14-01-012-DUP	P118A; Sample Dup	1470	NA	2350	NA
ISO19-14-01-012-FMS	P118A; FMS	2360	89.1	3470	NC
Average Concentration (ng/mL) ± %RPD		1460 ng/mL ± 1.4%		2350 ng/mL ± 0.0%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-012	P118A; Sample	141	NA	1.03	NA
ISO19-14-01-012-DUP	P118A; Sample Dup	140	NA	1.07	NA
ISO19-14-01-012-FMS	P118A; FMS	1150	100	9.96	89.1
Average Concentration (ng/mL) ± %RPD		141 ng/mL ± 0.71%		1.05 ng/mL ± 3.8%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration

Table 12. Location ID: P121

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-016	P121; Sample	0.534	NA	0.296	NA
ISO19-14-01-016-DUP	P121; Sample Dup	0.532	NA	0.310	NA
ISO19-14-01-016-FMS	P121; FMS	2.24	88.9	2.02	85.9
Average Concentration (ng/mL) ± %RPD		0.533 ng/mL ± 0.38%		0.303 ng/mL ± 4.6%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-016	P121; Sample	0.462	NA	0.0666	NA
ISO19-14-01-016-DUP	P121; Sample Dup	0.448	NA	0.0810	NA
ISO19-14-01-016-FMS	P121; FMS	1.97	81.9	1.79	85.8 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		0.455 ng/mL ± 3.1%		0.0738 ng/mL ± 20%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 13. Location ID: Blokkersdijkvijver standaard

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-019	Blokkersdijkvijver standaard; Sample	0.822	NA	0.476	NA
ISO19-14-01-019-DUP	Blokkersdijkvijver standaard; Sample Dup	0.796	NA	0.464	NA
ISO19-14-01-019-FMS	Blokkersdijkvijver standaard; FMS	9.20	83.9 ⁽¹⁾	9.02	85.5 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		0.809 ng/mL ± 3.2%		0.470 ng/mL ± 2.6%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-019	Blokkersdijkvijver standaard; Sample	1.07	NA	0.0378	NA
ISO19-14-01-019-DUP	Blokkersdijkvijver standaard; Sample Dup	1.03	NA	0.0394	NA
ISO19-14-01-019-FMS	Blokkersdijkvijver standaard; FMS	9.98	89.3	9.02	89.8 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		1.05 ng/mL ± 3.8%		0.0386 ng/mL ± 4.1%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 14. Location ID: P115

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-026	P115; Sample	2.88	NA	1.75	NA
ISO19-14-01-026-DUP	P115; Sample Dup	2.76	NA	1.61	NA
ISO19-14-01-026-FMS	P115; FMS	4.64	94.8	3.20	76.0
Average Concentration (ng/mL) ± %RPD		2.82 ng/mL ± 4.3%		1.68 ng/mL ± 8.3%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-026	P115; Sample	2.16	NA	<0.0250	NA
ISO19-14-01-026-DUP	P115; Sample Dup	1.68	NA	<0.0250	NA
ISO19-14-01-026-FMS	P115; FMS	3.46	83.2	1.76	88.0 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		1.92 ng/mL ± 25% ⁽²⁾		<0.0250 ng/mL	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 15. Location ID: PP01

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-029	PP01; Sample	164	NA	95.1	NA	116	NA
ISO19-14-01-029-DUP	PP01; Sample Dup	170	NA	97.0	NA	118	NA
ISO19-14-01-029-FMS	PP01; FMS	2140	96.2 ⁽¹⁾	148	104	2210	102 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		167 ng/mL ± 3.6%		96.1 ng/mL ± 2.0%		117 ng/mL ± 1.7%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-029	PP01; Sample	1270	NA	29.2	NA
ISO19-14-01-029-DUP	PP01; Sample Dup	1260	NA	25.8	NA
ISO19-14-01-029-FMS	PP01; FMS	3220	95.4	70.4	85.8
Average Concentration (ng/mL) ± %RPD		1270 ng/mL ± 0.79%		27.5 ng/mL ± 12%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 16. Location ID: PP06

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-033	PP06; Sample	241	NA	21.5	NA	78.3	NA
ISO19-14-01-033-DUP	PP06; Sample Dup	232	NA	21.5	NA	77.7	NA
ISO19-14-01-033-FMS	PP06; FMS	2180	94.8	75.8	109	2160	102 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		237 ng/mL ± 3.8%		21.5 ng/mL ± 0.0%		78.0 ng/mL ± 0.77%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-033	PP06; Sample	1730	NA	64.4	NA
ISO19-14-01-033-DUP	PP06; Sample Dup	1650	NA	59.6	NA
ISO19-14-01-033-FMS	PP06; FMS	3700	98.0	103	82.0
Average Concentration (ng/mL) ± %RPD		1690 ng/mL ± 4.7%		62.0 ng/mL ± 7.7%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 17. Location ID: Bemalingstation

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-041	Bemalingstation; Sample	17.2	NA	12.5	NA
ISO19-14-01-041-DUP	Bemalingstation; Sample Dup	16.1	NA	12.2	NA
ISO19-14-01-041-FMS	Bemalingstation; FMS	123	97.1	97.2	77.1
Average Concentration (ng/mL) ± %RPD		16.7 ng/mL ± 6.6%		12.4 ng/mL ± 2.4%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-041	Bemalingstation; Sample	29.6	NA	0.134	NA
ISO19-14-01-041-DUP	Bemalingstation; Sample Dup	30.2	NA	0.168	NA
ISO19-14-01-041-FMS	Bemalingstation; FMS	119	81.7	8.80	86.5
Average Concentration (ng/mL) ± %RPD		29.9 ng/mL ± 2.0%		0.151 ng/mL ± 23%⁽¹⁾	

NA = Not Applicable

(1) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%

Table 18. Location ID: P27

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-043	P27; Sample	234	NA	14900	NA	42.5	NA
ISO19-14-01-043-DUP	P27; Sample Dup	248	NA	15000	NA	41.6	NA
ISO19-14-01-043-FMS	P27; FMS	2210	96.0	NA ⁽²⁾	NA	2150	103 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		241 ng/mL ± 5.8%		15000 ng/mL ± 0.67%		42.1 ng/mL ± 2.1%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-043	P27; Sample	1720	NA	36.4	NA
ISO19-14-01-043-DUP	P27; Sample Dup	1700	NA	39.2	NA
ISO19-14-01-043-FMS	P27; FMS	3770	100	80.0	84.4
Average Concentration (ng/mL) ± %RPD		1710 ng/mL ± 1.2%		37.8 ng/mL ± 7.4%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) FMS concentration greater than 10 times the sample concentration.

(2) FMS not re-analyzed for PFBS as it was not an appropriate spike level.

Table 19. Location ID: P305

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-046	P305; Sample	152	NA	132	NA	185	NA
ISO19-14-01-046-DUP	P305; Sample Dup	137	NA	132	NA	183	NA
ISO19-14-01-046-FMS	P305; FMS	1210	106	1210	107	1250	106
Average Concentration (ng/mL) ± %RPD		145 ng/mL ± 10%		132 ng/mL ± 0.0%		184 ng/mL ± 1.1%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-046	P305; Sample	500	NA	10.4	NA
ISO19-14-01-046-DUP	P305; Sample Dup	473	NA	9.64	NA
ISO19-14-01-046-FMS	P305; FMS	1440	94.4	17.8	77.8
Average Concentration (ng/mL) ± %RPD		487 ng/mL ± 5.5%		10.0 ng/mL ± 7.6%	

NA = Not Applicable

Table 20. Location ID: L19

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-048	L19; Sample	253	NA	176	NA
ISO19-14-01-048-DUP	L19; Sample Dup	261	NA	175	NA
ISO19-14-01-048-FMS	L19; FMS	2170	93.3	2340	106 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		257 ng/mL ± 3.1%		176 ng/mL ± 0.57%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-048	L19; Sample	3950	NA	26.4	NA
ISO19-14-01-048-DUP	L19; Sample Dup	3960	NA	27.4	NA
ISO19-14-01-048-FMS	L19; FMS	6120	106	77.6	101
Average Concentration (ng/mL) ± %RPD		3960 ng/mL ± 0.25%		26.9 ng/mL ± 3.7%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 21. Location ID: P264

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-054	P264; Sample	339	NA	218	NA
ISO19-14-01-054-DUP	P264; Sample Dup	332	NA	223	NA
ISO19-14-01-054-FMS	P264; FMS	2260	93.9	2290	101
Average Concentration (ng/mL) ± %RPD		336 ng/mL ± 2.1%		221 ng/mL ± 2.3%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-054	P264; Sample	1490	NA	69.4	NA
ISO19-14-01-054-DUP	P264; Sample Dup	1500	NA	67.2	NA
ISO19-14-01-054-FMS	P264; FMS	3500	97.8	105	73.4
Average Concentration (ng/mL) ± %RPD		1500 ng/mL ± 0.67%		68.3 ng/mL ± 3.2%	

NA = Not Applicable

Table 22. Location ID: P371

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-059	P371; Sample	589	NA	488	NA
ISO19-14-01-059-DUP	P371; Sample Dup	621	NA	489	NA
ISO19-14-01-059-FMS	P371; FMS	2600	97.3	2500	98.1
Average Concentration (ng/mL) ± %RPD		605 ng/mL ± 5.3%		489 ng/mL ± 0.20%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-059	P371; Sample	1710	NA	10.3	NA
ISO19-14-01-059-DUP	P371; Sample Dup	1730	NA	10.3	NA
ISO19-14-01-059-FMS	P371; FMS	3650	94.1	55.0	89.4
Average Concentration (ng/mL) ± %RPD		1720 ng/mL ± 1.2%		10.3 ng/mL ± 0.0%	

NA = Not Applicable

Table 23. Location ID: Travel Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-064	Travel Blank	<0.192	NA	<0.200	NA	<0.100	NA
ISO19-14-01-064-FMS-LOW	Travel Blank FMS Low	1.77	92.2	1.97	98.5	1.76	88.0
ISO19-14-01-064-FMS-HIGH	Travel Blank FMS High	2100	102	54.9	110	2110	103

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-01-064	Travel Blank	<0.185	NA	<0.0250	NA
ISO19-14-01-064-FMS-LOW	Travel Blank FMS Low	1.68	90.8	1.80	90.0
ISO19-14-01-064-FMS-HIGH	Travel Blank FMS High	2100	102	45.6	91.2

NA = Not Applicable

Table 24. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]PFOA	[¹³ C ₄]PFOS	[¹³ C ₄]PFOS Re-analysis
ISO19-14-01-001	BD24-3; Sample	118	108	NA
ISO19-14-01-001-DUP	BD24-3; Sample Duplicate	106	106	NA
ISO19-14-01-001-FMS	BD24-3; Sample FMS	113	106	NA
ISO19-14-01-002	BD24-4; Sample	85.4 ⁽²⁾	92.6 ⁽²⁾	NA
ISO19-14-01-002-DUP	BD24-4; Sample Duplicate	91.0 ⁽²⁾	91.3 ⁽²⁾	NA
ISO19-14-01-003	D09; Sample	119	109	115
ISO19-14-01-003-DUP	D09; Sample Duplicate	118	108	111
ISO19-14-01-004	D10; Sample	97.2	107	NA
ISO19-14-01-004-DUP	D10; Sample Duplicate	95.9	107	NA
ISO19-14-01-005	D11; Sample	113	112	NA
ISO19-14-01-005-DUP	D11; Sample Duplicate	106	106	NA
ISO19-14-01-005-FMS	D11; Sample FMS	112	106	NA
ISO19-14-01-006	D14; Sample	91.8 ⁽²⁾	91.5 ⁽²⁾	NA
ISO19-14-01-006-DUP	D14; Sample Duplicate	98.4 ⁽²⁾	95.3 ⁽²⁾	NA
ISO19-14-01-007	D16; Sample	111	108	NA
ISO19-14-01-007-DUP	D16; Sample Duplicate	118	114	NA
ISO19-14-01-008	D17; Sample	116	113	NA
ISO19-14-01-008-DUP	D17; Sample Duplicate	120	117	NA
ISO19-14-01-009	D18; Sample	125	118	NA
ISO19-14-01-009-DUP	D18; Sample Duplicate	127	119	NA
ISO19-14-01-010	D5; Sample	117	117	NA
ISO19-14-01-010-DUP	D5; Sample Duplicate	120	116	NA
ISO19-14-01-011	ND7; Sample	107	108	NA
ISO19-14-01-011-DUP	ND7; Sample Duplicate	107	114	NA
ISO19-14-01-012	P118A; Sample	116	117	NA
ISO19-14-01-012-DUP	P118A; Sample Duplicate	118	117	NA
ISO19-14-01-012-FMS	P118A; Sample FMS	113	114	NA
ISO19-14-01-013	P118B; Sample	108	100	NA
ISO19-14-01-013-DUP	P118B; Sample Duplicate	114	105	NA
ISO19-14-01-014	P119A; Sample	112	111	NA
ISO19-14-01-014-DUP	P119A; Sample Duplicate	123	116	NA
ISO19-14-01-015	P119B; Sample	112	117	NA
ISO19-14-01-015-DUP	P119B; Sample Duplicate	105	111	NA
ISO19-14-01-016	P121; Sample	95.6 ⁽²⁾	90.7 ⁽²⁾	NA
ISO19-14-01-016-DUP	P121; Sample Duplicate	96.2 ⁽²⁾	93.8 ⁽²⁾	NA
ISO19-14-01-016-FMS	P121; Sample FMS	98.4 ⁽²⁾	90.3 ⁽²⁾	NA
ISO19-14-01-017	3M vijver; Sample	97.0 ⁽²⁾	97.8 ⁽²⁾	NA
ISO19-14-01-017-DUP	3M vijver; Sample Duplicate	96.0 ⁽²⁾	93.8 ⁽²⁾	NA
ISO19-14-01-018	Blokkersdijkvijver Noord; Sample	96.8 ⁽²⁾	90.1 ⁽²⁾	NA
ISO19-14-01-018-DUP	Blokkersdijkvijver Noord; Sample Duplicate	97.0 ⁽²⁾	93.8 ⁽²⁾	NA

NA = Not Applicable

(1) The surrogate recovery standards [¹³C₄]PFOA and [¹³C₄]PFOS were added to the samples during sample preparation unless noted otherwise.

(2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 24 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)	
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS
ISO19-14-01-019	Blokkersdijkvijver standaard; Sample	94.2 ⁽²⁾	92.8 ⁽²⁾
ISO19-14-01-019-DUP	Blokkersdijkvijver standaard; Sample Duplicate	89.6 ⁽²⁾	94.7 ⁽²⁾
ISO19-14-01-019-FMS	Blokkersdijkvijver standaard; Sample FMS	94.0 ⁽²⁾	91.5 ⁽²⁾
ISO19-14-01-020	P321; Sample	107	104
ISO19-14-01-020-DUP	P321; Sample Duplicate	117	107
ISO19-14-01-021	L21; Sample	82.4 ⁽²⁾	98.4 ⁽²⁾
ISO19-14-01-021-DUP	L21; Sample Duplicate	95.4 ⁽²⁾	91.1 ⁽²⁾
ISO19-14-01-022	L22; Sample	97.6 ⁽²⁾	89.6 ⁽²⁾
ISO19-14-01-022-DUP	L22; Sample Duplicate	96.6 ⁽²⁾	96.8 ⁽²⁾
ISO19-14-01-023	L31; Sample	86.4 ⁽²⁾	91.5 ⁽²⁾
ISO19-14-01-023-DUP	L31; Sample Duplicate	86.6 ⁽²⁾	93.4 ⁽²⁾
ISO19-14-01-024	L4; Sample	91.8 ⁽²⁾	95.1 ⁽²⁾
ISO19-14-01-024-DUP	L4; Sample Duplicate	93.8 ⁽²⁾	87.1 ⁽²⁾
ISO19-14-01-025	P114bis; Sample	88.0 ⁽²⁾	96.3 ⁽²⁾
ISO19-14-01-025-DUP	P114bis; Sample Duplicate	92.0 ⁽²⁾	89.8 ⁽²⁾
ISO19-14-01-026	P115; Sample	95.6 ⁽²⁾	91.1 ⁽²⁾
ISO19-14-01-026-DUP	P115; Sample Duplicate	94.2 ⁽²⁾	90.7 ⁽²⁾
ISO19-14-01-026-FMS	P115; Sample FMS	93.8 ⁽²⁾	95.7 ⁽²⁾
ISO19-14-01-027	P116; Sample	94.6 ⁽²⁾	92.4 ⁽²⁾
ISO19-14-01-027-DUP	P116; Sample Duplicate	88.8 ⁽²⁾	93.2 ⁽²⁾
ISO19-14-01-028	Effluent WWTP; Sample	91.4 ⁽²⁾	88.6 ⁽²⁾
ISO19-14-01-028-DUP	Effluent WWTP; Sample Duplicate	91.2 ⁽²⁾	92.4 ⁽²⁾
ISO19-14-01-029	PP01; Sample	119	113
ISO19-14-01-029-DUP	PP01; Sample Duplicate	131	115
ISO19-14-01-029-FMS	PP01; Sample FMS	111	105
ISO19-14-01-030	PP02; Sample	119	93.8
ISO19-14-01-030-DUP	PP02; Sample Duplicate	115	91.7
ISO19-14-01-031	PP04; Sample	112	97.5
ISO19-14-01-031-DUP	PP04; Sample Duplicate	112	95.4
ISO19-14-01-032	PP05; Sample	98.6	108
ISO19-14-01-032-DUP	PP05; Sample Duplicate	103	116
ISO19-14-01-033	PP06; Sample	112	101
ISO19-14-01-033-DUP	PP06; Sample Duplicate	115	105
ISO19-14-01-033-FMS	PP06; Sample FMS	99.5	99.0
ISO19-14-01-034	PP07; Sample	125	106
ISO19-14-01-034-DUP	PP07; Sample Duplicate	111	104
ISO19-14-01-035	PP08; Sample	117	93.3
ISO19-14-01-035-DUP	PP08; Sample Duplicate	119	95.0
ISO19-14-01-036	PP09; Sample	115	104
ISO19-14-01-036-DUP	PP09; Sample Duplicate	114	103
ISO19-14-01-037	PP10; Sample	116	101
ISO19-14-01-037-DUP	PP10; Sample Duplicate	123	103

NA = Not Applicable

(1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.

(2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 24 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO19-14-01-038	12; Sample	87.0 ⁽²⁾	91.9 ⁽²⁾	NA
ISO19-14-01-038-DUP	12; Sample Duplicate	95.6 ⁽²⁾	91.1 ⁽²⁾	NA
ISO19-14-01-039	13; Sample	88.8 ⁽²⁾	91.1 ⁽²⁾	NA
ISO19-14-01-039-DUP	13; Sample Duplicate	99.8 ⁽²⁾	91.7 ⁽²⁾	NA
ISO19-14-01-040	5; Sample	87.8 ⁽²⁾	92.4 ⁽²⁾	NA
ISO19-14-01-040-DUP	5; Sample Duplicate	89.0 ⁽²⁾	93.2 ⁽²⁾	NA
ISO19-14-01-041	Bemalingstation; Sample	106	94.5 ⁽²⁾	NA
ISO19-14-01-041-DUP	Bemalingstation; Sample Duplicate	95.3	92.8 ⁽²⁾	NA
ISO19-14-01-041-FMS	Bemalingstation; Sample FMS	104	91.7 ⁽²⁾	NA
ISO19-14-01-042	Collector put; Sample	90.0 ⁽²⁾	88.2 ⁽²⁾	107
ISO19-14-01-042-DUP	Collector put; Sample Duplicate	91.4 ⁽²⁾	93.2 ⁽²⁾	108
ISO19-14-01-043	P27; Sample	101	102	113
ISO19-14-01-043-DUP	P27; Sample Duplicate	100	99.7	115
ISO19-14-01-043-FMS	P27; Sample FMS	96.9	96.1	NA
ISO19-14-01-044	P21B; Sample	95.8	90.9	101
ISO19-14-01-044-DUP	P21B; Sample Duplicate	94.9	91.4	107
ISO19-14-01-045	P304; Sample	104	113	NA
ISO19-14-01-045-DUP	P304; Sample Duplicate	91.0	97.3	NA
ISO19-14-01-046	P305; Sample	114	113	NA
ISO19-14-01-046-DUP	P305; Sample Duplicate	102	112	NA
ISO19-14-01-046-FMS	P305; Sample FMS	105	108	NA
ISO19-14-01-047	P42; Sample	107	101	NA
ISO19-14-01-047-DUP	P42; Sample Duplicate	108	101	NA
ISO19-14-01-048	L19; Sample	104	95.9	NA
ISO19-14-01-048-DUP	L19; Sample Duplicate	108	94.1	NA
ISO19-14-01-048-FMS	L19; Sample FMS	87.6	86.7	NA
ISO19-14-01-049	M4; Sample	113	115	NA
ISO19-14-01-049-DUP	M4; Sample Duplicate	110	114	NA
ISO19-14-01-050	P118C; Sample	98.9	97.4	NA
ISO19-14-01-050-DUP	P118C; Sample Duplicate	104	105	NA
ISO19-14-01-051	P119C; Sample	95.1	78.0	108
ISO19-14-01-051-DUP	P119C; Sample Duplicate	101	84.5	108
ISO19-14-01-053	P263; Sample	112	111	NA
ISO19-14-01-053-DUP	P263; Sample Duplicate	106	113	NA
ISO19-14-01-054	P264; Sample	102	105	NA
ISO19-14-01-054-DUP	P264; Sample Duplicate	104	107	NA
ISO19-14-01-054-FMS	P264; Sample FMS	93.2	98.2	NA
ISO19-14-01-055	P265B; Sample	103	106	NA
ISO19-14-01-055-DUP	P265B; Sample Duplicate	98.6	107	NA
ISO19-14-01-056	P340; Sample	110	111	NA
ISO19-14-01-056-DUP	P340; Sample Duplicate	104	102	NA

NA = Not Applicable

(1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.

(2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 24 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)	
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS
ISO19-14-01-057	P341; Sample	97.8	90.6
ISO19-14-01-057-DUP	P341; Sample Duplicate	104	91.7
ISO19-14-01-058	P343; Sample	87.9	73.9
ISO19-14-01-058-DUP	P343; Sample Duplicate	96.1	88.5
ISO19-14-01-059	P371; Sample	96.3	99.5
ISO19-14-01-059-DUP	P371; Sample Duplicate	94.4	102
ISO19-14-01-059-FMS	P371; Sample FMS	105	97.2
ISO19-14-01-060	P374; Sample	98.1	107
ISO19-14-01-060-DUP	P374; Sample Duplicate	104	107
ISO19-14-01-061	P379; Sample	106	110
ISO19-14-01-061-DUP	P379; Sample Duplicate	100	108
ISO19-14-01-062	P380; Sample	99.6	105
ISO19-14-01-062-DUP	P380; Sample Duplicate	95.1	101
ISO19-14-01-063	P372; Sample	100	99.1
ISO19-14-01-063-DUP	P372; Sample Duplicate	90.7	97.6
ISO19-14-01-064	Travel Blank	92.0 ⁽²⁾	90.9 ⁽²⁾
ISO19-14-01-064-FMS-LOW	Travel Blank FMS Low	98.6 ⁽²⁾	93.6 ⁽²⁾
ISO19-14-01-064-FMS-HIGH	Travel Blank FMS High	100	105

NA = Not Applicable

(1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.

(2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 9-24 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M EHS Laboratory standard operating procedures.

7 Attachments

Chain of Custody Form

8 Signatures

Chelsie Grochow, 3M Report Author



Susan T. Wolf, 3M Principal Analytical Investigator



Brian T. Mader, Ph.D., 3M EHS Laboratory Manager



The 3M EHS Laboratory's Quality Assurance Unit has audited the data and report for this project.



Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M EHS Laboratory.

3M EHS LABORATORY
Chain-of-Custody

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Project: ISO19-14-01

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/9/2019

Project Description: 3M Antwerp Water Sampling for PFCs - January 2019

Comments:

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO19-14-01-001	BD24-3; Sample	31/01/2019	WG	✓
ISO19-14-01-001-DUP	BD24-3; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-001-FMS	BD24-3; Sample FMS	31/01/2019	WQ	✓
ISO19-14-01-002	BD24-4; Sample	31/01/2019	WG	✓
ISO19-14-01-002-DUP	BD24-4; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-003	D09; Sample	31/01/2019	WG	✓
ISO19-14-01-003-DUP	D09; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-004	D10; Sample	30/01/2019	WG	✓
ISO19-14-01-004-DUP	D10; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-005	D11; Sample	30/01/2019	WG	✓
ISO19-14-01-005-DUP	D11; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-005-FMS	D11; Sample FMS	30/01/2019	WQ	✓
ISO19-14-01-006	D14; Sample	30/01/2019	WG	✓
ISO19-14-01-006-DUP	D14; Sample Duplicate	30/01/2019	WG	✓

ISO19-14-01-001	BD24-3; Sample	31/01/2019	WG	✓
ISO19-14-01-001-DUP	BD24-3; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-001-FMS	BD24-3; Sample FMS	31/01/2019	WQ	✓
ISO19-14-01-002	BD24-4; Sample	31/01/2019	WG	✓
ISO19-14-01-002-DUP	BD24-4; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-003	D09; Sample	31/01/2019	WG	✓
ISO19-14-01-003-DUP	D09; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-004	D10; Sample	30/01/2019	WG	✓
ISO19-14-01-004-DUP	D10; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-005	D11; Sample	30/01/2019	WG	✓
ISO19-14-01-005-DUP	D11; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-005-FMS	D11; Sample FMS	30/01/2019	WQ	✓
ISO19-14-01-006	D14; Sample	30/01/2019	WG	✓
ISO19-14-01-006-DUP	D14; Sample Duplicate	30/01/2019	WG	✓

(LE) CJG 4/1/19 All samples (pages 1-9) were received at room temp and were stored at 4°C in WRI.

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Vrijsel Borgst (VBO) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
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14	Vrijsel Borgst	04/02/19	10:00	FedEx	Joseph Tamm	2-6-19	1310

3M EHS LABORATORY
Chain-of-Custody

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Project: ISO19-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/9/2019

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs - January 2019

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
-------------------------	---------------------------	--------------------------	---------------	----------------

ISO19-14-01-007	D16; Sample	31/01/2019	WG	✓
ISO19-14-01-007-DUP	D16; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-008	D17; Sample	31/01/2019	WG	✓
ISO19-14-01-008-DUP	D17; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-009	D18; Sample	30/01/2019	WG	✓
ISO19-14-01-009-DUP	D18; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-010	D5; Sample	30/01/2019	WG	✓
ISO19-14-01-010-DUP	D5; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-011	ND7; Sample	30/01/2019	WG	✓
ISO19-14-01-011-DUP	ND7; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-012	P118A; Sample	31/01/2019	WG	✓
ISO19-14-01-012-DUP	P118A; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-012-FMS	P118A; Sample FMS	31/01/2019	WQ	✓
ISO19-14-01-013	P118B; Sample	31/01/2019	WG	✓
ISO19-14-01-013-DUP	P118B; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-014	P119A; Sample	30/01/2019	WG	✓
ISO19-14-01-014-DUP	P119A; Sample Duplicate	30/01/2019	WG	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Kristof Bagout

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
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17	Kristof Bagout	01/02/19	10:00	Fedex	Tress Tamm	2-6-19	1310

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [REDACTED]
Alt. Ph: [REDACTED]
Fax: [REDACTED]

Project: ISO19-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)

Completion Date:

Department: 832202 Site Source: 01WJWT10

Project Lead: Susan T. Wolf

Project Number:

Phone Number: [REDACTED]

Date Created: 1/9/2019

Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs - January 2019

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
-------------------------	---------------------------	--------------------------	---------------	----------------

ISO19-14-01-015	P119B; Sample	30/01/2019	WG	✓
ISO19-14-01-015-DUP	P119B; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-016	P121; Sample	01/02/2019	WG	✓
ISO19-14-01-016-DUP	P121; Sample Duplicate	01/02/2019	WG	✓
ISO19-14-01-016-FMS	P121; Sample FMS	01/02/2019	WQ	✓
ISO19-14-01-017	3M vijver; Sample	31/01/2019	WG	✓
ISO19-14-01-017-DUP	3M vijver; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-018	Blokkersdijkvijver Noord; Sample	31/01/2019	WG	✓
ISO19-14-01-018-DUP	Blokkersdijkvijver Noord; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-019	Blokkersdijkvijver standaard; Sample	31/01/2019	WG	✓
ISO19-14-01-019-DUP	Blokkersdijkvijver standaard; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-019-FMS	Blokkersdijkvijver standaard; Sample FMS	31/01/2019	WQ	✓
ISO19-14-01-020	P321; Sample	30/01/2019	WG	✓
ISO19-14-01-020-DUP	P321; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-021	L21; Sample	31/01/2019	WG	✓
ISO19-14-01-021-DUP	L21; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-022	L22; Sample	30/01/2019	WG	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Wristop Borgent (WBO) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
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17	Wristop Borgent	04/02/19	10:00	FedEx	Susan T. Wolf	2-6-19	1310

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [REDACTED]
Alt. Ph: [REDACTED]
Fax: [REDACTED]

Project: ISO19-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)

Completion Date:

Department: 832202 Site Source: 01WJWT10

Project Lead: Susan T. Wolf

Project Number:

Phone Number: [REDACTED]

Date Created: 1/9/2019

Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs - January 2019

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
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ISO19-14-01-022-DUP	L22; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-023	L31; Sample	31/01/2019	WG	✓
ISO19-14-01-023-DUP	L31; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-024	L4; Sample	31/01/2019	WG	✓
ISO19-14-01-024-DUP	L4; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-025	P114bis; Sample	01/02/2019	WG	✓
ISO19-14-01-025-DUP	P114bis; Sample Duplicate	01/02/2019	WG	✓
ISO19-14-01-026	P115; Sample	01/02/2019	WG	✓
ISO19-14-01-026-DUP	P115; Sample Duplicate	01/02/2019	WG	✓
ISO19-14-01-026-FMS	P115; Sample FMS	01/02/2019	WQ	✓
ISO19-14-01-027	P116; Sample	01/02/2019	WG	✓
ISO19-14-01-027-DUP	P116; Sample Duplicate	01/02/2019	WG	✓
ISO19-14-01-028	Effluent WWTP; Sample	30/01/2019	WG	✓
ISO19-14-01-028-DUP	Effluent WWTP; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-029	PP01; Sample	30/01/2019	WG	✓
ISO19-14-01-029-DUP	PP01; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-029-FMS	PP01; Sample FMS	30/01/2019	WQ	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Wristof Bogart

Collector's signature: WB

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
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17	Wristof Bogart	01/02/19	10:00	FedEx	Jerry Timman	26-19	1310

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [REDACTED]
Alt. Phone: [REDACTED]
Fax: (651) 733-1111

Project: ISO19-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/9/2019

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs - January 2019

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
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ISO19-14-01-030	PP02; Sample	30/01/2019	WG	✓
ISO19-14-01-030-DUP	PP02; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-031	PP04; Sample	30/01/2019	WG	✓
ISO19-14-01-031-DUP	PP04; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-032	PP05; Sample	30/01/2019	WG	✓
ISO19-14-01-032-DUP	PP05; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-033	PP06; Sample	30/01/2019	WG	✓
ISO19-14-01-033-DUP	PP06; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-033-FMS	PP06; Sample FMS	30/01/2019	WQ	✓
ISO19-14-01-034	PP07; Sample	30/01/2019	WG	✓
ISO19-14-01-034-DUP	PP07; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-035	PP08; Sample	30/01/2019	WG	✓
ISO19-14-01-035-DUP	PP08; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-036	PP09; Sample	30/01/2019	WG	✓
ISO19-14-01-036-DUP	PP09; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-037	PP10; Sample	30/01/2019	WG	✓
ISO19-14-01-037-DUP	PP10; Sample Duplicate	30/01/2019	WG	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Kristof Begout

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
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17	<u>Kristof Begout</u>	<u>01/02/19</u>	<u>10:00</u>	<u>FedEx</u>	<u>SUSAN TILMAN</u>	<u>2-6-19</u>	<u>1310</u>

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [REDACTED]
Alt. Ph: [REDACTED]
Fax: [REDACTED]

Project: ISO19-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/9/2019

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs - January 2019

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
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ISO19-14-01-038	12; Sample	31/01/2019	WG	✓
ISO19-14-01-038-DUP	12; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-039	13; Sample	31/01/2019	WG	✓
ISO19-14-01-039-DUP	13; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-040	5; Sample	31/01/2019	WG	✓
ISO19-14-01-040-DUP	5; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-041	Bemalingstation; Sample	31/01/2019	WG	✓
ISO19-14-01-041-DUP	Bemalingstation; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-041-FMS	Bemalingstation; Sample FMS	31/01/2019	WQ	✓
ISO19-14-01-042	Collector put; Sample	30/01/2019	WG	✓
ISO19-14-01-042-DUP	Collector put; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-043	P27; Sample	30/01/2019	WG	✓
ISO19-14-01-043-DUP	P27; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-043-FMS	P27; Sample FMS	30/01/2019	WQ	✓
ISO19-14-01-044	P21B; Sample	30/01/2019	WG	✓
ISO19-14-01-044-DUP	P21B; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-045	P304; Sample	31/01/2019	WG	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Kristof Bogart

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	Kristof Bogart	04/02/19	10:00	FedEx	Joseph Timman	1/26/19	1310

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [REDACTED]
Alt. Phone: [REDACTED]
Fax: [REDACTED]

Project: ISO19-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/9/2019

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs - January 2019

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
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ISO19-14-01-045-DUP	P304; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-046	P305; Sample	30/01/2019	WG	✓
ISO19-14-01-046-DUP	P305; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-046-FMS	P305; Sample FMS	30/01/2019	WQ	✓
ISO19-14-01-047	P42; Sample	30/01/2019	WG	✓
ISO19-14-01-047-DUP	P42; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-048	L19; Sample	31/01/2019	WG	✓
ISO19-14-01-048-DUP	L19; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-048-FMS	L19; Sample FMS	31/01/2019	WQ	✓
ISO19-14-01-049	M4; Sample	30/01/2019	WG	✓
ISO19-14-01-049-DUP	M4; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-050	P118C; Sample	31/01/2019	WG	✓
ISO19-14-01-050-DUP	P118C; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-051	P119C; Sample	30/01/2019	WG	✓
ISO19-14-01-051-DUP	P119C; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-052	P262; Sample (empty)	Blocked, not sampled	WG	✓
ISO19-14-01-052-DUP	P262; Sample Duplicate (empty)	Sampled	WG	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Kristof Bogaert

Collector's signature: K.B.

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
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17	Kristof Bogaert	04/01/19	10:00	FedEx	JOSEPH TUMAN	2-6-19	1310

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [REDACTED]
Alt. Ph: [REDACTED]
Fax: [REDACTED]

Project: ISO19-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)

Completion Date:

Department: 832202 Site Source: 01WJWT10

Project Lead: Susan T. Wolf

Project Number:

Phone Number: [REDACTED]

Date Created: 1/9/2019

Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs - January 2019

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
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ISO19-14-01-053	P263; Sample	31/01/2019	WG	✓
ISO19-14-01-053-DUP	P263; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-054	P264; Sample	31/01/2019	WG	✓
ISO19-14-01-054-DUP	P264; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-054-FMS	P264; Sample FMS	31/01/2019	WQ	✓
ISO19-14-01-055	P265C; Sample <i>P265B</i>	31/01/2019	WG	✓
ISO19-14-01-055-DUP	P265C; Sample Duplicate <i>P265B</i>	31/01/2019	WG	✓
ISO19-14-01-056	P340; Sample	30/01/2019	WG	✓
ISO19-14-01-056-DUP	P340; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-057	P341; Sample	30/01/2019	WG	✓
ISO19-14-01-057-DUP	P341; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-058	P343; Sample	31/01/2019	WG	✓
ISO19-14-01-058-DUP	P343; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-059	P371; Sample	30/01/2019	WG	✓
ISO19-14-01-059-DUP	P371; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-059-FMS	P371; Sample FMS	30/01/2019	WQ	✓
ISO19-14-01-060	P374; Sample	31/01/2019	WG	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Kristen Bagant

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
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17	Kristen Bagant	04/01/19	10:00	FedEx	Joseph Fillman	2-6-19	1510

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone:
Alt. P.
Fax:

Project: ISO19-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/9/2019

Project Description: 3M Antwerp Water Sampling for PFCs - January 2019

Completion Date:
Project Lead: Susan T. Wolf
Phone Number:
Email Address:

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO19-14-01-060-DUP	P374; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-061	P379; Sample	31/01/2019	WG	✓
ISO19-14-01-061-DUP	P379; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-062	P380; Sample	31/01/2019	WG	✓
ISO19-14-01-062-DUP	P380; Sample Duplicate	31/01/2019	WG	✓
ISO19-14-01-063	P372; Sample	30/01/2019	WG	✓
ISO19-14-01-063-DUP	P372; Sample Duplicate	30/01/2019	WG	✓
ISO19-14-01-064	Travel Blank		WQ	✓ *
ISO19-14-01-064-FMS-HIGH	Travel Blank FMS High		WQ	✓ *
ISO19-14-01-064-FMS-LOW	Travel Blank FMS Low		WQ	✓ *

* Travel Blank samples prepared by the 3M EHS Laboratory with the bottle order.

Sample Condition Upon Receipt: Acceptable All items accounted for
Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Kristof Bagewit

Collector's signature: JWT

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
10	Kristof Bagewit	04/01/19	10:00	Faster,	SUSAN T. WOLF	2-6-19	1310



3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC19-00850-002

3M Lab Request Number: E19-0174

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: May 28th, 2019

Requester

Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000

SGS Belgium NV Institute for Applied Chromatography Haven 407 Polderdijkweg 16 B-2030 Antwerpen
t +32 (0)3 545 85 90 f +32 (0)3 545 85 99 e be_iac@sgs.com url www.sgs.be

Member of the SGS Group (Société Générale de Surveillance)

Registered office: Noorderlaan 87 B-2030 Antwerpen H.R. Antwerpen 141.810 BTW BE 404.882.750 Citibank BE87 5701 3412 5594
All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction - Summary.

At the request (Lab Request Number: E19-0174) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, one water sample was collected on May 3rd 2019 from 3 locations and analyzed for the following perfluorinated compounds:

- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$) (and its ^{13}C -labeled analogues)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$) (and its ^{13}C -labeled analogues)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$) (and its ^{13}C -labeled analogues)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$) (and its ^{13}C -labeled analogue)
- ^{13}C -labeled analogue of PFUdA ($C_4F_{21}^{13}C_6F_2^{13}COO^-$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared one sample container for each sampling location under the direction of Sven Herremans. Each empty container was marked with a "fill to here" line and was fortified with a surrogate recovery spike and an internal standard spike, prior to being sent to the field for sample collection.

Table 1 summarizes the sample results with their uncertainty. All PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$ unless noted otherwise. See Section 4 of the report for additional information on the method QC used to assess method uncertainty.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)			
	PFHS	PFOA	PFOS	FOSA
3M Vijver	0.421	1.290	3.23	0.0360 (a)
Blokkersdijkvijver Noord	0.454	1.06	2.92	0.0420
Blokkersdijkvijver standaard (filter)	0.266	0.933	<0.025	<0.025 (b)
Blokkersdijkvijver standaard (nt filter)	0.390	0.999	0.684	<0.025

(a): The recovery of FOSA for 3M vijver is $\pm 75\%$.

(b): The recovery of FOSA for Blokkersdijkvijver standard (filter) is $\pm 70\%$.

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

The sample was collected in a polyethylene bottle prepared at the SGS Belgium NV, Division IAC Laboratory. Based on the concentrations as reported in previous reports, the bottle was spiked, prior to sample collection, in the laboratory with a known volume of a surrogate recovery solution ($^{13}\text{C}_2\text{-PFHS}$, $^{13}\text{C}_4\text{-PFOA}$, $^{13}\text{C}_4\text{-PFOS}$, $^{13}\text{C}_7\text{-PFUdA}$) and internal standard solution ($^{13}\text{C}_3\text{-PFHS}$, $^{13}\text{C}_8\text{-PFOA}$, $^{13}\text{C}_8\text{-PFOS}$, $^{13}\text{C}_8\text{-FOSA}$). Table 2 below details the sample collected and spikes added to the bottle.

Table 2. Sample Collection and Spike Information.

Sample Identification	Nominal Final Volume Collected (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Final Nominal Spike Concentration (ng/mL)	
				$^{13}\text{C-PFC-SS}$	$^{13}\text{C-PFC-IS}$
3M vijver	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
Blokkersdijkvijver Noord	400	0.1	Solution A	0.25	-
		0.25	Solution B	-	1.0
Blokkersdijkvijver standaard (filter)	400	0.1	Solution A	0.25	-
		0.25	Solution B	-	1.0
Blokkersdijkvijver standaard (nt filter)	400	0.1	Solution A	0.25	-
		0.25	Solution B	-	1.0

Solution A = 1000 ng/mL (nominal) $^{13}\text{C-PFC}$ Surrogate Recovery Standards

Solution B = 1500 ng/mL (nominal) $^{13}\text{C-PFC}$ Internal Standards

2.2. Extraction.

All samples, calibration standards, and associated quality control samples were extracted using a modified procedure of ECO/AV/IAC/064 “Bepaling van pergefloreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)”. Briefly, an amount of sample (see table 3) was loaded, onto a pre-conditioned Waters tC18 solid phase extraction (SPE) cartridge (Sep-Pak, 1g, 6cc) using a vacuum manifold. The loaded SPE cartridges were then eluted with 5 mL of methanol using vacuum.

Table 3. Sample amount used.

Sample	Amount of Sample used (mL)	Concentration Factor
3M Vijver	40	8
Blokkersdijkvijver Noord	40	8
Blokkersdijkvijver standard (filter)	40	8
Blokkersdijkvijver standard (nt filter)	40	8
Field Trip Blank	40	8

2.3. Determination of suspended solids in water.

To avoid the influence of algae on the analytical result, filtration is proposed prior to extraction at the sampling locations Blokkersdijkvijver - standard.

The filtration was done by using Whatman GF/C glass-fiber filters, with a pore size of 1.2 µm.

The total suspended solids of the samples is determined by pouring a measured volume of sample, typically 200 mL, through a pre-weighed filter then weighing the filter again after drying the filter overnight to remove all water. The gain in weight is a dry weight measure of the particulates present in the water sample. This is expressed in units calculated from the volume of water filtered, milligrams per liter. Table 4 summarizes the results.

Table 4. Suspended solids.

Sample Identification	Suspended solids/ volume (mg/L)
Blokkersdijkvijver - standard (filtered)	0.01

2.4. Analysis.

All solutions and extracts were analyzed for the PFCs (PFHS, PFOA, PFOS, FOSA) and the surrogate recovery standards ($^{13}\text{C}_2\text{-PFHS}$, $^{13}\text{C}_4\text{-PFOA}$, $^{13}\text{C}_4\text{-PFOS}$, $^{13}\text{C}_7\text{-PFUdA}$) and internal standards ($^{13}\text{C}_3\text{-PFHS}$, $^{13}\text{C}_8\text{-PFOA}$, $^{13}\text{C}_8\text{-PFOS}$, $^{13}\text{C}_8\text{-FOSA}$) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetrie C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C ₃ -PFHS	2.00 - 3.00	402.0 /80.0	60	38	51
¹³ C ₂ -PFHS	2.30 - 3.30	403.0/84.0	60	38	51
¹³ C ₈ -PFOS	3.00 - 4.00	507.0/80.0	60	48	56
¹³ C ₄ -PFOA	2.30 - 3.50	417.0 /372.0	100	11	14
¹³ C ₈ PFOA	2.30 - 3.50	421.0 /376.0	60	11	14
PFHS	2.00 - 3.00	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.30 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
¹³ C ₄ -PFOS	3.00 - 4.00	503.0 /80.0	60	48	56
¹³ C ₇ PFUD _A	4.30 - 5.60	570.0 /525.0	60	12	17
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
¹³ C ₈ FOSA	4.30 - 5.70	506.0/78.0	60	34	44

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Extracted Calibration Standard.

Extracted calibration standards were prepared by spiking known amounts of stock solutions containing PFHS, PFOA, PFOS, FOSA and ^{13}C -labeled analogues into 40 mL of HPLC water. Each spiked water standard was then extracted in the same manner as the collected samples. A total of 12 spiked standards ranging from 0.005 ng/mL to 100 ng/mL (nominal) were prepared. Each curve point contains the mixture of internal standards at a nominal concentration of 1 ng/mL. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. The calibration curve will be generated by taking the ratio of the standard peak area counts over the internal standard peak area counts to fit the data for each analyte. Each extracted calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of $\pm 25\%$ ($\pm 30\%$ for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The 0.025ng/mL (nominal) reporting limit is a practical quantitation limit (PQL) required by the requester and it is possible that the samples contain target analytes at quantifiable concentrations below the PQL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within $\pm 25\%$, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by loading 40 mL of HPLC water onto a Waters tC18 solid phase extraction (SPE) cartridge (SEP-Pak, 1g, 6cc) and eluting with 5 mL of methanol using the same extraction procedure as the samples. Method blanks were prepared to evaluate the levels of background contamination in the overall extraction process (glassware, SPE cartridges, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carry over.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of HPLC water, spiked with the surrogate recovery standards and internal standards, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCSs)

Low (0.125 ng/mL nominal concentration) and high (2.5 ng/mL nominal concentration) lab control spikes were prepared and analyzed in duplicate. LCSs were prepared by spiking known amounts of the analytes and surrogates into 40 mL of HPLC water to produce the desired concentration. The spiked water samples were extracted and analyzed in the same manner as the samples.

All LCSs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

All LCSs produced recoveries within the method acceptance criteria of $\pm 15\%$ RPD for precision, except for 13C4PFOS.

Table 8 summarize the LCS recovery results.

Table 7. Lab Control Spike Results.

Extraction date	Description	Nominal Spike Level (ng/mL)	Percent Recovery							
			¹³ C ₄ -PFOA	¹³ C ₄ -PFOS	¹³ C ₂ -PFHS	¹³ C ₇ -PFuDA	PFHS	PFOA	PFOS	FOSA
20 May 2019	LCSL01	0.125	89.1	111	105	88.5	91.7	96.8	97.7	90.9
	LCSL02	0.125	90.2	93.8	105	76.2	84.6	113	94.6	90.4
	Average		89.7	102	105	82.4	88.2	105	96.2	90.7
	%RPD		1.2	17 (a)	0.5	15	8.1	15	3.2	0.6
	LCSH 01	2.5	93.6	101	103	90.3	87.0	107	99.5	94.3
	LCSH 02	2.5	97.9	108	110	92.4	96.1	116	113	109
	Average		95.8	105	106	91.4	91.6	111	106	101
	%RPD		4.5	7.0	6.7	2.3	10	7.8	13	14

(a) The recovery of the RPD fell outside the method acceptance criterion of $\pm 15\%$.

3.6. Surrogates.

Surrogate recovery standards were added to all samples to evaluate overall method performance.

3.7. Internal Standards.

Internal standards were added to all samples to calculate the concentration of PFCs in the samples by using internal standard calibration.

3.8. Equations.

Recovery.

$$\text{Recovery (\%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

Tabel 8 and 9 summarizes the results for the sample locations.

Table 8. Sample Results PFHS and PFOA.

	PFHS ng/ml	¹³ C ₂ -PFHS %Rec	PFOA ng/ml	¹³ C ₄ -PFOA %Rec
Field Trip Blank	<0.025	94.1	<0.025	86.3
3M vijver	0.421	84.0	1.290	93.4
Blokkersdijkvijver Noord	0.454	97.2	1.06	99.1
Blokkersdijkvijver standaard (filter)	0.266	92.7	0.933	96.1
Blokkersdijkvijver standaard (nt filter)	0.390	85.5	0.999	100

Table 9. Sample Results PFOS and FOSA.

	PFOS ng/ml	¹³ C ₄ -PFOS %Rec	FOSA ng/ml	¹³ C ₇ -PFuDA %Rec
Field Trip Blank	<0.025	99.8	<0.025	104
3M vijver	3.23	107	0.0360	29.2 (a)
Blokkersdijkvijver Noord	2.92	109	0.0420	74.2
Blokkersdijkvijver standaard (filter)	<0.025	95.0	<0.025	33.5 (a)
Blokkersdijkvijver standaard (nt filter)	0.684	107	<0.025	76.7

(a): The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130%.

All PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$, except for FOSA for the following sample locations:

- (a): The recovery of FOSA for 3M vijver is $\pm 75\%$.
- (b): The recovery of FOSA for Blokkersdijkvijver standard (filter) is $\pm 70\%$.

5. Conclusion.

- The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130% for two sample locations.
- Lab control spike recoveries fell within the method acceptance criteria of 25%.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

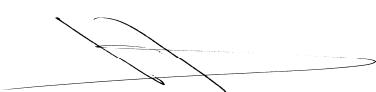
7. Addendum.

Addendum: e-mail from Kris Bogaert (ERM Belgium).

8. Signatures.

Sven Herremans,
Technical Manager

Date May 28th, 2019



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date May 28th, 2019

I.A.C.
A Division of SGS Belgium NV

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The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.



3M Corporate Environmental Programs.
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ANALYTICAL REPORT

IAC19-00850-001

3M Lab Request Number: E19-0174

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

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1. Introduction - Summary.

At the request (Lab Request Number: E19-0174) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected in April 2019 from 22 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOA ($C_6F_{13}^{13}CF_2^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOS ($C_7F_{15}^{13}CF_2SO_3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Sven Herremans. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles.

Table 1 summarizes the sample results. All PFBS, PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
L21		<0.182	0.258	3.09	<0.180
L22		0.250	0.480	1.80	<0.180
L31		1.51	0.369	3.85	<0.180
L4		2.04	2.01	18.8	0.800
P114bis		1.78	3.97	5.97	<0.180
P115		2.15	3.18	1.51	<0.180
P116		0.58	0.929	6.46	<0.180
Effluent WWTP		10.6	10.4	22.1	0.223
PP11	54.7	137	80.8	551	24.8
PP02	30.5	3628	514	16987	<20.5
PP04	978	234	576	4335	32.7
PP05	5068	3290	1028	335	50.3
PP06	26.0	56.1	246	890	48.0
PP07	197	89.9	203	1944	45.3
PP08	12.5	164	343	4186	25.8
PP09	21.0	304	512	2088	10.2
PP10	11.8	155	369	2590	11.5
12		16.3	48.8	223	<0.537
13		18.8	47.7	206	<0.537
5		27.6	27.2	35.6	<0.180
Bemalingsstation		22.5	20.7	29.8	<0.180
Collector Put		35.6	79.2	218	11.2

2. Methods – Analytical and Preparatory.**2.1. Sample collection.**

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
L21	100
L22	100
L31	100
L4	100
P114bis	100
P115	100
P116	100
Effluent WWTP	100
PP11	100
PP02	100
PP04	100
PP05	100
PP06	100
PP07	100
PP08	100
PP09	100
PP10	100
12	100
13	100
5	100
Bemalingssstation	100
Collector Put	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
L21	1.0	0.05	Solution A	1.0	-
L22	1.0	0.05	Solution A	1.0	-
L31	1.0	0.05	Solution A	1.0	-
L4	1.0	0.05	Solution A	1.0	-
P114bis	1.0	0.05	Solution A	1.0	-
P115	1.0	0.05	Solution A	1.0	-
P116	1.0	0.05	Solution A	1.0	-
Effluent WWTP	1.0	0.05	Solution A	1.0	-
PP11	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
PP02	1.0	0.1	Solution A	10.0	0.5 mL extract + 10.0 mL MeOH (*)
PP04	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
PP05	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
PP06	1.0	0.1	Solution A	10.0	1.0 mL extract + 1.0 mL MeOH (*)
PP07	1.0	0.1	Solution A	10.0	1.0 mL extract + 5.0 mL MeOH (*)
PP08	1.0	0.1	Solution A	10.0	1.0 mL extract + 5.0 mL MeOH (*)
PP09	1.0	0.1	Solution A	10.0	1.0 mL extract + 5.0 mL MeOH (*)
PP10	1.0	0.1	Solution A	10.0	1.0 mL extract + 5.0 mL MeOH (*)
12	1.0	0.1	Solution A	5.0	-
13	1.0	0.1	Solution A	5.0	-
5	1.0	0.05	Solution A	1.0	-
Bemalingsstation	1.0	0.05	Solution A	1.0	-
Collector Put	1.0	0.05	Solution A	1.0	-

(*): MeOH:LCMS-water (60:40)
 Solution A = 1000 ng/mL (nominal) ^{13}C -PFC Surrogate Standards

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ^{13}C -PFOA, PFOS, FOSA and ^{13}C -PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetre C18 50 x 2.1mm, 3.5 μm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7 μm
Injection volume	5 μL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate ($\mu\text{L}/\text{min}$)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
PFBS	2.08 - 2.50	298.93/79.82	40	28	45
		298.93/98.78	40	29	45
PFHS	2.45 - 3.05	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.80 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
¹³ C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of \pm 25% (\pm 30% for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.0880 to 0.121 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.18 to 28.2 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within \pm 25%, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by diluting 1 mL water with 10.0 mL MeOH:LCMS-water (60:40). Method blanks were prepared to evaluate the levels of background contamination in the overall dilution process (glassware, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carryover.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of LCMS-water, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCS)

One lab control spike was prepared and analyzed.

LCS was prepared by spiking known amounts of the analytes into 1.0 mL of LCMS-water to produce the desired concentration.

The spiked water sample was diluted with 10.0 mL MeOH:LCMS-water (60:40) and analyzed in the same manner as the samples.

The LCS produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 7 summarizes the LCS recovery results

Table 7. Lab Control Spike Results.

Dilution date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
20 May 2019	LCS1	80	92.4	96.5	77.1	106	103	105	106

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing PFBS, PFHS, PFOA, ^{13}C -PFOA, PFOS, FOSA and ^{13}C -PFOS (QC Lab Low and QC Lab High).

The QCs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 8 summarizes the QCs recovery results.

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
25 Jan 2018	QC Lab Low inj1	10	102	99.3	91.5	124	107	97.7	108
	QC Lab High inj1	100	83.9	79.4	93.1	94.4	97.2	98.6	106
	QC Lab Low inj2	10	96.4	99.7	78.5	82.7	109	107	119
	QC Lab High inj2	100	79.0	76.9	77.7	82.0	96.2	97.5	122

3.7. Equations.Recovery.

$$\text{Recovery (\%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

The following tables summarize the results for the sample locations.

Table 9. Sample Results PFBS, PFHS, PFOA, PFOS and FOSA

	PFBS ng/ml	PFHS ng/ml	PFOA ng/ml	PFOS ng/ml	FOSA ng/ml
L21		<0.182	0.258	3.09	<0.180
L22		0.250	0.480	1.80	<0.180
L31		1.51	0.369	3.85	<0.180
L4		2.04	2.01	18.8	0.800
P114bis		1.78	3.97	5.97	<0.180
P115		2.15	3.18	1.51	<0.180
P116		0.58	0.929	6.46	<0.180
Effluent WWTP		10.6	10.4	22.1	0.223
PP11	54.7	137	80.8	551	24.8
PP02	30.5	3628	514	16987	<20.5
PP04	978	234	576	4335	32.7
PP05	5068	3290	1028	335	50.3
PP06	26.0	56.1	246	890	48.0
PP07	197	89.9	203	1944	45.3
PP08	12.5	164	343	4186	25.8
PP09	21.0	304	512	2088	10.2
PP10	11.8	155	369	2590	11.5
12		16.3	48.8	223	<0.537
13		18.8	47.7	206	<0.537
5		27.6	27.2	35.6	<0.180
Bemalingsstation		22.5	20.7	29.8	<0.180
Collector Put		35.6	79.2	218	11.2
Field Trip Blank	<1.01	<0.988	<1.34	<1.08	<0.977

Table 10. Sample Results ^{13}C -PFOA and ^{13}C -PFOS

	^{13}C -PFOA		^{13}C -PFOS	
	ng/ml	% Rec	ng/ml	% Rec
L21	62.2	108	56.5	91.8
L22	64.1	112	54.8	89.0
L31	64.8	113	54.3	88.2
L4	62.2	108	56.6	91.9
P114bis	65.1	113	54.0	87.7
P115	60.3	105	58.3	94.8
P116	66.1	115	53.2	86.3
Effluent WWTP	59.7	104	59.0	95.8
PP11	99.6	86.8	143	116
PP02	96.0	83.7	150	122
PP04	109	95.0	130	105
PP05	121	105	117	94.9
PP06	96.5	84.0	147	120
PP07	106	92.3	134	109
PP08	114	99.6	124	101
PP09	112	97.6	126	102
PP10	96.3	83.9	147	119
12	96.4	84.0	146	119
13	96.3	83.9	147	119
5	59.2	103	59.5	96.6
Bemalingsstation	57.1	99.5	61.6	100
Collector Put	61.7	108	57.0	92.6
Field Trip Blank	105	91.1	135	110

5. Conclusion.

- The recovery of ¹³C-PFOA and ¹³C-PFOS fell within the method acceptance criterion of 70-130%.
- Lab control spike recoveries fell within the method acceptance criteria for all compounds, indicating that the analysis method is in control,
- QC Lab Standards recoveries fell within the method acceptance criteria for all compounds.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Kristof Bogaert (ERM Belgium).

8. Signatures.

Sven Herremans,
Technical Manager

Date March 28th, 2019



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date March 28th, 2019

I.A.C.
A Division of SGS Belgium NV

Reports are established on behalf of and for the account of the principal, who expressly accepts that these reports purely represent the situation at a given time and that they must always be presented and/or mentioned in their totality and in their particular context. SGS Belgium N.V., issuer of the reports, cannot be held liable for errors or modification of results during electronic or fax transmission. Only the originally signed report is binding.

The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.

Final Report

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

April 2019 Sampling

Laboratory Request Number: ISO19-14-02

Report Date – Date of Last Signature

Testing Laboratory

3M Environment, Health, and Safety
3M EHS Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Charlotte Tack
3M Belgium EHS Operations
3M Belgium; ZW019/0/01
Email: [REDACTED]



The testing reported herein meet the requirements of ISO/IEC 17025:2017 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M EHS Laboratory

3M EHS Laboratory Manager: Brian T. Mader
3M Principal Analytical Investigator: Susan Wolf
Report Author: Chelsie Grochow

Analytical Report ISO19-14-02

Water Sample Analysis at 3M Antwerp, Belgium
April 2019 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M EHS Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. All samples were collected on April 23-25, 2019, and returned to the 3M EHS Laboratory on May 3, 2019, at ambient temperature. The results in this report apply to the samples as received from ERM. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS) (select locations only), Perfluorohexane sulfonate (PFHS), Perfluoroctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO19-14-02.

The 3M EHS Laboratory prepared sample containers for twenty-nine sampling locations. Each sample set consisted of a field sample and field sample duplicate. Seven locations also included a target analyte field matrix spike. Each empty container was marked with a “fill to here” line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with a mass-labeled internal standard mix and select sample bottles were fortified with surrogate recovery standards [$^{13}\text{C}_4$]-PFOA and [$^{13}\text{C}_4$]-PFOS prior to being sent to the field for sample collection. During sample collection, sample location PP01 was not sampled and it was indicated on the chain of custody that samples were collected from location PP11 instead.

Samples were prepared and analyzed using method ETS-8-044.3 “Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis”. Select internal standards were used to aid in the data quality objectives.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ISO/IEC 17025:2017 “General Requirements for the Competence of Testing and Calibration Laboratories”, in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary ⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA
Zone: 2nd Aquifer					
ISO19-14-02-001	D16; Sample	880	393	3420	4.22
ISO19-14-02-001-DUP	D16; Sample Dup	777	329	3310	2.64
		Average	829 ⁽²⁾	361 ⁽²⁾	3370 ⁽²⁾
		%RPD Sample/Sample Dup	12	18	3.3
ISO19-14-02-002	P321; Sample	1930	448	5220	0.208
ISO19-14-02-002-DUP	P321; Sample Dup	1970	463	5770	0.204
		Average	1950 ⁽²⁾	456 ⁽²⁾	5500 ⁽²⁾
		%RPD Sample/Sample Dup	2.1	3.3	10
ISO19-14-02-003	P121; Sample	2.68	1.83	0.748	0.0966
ISO19-14-02-003-DUP	P121; Sample Dup	2.98	1.93	0.618	0.0892
		Average	2.83	1.88	0.683
		%RPD Sample/Sample Dup	11	5.3	19
Zone: Blokkersdijk Nature Reserve					
ISO19-14-02-004	3M vijver; Sample	1.04	0.580	3.20	0.0736
ISO19-14-02-004-DUP	3M vijver; Sample Dup	1.16	0.592	3.18	0.0698
		Average	1.10	0.586	3.19
		%RPD Sample/Sample Dup	11	2.0	0.63
ISO19-14-02-005	Blokkersdijkvijver - Noord; Sample	0.970	0.558	2.98	0.0806
ISO19-14-02-005-DUP	Blokkersdijkvijver - Noord; Sample Dup	1.17	0.630	3.48	0.117
		Average	1.07	0.594	3.23
		%RPD Sample/Sample Dup	19	12	15
ISO19-14-02-006	Blokkersdijkvijver - standard; Sample	0.910	0.604	0.852	0.0402
ISO19-14-02-006-DUP	Blokkersdijkvijver - standard; Sample Dup	0.844	0.516	0.758	0.0348
		Average	0.877	0.560	0.805
		%RPD Sample/Sample Dup	7.5	16	12
ISO19-14-02-007	L21; Sample	0.152	0.103	4.14	0.0494
ISO19-14-02-007-DUP	L21; Sample Dup	0.146	0.0916	3.96	0.0478
		Average	0.149	0.0973	4.05
		%RPD Sample/Sample Dup	4.0	12	4.4
ISO19-14-02-008	L22; Sample	0.348	0.284	2.76	0.0570
ISO19-14-02-008-DUP	L22; Sample Dup	0.344	0.262	2.24	0.0358
		Average	0.346	0.273	2.50
		%RPD Sample/Sample Dup	1.2	8.1	21 ⁽³⁾
ISO19-14-02-009	L31; Sample	0.390	1.17	4.92	0.0544
ISO19-14-02-009-DUP	L31; Sample Dup	0.304	1.07	4.22	0.0592
		Average	0.347	1.12	4.57
		%RPD Sample/Sample Dup	25 ⁽³⁾	8.9	15
					8.5

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using internal standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 16%, PFHS \pm 14%, PFOS \pm 43%, and PFOSA \pm 19%.
- (2) Sample set reported using external standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 18%, PFBS \pm 10%, PFHS \pm 12%, PFOS \pm 20%, and PFOSA \pm 24%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary ⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA
Zone: Blokkersdijk Nature Reserve					
ISO19-14-02-010	L4; Sample	2.10	1.87	24.8	1.88
ISO19-14-02-010-DUP	L4; Sample Dup	2.04	2.06	25.8	2.32
		Average	2.07	1.97	25.3
		%RPD Sample/Sample Dup	2.9	9.7	4.0
					21 ⁽³⁾
ISO19-14-02-011	P114bis; Sample	3.40	1.54	6.50	0.114
ISO19-14-02-011-DUP	P114bis; Sample Dup	3.70	1.68	6.96	0.119
		Average	3.55	1.61	6.73
		%RPD Sample/Sample Dup	8.5	8.7	6.8
					0.117
ISO19-14-02-012	P115; Sample	3.64	2.16	2.28	0.0582
ISO19-14-02-012-DUP	P115; Sample Dup	3.82	2.08	2.26	0.0402
		Average	3.73	2.12	2.27
		%RPD Sample/Sample Dup	4.8	3.8	0.88
					37 ⁽³⁾
ISO19-14-02-013	P116; Sample	0.736	0.498	8.42	0.123
ISO19-14-02-013-DUP	P116; Sample Dup	0.780	0.550	7.58	0.170
		Average	0.758	0.524	8.00
		%RPD Sample/Sample Dup	5.8	9.9	11
					0.147
					32 ⁽³⁾
Zone: Effluent WWTP					
ISO19-14-02-014	Effluent WWTP; Sample	11.4	10.6	29.6	0.584
ISO19-14-02-014-DUP	Effluent WWTP; Sample Dup	10.6	10.2	27.6	0.520
		Average	11.0	10.4	28.6
		%RPD Sample/Sample Dup	7.3	3.8	7.0
					0.552
Zone: Palingbeek & Tophatgracht					
ISO19-14-02-024	12; Sample	62.9	18.2	296	0.558
ISO19-14-02-024-DUP	12; Sample Dup	65.9	18.3	292	0.538
		Average	64.4 ⁽²⁾	18.3 ⁽²⁾	294 ⁽²⁾
		%RPD Sample/Sample Dup	4.7	0.55	1.4
					0.548
ISO19-14-02-025	13; Sample	69.8	22.4	274	0.568
ISO19-14-02-025-DUP	13; Sample Dup	68.9	22.6	285	0.574
		Average	69.4 ⁽²⁾	22.5 ⁽²⁾	280 ⁽²⁾
		%RPD Sample/Sample Dup	1.3	0.89	3.9
					0.571
ISO19-14-02-026	5; Sample	28.0	26.4	42.8	0.177
ISO19-14-02-026-DUP	5; Sample Dup	28.6	25.4	43.2	0.179
		Average	28.3	25.9	43.0
		%RPD Sample/Sample Dup	2.1	3.9	0.93
					0.178
ISO19-14-02-027	Bemalingsstation; Sample	24.4	23.0	38.2	0.206
ISO19-14-02-027-DUP	Bemalingsstation; Sample Dup	22.4	23.2	38.8	0.189
		Average	23.4	23.1	38.5
		%RPD Sample/Sample Dup	8.5	0.87	1.6
					0.198

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using internal standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 16%, PFHS \pm 14%, PFOS \pm 43%, and PFOSA \pm 19%.
- (2) Sample set reported using external standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 18%, PFBS \pm 10%, PFHS \pm 12%, PFOS \pm 20%, and PFOSA \pm 24%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA
Zone: Sewer					
ISO19-14-02-028	Collector put; Sample	85.6	31.2	269	24.4
ISO19-14-02-028-DUP	Collector put; Sample Dup	72.4	29.6	267	24.2
Average		79.0	30.4	268⁽²⁾	24.3
%RPD Sample/Sample Dup		17	5.3	0.75	0.82

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
Zone: Extraction Wells P&T						
ISO19-14-02-015	PP11; Sample	104	49.4	144	672	38.8
ISO19-14-02-015-DUP	PP11; Sample Dup	103	49.3	145	704	36.4
Average		104⁽²⁾	49.4⁽²⁾	145⁽²⁾	688⁽²⁾	37.6⁽²⁾
%RPD Sample/Sample Dup		0.97	0.20	0.69	4.7	6.4
ISO19-14-02-016	PP02; Sample	562	38.1	4080	21500	35.8
ISO19-14-02-016-DUP	PP02; Sample Dup	622	39.9	4030	20400	27.8
Average		592⁽²⁾	39.0⁽²⁾	4060⁽²⁾	21000⁽²⁾	31.8⁽²⁾
%RPD Sample/Sample Dup		10	4.6	1.2	5.3	25⁽³⁾
ISO19-14-02-017	PP04; Sample	680	1080	272	5070	58.4
ISO19-14-02-017-DUP	PP04; Sample Dup	698	1100	279	5030	60.2
Average		689⁽²⁾	1090⁽²⁾	276⁽²⁾	5050⁽²⁾	59.3⁽²⁾
%RPD Sample/Sample Dup		2.6	1.8	2.5	0.79	3.0
ISO19-14-02-018	PP05; Sample	855	5850	4350	456	102
ISO19-14-02-018-DUP	PP05; Sample Dup	819	6280	4430	423	109
Average		837⁽²⁾	6070⁽²⁾	4390⁽²⁾	440⁽²⁾	106⁽²⁾
%RPD Sample/Sample Dup		4.3	7.1	1.8	7.5	6.6
ISO19-14-02-019	PP06; Sample	337	35.7	68.0	1060	87.4
ISO19-14-02-019-DUP	PP06; Sample Dup	340	31.2	67.9	1160	87.2
Average		339⁽²⁾	33.5⁽²⁾	68.0⁽²⁾	1110⁽²⁾	87.3⁽²⁾
%RPD Sample/Sample Dup		0.89	13	0.15	9.0	0.23
ISO19-14-02-020	PP07; Sample	265	251	97.4	2280	79.6
ISO19-14-02-020-DUP	PP07; Sample Dup	255	233	90.1	2150	86.4
Average		260⁽²⁾	242⁽²⁾	93.8⁽²⁾	2220⁽²⁾	83.0⁽²⁾
%RPD Sample/Sample Dup		3.8	7.4	7.8	5.9	8.2
ISO19-14-02-021	PP08; Sample	416	20.4	173	5260	46.6
ISO19-14-02-021-DUP	PP08; Sample Dup	416	19.0	172	5300	49.6
Average		416⁽²⁾	19.7⁽²⁾	173⁽²⁾	5280⁽²⁾	48.1⁽²⁾
%RPD Sample/Sample Dup		0.0	7.1	0.58	0.76	6.2

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using internal standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 16%, PFHS \pm 14%, PFOS \pm 43%, and PFOSA \pm 19%.
- (2) Sample set reported using external standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 18%, PFBS \pm 10%, PFHS \pm 12%, PFOS \pm 20%, and PFOSA \pm 24%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
Zone: Extraction Wells P&T						
ISO19-14-02-022	PP09; Sample	596	20.8	335	2410	13.6
ISO19-14-02-022-DUP	PP09; Sample Dup	627	20.1	324	2390	14.3
Average %RPD Sample/Sample Dup		612⁽²⁾	20.5⁽²⁾	330⁽²⁾	2400⁽²⁾	14.0⁽²⁾
%RPD Sample/Sample Dup		5.1	3.4	3.3	0.83	5.0
ISO19-14-02-023	PP10; Sample	489	14.6	186	3430	24.0
ISO19-14-02-023-DUP	PP10; Sample Dup	503	16.2	182	3310	20.4
Average %RPD Sample/Sample Dup		496⁽²⁾	15.4⁽²⁾	184⁽²⁾	3370⁽²⁾	22.2⁽²⁾
%RPD Sample/Sample Dup		2.8	10	2.2	3.6	16
Zone: Source Area – Building 16						
ISO19-14-02-029	P21B; Sample	9340	7730	11900	60600	6.56
ISO19-14-02-029-DUP	P21B; Sample Dup	9190	7280	11400	61000	6.58
Average %RPD Sample/Sample Dup		9270⁽²⁾	7510⁽²⁾	11700⁽²⁾	60800⁽²⁾	6.57
%RPD Sample/Sample Dup		1.6	6.0	4.3	0.66	0.30
ISO19-14-02-030	Travel Blank	<0.0958	<0.200 ⁽²⁾	<0.0500	<0.185	<0.0250

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using internal standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 16%, PFHS ± 14%, PFOS ± 43%, and PFOSA ± 19%.
- (2) Sample set reported using external standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 18%, PFBS ± 10%, PFHS ± 12%, PFOS ± 20%, and PFOSA ± 24%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M EHS Laboratory method ETS-8-044.3 “Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis”.

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected on April 23-25, 2019, in Nalgene™ (high-density polyethylene) bottles prepared at the 3M EHS Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. All sample locations were spiked with a mass-labeled internal standard mix at a nominal concentration of 1 ng/mL and select samples were fortified with surrogate recovery standards (SRSs) at a nominal concentration of 0.1 ng/mL prior to sample collection. Samples were received on May 3, 2019.

2.3 Sample Preparation

Sample locations pre-spiked with surrogate recovery standards were prepared for PFOA, PFHS, and PFOS, and all samples were prepared for PFOSA by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples along with the method blanks.

Samples that required further dilution were prepared for PFOA, PFHS, PFOS and PFBS (select locations only) using a direct injection by methanol dilution method. Samples that required a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples that required a 1:100 dilution were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol. Samples that required a 1:500 dilution were prepared by diluting 0.02 mL of a well-mixed sample with 10.0 mL of methanol. Samples that required a 1:1000 dilution were prepared by first diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol, followed by a second dilution prepared by diluting 1 mL of the diluted sample into 9 mL of methanol. Diluted samples were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

2.4 Analysis

All samples and quality control samples were analyzed for the target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). SRSs were added and included in the analysis of these samples. Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.2. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

The following analytical runs were used to report the results herein:

5/9/19 (ETS Kirk) Internal Standard Calibration Analysis:

- All sample locations pre-spiked with surrogate recovery standards were analyzed for PFOA, PFHS, PFOS, and PFOSA. All sample results were reported **except** for the following: Collector Put (PFOS).

5/10/19 (ETS DaVinci) Internal Standard Calibration Analysis:

- All remaining sample locations were analyzed for PFOSA. All sample results were reported **except** for PP02 – PP11.

5/15/19 (ETS Tesla) External Standard Calibration Analysis:

- All remaining sample locations were analyzed for PFOA, PFHS, and PFOS with select locations analyzed for PFBS. In addition, sample location Collector Put was re-analyzed for PFOS. All sample results were reported **except** for the following: P21B (PFOS).

5/22/19 (ETS Kirk) External Standard Calibration Analysis:

- Sample location P21B was analyzed and reported for PFOS.

5/23/19 (ETS Kirk) External Standard Calibration Analysis:

- Sample locations PP02 – PP11 were re-analyzed for PFOSA. All sample results were reported **except** for PP07.

5/29/19 (ETS Kirk) External Standard Calibration Analysis:

- Sample location PP07 was re-analyzed and reported for PFOSA.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk	ETS DaVinci	ETS Tesla
Liquid Chromatograph	Agilent 1260	Agilent 1200	Agilent 1260
Analysis Method	ETS-8-044.3	ETS-8-044.3	ETS-8-044.3
Analysis Date	5/9/19, 5/23/19, 5/29/19	5/10/19	5/15/19
Guard column	Prism RP (2.1 mm X 50 mm), 5 μ	Prism RP (2.1 mm X 50 mm), 5 μ	Prism RP (2.1 mm X 50 mm), 5 μ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ
Injection Volume	1, 2 or 5 μ L	10 μ L	5 μ L
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 6500	AB Sciex Triple Quad 6500+
Ion Source	Turbo Spray	Turbo Spray	Turbo Spray
Polarity	Negative	Negative	Negative
Software	Analyst 1.6.3	Analyst 1.6.3	Analyst 1.6.3

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis				
Step Number	Total Time (min)	Flow Rate (μ L/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0.00	300	90.0	10.0
1	0.50	300	90.0	10.0
2	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard ⁽¹⁾	Mass Transition Q1/Q3
PFOA	413/369	$[^{13}\text{C}_8]\text{-PFOA}$	421/376
	413/219		
	413/169		
PFBS	299/99	$[^{18}\text{O}_2]\text{-PFBS}$	303/84
	299/80		
PFHS	399/99	$[^{13}\text{C}_3]\text{-PFHS}$	402/80
	399/80		
PFOS	499/99	$[^{13}\text{C}_8]\text{-PFOS}$	507/80
	499/80		
	499/130		
PFOSA	498/78	$[^{13}\text{C}_8]\text{-PFOSA}$	506/78
$[^{13}\text{C}_4]\text{-PFOA}$	417/372	$[^{13}\text{C}_8]\text{-PFOA}$	421/376
$[^{13}\text{C}_4]\text{-PFOS}$	503/80	$[^{13}\text{C}_8]\text{-PFOS}$	507/80

The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

(1) Internal standard was not used for the external calibration analyses on 5/15/19 and 5/22/19.

3 Data Analysis

3.1 Calibration

5/9/19 and 5/10/19 Analysis (Internal Standard Calibration): Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of fourteen calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed. The calibration standards also contained surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

5/15/19 and 5/23/18 Analysis (External Standard Calibration): Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL to 25 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

5/23/19 and 5/29/19 Analysis of PFOSA (External Standard Calibration): Samples were analyzed for PFOSA against an external standard matrix-matched calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into laboratory reagent water. Calibration standards ranging from 0.025 ng/mL to 300 ng/mL (nominal) were analyzed. Prior to analysis, the calibration standards were diluted 2x by removing a 0.4 mL aliquot and diluting it with 0.4 mL of methanol. A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of $100\pm 25\%$ ($100\pm 30\%$ for the lowest curve point) were met for all analytes. The correlation coefficient (r) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes except for the following:

- On 5/29/19, area ratio RSD was 9.3% for FOSA. Other QC elements were used to determine the reportability of the sample results, including laboratory control samples and field matrix spikes. These other QC elements are discussed in sections 3.6 and 4 of the report.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.3 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the **Table 6** below.

Table 6. LOQ

Analyte	LOQ, ng/mL ⁽¹⁾ 5/9/19 Analysis	LOQ, ng/mL ⁽¹⁾ 5/10/19 Analysis	LOQ, ng/mL ⁽²⁾ 5/15/19 Analysis	LOQ, ng/mL ⁽²⁾ 5/23/19 Analysis	LOQ, ng/mL ⁽¹⁾ 5/23/19 Analysis	LOQ, ng/mL ⁽¹⁾ 5/29/19 Analysis
PFOA	0.0958	NA	0.0479	NA	NA	NA
PFBS	NA	NA	0.0200	NA	NA	NA
PFHS	0.0500	NA	0.0200	NA	NA	NA
PFOS	0.185	NA	0.0464	0.0185	NA	NA
PFOSA	0.0250	0.0500	NA	NA	0.0500	0.500

NA = Not Applicable

(1) A dilution factor of 2 was applied to the LOQ.

(2) A dilution factor was not applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of $100\% \pm 25\%$.

3.5 Blanks

Two types of blanks were prepared and analyzed with the samples: method/solvent blanks and field/trip blanks. Each blank result was reviewed and used to evaluate method performance. The method blanks were used to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. LCSs were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. Surrogates [$^{13}\text{C}_4$]-PFOA and [$^{13}\text{C}_4$]-PFOS were added post dilution when analyzed by external standard.

- Target analyte LCSs analyzed on 5/9/19 and 5/10/19 were prepared at nominal concentrations of 0.2 ng/mL, 20 ng/mL, and 140 ng/mL.
- Target analyte LCSs analyzed on 5/15/19 were prepared at nominal concentrations of 100 ng/mL, 5000 ng/mL, and 35000 ng/mL.
- Target analyte LCSs analyzed on 5/22/19 were prepared at nominal concentrations of 200 ng/mL, 20000 ng/mL, and 70000 ng/mL.
- Target analyte LCSs analyzed on 5/23/19 and 5/29/19 were prepared at nominal concentrations of 0.2 ng/mL, 20 ng/mL, and 200 ng/mL.

Method ETS-8-044.3 states that the average recovery of LCSs at each spiking level must be within 80%-120% with an RSD $\leq 20\%$. All LCS samples met criteria with the following exception:

- 5/9/19: Two low-level LCSs (0.2 ng/mL) for PFOS were below the resulting LOQ (0.185 ng/mL) and the third had a recovery of 143%. The Mid and High-level LCSs met method acceptance criteria for PFOS. Sample results for PFOS that were within the calibration range were reported from this run. A method deviation for the high LCS recovery for the low-level LCS is included with the raw data. High-level LCSs (140 ng/mL) for PFOSA were spiked above the resulting ULOQ. The Low and Mid-level LCSs were appropriate for the sample concentrations and the data were reported.
- 5/23/19: Low-level LCSs (0.2 ng/mL) for PFOSA had an average recovery of 124% using external calibration. The High-level LCSs (200 ng/mL) for PFOSA were spiked above the resulting ULOQ. The High-level LCSs were reinjected on 5/29/19 and met acceptance criteria.
- 5/29/19: Low-level LCSs (0.2 ng/mL) for PFOSA were spiked below the results LLOQ. The Mid and High-level LCSs were appropriate for the sample concentrations and the data were reported.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report. Individual LCS results are included in the raw data and available upon request.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.4. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in **Table 7** below.

- The data uncertainty for PFOS using internal calibration was calculated at $\pm 23\%$ following ETS-12-012.4; however, the data uncertainty was increased to $\pm 43\%$ based on the PFOS recovery of the reportable low-level LCS from analysis on 5/9/19.
- The data uncertainty for PFOSA using external calibration was calculated at $\pm 21\%$ following ETS-12-012.4; however, the data uncertainty was increased to $\pm 24\%$ based on the PFOSA recovery of the low-level LCSs from analysis on 5/23/19.

Table 7. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	8.98	$\pm 18\%$
PFBS	External	5.19	$\pm 10\%$
PFHS	External	6.15	$\pm 12\%$
PFOS	External	9.84	$\pm 20\%$
PFOSA	External	NA	$\pm 24\%$
PFOA	Internal	8.20	$\pm 16\%$
PFHS	Internal	6.97	$\pm 14\%$
PFOS	Internal	NA	$\pm 43\%$
PFOSA	Internal	9.26	$\pm 19\%$

NA = Not Applicable

3.8 Field Matrix Spikes (FMS)

Target analyte field matrix spike samples were collected for select sampling points to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions at 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOA and PFOS. The standards used for the preparation of the field matrix spiking solutions greater than 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, select sample bottles contained stable isotope surrogate recovery spikes of [¹³C₄]-PFOA and [¹³C₄]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles prior to sample collection. The [¹³C₄]-PFOA was selected to represent the C8 perfluorocarboxylic acid. The [¹³C₄]-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

The following calculation was used to generate data in section 4 of the report.

$$FMS\ Recovery = \frac{Sample\ Concentration\ of\ FMS - Average\ Concentration\ (Field\ Sample\ & Field\ Sample\ Dup)}{Spike\ Concentration} \times 100\%$$

Table 8. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
Blokkersdijkvijver - Noord	FMS	1.92	2.00	2.00	1.85	2.00
P114bis, P21B	FMS	9.58	10.0	10.0	9.27	9.99
5	FMS	102	102	102	102	2.00
D16, PP05, PP08	FMS	2050	2050	2050	2050	50.0
Trip Blank	Low	1.92	2.00	2.00	1.85	2.00
	High	2050	2050	2050	2050	50.0

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of $\pm 30\%$, demonstrate that the method is appropriate for the given matrix.

The method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. At times, the spike level exceeded the recommended upper limit of 10 times the analyte concentration. In these instances, the FMS recovery was reported and flagged as above 10 times the sample concentration.

For PFOA, PFBS, PFHS, and PFOS, when the field matrix spike level was not appropriate as compared to the endogenous sample concentration, the surrogate recovery standards were used to assess method accuracy. All field matrix spike and surrogate recovery standard recoveries met acceptance criteria.

Table 9. Location ID: D16

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-001	D16; Sample	880	NA	393	NA
ISO19-14-02-001-DUP	D16; Sample Duplicate	777	NA	329	NA
ISO19-14-02-001-FMS	D16; Sample FMS	2580	85.4	2220	90.7
Average Concentration (ng/mL) ± %RPD		829 ng/mL ± 12%		361 ng/mL ± 18%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-001	D16; Sample	3420	NA	4.22	NA
ISO19-14-02-001-DUP	D16; Sample Duplicate	3310	NA	2.64	NA
ISO19-14-02-001-FMS	D16; Sample FMS	5330	95.9	50.6	94.3 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		3370 ng/mL ± 3.3%		3.43 ng/mL ± 46% ⁽²⁾	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 7. Location ID: Blokkersdijkvijver - Noord

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-005	Blokkersdijkvijver - Noord; Sample	0.970	NA	0.558	NA
ISO19-14-02-005-DUP	Blokkersdijkvijver - Noord; Sample Dup	1.17	NA	0.630	NA
ISO19-14-02-005-FMS	Blokkersdijkvijver - Noord; FMS	2.96	98.4	2.68	104
Average Concentration (ng/mL) ± %RPD		1.07 ng/mL ± 19%		0.594 ng/mL ± 12%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-005	Blokkersdijkvijver - Noord; Sample	2.98	NA	0.0806	NA
ISO19-14-02-005-DUP	Blokkersdijkvijver - Noord; Sample Dup	3.48	NA	0.117	NA
ISO19-14-02-005-FMS	Blokkersdijkvijver - Noord; FMS	5.32	113	2.06	98.1 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		3.23 ng/mL ± 15%		0.0988 ng/mL ± 37% ⁽²⁾	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 12. Location ID: P114bis

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-011	P114bis; Sample	3.40	NA	1.54	NA
ISO19-14-02-011-DUP	P114bis; Sample Duplicate	3.70	NA	1.68	NA
ISO19-14-02-011-FMS	P114bis; FMS	13.9	108	11.9	103
Average Concentration (ng/mL) ± %RPD		3.55 ng/mL ± 8.5%		1.61 ng/mL ± 8.7%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-011	P114bis; Sample	6.50	NA	0.114	NA
ISO19-14-02-011-DUP	P114bis; Sample Duplicate	6.96	NA	0.119	NA
ISO19-14-02-011-FMS	P114bis; FMS	15.8	97.8	10.4	103 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		6.73 ng/mL ± 6.8%		0.117 ng/mL ± 4.3%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 13. Location ID: PP05

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-018	PP05; Sample	855	NA	5850	NA	4350	NA
ISO19-14-02-018-DUP	PP05; Sample Duplicate	819	NA	6280	NA	4430	NA
ISO19-14-02-018-FMS	PP05; FMS	2570	84.5	7720	NC	6430	NC
Average Concentration (ng/mL) ± %RPD		837 ng/mL ± 4.3%		6070 ng/mL ± 7.1%		4390 ng/mL ± 1.8%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-018	PP05; Sample	456	NA	102	NA
ISO19-14-02-018-DUP	PP05; Sample Duplicate	423	NA	109	NA
ISO19-14-02-018-FMS	PP05; FMS	1980	75.1	NA ⁽¹⁾	NA ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		440 ng/mL ± 7.5%		106 ng/mL ± 6.6%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) FMS not re-analyzed for analyte due to inappropriate spike level.

Table 14. Location ID: PP08

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-021	PP08; Sample	416	NA	20.4	NA	173	NA
ISO19-14-02-021-DUP	PP08; Sample Duplicate	416	NA	19.0	NA	172	NA
ISO19-14-02-021-FMS	PP08; FMS	2220	88.0	1970	95.1 ⁽¹⁾	2200	98.9 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		416 ng/mL ± 0.0%		19.7 ng/mL ± 7.1%		173 ng/mL ± 0.58%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-021	PP08; Sample	5260	NA	46.6	NA
ISO19-14-02-021-DUP	PP08; Sample Duplicate	5300	NA	49.6	NA
ISO19-14-02-021-FMS	PP08; FMS	7480	NC	87.6	79.0
Average Concentration (ng/mL) ± %RPD		5280 ng/mL ± 0.76%		48.1 ng/mL ± 6.2%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) FMS concentration greater than 10 times the sample concentration.

Table 15. Location ID: 5

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-026	5; Sample	28.0	NA	26.4	NA
ISO19-14-02-026-DUP	5; Sample Duplicate	28.6	NA	25.4	NA
ISO19-14-02-026-FMS	5; Sample FMS	107	77.2	102	74.6
Average Concentration (ng/mL) ± %RPD		28.3 ng/mL ± 2.1%		25.9 ng/mL ± 3.9%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-026	5; Sample	42.8	NA	0.177	NA
ISO19-14-02-026-DUP	5; Sample Duplicate	43.2	NA	0.179	NA
ISO19-14-02-026-FMS	5; Sample FMS	126	81.4	2.12	97.1 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		43.0 ng/mL ± 0.93%		0.178 ng/mL ± 1.1%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 16. Location ID: P21B

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-029	P21B; Sample	9340	NA	7730	NA	11900	NA
ISO19-14-02-029-DUP	P21B; Sample Duplicate	9190	NA	7280	NA	11400	NA
ISO19-14-02-029-FMS	P21B; FMS	9290	NC	7630	NC	11800	NC
Average Concentration (ng/mL) ± %RPD		9270 ng/mL ± 1.6%		7510 ng/mL ± 6.0%		11700 ng/mL ± 4.3%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-029	P21B; Sample	60600	NA	6.56	NA
ISO19-14-02-029-DUP	P21B; Sample Duplicate	61000	NA	6.58	NA
ISO19-14-02-029-FMS	P21B; FMS	NA ⁽¹⁾	NA ⁽¹⁾	15.0	84.4
Average Concentration (ng/mL) ± %RPD		60800 ng/mL ± 0.66%		6.57 ng/mL ± 0.30%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) FMS not re-analyzed for analyte due to inappropriate spike level.

Table 17. Location ID: Travel Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-030	Travel Blank	<0.0958	NA	<0.200	NA	<0.0500	NA
ISO19-14-02-030-FMS-LOW	Travel Blank FMS Low	2.02	105	NA ⁽¹⁾	NA ⁽¹⁾	1.92	96.0
ISO19-14-02-030-FMS-HIGH	Travel Blank FMS High	1740	84.9	1810	88.3	1890	92.2

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-02-030	Travel Blank	<0.185	NA	<0.0250	NA
ISO19-14-02-030-FMS-LOW	Travel Blank FMS Low	1.83	98.9	1.93	96.5
ISO19-14-02-030-FMS-HIGH	Travel Blank FMS High	1800	87.8	50.2	100

NA = Not Applicable

(1) FMS Low not analyzed for PFBS due to the spike level not used for any samples analyzed at that level.

Table 18. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO19-14-02-001	D16; Sample	104 ⁽²⁾	103 ⁽²⁾	NA
ISO19-14-02-001-DUP	D16; Sample Duplicate	105 ⁽²⁾	96.3 ⁽²⁾	NA
ISO19-14-02-001-FMS	D16; FMS	100 ⁽²⁾	93.6 ⁽²⁾	NA
ISO19-14-02-002	P321; Sample	110 ⁽²⁾	99.9 ⁽²⁾	NA
ISO19-14-02-002-DUP	P321; Sample Duplicate	116 ⁽²⁾	100 ⁽²⁾	NA
ISO19-14-02-003	P121; Sample	100	107	NA
ISO19-14-02-003-DUP	P121; Sample Duplicate	90.4	100	NA
ISO19-14-02-004	3M vijver; Sample	106	93.6	NA
ISO19-14-02-004-DUP	3M vijver; Sample Duplicate	101	93.1	NA
ISO19-14-02-005	Blokkersdijkvijver - Noord; Sample	95.0	94.0	NA
ISO19-14-02-005-DUP	Blokkersdijkvijver - Noord; Sample Dup	103	96.8	NA
ISO19-14-02-005-FMS	Blokkersdijkvijver - Noord; FMS	98.8	111	NA
ISO19-14-02-006	Blokkersdijkvijver-standard; Sample	93.8	95.3	NA
ISO19-14-02-006-DUP	Blokkersdijkvijver-standard; Sample Dup	96.6	96.3	NA
ISO19-14-02-007	L21; Sample	94.4	97.4	NA
ISO19-14-02-007-DUP	L21; Sample Duplicate	107	98.6	NA
ISO19-14-02-008	L22; Sample	103	97.0	NA
ISO19-14-02-008-DUP	L22; Sample Duplicate	97.6	93.8	NA
ISO19-14-02-009	L31; Sample	92.8	95.5	NA
ISO19-14-02-009-DUP	L31; Sample Duplicate	89.8	99.1	NA
ISO19-14-02-010	L4; Sample	99.8	102	NA
ISO19-14-02-010-DUP	L4; Sample Duplicate	95.8	99.5	NA
ISO19-14-02-011	P114bis; Sample	103	97.8	NA
ISO19-14-02-011-DUP	P114bis; Sample Duplicate	102	94.9	NA
ISO19-14-02-011-FMS	P114bis; FMS	97.4	99.5	NA
ISO19-14-02-012	P115; Sample	95.0	89.4	NA
ISO19-14-02-012-DUP	P115; Sample Duplicate	105	92.1	NA
ISO19-14-02-013	P116; Sample	96.4	97.4	NA
ISO19-14-02-013-DUP	P116; Sample Duplicate	103	97.2	NA
ISO19-14-02-014	Effluent WWTP; Sample	98.4	92.1	NA
ISO19-14-02-014-DUP	Effluent WWTP; Sample Duplicate	92.6	98.8	NA
ISO19-14-02-015	PP11; Sample	104 ⁽²⁾	101 ⁽²⁾	NA
ISO19-14-02-015-DUP	PP11; Sample Duplicate	112 ⁽²⁾	104 ⁽²⁾	NA
ISO19-14-02-016	PP02; Sample	102 ⁽²⁾	101 ⁽²⁾	NA
ISO19-14-02-016-DUP	PP02; Sample Duplicate	102 ⁽²⁾	91.4 ⁽²⁾	NA
ISO19-14-02-017	PP04; Sample	88.0 ⁽²⁾	84.3 ⁽²⁾	NA
ISO19-14-02-017-DUP	PP04; Sample Duplicate	94.8 ⁽²⁾	89.3 ⁽²⁾	NA
ISO19-14-02-018	PP05; Sample	102 ⁽²⁾	78.2 ⁽²⁾	NA
ISO19-14-02-018-DUP	PP05; Sample Duplicate	93.5 ⁽²⁾	73.2 ⁽²⁾	NA
ISO19-14-02-018-FMS	PP05; FMS	94.1 ⁽²⁾	79.9 ⁽²⁾	NA

NA = Not Applicable

- (1) The surrogate recovery standards were added to the sample bottle prior to the sampling event unless noted otherwise.
(2) The surrogate recovery standards were added to the samples during sample preparation.

Table 18 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO19-14-02-019	PP06; Sample	108 ⁽²⁾	95.4 ⁽²⁾	NA
ISO19-14-02-019-DUP	PP06; Sample Duplicate	108 ⁽²⁾	98.8 ⁽²⁾	NA
ISO19-14-02-020	PP07; Sample	112 ⁽²⁾	104 ⁽²⁾	NA
ISO19-14-02-020-DUP	PP07; Sample Duplicate	100 ⁽²⁾	92.7 ⁽²⁾	NA
ISO19-14-02-021	PP08; Sample	107 ⁽²⁾	99.7 ⁽²⁾	NA
ISO19-14-02-021-DUP	PP08; Sample Duplicate	106 ⁽²⁾	96.6 ⁽²⁾	NA
ISO19-14-02-021-FMS	PP08; FMS	99.7 ⁽²⁾	98.4 ⁽²⁾	NA
ISO19-14-02-022	PP09; Sample	104 ⁽²⁾	92.3 ⁽²⁾	NA
ISO19-14-02-022-DUP	PP09; Sample Duplicate	101 ⁽²⁾	81.3 ⁽²⁾	NA
ISO19-14-02-023	PP10; Sample	108 ⁽²⁾	99.2 ⁽²⁾	NA
ISO19-14-02-023-DUP	PP10; Sample Duplicate	101 ⁽²⁾	94.6 ⁽²⁾	NA
ISO19-14-02-024	12; Sample	108 ⁽²⁾	96.4 ⁽²⁾	NA
ISO19-14-02-024-DUP	12; Sample Duplicate	98.3 ⁽²⁾	88.1 ⁽²⁾	NA
ISO19-14-02-025	13; Sample	110 ⁽²⁾	98.0 ⁽²⁾	NA
ISO19-14-02-025-DUP	13; Sample Duplicate	113 ⁽²⁾	101 ⁽²⁾	NA
ISO19-14-02-026	5; Sample	94.2	94.5	NA
ISO19-14-02-026-DUP	5; Sample Duplicate	97.4	86.9	NA
ISO19-14-02-026-FMS	5; FMS	100	97.6	NA
ISO19-14-02-027	Bemalingsstation; Sample	99.8	96.3	NA
ISO19-14-02-027-DUP	Bemalingsstation; Sample Dup	95.0	90.9	NA
ISO19-14-02-028	Collector put; Sample	103	94.5	96.2 ⁽²⁾
ISO19-14-02-028-DUP	Collector put; Sample Duplicate	99.4	92.4	100 ⁽²⁾
ISO19-14-02-029	P21B; Sample	98.1 ⁽²⁾	97.7 ⁽²⁾	115 ⁽²⁾
ISO19-14-02-029-DUP	P21B; Sample Duplicate	103 ⁽²⁾	96.6 ⁽²⁾	114 ⁽²⁾
ISO19-14-02-029-FMS	P21B; FMS	109 ⁽²⁾	103 ⁽²⁾	NA
ISO19-14-02-030	Travel Blank	96.8	93.0	NA
ISO19-14-02-030-FMS-LOW	Travel Blank FMS Low	103	102	NA
ISO19-14-02-030-FMS-HIGH	Travel Blank FMS High	101 ⁽²⁾	92.4 ⁽²⁾	NA

NA = Not Applicable

- (1) The surrogate recovery standards were added to the sample bottle prior to the sampling event unless noted otherwise.
(2) The surrogate recovery standards were added to the samples during sample preparation.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 10-18 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M EHS Laboratory standard operating procedures.

7 Attachment

Chain of Custody Form

8 Signatures

Chelsie Grochow, 3M Report Author

Susan T. Wolf, 3M Principal Analytical Investigator

Brian T. Mader, Ph.D., 3M EHS Laboratory Manager

The 3M EHS Laboratory's Quality Assurance Unit has audited the data and report for this project.

Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M EHS Laboratory.

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: (651) [REDACTED]
Alt. Phone: [REDACTED]
Fax: (651) [REDACTED]

Project: ISO19-14-02

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 3/20/2019
Project Description: 3M Antwerp Water Sampling for PFCs
Comments:

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
✓ ISO19-14-02-001	D16; Sample	24104/19 - 7:25 AM		
✓ ISO19-14-02-001-DUP	D16; Sample Duplicate	24104/19 - 7:25 AM		
✓ ISO19-14-02-001-FMS	D16; FMS	24104/19 - 7:25 AM		
✓ ISO19-14-02-002	P321; Sample	24104/19 - 8:25 AM		
✓ ISO19-14-02-002-DUP	P321; Sample Duplicate	24104/19 - 8:25 AM		
✓ ISO19-14-02-003	P121; Sample	24104/19 - 11:10 AM		
✓ ISO19-14-02-003-DUP	P121; Sample Duplicate	24104/19 - 11:10 AM		
✓ ISO19-14-02-004	3M vijver; Sample	25104/19 - 9:30 AM		
✓ ISO19-14-02-004-DUP	3M vijver; Sample Duplicate	25104/19 - 9:30 AM		
✓ ISO19-14-02-005	Blokkersdijkvijver - Noord; Sample	25104/19 - 9:30 AM		
✓ ISO19-14-02-005-DUP	Blokkersdijkvijver - Noord; Sample Dup	25104/19 - 9:30 AM		
✓ ISO19-14-02-005-FMS	Blokkersdijkvijver - Noord; FMS	25104/19 - 9:30 AM		

Sample Condition Upon Receipt:

Acceptable

All items accounted for

Stored in WRI: 3.4°C

Temperature:

10

Deg C

Received on Ice

Other:

Collected by (print): Julie Fichjet (JFI)

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
--------	------------------	------	------	-------------	--------------	------	------

12	Julie Fichjet	29/04/19	10:00	Fedex	Will Anette WA	5/3/19	11:00

3M EHS LABORATORY
Chain-of-Custody

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3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: ()
Alt. Phone:
Fax: (65)

Project: ISO19-14-02 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 3/20/2019

Project Description: 3M Antwerp Water Sampling for PFCs

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO19-14-02-006	Blokkersdijkvijver-standard; Sample	25/04/19 - 10:25 AM		
ISO19-14-02-006-DUP	Blokkersdijkvijver-standard; Sample Dup	25/04/19 - 10:25 AM		
ISO19-14-02-007	L21; Sample	24/04/19 - 10:15 AM		
ISO19-14-02-007-DUP	L21; Sample Duplicate	24/04/19 - 10:15 AM		
ISO19-14-02-008	L22; Sample	24/04/19 - 11:30 AM		
ISO19-14-02-008-DUP	L22; Sample Duplicate	24/04/19 - 11:30 AM		
ISO19-14-02-009	L31; Sample	24/04/19 - 10:48 AM		
ISO19-14-02-009-DUP	L31; Sample Duplicate	24/04/19 - 10:48 AM		
ISO19-14-02-010	L4; Sample	24/04/19 - 10:40 AM		
ISO19-14-02-010-DUP	L4; Sample Duplicate	24/04/19 - 10:40 AM		
ISO19-14-02-011	P114bis; Sample	24/04/19 - 12:05 PM		
ISO19-14-02-011-DUP	P114bis; Sample Duplicate	24/04/19 - 12:05 PM		
ISO19-14-02-011-FMS	P114bis; FMS	24/04/19 - 12:05 PM		
ISO19-14-02-012	P115; Sample	24/04/19 - 1 PM		

Sample Condition Upon Receipt: Acceptable All items accounted for

Sample in WR1: 3.4 °C

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Juli Fichjet (JF)

Collector's signature: JF

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
--------	------------------	------	------	-------------	--------------	------	------

14	Juli Fichjet	29/04/19	10:00	FedEx	Will Amets	5/3/19	11:00

3M EHS LABORATORY
Chain-of-Custody

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Fax: [REDACTED]

Project: ISO19-14-02 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 3/20/2019

Project Description: 3M Antwerp Water Sampling for PFCs

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO19-14-02-012-DUP	P115, Sample Duplicate	24104119 - 1PM		
ISO19-14-02-013	P116, Sample	24104119 - 12:56 PM		
ISO19-14-02-013-DUP	P116, Sample Duplicate	24104119 - 12:56 PM		
ISO19-14-02-014	Effluent WWTP; Sample	23104119 - 01:25 PM		
ISO19-14-02-014-DUP	Effluent WWTP; Sample Duplicate	23104119 - 01:25 PM		
ISO19-14-02-015	PP01; Sample <i>PPM</i>	24104119 - 10:30 AM		
ISO19-14-02-015-DUP	PP01; Sample Duplicate <i>PPM</i>	24104119 - 10:30 AM		
ISO19-14-02-016	PP02; Sample	23104119 - 1:35 PM		
ISO19-14-02-016-DUP	PP02; Sample Duplicate	23104119 - 1:35 PM		
ISO19-14-02-017	PP04; Sample	24104119 - 10:15 AM		
ISO19-14-02-017-DUP	PP04; Sample Duplicate	24104119 - 10:15 AM		
ISO19-14-02-018	PP05; Sample	24104119 - 10:45 AM		
ISO19-14-02-018-DUP	PP05; Sample Duplicate	24104119 - 10:45 AM		
ISO19-14-02-018-FMS	PP05; FMS	24104119 - 10:45 AM		

Sample Condition Upon Receipt: Acceptable All items accounted for

Sample in WR1: 3.40

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): *Julie Fichfut (JFI)*

Collector's signature: *[Signature]*

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
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14	<i>Julie Fichfut</i>	29104119	10:00	FedEx	<i>Will Amett</i>	5/3/17	11:00

3M EHS LABORATORY
Chain-of-Custody

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St. Paul, MN 55144

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Fax: (65)

Project: ISO19-14-02 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10

Project Number:

Date Created: 3/20/2019

Project Description: 3M Antwerp Water Sampling for PFCs

Completion Date:

Project Lead: Susan T. Wolf

Phone Number

Email Address

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
✓ ISO19-14-02-019	PP06, Sample	23104119 - 02:40 PM		
✓ ISO19-14-02-019-DUP	PP06, Sample Duplicate	23104119 - 02:40 PM		
✓ ISO19-14-02-020	PP07, Sample	23104119 - 01:49 PM		
✓ ISO19-14-02-020-DUP	PP07, Sample Duplicate	23104119 - 01:49 PM		
✓ ISO19-14-02-021	PP08, Sample	24104119 - 11:20 AM		
✓ ISO19-14-02-021-DUP	PP08, Sample Duplicate	24104119 - 11:20 AM		
✓ ISO19-14-02-021-FMS	PP08, FMS	24104119 - 11:20 AM		
✓ ISO19-14-02-022	PP09, Sample	23104119 - 02:40 PM		
✓ ISO19-14-02-022-DUP	PP09, Sample Duplicate	23104119 - 02:40 PM		
✓ ISO19-14-02-023	PP10, Sample	23104119 - 01:30 PM		
✓ ISO19-14-02-023-DUP	PP10, Sample Duplicate	23104119 - 01:30 PM		
✓ ISO19-14-02-024	12; Sample	25104119 - 11:00 AM		
✓ ISO19-14-02-024-DUP	12; Sample Duplicate	25104119 - 11:00 AM		
✓ ISO19-14-02-025	13; Sample	25104119 - 11:05 AM		

Sample Condition Upon Receipt: Acceptable All items accounted for

Stored in wRI: 3.4 °C

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Julie Fichfeld (JFI)

Collector's signature: 

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
--------	------------------	------	------	-------------	--------------	------	------

14	Julie Fichfeld	29104119	10:00	FedEx	Will Amett WT	5/3/19	11:00

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
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Project: ISO19-14-02 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)

Department: 832202 Site Source: 01WJWT10

Project Number:

Date Created: 3/20/2019

Project Description: 3M Antwerp Water Sampling for PFCs

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
✓ ISO19-14-02-025-DUP	13; Sample Duplicate	25104119 - M: 05 AM		
✓ ISO19-14-02-026	5; Sample	25104119 - M: 30 AM		
✓ ISO19-14-02-026-DUP	5; Sample Duplicate	25104119 - M: 30 AM		
✓ ISO19-14-02-026-FMS	5; FMS	25104119 - M: 30 AM		
✓ ISO19-14-02-027	Bemalingsstation; Sample	25104119 - M: 20 AM		
✓ ISO19-14-02-027-DUP	Bemalingsstation; Sample Dup	25104119 - M: 20 AM		
✓ ISO19-14-02-028	Collector put; Sample	23104119 - 02: 45 PM		
✓ ISO19-14-02-028-DUP	Collector put; Sample Duplicate	23104119 - 02: 45 PM		
✓ ISO19-14-02-029	P21B; Sample	24104119 - 01: 55 PM		
✓ ISO19-14-02-029-DUP	P21B; Sample Duplicate	24104119 - 01: 55 PM		
✓ ISO19-14-02-029-FMS	P21B; FMS	24104119 - 01: 55 PM		
✓ ISO19-14-02-030	Travel Blank	3/25/19 1:45pm CJG	*	
✓ ISO19-14-02-030-FMS-LOW	Travel Blank FMS Low	3/25/19 1:45pm CJG	*	
✓ ISO19-14-02-030-FMS-HIGH	Travel Blank FMS High	3/25/19 1:45pm CJG	*	

* Travel Blank samples prepared by the 3M EHS Laboratory with the bottle order.

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Stored in wr1: 3.4°C

Collected by (print): Julie Fidcht (JFI)

Collector's signature: JF

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
--------	------------------	------	------	-------------	--------------	------	------

14	Julie Fidcht	29104119	10:00	Fidcht	Will Anette WAA	5/3/19	11:00



3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC19-03560-001

3M Lab Request Number: E19-0339

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: September 5th, 2019

Requester

Jim Kotsmith
3M Center, Bldg 224-5w-17
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Member of the SGS Group (Société Générale de Surveillance)

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All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction - Summary.

At the request (Lab Request Number: E19-0339) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected in July and August 2019 from 3 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOA ($C_6F_{13}^{13}CF_2^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOS ($C_7F_{15}^{13}CF_2SO_3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Sven Herremans. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles.

Table 1 summarizes the sample results. All PFBS, PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
Effluent WWTP		<0.152	<0.252	<0.185	<0.256
Bemalingsstation		60.8	56.3	52.9	<0.256
Collector Put		34.1	82.3	185	9.10

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
Effluent WWTP	100
Bemalingsstation	100
Collector Put	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
Effluent WWTP	1.0	0.05	Solution A	1.0	-
Bemalingsstation	1.0	0.05	Solution A	1.0	-
Collector Put	1.0	0.05	Solution A	1.0	-

(*): MeOH:LCMS-water (60:40)
Solution A = 1000 ng/mL (nominal) ^{13}C -PFC Surrogate Standards

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ^{13}C -PFOA, PFOS, FOSA and ^{13}C -PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetre C18 50 x 2.1mm, 3.5 μm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7 μm
Injection volume	5 μL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate ($\mu\text{L}/\text{min}$)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
PFBS	2.08 - 2.50	298.93/79.82	40	28	45
		298.93/98.78	40	29	45
PFHS	2.45 - 3.05	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.80 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
¹³ C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of $\pm 25\%$ ($\pm 30\%$ for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.0740 to 0.125 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.152 to 0.256 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within $\pm 25\%$, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by diluting 1 mL water with 10.0 mL MeOH:LCMS-water (60:40). Method blanks were prepared to evaluate the levels of background contamination in the overall dilution process (glassware, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carryover.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of LCMS-water, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCS)

One lab control spike was prepared and analyzed.

LCS was prepared by spiking known amounts of the analytes into 1.0 mL of LCMS-water to produce the desired concentration.

The spiked water sample was diluted with 10.0 mL MeOH:LCMS-water (60:40) and analyzed in the same manner as the samples.

The LCS produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 7 summarizes the LCS recovery results

Table 7. Lab Control Spike Results.

Dilution date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
7 August 2019	LCS1	80		99.0	78.7	107	108	112	108

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing PFBS, PFHS, PFOA, ^{13}C -PFOA, PFOS, FOSA and ^{13}C -PFOS (QC Lab Low and QC Lab High).

The QCs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 8 summarizes the QCs recovery results.

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
25 Jan 2018	QC Lab Low inj1	10		99.1	90.1	124	108	90.4	111
	QC Lab High inj1	100		77.1	90.2	92.9	94.7	108	107
	QC Lab Low inj2	10		99.4	88.8	87.1	109	91.0	114
	QC Lab High inj2	100		76.3	88.9	90.7	94.0	114	109

3.7. Equations.

Recovery.

$$\text{Recovery (\%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

The following tables summarize the results for the sample locations.

Table 9. Sample Results PFBS, PFHS, PFOA, PFOS and FOSA

	PFBS	PFHS	PFOA	PFOS	FOSA
	ng/ml	ng/ml	ng/ml	ng/ml	ng/ml
Effluent WWTP		<0.152	<0.252	<0.185	<0.256
Bemalingsstation		60.8	56.3	52.9	<0.256
Collector Put		34.1	82.3	185	9.10
Field Trip Blank		<0.821	<1.37	<1.00	<1.39

Table 10. Sample Results ^{13}C -PFOA and ^{13}C -PFOS

	^{13}C -PFOA		^{13}C -PFOS	
	ng/ml	% Rec	ng/ml	% Rec
Effluent WWTP	66.8	114	54.3	86.5
Bemalingsstation	63.9	109	56.8	90.5
Collector Put	69.8	119	51.9	82.7
Field Trip Blank	103	87.7	141	113

5. Conclusion.

- The recovery of ¹³C-PFOA and ¹³C-PFOS fell within the method acceptance criterion of 70-130%.
- Lab control spike recoveries fell within the method acceptance criteria for all compounds, indicating that the analysis method is in control,
- QC Lab Standards recoveries fell within the method acceptance criteria for all compounds.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Erik Broeckx (ERM Belgium).

8. Signatures.

Sven Herremans,
Technical Manager

Date September 5th, 2019



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date September 5th, 2019

I.A.C.
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Reports are established on behalf of and for the account of the principal, who expressly accepts that these reports purely represent the situation at a given time and that they must always be presented and/or mentioned in their totality and in their particular context. SGS Belgium N.V., issuer of the reports, cannot be held liable for errors or modification of results during electronic or fax transmission. Only the originally signed report is binding.

The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.



3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC19-03560_002

3M Lab Request Number: E19-0339

Analysis of Total Petroleum Hydrocarbons in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: September 5th, 2019

Requester

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All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction.

At the request (Lab Request Number: E19-0339) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected between July and August, 2018 from 2 locations and analyzed for:

- Total Petroleum Hydrocarbons (TPH)

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

The sample was collected in a 1L glass container containing HCl as a preservative. The bottle was sent by SGS Belgium NV, Division IAC Laboratory.

2.2. Analysis of mineral oil.

The samples was analyzed according to WAC/IV/B/025, the official Dutch method.

3. Results.

Table 1 summarizes the sample results.

Table 1. Mineral oil results.

Sample identification	Concentration (in µg/L)				
	Fraction C-10 -C-12	Fraction C-12 -C-20	Fraction C-20 -C-30	Fraction C-30 -C-40	Mineral oil
P18	170	1500	1200	120	3000
P28	110	1000	710	51	1900

4. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

5. Signatures.

Sven Herremans,
Technical Manager

Date September 5th, 2019



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date September 5th, 2019

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The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.

Final Report

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

July 2019 Sampling

Laboratory Request Number: ISO19-14-03

Report Date – Date of Last Signature

Testing Laboratory

3M Environment, Health, Safety & Medical
3M EHS Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Charlotte Tack
3M Belgium; EHS Project Supervisor
3M Belgium; ZW019/0/01
[REDACTED]



The testing reported herein meet the requirements of ISO/IEC 17025:2017 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M EHS Laboratory

3M EHS Laboratory Manager: Brian T. Mader
3M Principal Analytical Investigator: Susan Wolf
Report Author: Chelsie Grochow

Analytical Report ISO19-14-03

Water Sample Analysis at 3M Antwerp, Belgium
July 2019 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M EHS Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. All samples were collected July 30 – August 1, 2019 and returned to the 3M EHS Laboratory on August 6, 2019, at ambient temperature. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS) (select locations only), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO19-14-03.

The 3M EHS Laboratory prepared sample containers for seventy-four sampling locations. Each sample set consisted of a field sample and field sample duplicate. Fourteen locations also included a target analyte field matrix spike. Each empty container was marked with a “fill to here” line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with a mass-labeled internal standard mix and select sample bottles were fortified with surrogate recovery standards [$^{13}\text{C}_4$]-PFOA and [$^{13}\text{C}_4$]-PFOS prior to being sent to the field for sample collection. During sample collection, sample locations D09 and PP09 were not sampled.

Samples were prepared and analyzed using method ETS-8-044.3 “Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis”. Select internal standards were used to aid in the data quality objectives.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ISO/IEC 17025:2017 “General Requirements for the Competence of Testing and Calibration Laboratories”, in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary ⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: 2nd Aquifer					
ISO19-14-03-001	BD24-3; Sample	25.3	27.8	856	4.58
ISO19-14-03-001-DUP	BD24-3; Sample Dup	23.5	25.4	828	4.08
		Average	24.4	26.6	842
		%RPD Sample/Sample Dup	7.4	9.0	3.3
ISO19-14-03-002	BD24-4; Sample	83.2	47.2	0.768	0.526
ISO19-14-03-002-DUP	BD24-4; Sample Dup	81.2	43.4	0.626	0.0930
		Average	82.2 ⁽²⁾	45.3 ⁽²⁾	0.697 ⁽²⁾
		%RPD Sample/Sample Dup	2.4	8.4	20
ISO19-14-03-004	D10; Sample	663	277	52.4	<0.100
ISO19-14-03-004-DUP	D10; Sample Dup	654	287	54.2	<0.100
		Average	659	282	53.3
		%RPD Sample/Sample Dup	1.4	3.5	3.4
ISO19-14-03-005	D11; Sample	309	152	1270	16.7
ISO19-14-03-005-DUP	D11; Sample Dup	1060	980	2030	63.4
		Average	685	566	1650
		%RPD Sample/Sample Dup	110 ⁽³⁾	146 ⁽³⁾	46 ⁽³⁾
ISO19-14-03-006	D14; Sample	2.44	1.43	0.810	0.105
ISO19-14-03-006-DUP	D14; Sample Dup	2.30	1.45	0.800	0.0832
		Average	2.37 ⁽²⁾	1.44 ⁽²⁾	0.805 ⁽²⁾
		%RPD Sample/Sample Dup	5.9	1.4	1.2
ISO19-14-03-007	D16; Sample	728	334	2630	7.36
ISO19-14-03-007-DUP	D16; Sample Dup	731	342	2650	7.68
		Average	730	338	2640
		%RPD Sample/Sample Dup	0.41	2.4	0.76
ISO19-14-03-008	D17; Sample	667	155	307	0.256
ISO19-14-03-008-DUP	D17; Sample Dup	612	149	313	0.282
		Average	640	152	310
		%RPD Sample/Sample Dup	8.6	3.9	1.9
ISO19-14-03-009	D18; Sample	179	155	64.6	2.06
ISO19-14-03-009-DUP	D18; Sample Dup	186	156	84.1	1.90
		Average	183	156	74.4
		%RPD Sample/Sample Dup	3.8	0.64	26 ⁽³⁾
ISO19-14-03-010	D2; Sample	<0.0240	<0.0250	0.184	0.0846
ISO19-14-03-010-DUP	D2; Sample Dup	<0.0240	<0.0250	0.210	0.0920
		Average	<0.0240 ⁽²⁾	<0.0250 ⁽²⁾	0.197 ⁽²⁾
		%RPD Sample/Sample Dup	NA	NA	13
					0.0883
					8.4

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 24%, PFBS \pm 15%, PFHS \pm 13%, and PFOS \pm 19%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 22%, PFBS \pm 12%, PFHS \pm 14%, PFOS \pm 13%, and PFOSA \pm 30%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) The analytical data uncertainty has been further expanded for location P379 for PFOA to \pm 48% based on field matrix spike recovery.

Table 1 continued. Sample Results Summary⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: 2nd Aquifer					
ISO19-14-03-011	D5; Sample	409	168	449	12.5
ISO19-14-03-011-DUP	D5; Sample Dup	408	164	443	11.1 ⁽³⁾
		Average	409	166	446
		%RPD Sample/Sample Dup	0.24	2.4	1.3
ISO19-14-03-012	ND7; Sample	181	43.4	100	10.2
ISO19-14-03-012-DUP	ND7; Sample Dup	182	43.1	105	9.92
		Average	182	43.3	103
		%RPD Sample/Sample Dup	0.55	0.69	4.9
		Average	1350	2180	61.2
		%RPD Sample/Sample Dup	5.2	3.7	4.7
ISO19-14-03-014	P118A; Sample	1600	748	6980	4.64
ISO19-14-03-014-DUP	P118A; Sample Dup	1640	763	7630	4.70
		Average	1620	756	7310
		%RPD Sample/Sample Dup	2.5	2.0	8.9
		Average	12.6⁽²⁾	4.71⁽²⁾	1.16⁽²⁾
		%RPD Sample/Sample Dup	4.8	1.3	6.9
ISO19-14-03-015	P119A; Sample	2600	1470	381	0.476
ISO19-14-03-015-DUP	P119A; Sample Dup	2600	1450	386	0.442
		Average	2600	1460	384
		%RPD Sample/Sample Dup	0.0	1.4	1.3
		Average	0.840	1.08	2.34
		%RPD Sample/Sample Dup	0.866	<1.00	2.14
		Average	0.853	1.08	2.24
		%RPD Sample/Sample Dup	3.0	NA	0.330
		Average	2220	597	4670
		%RPD Sample/Sample Dup	2220	602	4720
		Average	2220	600	4700
		%RPD Sample/Sample Dup	0.0	0.83	0.426
		Average	0.0	1.1	0.94

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 24%, PFBS \pm 15%, PFHS \pm 13%, and PFOS \pm 19%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 22%, PFBS \pm 12%, PFHS \pm 14%, PFOS \pm 13%, and PFOSA \pm 30%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) The analytical data uncertainty has been further expanded for location P379 for PFOA to \pm 48% based on field matrix spike recovery.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Blokkersdijk Nature Reserve					
ISO19-14-03-018	3M vijver; Sample	1.67	0.912	14.2	0.204
ISO19-14-03-018-DUP	3M vijver; Sample Dup	1.62	0.922	13.9	0.168
		Average	1.65⁽²⁾	0.917⁽²⁾	14.1⁽²⁾
		%RPD Sample/Sample Dup	3.0	1.1	2.1
ISO19-14-03-019	Blokkersdijkvijver Noord; Sample	1.26	0.754	1.66	0.0910
ISO19-14-03-019-DUP	Blokkersdijkvijver Noord; Sample Dup	1.27	0.812	1.71	0.0896
		Average	1.27⁽²⁾	0.783⁽²⁾	1.69⁽²⁾
		%RPD Sample/Sample Dup	0.79	7.4	3.0
ISO19-14-03-020	Blokkersdijkvijver standard; Sample	1.13	0.776	1.10	0.112
ISO19-14-03-020-DUP	Blokkersdijkvijver standard; Sample Dup	1.16	0.770	1.06	0.134
		Average	1.15⁽²⁾	0.773⁽²⁾	1.08⁽²⁾
		%RPD Sample/Sample Dup	2.6	0.78	3.7
ISO19-14-03-022	L21; Sample	0.170	0.136	4.24	0.0616
ISO19-14-03-022-DUP	L21; Sample Dup	0.170	0.139	4.62	0.0608
		Average	0.170⁽²⁾	0.138⁽²⁾	4.43⁽²⁾
		%RPD Sample/Sample Dup	0.0	2.2	8.6
ISO19-14-03-023	L22; Sample	0.434	0.348	3.00	0.104
ISO19-14-03-023-DUP	L22; Sample Dup	0.444	0.368	3.24	0.110
		Average	0.439⁽²⁾	0.358⁽²⁾	3.12⁽²⁾
		%RPD Sample/Sample Dup	2.3	5.6	7.7
ISO19-14-03-024	L31; Sample	0.418	0.774	7.68	0.148
ISO19-14-03-024-DUP	L31; Sample Dup	0.406	0.868	7.50	0.149
		Average	0.412⁽²⁾	0.821⁽²⁾	7.59⁽²⁾
		%RPD Sample/Sample Dup	2.9	11	2.4
ISO19-14-03-025	L4; Sample	3.68	2.82	29.0	0.468
ISO19-14-03-025-DUP	L4; Sample Dup	4.08	3.08	30.0	0.442
		Average	3.88⁽²⁾	2.95⁽²⁾	29.5⁽²⁾
		%RPD Sample/Sample Dup	10	8.8	3.4
ISO19-14-03-026	P114bis; Sample	4.08	1.79	5.52	0.0920
ISO19-14-03-026-DUP	P114bis; Sample Dup	3.68	1.58	5.16	0.0722
		Average	3.88⁽²⁾	1.69⁽²⁾	5.34⁽²⁾
		%RPD Sample/Sample Dup	10	12	6.7
ISO19-14-03-027	P115; Sample	4.16	2.40	3.20	0.0636
ISO19-14-03-027-DUP	P115; Sample Dup	4.16	2.32	2.84	<0.0500
		Average	4.16⁽²⁾	2.36⁽²⁾	3.02⁽²⁾
		%RPD Sample/Sample Dup	0.0	3.4	12
					NA

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 24%, PFBS ± 15%, PFHS ± 13%, and PFOS ± 19%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 22%, PFBS ± 12%, PFHS ± 14%, PFOS ± 13%, and PFOSA ± 30%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
- (4) The analytical data uncertainty has been further expanded for location P379 for PFOA to ±48% based on field matrix spike recovery.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Blokkersdijk Nature Reserve					
ISO19-14-03-028	P116; Sample	1.05	0.636	8.46	0.134
ISO19-14-03-028-DUP	P116; Sample Dup	1.08	0.632	8.26	0.107
Average %RPD Sample/Sample Dup		1.07 ⁽²⁾ 2.8	0.634 ⁽²⁾ 0.63	8.36 ⁽²⁾ 2.4	0.121 22 ⁽³⁾
Zone: Effluent WWTP					
ISO19-14-03-029	Effluent WWTP; Sample	<0.0240	<0.0250	<0.0464	<0.0500
ISO19-14-03-029-DUP	Effluent WWTP; Sample Dup	<0.0240	<0.0250	0.0520	<0.0500
Average %RPD Sample/Sample Dup		<0.0240 ⁽²⁾ NA	<0.0250 ⁽²⁾ NA	0.0520 ⁽²⁾ NA	<0.0500 NA

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
ISO19-14-03-030	PP02; Sample	663	45.2	3870	18300	23.4
ISO19-14-03-030-DUP	PP02; Sample Dup	633	45.2	3670	16300	21.2
Average %RPD Sample/Sample Dup		648 4.6	45.2 0.0	3770 5.3	17300 12	22.3 9.9
ISO19-14-03-031	PP04; Sample	230	383	108	3960	77.4
ISO19-14-03-031-DUP	PP04; Sample Dup	238	382	106	3940	69.8
Average %RPD Sample/Sample Dup		234 3.4	383 0.26	107 1.9	3950 0.51	73.6 10
ISO19-14-03-032	PP05; Sample	2430	15800	8710	90.8	18.4
ISO19-14-03-032-DUP	PP05; Sample Dup	2430	15900	9310	84.5	19.4
Average %RPD Sample/Sample Dup		2430 0.0	15900 0.63	9010 6.7	87.7 7.2	18.9 5.3
ISO19-14-03-033	PP06; Sample	189	22.8	88.9	947	52.0
ISO19-14-03-033-DUP	PP06; Sample Dup	193	28.3	89.7	966	53.4
Average %RPD Sample/Sample Dup		191 2.1	25.6 22 ⁽³⁾	89.3 0.90	957 2.0	52.7 2.7
ISO19-14-03-034	PP07; Sample	224	648	98.7	1810	58.4
ISO19-14-03-034-DUP	PP07; Sample Dup	223	664	95.9	1860	59.8
Average %RPD Sample/Sample Dup		224 0.45	656 2.4	97.3 2.9	1840 2.7	59.1 2.4
ISO19-14-03-035	PP08; Sample	520	30.7	272	4660	67.2
ISO19-14-03-035-DUP	PP08; Sample Dup	545	29.9	278	4980	60.8
Average %RPD Sample/Sample Dup		533 4.7	30.3 2.6	275 2.2	4820 6.6	64.0 10

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 24%, PFBS \pm 15%, PFHS \pm 13%, and PFOS \pm 19%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 22%, PFBS \pm 12%, PFHS \pm 14%, PFOS \pm 13%, and PFOSA \pm 30%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) The analytical data uncertainty has been further expanded for location P379 for PFOA to \pm 48% based on field matrix spike recovery.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
ISO19-14-03-037	PP10; Sample	476	16.0	197	3470	23.4
ISO19-14-03-037-DUP	PP10; Sample Dup	455	13.3	192	3350	22.8
Average		466	14.7	195	3410	23.1
%RPD Sample/Sample Dup		4.5	18	2.6	3.5	2.6
ISO19-14-03-063	PP11; Sample	325	174	143	2520	26.2
ISO19-14-03-063-DUP	PP11; Sample Dup	322	169	139	2410	27.8
Average		324	172	141	2470	27.0
%RPD Sample/Sample Dup		0.93	2.9	2.8	4.5	5.9

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Palingbeek & Tophatgracht					
ISO19-14-03-038	12; Sample	56.1	17.8	224	0.372
ISO19-14-03-038-DUP	12; Sample Dup	57.2	17.5	223	0.346
Average		56.7	17.7	224	0.359
%RPD Sample/Sample Dup		1.9	1.7	0.45	7.2
ISO19-14-03-039	13; Sample	61.0	23.0	220	0.474
ISO19-14-03-039-DUP	13; Sample Dup	60.0	23.8	232	0.418
Average		60.5 ⁽²⁾	23.4 ⁽²⁾	226	0.446
%RPD Sample/Sample Dup		1.7	3.4	5.3	13
ISO19-14-03-040	5; Sample	65.6	86.0	110	0.324
ISO19-14-03-040-DUP	5; Sample Dup	67.4	84.4	109	0.318
Average		66.5 ⁽²⁾	85.2 ⁽²⁾	110 ⁽²⁾	0.321
%RPD Sample/Sample Dup		2.7	1.9	0.91	1.9
ISO19-14-03-041	Bemalingsstation; Sample	64.4	78.4	90.8	0.230
ISO19-14-03-041-DUP	Bemalingsstation; Sample Dup	57.2	69.2	84.2	0.214
Average		60.8 ⁽²⁾	73.8 ⁽²⁾	87.5 ⁽²⁾	0.222
%RPD Sample/Sample Dup		12	12	7.5	7.2
Zone: Sewer					
ISO19-14-03-042	Collector put; Sample	66.0	29.6	229	37.8
ISO19-14-03-042-DUP	Collector put; Sample Dup	68.2	29.0	231	38.4
Average		67.1 ⁽²⁾	29.3 ⁽²⁾	230	38.1
%RPD Sample/Sample Dup		3.3	2.0	0.87	1.6

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 24%, PFBS \pm 15%, PFHS \pm 13%, and PFOS \pm 19%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 22%, PFBS \pm 12%, PFHS \pm 14%, PFOS \pm 13%, and PFOSA \pm 30%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) The analytical data uncertainty has been further expanded for location P379 for PFOA to \pm 48% based on field matrix spike recovery.

Table 1 continued. Sample Results Summary ⁽¹⁾

		Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - Building 16						
ISO19-14-03-043	K3; Sample	84.3	285	14500	6580	12.4
ISO19-14-03-043-DUP	K3; Sample Dup	77.8	290	15200	6830	13.5
		Average	81.1	288	14900	6710
		%RPD Sample/Sample Dup	8.0	1.7	4.7	3.7
ISO19-14-03-044	P27; Sample	183	4460	35.7	1660	76.4
ISO19-14-03-044-DUP	P27; Sample Dup	194	4660	34.3	1700	73.4
		Average	189	4560	35.0	1680
		%RPD Sample/Sample Dup	5.8	4.4	4.0	2.4
ISO19-14-03-045	P21B; Sample	9290	8030	13300	71700	15.1
ISO19-14-03-045-DUP	P21B; Sample Dup	9090	7830	13000	71800	13.6
		Average	9190	7930	13200	71800
		%RPD Sample/Sample Dup	2.2	2.5	2.3	0.14
ISO19-14-03-046	P304; Sample	453	298	149	1160	11.1
ISO19-14-03-046-DUP	P304; Sample Dup	481	298	150	1210	10.8
		Average	467	298	150	1190
		%RPD Sample/Sample Dup	6.0	0.0	0.67	4.2
ISO19-14-03-047	P305; Sample	166	229	234	454	9.40
ISO19-14-03-047-DUP	P305; Sample Dup	169	231	241	463	10.2
		Average	168	230	238	459
		%RPD Sample/Sample Dup	1.8	0.87	2.9	2.0
ISO19-14-03-048	P42; Sample	229	47.3	372	2280	12.0
ISO19-14-03-048-DUP	P42; Sample Dup	232	41.0	357	2410	11.1
		Average	231	44.2	365	2350
		%RPD Sample/Sample Dup	1.3	14	4.1	5.5
						11.6
						7.8

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - WWTP					
ISO19-14-03-049	L19; Sample	245	166	3300	48.4
ISO19-14-03-049-DUP	L19; Sample Dup	238	152	3330	45.8
		Average	242	159	3320
		%RPD Sample/Sample Dup	2.9	8.8	47.1
ISO19-14-03-050	M4; Sample	138	60.7	1150	112
ISO19-14-03-050-DUP	M4; Sample Dup	130	51.7	1170	114
		Average	134	56.2	1160
		%RPD Sample/Sample Dup	6.0	16	113
					1.8

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 24%, PFBS \pm 15%, PFHS \pm 13%, and PFOS \pm 19%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 22%, PFBS \pm 12%, PFHS \pm 14%, PFOS \pm 13%, and PFOSA \pm 30%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) The analytical data uncertainty has been further expanded for location P379 for PFOA to \pm 48% based on field matrix spike recovery.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - WWTP					
ISO19-14-03-051	P118C; Sample	166	84.7	1700	78.6
ISO19-14-03-051-DUP	P118C; Sample Dup	166	83.2	1670	67.6
		Average	166	84.0	1690
		%RPD Sample/Sample Dup	0.0	1.8	1.8
ISO19-14-03-052	P119C; Sample	1340	490	7980	92.2
ISO19-14-03-052-DUP	P119C; Sample Dup	1340	486	8030	98.8
		Average	1340	488	8010
		%RPD Sample/Sample Dup	0.0	0.82	0.62
ISO19-14-03-053	P263; Sample	964	344	7380	12.6
ISO19-14-03-053-DUP	P263; Sample Dup	889	339	7180	14.1
		Average	927	342	7280
		%RPD Sample/Sample Dup	8.1	1.5	2.7
ISO19-14-03-054	P264; Sample	384	264	1320	59.2
ISO19-14-03-054-DUP	P264; Sample Dup	399	274	1380	53.4
		Average	392	269	1350
		%RPD Sample/Sample Dup	3.8	3.7	4.4
ISO19-14-03-055	P340; Sample	390	228	2530	47.0
ISO19-14-03-055-DUP	P340; Sample Dup	406	237	2590	59.8
		Average	398	233	2560
		%RPD Sample/Sample Dup	4.0	3.9	2.3
					24 ⁽³⁾
ISO19-14-03-056	P341; Sample	691	217	5500	55.0
ISO19-14-03-056-DUP	P341; Sample Dup	713	215	5350	55.0
		Average	702	216	5430
		%RPD Sample/Sample Dup	3.1	0.93	2.8
					0.0
ISO19-14-03-057	P374; Sample	220	291	1240	41.4
ISO19-14-03-057-DUP	P374; Sample Dup	230	298	1280	43.4
		Average	225	295	1260
		%RPD Sample/Sample Dup	4.4	2.4	3.2
					4.7
ISO19-14-03-058	P379; Sample	68.4	20.5	749	58.2
ISO19-14-03-058-DUP	P379; Sample Dup	67.6	19.1	749	60.6
		Average	68.0 ⁽⁴⁾	19.8	749
		%RPD Sample/Sample Dup	1.2	7.1	0.0
					4.0
ISO19-14-03-059	P380; Sample	555	562	1930	35.2
ISO19-14-03-059-DUP	P380; Sample Dup	551	570	1920	38.8
		Average	553	566	1930
		%RPD Sample/Sample Dup	0.72	1.4	0.52
					9.7

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 24%, PFBS \pm 15%, PFHS \pm 13%, and PFOS \pm 19%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 22%, PFBS \pm 12%, PFHS \pm 14%, PFOS \pm 13%, and PFOSA \pm 30%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) The analytical data uncertainty has been further expanded for location P379 for PFOA to \pm 48% based on field matrix spike recovery.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - WWTP					
ISO19-14-03-060	P381; Sample	521	166	1120	11.9
ISO19-14-03-060-DUP	P381; Sample Dup	495	162	1100	11.2
		Average	508	164	1110
		%RPD Sample/Sample Dup	5.1	2.4	1.8
ISO19-14-03-061	P382; Sample	622	208	917	111
ISO19-14-03-061-DUP	P382; Sample Dup	703	234	960	90.6
		Average	663	221	939
		%RPD Sample/Sample Dup	12	12	4.6
ISO19-14-03-064	P262bis; Sample	523	322	5400	8.80
ISO19-14-03-064-DUP	P262bis; Sample Dup	541	327	5530	9.10
		Average	532	325	5470
		%RPD Sample/Sample Dup	3.4	1.5	2.4
ISO19-14-03-065	P265B; Sample	108	43.6	453	149
ISO19-14-03-065-DUP	P265B; Sample Dup	112	42.5	455	127
		Average	110	43.1	454
		%RPD Sample/Sample Dup	3.6	2.6	0.44
ISO19-14-03-066	P371; Sample	609	399	3400	19.2
ISO19-14-03-066-DUP	P371; Sample Dup	619	394	3460	17.5
		Average	614	397	3430
		%RPD Sample/Sample Dup	1.6	1.3	1.7
Zone: Southern Site Boundary					
ISO19-14-03-062	P372; Sample	85.6	77.3	1070	41.4
ISO19-14-03-062-DUP	P372; Sample Dup	84.7	73.7	1080	38.8
		Average	85.2	75.5	1080
		%RPD Sample/Sample Dup	1.1	4.8	0.93

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
ISO19-14-03-067	Travel Blank	<0.0240 ⁽²⁾	<0.0250 ⁽²⁾	<0.0250 ⁽²⁾	<0.0464 ⁽²⁾	<0.0500

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 24%, PFBS \pm 15%, PFHS \pm 13%, and PFOS \pm 19%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 22%, PFBS \pm 12%, PFHS \pm 14%, PFOS \pm 13%, and PFOSA \pm 30%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) The analytical data uncertainty has been further expanded for location P379 for PFOA to \pm 48% based on field matrix spike recovery.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M EHS Laboratory method ETS-8-044.3 “Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis”.

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluoroctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluoroctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected on July 30 – August 1, 2019, in Nalgene™ (high-density polyethylene) bottles prepared at the 3M EHS Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. All sample locations were spiked with a mass-labeled internal standard mix at a nominal concentration of 1 ng/mL and select samples were fortified with surrogate recovery standards (SRSs) at a nominal concentration of 0.1 ng/mL prior to sample collection. Samples were received on August 6, 2019.

2.3 Sample Preparation

Sample locations pre-spiked with internal standard and/or surrogates were analyzed for all analytes (PFBS for select locations) by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples along with the method blanks.

Samples requiring dilutions were prepared for PFOA, PFHS, PFOS and PFBS (select locations only) using a direct injection by methanol dilution method. Samples that required a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples that required a 1:100 dilution were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol. Samples that required a 1:500 dilution were prepared by diluting 0.02 mL of a well-mixed sample with 10.0 mL of methanol. Samples that required a 1:1000 dilution were prepared by first diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol, followed by a second dilution prepared by diluting 1 mL of the diluted sample into 9 mL of methanol. Diluted samples and LCSs were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

2.4 Analysis

All samples and quality control samples were analyzed for the target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). SRSs were added and included in the analysis of these samples. Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.2. The consistency of the laboratory's integration is ensured through the training of laboratory personnel,

the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

The following analytical runs were used to report the results herein:

8/21/19 (ETS Kirk) Internal Standard Calibration Analysis:

- Sample locations pre-spiked with surrogates were analyzed for PFOA, PFHS, PFOS, and PFOSA with select locations being analyzed for PFBS. All sample results were reported **except** for the following sample locations: 12 for PFOA, PFHS, and PFOS, 13 for PFOS, and Collector put for PFOS and PFOSA.

8/27/19 (ETS Kirk) External Standard Calibration Analysis:

- All sample locations were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported **except** for the following sample locations: PP02 and PP05 for PFBS and P21B for PFOS.

8/29/19 (ETS Kirk) External Standard Calibration Analysis:

- All sample locations were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported **except** for the following sample locations: D11, 5, and Bemalingstation for all analytes, 13 and Collector Put for PFOA and PFHS, P119C for PFOS, P379 for PFOA, PP02 and PP05 for PFOA, PFHS, and PFOS, and P21B for PFOA, PFBS, and PFHS.

9/4/19 (ETS Kirk) External Standard Calibration Analysis:

- All sample locations were analyzed for PFOA and PFOS. All sample results were reported **except** for the following sample locations: P119C for PFOA and P379 for PFOS.

9/7/19 (ETS Beethoven) Internal Standard Calibration Analysis:

- All sample locations were analyzed for PFOSA. All sample results were reported except for the following sample locations: BD24-4, D10, D17, P119B, P121, and P321.

9/9/19 (ETS Kirk) Internal Standard Calibration Analysis:

- All sample locations were analyzed for PFOSA. All sample results were reported except for the following sample locations: BD24-4 and D11.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk	ETS Beethoven
Liquid Chromatograph	Agilent 1260	Agilent 1260
Analysis Method	ETS-8-044.3	ETS-8-044.3
Analysis Date	8/21/19, 8/27/19, 8/29/19, 9/4/19, 9/9/19	9/7/19
Guard column	Prism RP (2.1 mm X 50 mm), 5 μ	Prism RP (2.1 mm X 50 mm), 5 μ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ
Injection Volume	2 or 5 μ L	60 μ L
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 4500
Ion Source	Turbo Spray	Turbo Spray
Polarity	Negative	Negative
Software	Analyst 1.6.3	Analyst 1.6.3

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis				
Step Number	Total Time (min)	Flow Rate ($\mu\text{L}/\text{min}$)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0.00	300	90.0	10.0
1	0.50	300	90.0	10.0
2	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard	Mass Transition Q1/Q3
PFOA	413/369	$[^{13}\text{C}_8]\text{-PFOA}$	421/376
	413/219		
	413/169		
PFBS	299/99	$[^{18}\text{O}_2]\text{-PFBS}$	303/84
	299/80		
PFHS	399/99	$[^{13}\text{C}_3]\text{-PFHS}$	402/80
	399/80		
PFOS	499/99	$[^{13}\text{C}_8]\text{-PFOS}$	507/80
	499/80		
	499/130		
PFOA	498/78	$[^{13}\text{C}_8]\text{-PFOA}$	506/78
$[^{13}\text{C}_4]\text{-PFOA}$	417/372	$[^{13}\text{C}_8]\text{-PFOA}$	421/376
$[^{13}\text{C}_4]\text{-PFOS}$	503/80	$[^{13}\text{C}_8]\text{-PFOS}$	507/80

The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

3 Data Analysis

3.1 Calibration

8/21/19, 9/7/19, and 9/9/19 Analysis (Internal Standard Calibration): Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of fourteen calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed. The calibration standards also contained surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

8/27/19, 8/29/19, and 9/4/19 Analysis (External Standard Calibration): Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards

ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL to 25 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±25% (100±30% for the lowest curve point) were met for all analytes. The correlation coefficient (*r*) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.3 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the **Table 6** below.

Table 6. LOQ

Analyte	LOQ, ng/mL ⁽²⁾ 8/21/19 Analysis	LOQ, ng/mL ⁽¹⁾ 8/27/19 Analysis	LOQ, ng/mL ⁽¹⁾ 8/29/19 Analysis	LOQ, ng/mL ⁽¹⁾ 9/4/19 Analysis	LOQ, ng/mL ⁽²⁾ 9/7/19 Analysis	LOQ, ng/mL ⁽²⁾ 9/9/19 Analysis
PFOA	0.0240	0.0479	0.0479	0.0958	NA	NA
PFBS	0.0250	0.100	0.100	NA	NA	NA
PFHS	0.0250	0.100	0.100	NA	NA	NA
PFOS	0.0464	0.0927	0.0464	0.0927	NA	NA
PFOSA	0.0500	NA	NA	NA	0.500	0.100

NA = Not Applicable

(1) A dilution factor was not applied to the LOQ.

(2) A dilution factor of 2 was applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of 100%±25%.

3.5 Blanks

Two types of blanks were prepared and analyzed with the samples: method/solvent blanks and field/trip blanks. Each blank result was reviewed and used to evaluate method performance. The method blanks were used to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. LCSs were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. Surrogates [¹³C₄]-PFOA and [¹³C₄]-PFOS were added post dilution when analyzed by external standard. The target analyte LCSs were spiked at the following nominal concentrations:

- LCSs analyzed on 8/21/19 were prepared at 0.2 ng/mL, 20 ng/mL, and 140 ng/mL.

- LCSs analyzed on 8/27/19 were prepared at 100 ng/mL, 5000 ng/mL, and 35000 ng/mL.
- LCSs analyzed on 8/29/19 were prepared at 200 ng/mL, 20000 ng/mL, and 70000 ng/mL.
- LCSs analyzed on 9/4/19 were prepared at 100 ng/mL, 5000 ng/mL, and 35000 ng/mL.
- LCSs analyzed on 9/7/19 and 9/9/19 were prepared at 0.2 ng/mL, 20 ng/mL, and 140 ng/mL.

Method ETS-8-044.3 states that the average recovery of LCSs at each spiking level must be within 80%-120% with a RSD ≤20%. All LCS samples met criteria with the following exceptions:

- 8/21/19: Low-level LCSs for PFOSA had an average recovery of 124%. High-level LCSs for PFBS, PFHS, and PFOSA were spiked above the resulting ULOQ (50 ng/mL or 100 ng/mL with dilution factor of 2 applied). Since more than 33% of the LCSs are outside the acceptance criteria for PFOSA, a method deviation is included in the raw data.
- 8/27/19: Low-level LCSs had an RSD of 32% for PFOA.
- 9/4/19: Low-level LCSs had an RSD of 23% for PFOS.
- 9/7/19: Low-level LCSs for PFOSA were spiked below the results LLOQ (0.500 ng/mL with dilution factor of 2 applied).
- 9/9/19: Low-level LCSs for PFOSA had an average recovery of 130%. High-level LCSs for PFOSA were spiked above the resulting ULOQ (100 ng/mL with dilution factor of 2 applied). Since more than 33% of the LCSs are outside the acceptance criteria for PFOSA, a method deviation is included in the raw data.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report. Individual LCS results are included in the raw data and available upon request.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.4. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in **Table 7** below.

- The data uncertainty for PFOSA using internal calibration was calculated at ±25% following ETS-12-012.4; however, the data uncertainty was increased to ±30% based on the PFOSA recovery of the low-level LCS from analysis on 9/9/19.

Table 7. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	12.0	±24%
PFBS	External	7.64	±15%
PFHS	External	6.62	±13%
PFOS	External	9.48	±19%
PFOA	Internal	11.0	±22%
PFBS	Internal	6.24	±12%
PFHS	Internal	6.90	±14%
PFOS	Internal	6.56	±13%
PFOSA	Internal	NA	±30%

NA = Not Applicable

3.8 Field Matrix Spikes (FMS)

Target analyte field matrix spike samples were collected for select sampling points to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions at 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOA and PFOS. The standards used for the preparation of the field matrix spiking solutions greater than 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, select sample bottles contained stable isotope surrogate recovery spikes of [¹³C₄]-PFOA and [¹³C₄]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles prior to sample collection. The [¹³C₄]-PFOA was selected to represent the C8 perfluorocarboxylic acid. The [¹³C₄]-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

The following calculation was used to generate data in section 4 of the report.

$$FMS\ Recovery = \frac{Sample\ Concentration\ of\ FMS - Average\ Concentration\ (Field\ Sample\ & Field\ Sample\ Dup)}{Spike\ Concentration} \times 100\%$$

Table 8. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
BD24-4, L21, L4	FMS	9.58	10.0	10.0	9.27	9.99
D18, 12	FMS	255	255	255	255	5.00
P379	FMS	550	50.0	550	550	50.0
D09, P119B, PP09, P42, P262bis, P372	FMS	1050	50.0	1050	1050	50.0
PP02 ⁽¹⁾	FMS	897	42.7	897	897	42.7
P341 ⁽¹⁾	FMS	942	44.8	942	942	44.8
Trip Blank	Low	9.58	10.0	10.0	9.27	9.99
	High	1050	50.0	1050	1050	50.0

(1) Sample container for the FMS sample was overfilled by 10%. The FMS true values were adjusted accordingly.

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of $\pm 30\%$, demonstrate that the method is appropriate for the given matrix.

The method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. At times, the spike level exceeded the recommended upper limit of 10 times the analyte concentration. In these instances, the FMS recovery was reported and flagged as above 10 times the sample concentration.

For PFOA, PFBS, PFHS, and PFOS, when the field matrix spike level was not appropriate as compared to the endogenous sample concentration, the surrogate recovery standards were used to assess method accuracy. All field matrix spike and surrogate recovery standard recoveries met acceptance criteria with the following exception:

BD24-4: The sample/sample duplicate RPD was 140% for PFOSA. The two subsequent re-analyses showed similar results, confirming the high RPD value. The results from the initial analysis are reported.

D11: The sample/sample duplicate RPDs were 110% for PFOA, 146% for PFHS, 46% for PFOS, and 117% for PFOSA. The two subsequent re-analyses showed similar results, confirming the high RPD values. The results from the initial analysis are reported.

P372: The FMS sample does not appear to have been spiked with the target analyte spiking solution or was spiked incorrectly at the time of the bottle preparation. The sample did contain internal standard demonstrating there was no issue with the sample collection.

P379: The initial analysis of PFOA had an FMS recovery of 46.2%. Since the FMS did not meet acceptance criteria of $100 \pm 50\%$ and the results would be not reportable, the sample set was re-analyzed for PFOA. The re-analysis had an FMS recovery of 52.0%. Since the re-analysis has a reportable QC result, the results from the re-analysis were reported and the analytical data uncertainty was further expanded to $\pm 48\%$ for PFOA based on the FMS %bias.

Table 9. Location ID: BD24-4

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-002	BD24-4; Sample	83.2	NA	47.2	NA
ISO19-14-03-002-DUP	BD24-4; Sample Duplicate	81.2	NA	43.4	NA
ISO19-14-03-002-FMS	BD24-4; Sample FMS	100	NC	68.4	NC
Average Concentration (ng/mL) ± %RPD		82.2 ng/mL ± 2.4%		45.3 ng/mL ± 8.4%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-002	BD24-4; Sample	0.768	NA	0.526	NA
ISO19-14-03-002-DUP	BD24-4; Sample Duplicate	0.626	NA	0.0930	NA
ISO19-14-03-002-FMS	BD24-4; Sample FMS	11.3	114 ⁽¹⁾	11.5	112 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		0.697 ng/mL ± 20%		0.310 ng/mL ± 140% ⁽²⁾	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration

(1) FMS concentration greater than 10 times the sample concentration.

(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 10. Location ID: D18

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-009	D18; Sample	179	NA	155	NA
ISO19-14-03-009-DUP	D18; Sample Duplicate	186	NA	156	NA
ISO19-14-03-009-FMS	D18; Sample FMS	399	85.0	398	95.1
Average Concentration (ng/mL) ± %RPD		183 ng/mL ± 3.8%		156 ng/mL ± 0.64%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-009	D18; Sample	64.6	NA	2.06	NA
ISO19-14-03-009-DUP	D18; Sample Duplicate	84.1	NA	1.90	NA
ISO19-14-03-009-FMS	D18; Sample FMS	299	88.2	8.46	130
Average Concentration (ng/mL) ± %RPD		74.4 ng/mL ± 26% ⁽¹⁾		1.98 ng/mL ± 8.1%	

NA = Not Applicable

(1) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 7. Location ID: P119B

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-016	P119B; Sample	2600	NA	1470	NA
ISO19-14-03-016-DUP	P119B; Sample Duplicate	2600	NA	1450	NA
ISO19-14-03-016-FMS	P119B; Sample FMS	3490	NC	2530	102
Average Concentration (ng/mL) ± %RPD		2600 ng/mL ± 0.0%		1460 ng/mL ± 1.4%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-016	P119B; Sample	381	NA	0.476	NA
ISO19-14-03-016-DUP	P119B; Sample Duplicate	386	NA	0.442	NA
ISO19-14-03-016-FMS	P119B; Sample FMS	1300	87.3	60.4	120 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		384 ng/mL ± 1.3%		0.459 ng/mL ± 7.4%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration

(1) FMS concentration greater than 10 times the sample concentration.

Table 8. Location ID: L21

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-022	L21; Sample	0.170	NA	0.136	NA
ISO19-14-03-022-DUP	L21; Sample Duplicate	0.170	NA	0.139	NA
ISO19-14-03-022-FMS	L21; Sample FMS	10.9	112 ⁽¹⁾	11.7	116 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		0.170 ng/mL ± 0.0%		0.138 ng/mL ± 2.2%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-022	L21; Sample	4.24	NA	0.0616	NA
ISO19-14-03-022-DUP	L21; Sample Duplicate	4.62	NA	0.0608	NA
ISO19-14-03-022-FMS	L21; Sample FMS	15.7	122	11.6	116 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		4.43 ng/mL ± 8.6%		0.0612 ng/mL ± 1.3%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 9. Location ID: L4

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-025	L4; Sample	3.68	NA	2.82	NA
ISO19-14-03-025-DUP	L4; Sample Duplicate	4.08	NA	3.08	NA
ISO19-14-03-025-FMS	L4; Sample FMS	14.2	108	14.3	114
Average Concentration (ng/mL) ± %RPD		3.88 ng/mL ± 10%		2.95 ng/mL ± 8.8%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-025	L4; Sample	29.0	NA	0.468	NA
ISO19-14-03-025-DUP	L4; Sample Duplicate	30.0	NA	0.442	NA
ISO19-14-03-025-FMS	L4; Sample FMS	38.6	NC	11.7	113 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		29.5 ng/mL ± 3.4%		0.455 ng/mL ± 5.7%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

(1) FMS concentration greater than 10 times the sample concentration.

Table 10. Location ID: PP02

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-030	PP02; Sample	663	NA	45.2	NA	3870	NA
ISO19-14-03-030-DUP	PP02; Sample Duplicate	633	NA	45.2	NA	3670	NA
ISO19-14-03-030-FMS	PP02; Sample FMS	1580	104	85.8	95.0	4970	NC
Average Concentration (ng/mL) ± %RPD		648 ng/mL ± 4.6%		45.2 ng/mL ± 0.0%		3770 ng/mL ± 5.3%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-030	PP02; Sample	18300	NA	23.4	NA
ISO19-14-03-030-DUP	PP02; Sample Duplicate	16300	NA	21.2	NA
ISO19-14-03-030-FMS	PP02; Sample FMS	19600	NC	67.4	106
Average Concentration (ng/mL) ± %RPD		17300 ng/mL ± 12%		22.3 ng/mL ± 9.9%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 15. Location ID: 12

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-038	12; Sample	56.1	NA	17.8	NA
ISO19-14-03-038-DUP	12; Sample Duplicate	57.2	NA	17.5	NA
ISO19-14-03-038-FMS	12; Sample FMS	284	89.2	263	96.2 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		56.7 ng/mL ± 1.9%		17.7 ng/mL ± 1.7%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-038	12; Sample	224	NA	0.372	NA
ISO19-14-03-038-DUP	12; Sample Duplicate	223	NA	0.346	NA
ISO19-14-03-038-FMS	12; Sample FMS	444	86.6	5.92	111 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		224 ng/mL ± 0.45%		0.359 ng/mL ± 7.2%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 16. Location ID: P42

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-048	P42; Sample	229	NA	47.3	NA	372	NA
ISO19-14-03-048-DUP	P42; Sample Duplicate	232	NA	41.0	NA	357	NA
ISO19-14-03-048-FMS	P42; Sample FMS	1080	80.9	89.4	90.5	1320	91.0
Average Concentration (ng/mL) ± %RPD		231 ng/mL ± 1.3%		44.2 ng/mL ± 14%		365 ng/mL ± 4.1%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-048	P42; Sample	2280	NA	12.0	NA
ISO19-14-03-048-DUP	P42; Sample Duplicate	2410	NA	11.1	NA
ISO19-14-03-048-FMS	P42; Sample FMS	2960	NC	66.4	110
Average Concentration (ng/mL) ± %RPD		2350 ng/mL ± 5.5%		11.6 ng/mL ± 7.8%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 17. Location ID: P341

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-056	P341; Sample	691	NA	217	NA
ISO19-14-03-056-DUP	P341; Sample Duplicate	713	NA	215	NA
ISO19-14-03-056-FMS	P341; Sample FMS	1580	93.2	1180	102
Average Concentration (ng/mL) ± %RPD		702 ng/mL ± 3.1%		216 ng/mL ± 0.93%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-056	P341; Sample	5500	NA	55.0	NA
ISO19-14-03-056-DUP	P341; Sample Duplicate	5350	NA	55.0	NA
ISO19-14-03-056-FMS	P341; Sample FMS	6560	NC	111	125
Average Concentration (ng/mL) ± %RPD		5430 ng/mL ± 2.8%		55.0 ng/mL ± 0.0%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 18. Location ID: P379

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-058	P379; Sample	68.4	NA	20.5	NA
ISO19-14-03-058-DUP	P379; Sample Duplicate	67.6	NA	19.1	NA
ISO19-14-03-058-FMS	P379; Sample FMS	354	52.0 ⁽¹⁾	509	88.9 ⁽³⁾
Average Concentration (ng/mL) ± %RPD		68.0 ng/mL ± 1.2% ⁽²⁾		19.8 ng/mL ± 7.1%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-058	P379; Sample	749	NA	58.2	NA
ISO19-14-03-058-DUP	P379; Sample Duplicate	749	NA	60.6	NA
ISO19-14-03-058-FMS	P379; Sample FMS	1200	82.0	114	109
Average Concentration (ng/mL) ± %RPD		749 ng/mL ± 0.0%		59.4 ng/mL ± 4.0%	

NA = Not Applicable

(1) FMS did not meet acceptance criteria of $100 \pm 30\%$.

(2) Analytical data uncertainty has been expanded to $\pm 48\%$.

(3) FMS concentration greater than 10 times the sample concentration.

Table 19. Location ID: P262bis

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-064	P262bis Sample	523	NA	322	NA
ISO19-14-03-064-DUP	P262bis Sample Duplicate	541	NA	327	NA
ISO19-14-03-064-FMS	P262bis FMS	1360	78.9	1250	88.1
Average Concentration (ng/mL) ± %RPD		532 ng/mL ± 3.4%		325 ng/mL ± 1.5%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-064	P262bis Sample	5400	NA	8.80	NA
ISO19-14-03-064-DUP	P262bis Sample Duplicate	5530	NA	9.10	NA
ISO19-14-03-064-FMS	P262bis FMS	6570	NC	65.6	113
Average Concentration (ng/mL) ± %RPD		5470 ng/mL ± 2.4%		8.95 ng/mL ± 3.4%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 20. Location ID: Travel Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-067	Travel Blank	<0.0240	NA	<0.0250	NA	<0.0250	NA
ISO19-14-03-067-FMS-LOW	Travel Blank FMS Low	10.1	105	11.2	112	11.0	110
ISO19-14-03-067-FMS-HIGH	Travel Blank FMS High	948	90.3	55.9	112	1030	98.1

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-03-067	Travel Blank	<0.0464	NA	<0.0500	NA
ISO19-14-03-067-FMS-LOW	Travel Blank FMS Low	10.1	109	10.8	108
ISO19-14-03-067-FMS-HIGH	Travel Blank FMS High	923	87.9	46.8	93.6

NA = Not Applicable

Table 21. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO19-14-03-001	BD24-3; Sample	110	109	NA
ISO19-14-03-001-DUP	BD24-3; Sample Duplicate	108	111	NA
ISO19-14-03-002	BD24-4; Sample	104 ⁽²⁾	105 ⁽²⁾	NA
ISO19-14-03-002-DUP	BD24-4; Sample Duplicate	108 ⁽²⁾	107 ⁽²⁾	NA
ISO19-14-03-002-FMS	BD24-4; Sample FMS	102 ⁽²⁾	107 ⁽²⁾	NA
ISO19-14-03-004	D10; Sample	112	115	NA
ISO19-14-03-004-DUP	D10; Sample Duplicate	106	114	NA
ISO19-14-03-005	D11; Sample	110	110	NA
ISO19-14-03-005-DUP	D11; Sample Duplicate	110	111	NA
ISO19-14-03-006	D14; Sample	109 ⁽²⁾	109 ⁽²⁾	NA
ISO19-14-03-006-DUP	D14; Sample Duplicate	108 ⁽²⁾	108 ⁽²⁾	NA
ISO19-14-03-007	D16; Sample	111	110	NA
ISO19-14-03-007-DUP	D16; Sample Duplicate	108	112	NA
ISO19-14-03-008	D17; Sample	109	109	NA
ISO19-14-03-008-DUP	D17; Sample Duplicate	111	113	NA
ISO19-14-03-009	D18; Sample	105	114	NA
ISO19-14-03-009-DUP	D18; Sample Duplicate	111	112	NA
ISO19-14-03-009-FMS	D18; Sample FMS	112	111	NA
ISO19-14-03-010	D2; Sample	101 ⁽²⁾	111 ⁽²⁾	NA
ISO19-14-03-010-DUP	D2; Sample Duplicate	104 ⁽²⁾	103 ⁽²⁾	NA
ISO19-14-03-011	D5; Sample	116	118	NA
ISO19-14-03-011-DUP	D5; Sample Duplicate	112	113	NA
ISO19-14-03-012	ND7; Sample	106	111	NA
ISO19-14-03-012-DUP	ND7; Sample Duplicate	109	111	NA
ISO19-14-03-013	P118A; Sample	113	118	NA
ISO19-14-03-013-DUP	P118A; Sample Duplicate	108	114	NA
ISO19-14-03-014	P118B; Sample	107	110	NA
ISO19-14-03-014-DUP	P118B; Sample Duplicate	108	112	NA
ISO19-14-03-015	P119A; Sample	103 ⁽²⁾	108 ⁽²⁾	NA
ISO19-14-03-015-DUP	P119A; Sample Duplicate	110 ⁽²⁾	110 ⁽²⁾	NA
ISO19-14-03-016	P119B; Sample	109	114	NA
ISO19-14-03-016-DUP	P119B; Sample Duplicate	112	114	NA
ISO19-14-03-016-FMS	P119B; Sample FMS	112	117	NA
ISO19-14-03-017	P121; Sample	107	112	110
ISO19-14-03-017-DUP	P121; Sample Duplicate	105	111	109
ISO19-14-03-018	3M vijver; Sample	110 ⁽²⁾	111 ⁽²⁾	NA
ISO19-14-03-018-DUP	3M vijver; Sample Duplicate	104 ⁽²⁾	117 ⁽²⁾	NA
ISO19-14-03-019	Blokkersdijkvijver Noord; Sample	109 ⁽²⁾	111 ⁽²⁾	NA
ISO19-14-03-019-DUP	Blokkersdijkvijver Noord; Sample Duplicate	106 ⁽²⁾	108 ⁽²⁾	NA
ISO19-14-03-020	Blokkersdijkvijver standard; Sample	105 ⁽²⁾	110 ⁽²⁾	NA
ISO19-14-03-020-DUP	Blokkersdijkvijver standard; Sample Duplicate	109 ⁽²⁾	111 ⁽²⁾	NA

NA = Not Applicable

(1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.

(2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 21 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]PFOA	[¹³ C ₄]PFOS	[¹³ C ₄]PFOS Re-analysis
ISO19-14-03-021	P321; Sample	113	113	NA
ISO19-14-03-021-DUP	P321; Sample Duplicate	113	112	NA
ISO19-14-03-022	L21; Sample	101 ⁽²⁾	111 ⁽²⁾	NA
ISO19-14-03-022-DUP	L21; Sample Duplicate	108 ⁽²⁾	108 ⁽²⁾	NA
ISO19-14-03-022-FMS	L21; Sample FMS	106 ⁽²⁾	117 ⁽²⁾	NA
ISO19-14-03-023	L22; Sample	108 ⁽²⁾	109 ⁽²⁾	NA
ISO19-14-03-023-DUP	L22; Sample Duplicate	109 ⁽²⁾	110 ⁽²⁾	NA
ISO19-14-03-024	L31; Sample	102 ⁽²⁾	102 ⁽²⁾	NA
ISO19-14-03-024-DUP	L31; Sample Duplicate	107 ⁽²⁾	108 ⁽²⁾	NA
ISO19-14-03-025	L4; Sample	110 ⁽²⁾	109 ⁽²⁾	NA
ISO19-14-03-025-DUP	L4; Sample Duplicate	114 ⁽²⁾	114 ⁽²⁾	NA
ISO19-14-03-025-FMS	L4; Sample FMS	106 ⁽²⁾	104 ⁽²⁾	NA
ISO19-14-03-026	P114bis; Sample	113 ⁽²⁾	112 ⁽²⁾	NA
ISO19-14-03-026-DUP	P114bis; Sample Duplicate	107 ⁽²⁾	114 ⁽²⁾	NA
ISO19-14-03-027	P115; Sample	114 ⁽²⁾	107 ⁽²⁾	NA
ISO19-14-03-027-DUP	P115; Sample Duplicate	113 ⁽²⁾	107 ⁽²⁾	NA
ISO19-14-03-028	P116; Sample	99.0 ⁽²⁾	114 ⁽²⁾	NA
ISO19-14-03-028-DUP	P116; Sample Duplicate	105 ⁽²⁾	114 ⁽²⁾	NA
ISO19-14-03-029	Effluent WWTP; Sample	104 ⁽²⁾	108 ⁽²⁾	NA
ISO19-14-03-029-DUP	Effluent WWTP; Sample Duplicate	112 ⁽²⁾	112 ⁽²⁾	NA
ISO19-14-03-030	PP02; Sample	115	115	113
ISO19-14-03-030-DUP	PP02; Sample Duplicate	112	112	110
ISO19-14-03-030-FMS	PP02; Sample FMS	112	114	111
ISO19-14-03-031	PP04; Sample	108	107	NA
ISO19-14-03-031-DUP	PP04; Sample Duplicate	108	105	NA
ISO19-14-03-032	PP05; Sample	106	101	120
ISO19-14-03-032-DUP	PP05; Sample Duplicate	110	108	118
ISO19-14-03-033	PP06; Sample	108	111	NA
ISO19-14-03-033-DUP	PP06; Sample Duplicate	110	111	NA
ISO19-14-03-034	PP07; Sample	109	110	NA
ISO19-14-03-034-DUP	PP07; Sample Duplicate	110	112	NA
ISO19-14-03-035	PP08; Sample	110	111	NA
ISO19-14-03-035-DUP	PP08; Sample Duplicate	111	110	NA
ISO19-14-03-037	PP10; Sample	111	112	NA
ISO19-14-03-037-DUP	PP10; Sample Duplicate	105	109	NA
ISO19-14-03-038	12; Sample	105	110	NA
ISO19-14-03-038-DUP	12; Sample Duplicate	107	108	NA
ISO19-14-03-038-FMS	12; Sample FMS	110	111	NA
ISO19-14-03-039	13; Sample	108 ⁽²⁾	108 ⁽²⁾	114
ISO19-14-03-039-DUP	13; Sample Duplicate	100 ⁽²⁾	103 ⁽²⁾	118
ISO19-14-03-040	5; Sample	113 ⁽²⁾	107 ⁽²⁾	NA
ISO19-14-03-040-DUP	5; Sample Duplicate	104 ⁽²⁾	104 ⁽²⁾	NA

NA = Not Applicable

(1) The surrogate recovery standards [¹³C₄]PFOA and [¹³C₄]PFOS were added to the samples during sample preparation unless noted otherwise.

(2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 21 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO19-14-03-041	Bemalingstation; Sample	117 ⁽²⁾	106 ⁽²⁾	NA
ISO19-14-03-041-DUP	Bemalingstation; Sample Duplicate	103 ⁽²⁾	116 ⁽²⁾	NA
ISO19-14-03-042	Collector put; Sample	109 ⁽²⁾	106 ⁽²⁾	114
ISO19-14-03-042-DUP	Collector put; Sample Duplicate	107 ⁽²⁾	109 ⁽²⁾	113
ISO19-14-03-043	K3; Sample	108	112	NA
ISO19-14-03-043-DUP	K3; Sample Duplicate	113	118	NA
ISO19-14-03-044	P27; Sample	112	114	NA
ISO19-14-03-044-DUP	P27; Sample Duplicate	118	120	NA
ISO19-14-03-045	P21B; Sample	116	116	119
ISO19-14-03-045-DUP	P21B; Sample Duplicate	114	119	116
ISO19-14-03-046	P304; Sample	118	114	NA
ISO19-14-03-046-DUP	P304; Sample Duplicate	114	117	NA
ISO19-14-03-047	P305; Sample	108	112	NA
ISO19-14-03-047-DUP	P305; Sample Duplicate	110	113	NA
ISO19-14-03-048	P42; Sample	106	113	NA
ISO19-14-03-048-DUP	P42; Sample Duplicate	115	118	NA
ISO19-14-03-048-FMS	P42; Sample FMS	101	109	NA
ISO19-14-03-049	L19; Sample	112	115	NA
ISO19-14-03-049-DUP	L19; Sample Duplicate	113	116	NA
ISO19-14-03-050	M4; Sample	109	111	NA
ISO19-14-03-050-DUP	M4; Sample Duplicate	111	113	NA
ISO19-14-03-051	P118C; Sample	116	121	NA
ISO19-14-03-051-DUP	P118C; Sample Duplicate	109	113	NA
ISO19-14-03-052	P119C; Sample	101	109	112
ISO19-14-03-052-DUP	P119C; Sample Duplicate	116	121	119
ISO19-14-03-053	P263; Sample	110	116	NA
ISO19-14-03-053-DUP	P263; Sample Duplicate	107	113	NA
ISO19-14-03-054	P264; Sample	107	110	NA
ISO19-14-03-054-DUP	P264; Sample Duplicate	113	119	NA
ISO19-14-03-055	P340; Sample	113	117	NA
ISO19-14-03-055-DUP	P340; Sample Duplicate	110	117	NA
ISO19-14-03-056	P341; Sample	116	121	NA
ISO19-14-03-056-DUP	P341; Sample Duplicate	112	116	NA
ISO19-14-03-056-FMS	P341; Sample FMS	113	118	NA
ISO19-14-03-057	P374; Sample	113	119	NA
ISO19-14-03-057-DUP	P374; Sample Duplicate	119	123	NA
ISO19-14-03-058	P379; Sample	121	118	NA
ISO19-14-03-058-DUP	P379; Sample Duplicate	112	117	NA
ISO19-14-03-058-FMS	P379; Sample FMS	125	118	NA
ISO19-14-03-059	P380; Sample	114	118	NA
ISO19-14-03-059-DUP	P380; Sample Duplicate	113	119	NA

NA = Not Applicable

(1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.

(2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 21 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]PFOA	[¹³ C ₄]PFOS	[¹³ C ₄]PFOS Re-analysis
ISO19-14-03-060	P381; Sample	115	117	NA
ISO19-14-03-060-DUP	P381; Sample Duplicate	107	118	NA
ISO19-14-03-061	P382; Sample	107	111	NA
ISO19-14-03-061-DUP	P382; Sample Duplicate	105	111	NA
ISO19-14-03-062	P372; Sample	104	112	NA
ISO19-14-03-062-DUP	P372; Sample Duplicate	111	117	NA
ISO19-14-03-062-FMS	P372; Sample FMS	105	114	NA
ISO19-14-03-063	PP11 Samples	108	117	NA
ISO19-14-03-063-DUP	PP11 Sample Duplicate	109	114	NA
ISO19-14-03-064	P262bis Sample	108	114	NA
ISO19-14-03-064-DUP	P262bis Sample Duplicate	110	114	NA
ISO19-14-03-064-FMS	P262bis FMS	112	118	NA
ISO19-14-03-065	P265B Sample	107	116	NA
ISO19-14-03-065-DUP	P265B Sample Duplicate	111	119	NA
ISO19-14-03-066	P371 Sample	100	110	NA
ISO19-14-03-066-DUP	P371 Sample Duplicate	111	118	NA
ISO19-14-03-067	Travel Blank	113 ⁽²⁾	111 ⁽²⁾	NA
ISO19-14-03-067-FMS-LOW	Travel Blank FMS Low	100 ⁽²⁾	110 ⁽²⁾	NA
ISO19-14-03-067-FMS-HIGH	Travel Blank FMS High	114	115	NA

NA = Not Applicable

(1) The surrogate recovery standards [¹³C₄]PFOA and [¹³C₄]PFOS were added to the samples during sample preparation unless noted otherwise.

(2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 9-21 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M EHS Laboratory standard operating procedures.

7 Attachments

Chain of Custody Form

8 Signatures

Chelsie Grochow, 3M Report Author

Susan T. Wolf, 3M Principal Analytical Investigator

Brian T. Mader, Ph.D., 3M EHS Laboratory Manager

The 3M EHS Laboratory's Quality Assurance Unit has audited the data and report for this project.

Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M EHS Laboratory.

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Project: ISO19-14-03

Phone:
Alt. Ph:
Fax: (651) 777-2100

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 7/1/2019
Project Description: 3M Antwerp Water Sampling for PFCs - July 2019
Comments:

Completion Date:
Project Lead: Susan T. Wolf
Phone Number:
Email Address:

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO19-14-03-001 ✓	BD24-3; Sample ✓	01/08/19-9:51AM	WG	
ISO19-14-03-001-DUP ✓	BD24-3; Sample Duplicate ✓	01/08/19-9:58AM	WG	
ISO19-14-03-002 ✓	BD24-4; Sample ✓	01/08/19-9:58AM	WG	
ISO19-14-03-002-DUP ✓	BD24-4; Sample Duplicate ✓	01/08/19-9:58AM	WG	
ISO19-14-03-002-FMS ✓	BD24-4; Sample FMS ✓	01/08/19-9:58AM	WQ	
ISO19-14-03-003 ✓	D09; Sample ✓		WG	not sampled
ISO19-14-03-003-DUP ✓	D09; Sample Duplicate ✓		WG	not sampled
ISO19-14-03-003-FMS ✓	D09; Sample FMS ✓		WQ	not sampled
ISO19-14-03-004 ✓	D10; Sample ✓	01/08/19-8:50AM	WG	
ISO19-14-03-004-DUP ✓	D10; Sample Duplicate ✓	01/08/19-8:50AM	WG	
ISO19-14-03-005 ✓	D11; Sample ✓	01/08/19-12:27AM	WG	
ISO19-14-03-005-DUP ✓	D11; Sample Duplicate ✓	01/08/19-12:27AM	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for (1)

Temperature: 20 Deg C Received on Ice Other:

Collected by (print): Julie Tichyjt Collector's signature: 

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
12	Julie Tichyjt	05/08/19	10:00	Fedex	Joseph Tuman	8-6-2019	11:00 AM

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
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St. Paul, MN 55144

Project: ISO19-14-03 (cont.)

Phone:
Alt. Pho
Fax: (6)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 7/1/2019

Project Description: 3M Antwerp Water Sampling for PFCs - July 2019

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO19-14-03-006	D14; Sample ✓	30/07/19-9:16 AM	WG	
ISO19-14-03-006-DUP	D14; Sample Duplicate ✓	30/07/19-9:20 AM	WG	
ISO19-14-03-007	D16; Sample ✓	30/07/19-9:40 AM	WG	
ISO19-14-03-007-DUP	D16; Sample Duplicate ✓	30/07/19-9:40 AM	WG	
ISO19-14-03-008	D17; Sample ✓	30/07/19-11:22 AM	WG	
ISO19-14-03-008-DUP	D17; Sample Duplicate ✓	30/07/19-11:22 AM	WG	
ISO19-14-03-009	D18; Sample ✓	31/07/19-11:00 AM	WG	
ISO19-14-03-009-DUP	D18; Sample Duplicate ✓	31/07/19-11:00 AM	WG	
ISO19-14-03-009-FMS	D18; Sample FMS ✓	31/07/19-11:00 AM	WQ	
ISO19-14-03-010	D2; Sample ✓	31/07/19-10:15 AM	WG	
ISO19-14-03-010-DUP	D2; Sample Duplicate ✓	31/07/19-10:15 AM	WG	
ISO19-14-03-011	D5; Sample ✓	30/07/19-1 PM	WG	
ISO19-14-03-011-DUP	D5; Sample Duplicate ✓	30/07/19-1 PM	WG	
ISO19-14-03-012	ND7; Sample ✓	30/07/19-12:20 AM	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 25 Deg C Received on Ice Other: ①

Collected by (print): Julie Tichijt

Collector's signature: J. Tichijt

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Julie Tichijt	05/08/19	10:00	FedEx	Jessica Tillman	8-6-19	11:00 AM

3M EHS LABORATORY
Chain-of-Custody

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Project: ISO19-14-03 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 7/1/2019

Project Description: 3M Antwerp Water Sampling for PFCs - July 2019

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO19-14-03-012-DUP	ND7; Sample Duplicate ✓	31/07/19-12:50 AM	WG	
ISO19-14-03-013	P118A; Sample ✓	31/07/19-11:40 AM	WG	
ISO19-14-03-013-DUP	P118A; Sample Duplicate ✓	31/07/19-11:40 AM	WG	
ISO19-14-03-014	P118B; Sample ✓	31/07/19-12:10 AM	WG	
ISO19-14-03-014-DUP	P118B; Sample Duplicate ✓	31/07/19-12:10 AM	WG	
ISO19-14-03-015	P119A; Sample ✓	01/08/19 10:40 AM	WG	
ISO19-14-03-015-DUP	P119A; Sample Duplicate ✓	01/08/19 10:40 AM	WG	
ISO19-14-03-016	P119B; Sample ✓	01/08/19-12:40 AM	WG	
ISO19-14-03-016-DUP	P119B; Sample Duplicate ✓	01/08/19-12:40 AM	WG	
ISO19-14-03-016-FMS	P119B; Sample FMS ✓	01/08/19-12:40 AM	WQ	
ISO19-14-03-017	P121; Sample ✓	31/07/19-12:40 PM	WG	
ISO19-14-03-017-DUP	P121; Sample Duplicate ✓	31/07/19-12:40 PM	WG	
ISO19-14-03-018	3M vijver; Sample ✓	30/07/19-10 AM	WG	
ISO19-14-03-018-DUP	3M vijver; Sample Duplicate ✓	30/07/19-10 AM	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 25 Deg C Received on Ice Other: (1)

Collected by (print): Julie Fichfjord

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Julie Fichfjord	05/08/19	10:00	FedEx	Susan T. Wolf	8-6-19	11:00 AM

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
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Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 7/1/2019

Project Description: 3M Antwerp Water Sampling for PFCs - July 2019

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO19-14-03-019 ✓	Blokkersdijkvijver Noord; Sample ✓	30/07/19-10:30 AM	WG	
ISO19-14-03-019-DUP •	Blokkersdijkvijver Noord; Sample Duplicate ✓	30/07/19-10:30 AM	WG	
ISO19-14-03-020 ✓	Blokkersdijkvijver standaard; Sample ✓	30/07/19-11:00 AM	WG	
ISO19-14-03-020-DUP •	Blokkersdijkvijver standaard; Sample Duplicate ✓	30/07/19-11:00 AM	WG	
ISO19-14-03-021 ✓	P321; Sample ✓	31/07/19-8:57 AM	WG	
ISO19-14-03-021-DUP •	P321; Sample Duplicate ✓	31/07/19-8:57 AM	WG	
ISO19-14-03-022 ✓	L21; Sample ✓	31/07/19-9:30 AM	WG	
ISO19-14-03-022-DUP •	L21; Sample Duplicate ✓	31/07/19-9:30 AM	WG	
ISO19-14-03-022-FMS ✓	L21; Sample FMS ✓	31/07/19-9:30 AM	WQ	
ISO19-14-03-023 ✓	L22; Sample ✓	31/07/19-12:30 PM	WG	
ISO19-14-03-023-DUP •	L22; Sample Duplicate ✓	31/07/19-12:30 PM	WG	
ISO19-14-03-024 ✓	L31; Sample ✓	31/07/19-9:50 AM	WG	
ISO19-14-03-024-DUP •	L31; Sample Duplicate ✓	31/07/19-9:50 AM	WG	
ISO19-14-03-025 ✓	L4; Sample ✓	31/07/19-9:55 AM	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 25 Deg C Received on Ice Other: ①

Collected by (print): Julie Tichydt

Collector's signature: JT

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Julie Tichydt	05/08/19	10:00	FedEx	JOSEPH TICHYDT	6-6-19	11:00 AM

3M EHS LABORATORY
Chain-of-Custody

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Project: ISO19-14-03 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)

Department: 832202 Site Source: 01WJWT10

Project Number:

Date Created: 7/1/2019

Project Description: 3M Antwerp Water Sampling for PFCs - July 2019

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO19-14-03-025-DUP *	L4; Sample Duplicate ✓	01/08/19-9:15 AM	WG	
ISO19-14-03-025-FMS *	L4; Sample FMS ✓	01/08/19-9:15 AM	WQ	
ISO19-14-03-026 *	P114bis; Sample ✓	31/07/19-1:30 PM	WG	
ISO19-14-03-026-DUP *	P114bis; Sample Duplicate ✓	31/07/19-1:30 PM	WG	
ISO19-14-03-027 *	P115; Sample ✓	31/07/19-9:10 PM	WG	
ISO19-14-03-027-DUP *	P115; Sample Duplicate ✓	31/07/19-9:10 PM	WG	
ISO19-14-03-028 *	P116; Sample ✓	01/08/19-10:50 AM	WG	
ISO19-14-03-028-DUP *	P116; Sample Duplicate ✓	01/08/19-10:50 AM	WG	
ISO19-14-03-029 *	Effluent WWTP; Sample ✓	30/07/19-11:00 PM	WT	
ISO19-14-03-029-DUP *	Effluent WWTP; Sample Duplicate ✓	30/07/19-11:00 PM	WT	
ISO19-14-03-030 *	PP02; Sample ✓	30/07/19-1:30 PM	WG	
ISO19-14-03-030-DUP *	PP02; Sample Duplicate ✓	30/07/19-1:30 PM	WG	
ISO19-14-03-030-FMS *	PP02; Sample FMS ✓	30/07/19-1:30 PM	WQ	
ISO19-14-03-031 *	PP04; Sample ✓	01/08/19-11:30 AM	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 25 Deg C Received on Ice Other: ①

Collected by (print): Julie Fichjet

Collector's signature: JF

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Julie Fichjet	08/08/19	10:00	FedEx	SUSAN TILLMAN	8-6-19	11:00 AM

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
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Project: ISO19-14-03 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)

Department: 832202 Site Source: 01WJWT10

Project Number:

Date Created: 7/1/2019

Project Description: 3M Antwerp Water Sampling for PFCs - July 2019

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO19-14-03-031-DUP	PP04; Sample Duplicate ✓	01/08/19-11:30 AM	WG	
ISO19-14-03-032	PP05; Sample ✓	01/08/19-11:15 AM	WG	
ISO19-14-03-032-DUP	PP05; Sample Duplicate ✓	01/08/19-11:15 AM	WG	
ISO19-14-03-033	PP06; Sample ✓	01/08/19-11:00 AM	WG	
ISO19-14-03-033-DUP	PP06; Sample Duplicate ✓	01/08/19-11:00 AM	WG	
ISO19-14-03-034	PP07; Sample ✓	30/07/19-11:30 PM	WG	
ISO19-14-03-034-DUP	PP07; Sample Duplicate ✓	30/07/19-11:30 PM	WG	
ISO19-14-03-035	PP08; Sample ✓	01/08/19-10:30 AM	WG	
ISO19-14-03-035-DUP	PP08; Sample Duplicate ✓	01/08/19-10:30 AM	WG	
ISO19-14-03-036	PP09; Sample ✓		WG	Not Sampled
ISO19-14-03-036-DUP	PP09; Sample Duplicate ✓		WG	not Sampled
ISO19-14-03-036-FMS	PP09; Sample FMS ✓		WQ	not Sampled
ISO19-14-03-037	PP10; Sample ✓	30/07/19-11:00 PM	WG	
ISO19-14-03-037-DUP	PP10; Sample Duplicate ✓	30/07/19-11:00 PM	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 25 Deg C Received on Ice Other: (1)

Collected by (print):

Julie Fichjet

Collector's signature: —

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
19	Julie Fichjet	05/08/19	10:00	FedEx	SUSAN T. WOLF	8-6-19	11:00 AM

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [REDACTED]
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Fax: (651) 753-2300

Project: ISO19-14-03 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)

Department: 832202 Site Source: 01WJWT10

Project Number:

Date Created: 7/1/2019

Project Description: 3M Antwerp Water Sampling for PFCs - July 2019

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO19-14-03-038	12; Sample ✓	30/07/19-11:30AM	WG	
ISO19-14-03-038-DUP	12; Sample Duplicate ✓	30/07/19-11:30AM	WG	
ISO19-14-03-038-FMS	12; Sample FMS ✓	30/07/19-11:30AM	WQ	
ISO19-14-03-039	13; Sample ✓	30/07/19-11:45AM	WG	
ISO19-14-03-039-DUP	13; Sample Duplicate ✓	30/07/19-11:45AM	WG	
ISO19-14-03-040	5; Sample ✓	30/07/19-12:00AM	WG	
ISO19-14-03-040-DUP	5; Sample Duplicate ✓	30/07/19-12:00AM	WG	
ISO19-14-03-041	Bemalingstation; Sample ✓	30/07/19-12:15AM	WG	
ISO19-14-03-041-DUP	Bemalingstation; Sample Duplicate ✓	30/07/19-12:15AM	WG	
ISO19-14-03-042	Collector put; Sample ✓	31/07/19-10:00AM	WG	
ISO19-14-03-042-DUP	Collector put; Sample Duplicate ✓	31/07/19-10:00AM	WG	
ISO19-14-03-043	K3; Sample ✓	30/07/19-10:08AM	WG	
ISO19-14-03-043-DUP	K3; Sample Duplicate ✓	30/07/19-10:08AM	WG	
ISO19-14-03-044	P27; Sample ✓	31/07/19-10:15AM	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 25 Deg C Received on Ice Other: ①

Collected by (print): Julie Fichter

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Julie Fichter	05/08/19	10:00	FedEx	JOSEPH TUMAN	8-6-19	11:00 AM

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Project: ISO19-14-03 (cont.)

Phone:
Alt. Pho
Fax: (65)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 7/1/2019

Project Description: 3M Antwerp Water Sampling for PFCs - July 2019

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO19-14-03-044-DUP	P27; Sample Duplicate ✓	31/07/19-10:25 AM	WG	
ISO19-14-03-045	P21B; Sample ✓	31/07/19-9:15 AM	WG	
ISO19-14-03-045-DUP	P21B; Sample Duplicate ✓	31/07/19-9:15 AM	WG	
ISO19-14-03-046	P304; Sample ✓	31/07/19-10:55 AM	WG	
ISO19-14-03-046-DUP	P304; Sample Duplicate ✓	31/07/19-10:55 AM	WG	
ISO19-14-03-047	P305; Sample ✓	31/07/19-9:00 AM	WG	
ISO19-14-03-047-DUP	P305; Sample Duplicate ✓	31/07/19-9:00 AM	WG	
ISO19-14-03-048	P42; Sample ✓	31/07/19-1:40 PM	WG	
ISO19-14-03-048-DUP	P42; Sample Duplicate ✓	31/07/19-1:40 PM	WG	
ISO19-14-03-048-FMS	P42; Sample FMS ✓	31/07/19-1:40 PM	WQ	
ISO19-14-03-049	L19; Sample ✓	31/07/19-9:30 AM	WG	
ISO19-14-03-049-DUP	L19; Sample Duplicate ✓	31/07/19-9:30 AM	WG	
ISO19-14-03-050	M4; Sample ✓	31/07/19-9:30 AM	WG	
ISO19-14-03-050-DUP	M4; Sample Duplicate ✓	31/07/19-9:30 AM	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 25 Deg C Received on Ice Other: O

Collected by (print): Julie Fischjet

Collector's signature: JF

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Julie Fischjet	05/08/19	10:00	FedEx	Joseph Tuomi	8-6-19	11:00 AM

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [REDACTED]
Alt. Phone: [REDACTED]
Fax: (651) [REDACTED]

Project: ISO19-14-03 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 7/1/2019

Project Description: 3M Antwerp Water Sampling for PFCs - July 2019

Completion Date:

Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

D-17-Y

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO19-14-03-051 ✓	P118C; Sample ✓	31/07/19 - 1:35 PM	WG	
ISO19-14-03-051-DUP ✓	P118C; Sample Duplicate ✓	31/07/19 - 1:35 PM	WG	
ISO19-14-03-052 ✓	P119C; Sample ✓	31/07/19 - 11:13 AM	WG	
ISO19-14-03-052-DUP ✓	P119C; Sample Duplicate ✓	31/07/19 - 11:13 AM	WG	
ISO19-14-03-053 ✓	P263; Sample ✓	31/07/19 - 10:30 AM	WG	
ISO19-14-03-053-DUP ✓	P263; Sample Duplicate ✓	31/07/19 - 10:30 AM	WG	
ISO19-14-03-054 ✓	P264; Sample ✓	31/07/19 - 12:10 AM	WG	
ISO19-14-03-054-DUP ✓	P264; Sample Duplicate ✓	31/07/19 - 12:10 AM	WG	
ISO19-14-03-055 ✓	P340; Sample ✓	31/07/19 - 2:45 PM	WG	
ISO19-14-03-055-DUP ✓	P340; Sample Duplicate ✓	31/07/19 - 2:45 PM	WG	
ISO19-14-03-056 ✓	P341; Sample ✓	31/07/19 - 3:41 PM	WG	
ISO19-14-03-056-DUP ✓	P341; Sample Duplicate ✓	31/07/19 - 3:41 PM	WG	
ISO19-14-03-056-FMS ✓	P341; Sample FMS ✓	31/07/19 - 1 PM	WQ	
ISO19-14-03-057 ✓	P374; Sample ✓	30/07/19 - 11:15 AM	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 25 Deg C Received on Ice Other: (1)

Collected by (print): Juli Tichy

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
19	Juli Tichy	05/08/19	10:00	FedEx	JOSEPH TILMAN	8/6/19	11:00 AM

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Project: ISO19-14-03 (cont.)

Phone:
Alt. Pho
Fax: (6)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 7/1/2019

Project Description: 3M Antwerp Water Sampling for PFCs - July 2019

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO19-14-03-057-DUP	P374; Sample Duplicate ✓	30/07/19-11:15 AM	WG	
ISO19-14-03-058	P379; Sample ✓	30/07/19-9:15 PM	WG	
ISO19-14-03-058-DUP	P379; Sample Duplicate ✓	30/07/19-2:15 PM	WG	
ISO19-14-03-058-FMS	P379; Sample FMS ✓	30/07/19-4:15 PM	WQ	
ISO19-14-03-059	P380; Sample ✓	30/07/19-11:30 AM	WG	
ISO19-14-03-059-DUP	P380; Sample Duplicate ✓	30/07/19-11:30 AM	WG	
ISO19-14-03-060	P381; Sample ✓	30/07/19 10:35 AM	WG	
ISO19-14-03-060-DUP	P381; Sample Duplicate ✓	30/07/19 10:35 AM	WG	
ISO19-14-03-061	P382; Sample ✓	30/07/19-10:35 AM	WG	
ISO19-14-03-061-DUP	P382; Sample Duplicate ✓	30/07/19-10:35 AM	WG	
ISO19-14-03-062	P372; Sample ✓	30/07/19-1:10 PM	WG	
ISO19-14-03-062-DUP	P372; Sample Duplicate ✓	30/07/19-1:10 PM	WG	
ISO19-14-03-062-FMS	P372; Sample FMS ✓	30/07/19-1:10 PM	WQ	
ISO19-14-03-063	PP11 Samples ✓	01/08/19-19:00 AM	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 25 Deg C Received on Ice Other: (1)

Collected by (print): Juli Tichjet

Collector's signature: J

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Juli Tichjet	05/08/19	10:00	Fedex	JOSEPH Trumm	8-6-19	11:00 AM

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Project: ISO19-14-03 (cont.)

Phone: [REDACTED]
Alt. Pho: [REDACTED]
Fax: (651) 773-2000

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 7/1/2019

Project Description: 3M Antwerp Water Sampling for PFCs - July 2019

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO19-14-03-063-DUP ✓	PP11 Sample Duplicate ✓	01/08/19-12:00AM	WG	
ISO19-14-03-064 ✓	P262bis Sample ✓	31/07/19-10:10AM	WG	
ISO19-14-03-064-DUP ✓	P262bis Sample Duplicate ✓	31/07/19-10:10AM	WG	
ISO19-14-03-064-FMS ✓	P262bis FMS ✓	31/07/19-10:10AM		
ISO19-14-03-065 ✓	P265B Sample ✓	31/07/19-1:19PM	WG	
ISO19-14-03-065-DUP ✓	P265B Sample Duplicate ✓	31/07/19-1:19PM	WG	
ISO19-14-03-066 ✓	P371 Sample ✓	31/07/19-12:50AM	WG	
ISO19-14-03-066-DUP ✓	P371 Sample Duplicate ✓	31/07/19-12:50AM	WG	
ISO19-14-03-067 ✓	Travel Blank ✓		WQ	(2)
ISO19-14-03-067-FMS-LOW ✓	Travel Blank FMS Low ✓		WQ	(2)
ISO19-14-03-067-FMS-HIGH ✓	Travel Blank FMS High ✓		WQ	(2)

(1) THE COLLECTOR ERRONEOUSLY FILLED OUT THIS SECTION WHEN THEY RECEIVED THE EMPTY BOTTLES. UPON RECEIPT IN THE EHS LAB, ALL SAMPLES WERE ACCEPTABLE, ACCOUNTED FOR AND AT RT. ST 8-6-19

(2) THESE SAMPLES WERE RECEIVED WITH ALL OTHER SAMPLES ON 8-6-19, BUT NO SAMPLING INFORMATION WAS INCLUDED FOR THEM. NOTE (1) STILL APPLIES. ST 8-6-19

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 25 Deg C Received on Ice Other: (1)

Collected by (print): Julie Fichter

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
M	Julie Fichter	05/08/19	10:00	FedEx	Jessica Truman	8-6-19	11:00 AM



3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC19-06111

3M Lab Request Number: E19-0756

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: November 28th, 2019

Requester

Jim Kotsmith
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Member of the SGS Group (Société Générale de Surveillance)

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All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction - Summary.

At the request (Lab Request Number: E19-0xxx) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected in October 23th and 24th 2019 from 3 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOA ($C_6F_{13}^{13}CF_2^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOS ($C_7F_{15}^{13}CF_2SO_3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Sven Herremans. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles.

Table 1 summarizes the sample results. All PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
Effluent WWTP	5.47	9.20	4.38	<0.248	
Bemalingsstation	22.7	28.6	31.6	<0.248	
Collector Put	26.5	65.3	144	7.81	

2. Methods – Analytical and Preparatory.**2.1. Sample collection.**

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
Effluent WWTP	100
Bemalingsstation	100
Collector Put	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefloreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
Effluent WWTP	1.0	0.05	Solution A	1.0	-
Bemalingsstation	1.0	0.05	Solution A	1.0	-
Collector Put	1.0	0.05	Solution A	1.0	-

(*): MeOH:LCMS-water (60:40)
Solution A = 1000 ng/mL (nominal) ^{13}C -PFC Surrogate Standards

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetri C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
PFBS	2.08 - 2.50	298.93/79.82	40	28	45
		298.93/98.78	40	29	45
PFHS	2.45 - 3.05	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.80 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
¹³ C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of $\pm 25\%$ ($\pm 30\%$ for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.0790 to 0.121 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.162 to 0.248 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within $\pm 25\%$, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by diluting 1 mL water with 10.0 mL MeOH:LCMS-water (60:40). Method blanks were prepared to evaluate the levels of background contamination in the overall dilution process (glassware, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carryover.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of LCMS-water, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCS)

One lab control spike was prepared and analyzed.

LCS was prepared by spiking known amounts of the analytes into 1.0 mL of LCMS-water to produce the desired concentration.

The spiked water sample was diluted with 10.0 mL MeOH:LCMS-water (60:40) and analyzed in the same manner as the samples.

The LCS produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 7 summarizes the LCS recovery results

Table 7. Lab Control Spike Results.

Dilution date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
7 August 2019	LCS1	80		99.1	82.1	102	97.9	100	104

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing PFBS, PFHS, PFOA, ^{13}C -PFOA, PFOS, FOSA and ^{13}C -PFOS (QC Lab Low and QC Lab High).

The QCs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 8 summarizes the QCs recovery results.

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
7 Nov 2019	QC Lab Low inj1	10		86.3	104	124	102	90.4	104
	QC Lab High inj1	100		80.8	107	105	102	108	100
	QC Lab Low inj2	10		84.4	109	101	102	91.0	104
	QC Lab High inj2	100		79.4	107	107	102	114	98.7

3.7. Equations.Recovery.

$$\text{Recovery(%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

The following tables summarize the results for the sample locations.

Table 9. Sample Results PFBS, PFHS, PFOA, PFOS and FOSA

	PFBS	PFHS	PFOA	PFOS	FOSA
	ng/ml	ng/ml	ng/ml	ng/ml	ng/ml
Effluent WWTP		5.47	9.20	4.38	<0.248
Bemalingsstation		22.7	28.6	31.6	<0.248
Collector Put		26.5	65.3	144	7.81
Field Trip Blank		<1.05	<0.877	<1.24	<1.34

Table 10. Sample Results ^{13}C -PFOA and ^{13}C -PFOS

	^{13}C -PFOA		^{13}C -PFOS	
	ng/ml	% Rec	ng/ml	% Rec
Effluent WWTP	71.1	122	52.5	83.7
Bemalingsstation	75.1	128	51.4	81.9
Collector Put	74.5	127	49.4	78.8
Field Trip Blank	120	103	128	102

5. Conclusion.

- The recovery of ¹³C-PFOA and ¹³C-PFOS fell within the method acceptance criterion of 70-130%.
- Lab control spike recoveries fell within the method acceptance criteria for all compounds, indicating that the analysis method is in control,
- QC Lab Standards recoveries fell within the method acceptance criteria for all compounds.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Julie Fichefet (ERM Belgium).

8. Signatures.

Sven Herremans,
Lab Operations Manager

Date December 28th, 2019



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date December 28th, 2019

I.A.C.
A Division of SGS Belgium NV

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The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.

Final Report

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

October 2019 Sampling

Laboratory Request Number: ISO19-14-04

Report Date – Date of Last Signature

Testing Laboratory

3M Environment, Health & Safety
3M EHS Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Charlotte Tack
3M Belgium; EHS Project Supervisor
3M Belgium; ZW019/0/01
[REDACTED]



The testing reported herein meet the requirements of ISO/IEC 17025:2017 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M EHS Laboratory

3M EHS Laboratory Manager: Brian T. Mader
3M Principal Analytical Investigator: Susan Wolf
Report Author: Scott Porcher

Analytical Report ISO19-14-04

Water Sample Analysis at 3M Antwerp, Belgium
October 2019 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M EHS Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. All samples were collected October 21 – 22, 2019 and returned to the 3M EHS Laboratory on October 28, 2019, at ambient temperature. The results in this report apply to the samples as received from Environmental Resources Management personnel. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS) (select locations only), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO19-14-04.

The 3M EHS Laboratory prepared sample containers for twenty-nine sampling locations. Each sample set consisted of a field sample and field sample duplicate. Six locations also included a target analyte field matrix spike. Each empty container was marked with a “fill to here” line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with a mass-labeled internal standard mix and select sample bottles were fortified with surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS prior to being sent to the field for sample collection. During sample collection, sample locations PP12 and PP13 were not sampled.

Samples were prepared and analyzed using method ETS-8-044.3 “Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis”. Select internal standards were used to aid in the data quality objectives.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ISO/IEC 17025:2017 “General Requirements for the Competence of Testing and Calibration Laboratories”, in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: 2nd Aquifer					
ISO19-14-04-001	D16; Sample	626	260	2100	14.0
ISO19-14-04-001-DUP	D16; Sample Dup	632	261	2150	11.0
		Average	629	261	2130
		%RPD Sample/Sample Dup	0.95	0.38	2.4
ISO19-14-04-005	P321; Sample	593	189	2970	14.0
ISO19-14-04-005-DUP	P321; Sample Dup	578	187	2940	15.2
		Average	586	188	2960
		%RPD Sample/Sample Dup	2.6	1.1	1.0
Zone: Blokkersdijk Nature Reserve					
ISO19-14-04-002	3M vijver; Sample	0.616	0.362	1.77	<0.0250
ISO19-14-04-002-DUP	3M vijver; Sample Dup	0.544	0.368	1.58	<0.0250
		Average	0.580⁽²⁾	0.365⁽²⁾	1.68⁽²⁾
		%RPD Sample/Sample Dup	12	1.6	11
ISO19-14-04-003	Blokkersdijkvijver Noord; Sample	1.14	0.744	2.64	0.100
ISO19-14-04-003-DUP	Blokkersdijkvijver Noord; Sample Dup	1.10	0.732	2.58	0.0972
		Average	1.12⁽²⁾	0.738⁽²⁾	2.61⁽²⁾
		%RPD Sample/Sample Dup	3.6	1.6	2.3
ISO19-14-04-004	Blokkersdijkvijver Standard; Sample	0.822	0.498	3.04	0.174
ISO19-14-04-004-DUP	Blokkersdijkvijver Standard; Sample Dup	0.912	0.498	3.08	0.161
		Average	0.867⁽²⁾	0.498⁽²⁾	3.06⁽²⁾
		%RPD Sample/Sample Dup	10	0.0	1.3
ISO19-14-04-006	L21; Sample	0.216	0.143	6.20	0.310
ISO19-14-04-006-DUP	L21; Sample Dup	0.212	0.145	5.96	0.278
		Average	0.214⁽²⁾	0.144⁽²⁾	6.08⁽²⁾
		%RPD Sample/Sample Dup	1.9	1.4	3.9
ISO19-14-04-007	L22; Sample	0.304	0.284	3.34	0.120
ISO19-14-04-007-DUP	L22; Sample Dup	0.404	0.340	2.60	0.172
		Average	0.354⁽²⁾	0.312⁽²⁾	2.97⁽²⁾
		%RPD Sample/Sample Dup	28⁽³⁾	18	25⁽³⁾
					0.146
ISO19-14-04-008	L31; Sample	0.360	0.694	8.22	0.144
ISO19-14-04-008-DUP	L31; Sample Dup	0.410	0.632	7.44	0.158
		Average	0.385⁽²⁾	0.663⁽²⁾	7.83⁽²⁾
		%RPD Sample/Sample Dup	13	9.4	10
					0.151
ISO19-14-04-009	L4; Sample	5.08	3.92	35.2	0.328
ISO19-14-04-009-DUP	L4; Sample Dup	5.94	4.36	38.0	0.326
		Average	5.51⁽²⁾	4.14⁽²⁾	36.6⁽²⁾
		%RPD Sample/Sample Dup	16	11	7.7
					0.327
					0.61

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 29%, PFBS \pm 17%, PFHS \pm 21%, and PFOS \pm 28%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 19%, PFBS \pm 16%, PFHS \pm 18%, PFOS \pm 16%, and PFOSA \pm 12%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of $\leq 20\%$.

Table 1 continued. Sample Results Summary⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Blokkersdijk Nature Reserve					
ISO19-14-04-010	P114bis; Sample	3.54	1.70	9.32	0.0492
ISO19-14-04-010-DUP	P114bis; Sample Dup	3.94	1.73	9.68	0.0684
		Average	3.74⁽²⁾	1.72⁽²⁾	9.50⁽²⁾
		%RPD Sample/Sample Dup	11	1.7	3.8
ISO19-14-04-011	P115; Sample	4.08	2.34	2.80	0.0458
ISO19-14-04-011-DUP	P115; Sample Dup	4.10	2.30	2.92	0.0500
		Average	4.09⁽²⁾	2.32⁽²⁾	2.86⁽²⁾
		%RPD Sample/Sample Dup	0.49	1.7	4.2
ISO19-14-04-012	P116; Sample	0.930	0.568	8.40	0.119
ISO19-14-04-012-DUP	P116; Sample Dup	1.05	0.604	9.84	0.125
		Average	0.990⁽²⁾	0.586⁽²⁾	9.12⁽²⁾
		%RPD Sample/Sample Dup	12	6.1	16
Zone: Effluent WWTP					
ISO19-14-04-013	Effluent WWTP; Sample	7.76	6.18	7.16	0.137
ISO19-14-04-013-DUP	Effluent WWTP; Sample Dup	7.90	6.04	7.02	0.130
		Average	7.83⁽²⁾	6.11⁽²⁾	7.09⁽²⁾
		%RPD Sample/Sample Dup	1.8	2.3	2.0
Zone: Palingbeek & Tophatgracht					
ISO19-14-04-024	12; Sample	53.2	17.8	167	0.220
ISO19-14-04-024-DUP	12; Sample Dup	53.8	17.0	166	0.244
		Average	53.5⁽²⁾	17.4⁽²⁾	167
		%RPD Sample/Sample Dup	1.1	4.6	0.60
ISO19-14-04-025	13; Sample	36.9	13.0	167	0.204
ISO19-14-04-025-DUP	13; Sample Dup	37.1	13.1	166	0.214
		Average	37.0	13.1	167
		%RPD Sample/Sample Dup	0.54	0.77	0.60
ISO19-14-04-026	5; Sample	26.2	25.4	53.2	0.163
ISO19-14-04-026-DUP	5; Sample Dup	25.4	23.8	47.2	0.122
		Average	25.8⁽²⁾	24.6⁽²⁾	50.2⁽²⁾
		%RPD Sample/Sample Dup	3.1	6.5	12

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 29%, PFBS \pm 17%, PFHS \pm 21%, and PFOS \pm 28%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 19%, PFBS \pm 16%, PFHS \pm 18%, PFOS \pm 16%, and PFOSA \pm 12%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Palingbeek & Tophatgracht					
ISO19-14-04-027	Bemalingstation; Sample	24.6	23.4	49.8	0.159
ISO19-14-04-027-DUP	Bemalingstation; Sample Dup	25.2	25.0	51.4	0.138
		Average	24.9⁽²⁾	24.2⁽²⁾	50.6⁽²⁾
		%RPD Sample/Sample Dup	2.4	6.6	3.2
Zone: Sewer					
ISO19-14-04-028	Collector put; Sample	34.9	17.2	127	12.7
ISO19-14-04-028-DUP	Collector put; Sample Dup	34.5	17.4	123	14.7
		Average	34.7	17.3	125
		%RPD Sample/Sample Dup	1.2	1.2	3.2

		Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Extraction Wells P&T						
ISO19-14-04-014	PP02; Sample	552	18.2	4780	17900	30.2
ISO19-14-04-014-DUP	PP02; Sample Dup	552	17.3	4790	17600	31.4
		Average	552	17.8	4790	17800
		%RPD Sample/Sample Dup	0.0	5.1	0.21	1.7
ISO19-14-04-015	PP04; Sample	1140	986	397	2530	113
ISO19-14-04-015-DUP	PP04; Sample Dup	1160	992	400	2560	106
		Average	1150	989	399	2550
		%RPD Sample/Sample Dup	1.7	0.61	0.75	1.2
ISO19-14-04-016	PP05; Sample	653	4600	2670	92.4	9.28
ISO19-14-04-016-DUP	PP05; Sample Dup	638	4550	2650	127	9.12
		Average	646	4580	2660	110
		%RPD Sample/Sample Dup	2.3	1.1	0.75	32⁽³⁾
ISO19-14-04-017	PP06; Sample	256	46.6	52.2	1660	67.0
ISO19-14-04-017-DUP	PP06; Sample Dup	252	46.0	51.1	1640	63.8
		Average	254	46.3	51.7	1650
		%RPD Sample/Sample Dup	1.6	1.3	2.1	1.2
ISO19-14-04-018	PP07; Sample	150	78.4	49.5	1400	64.6
ISO19-14-04-018-DUP	PP07; Sample Dup	149	80.1	49.5	1380	64.2
		Average	150	79.3	49.5	1390
		%RPD Sample/Sample Dup	0.67	2.1	0.0	1.4
ISO19-14-04-019	PP08; Sample	527	21.5	217	4980	57.6
ISO19-14-04-019-DUP	PP08; Sample Dup	537	21.3	215	4750	58.4
		Average	532	21.4	216	4870
		%RPD Sample/Sample Dup	1.9	0.93	0.93	4.7

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 29%, PFBS ± 17%, PFHS ± 21%, and PFOS ± 28%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 19%, PFBS ± 16%, PFHS ± 18%, PFOS ± 16%, and PFOSA ± 12%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 1 continued. Sample Results Summary⁽¹⁾

		Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Extraction Wells P&T						
ISO19-14-04-020	PP10; Sample	453	8.91	169	3240	23.8
ISO19-14-04-020-DUP	PP10; Sample Dup	446	8.75	169	3100	22.4
Average		450	8.83	169	3170	23.1
%RPD Sample/Sample Dup		1.6	1.8	0.0	4.4	6.1
ISO19-14-04-021	PP11; Sample	312	137	127	1780	33.4
ISO19-14-04-021-DUP	PP11; Sample Dup	316	140	130	1680	30.2
Average		314	139	129	1730	31.8
%RPD Sample/Sample Dup		1.3	2.2	2.3	5.8	10
Zone: Source Area – Building 16						
ISO19-14-04-029	P21B; Sample	6430	5320	8790	67600	5.36
ISO19-14-04-029-DUP	P21B; Sample Dup	6120	5070	8430	66200	5.98
Average		6275	5200	8610	66900	5.67
%RPD Sample/Sample Dup		4.9	4.8	4.2	2.1	11
		Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
ISO19-14-04-030	Travel Blank	<0.0480 ⁽²⁾	<0.100 ⁽²⁾	<0.0250 ⁽²⁾	<0.0232 ⁽²⁾	<0.0250

NA = Not Applicable

- (1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 29%, PFBS ± 17%, PFHS ± 21%, and PFOS ± 28%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 19%, PFBS ± 16%, PFHS ± 18%, PFOS ± 16%, and PFOSA ± 12%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M EHS Laboratory method ETS-8-044.3 “Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis”.

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected on October 21 – October 22, 2019, in Nalgene™ (high-density polyethylene) bottles prepared at the 3M EHS Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. All sample locations were spiked with a mass-labeled internal standard mix at a nominal concentration of 1 ng/mL and select samples were fortified with surrogate recovery standards (SRSs) at a nominal concentration of 0.1 ng/mL prior to sample collection. Samples were received on October 28, 2019.

2.3 Sample Preparation

Sample locations pre-spiked with internal standard and/or surrogates were analyzed for all analytes (PFBS for select locations) by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples along with the method blanks.

Samples requiring dilutions were prepared for PFOA, PFHS, PFOS and PFBS (select locations only) using a direct injection by methanol dilution method. Samples that required a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples that required a 1:100 dilution were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol. Samples that required a 1:500 dilution were prepared by diluting 0.02 mL of a well-mixed sample with 10.0 mL of methanol. Samples that required a 1:1000 dilution were prepared by first diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol, followed by a second dilution prepared by diluting 1 mL of the diluted sample into 9 mL of methanol. Diluted samples and LCSs were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

2.4 Analysis

All samples and quality control samples were analyzed for the target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). SRSs were added and included in the analysis of these samples. Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.2. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

The following analytical runs were used to report the results herein:

3/25/20 (ETS Athena) External Standard Calibration Analysis:

- All sample locations were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported **except** for the following sample locations: 3M vijver, Blokkersdijkvijver Noord, Blokkersdijkvijver standard, L21, L22, L31, L4, P114bis, P115, P116, Effluent WWTP, 5, Bemalingstation for all analytes. 12 for PFOA and PFHS. 13 and P21B for PFOS.

3/26/20 (ETS Tesla) Internal Standard Calibration Analysis:

- Select sample locations were analyzed for PFOSA. All sample results were reported.

3/27/20 (ETS Athena) Internal Standard Calibration Analysis:

- Select sample locations were analyzed for PFOA, PFHS, PFOS, and PFOSA. All sample results were reported, **except** for the following sample locations: 13 and Collector put for PFOA, PFHS and PFOS. 12 for PFOS

3/31/20 (ETS Kirk) External Standard Calibration Analysis:

- All sample locations were analyzed for PFOS. All sample results were reported.

Table 3. Instrument Parameters.

Instrument Name	ETS Athena	ETS Kirk	ETS Tesla
Liquid Chromatograph	Agilent 1260	Agilent 1260	Agilent 1260
Analysis Method	ETS-8-044.3	ETS-8-044.3	ETS-8-044.3
Analysis Date	3/25/20, 3/27/20	3/31/20	3/26/20
Guard column	Prism RP (2.1 mm X 50 mm), 5 μ	Betasil C8 (2.1 mm X 50 mm), 5 μ	Betasil C8 (2.1 mm X 50 mm), 5 μ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ
Injection Volume	5 μ L or 15 μ L	5 μ L	5 μ L
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 6500
Ion Source	Turbo Spray	Turbo Spray	Turbo Spray
Polarity	Negative	Negative	Negative
Software	Analyst 1.7.1	Analyst 1.7.1	Analyst 1.7.1

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis				
Step Number	Total Time (min)	Flow Rate (μ L/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0.00	300	90.0	10.0
1	0.50	300	90.0	10.0
2	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard	Mass Transition Q1/Q3
PFOA	413/369	[¹³ C ₈]-PFOA	421/376
	413/219		
	413/169		
PFBS	299/99	[¹⁸ O ₂]-PFBS	303/84
	299/80		
PFHS	399/99	[¹³ C ₃]-PFHS	402/80
	399/80		
PFOS	499/99	[¹³ C ₈]-PFOS	507/80
	499/80		
	499/130		
PFOSA	498/78	[¹³ C ₈]-PFOSA	506/78
[¹³ C ₄]-PFOA	417/372	[¹³ C ₈]-PFOA	421/376
[¹³ C ₄]-PFOS	503/80	[¹³ C ₈]-PFOS	507/80
The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.			
Internal standards were not used for samples analyzed on 3/25/20 and 3/31/20.			

3 Data Analysis

3.1 Calibration

3/26/20 and 3/27/20 Analysis (Internal Standard Calibration): Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of fourteen calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed. The calibration standards also contained surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

3/25/20 and 3/31/20 Analysis (External Standard Calibration): Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL to 25 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±25% (100±30% for the lowest curve point) were met for all analytes. The correlation coefficient (*r*) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.3 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the **Table 6** below.

Table 6. LOQ

Analyte	LOQ, ng/mL 3/25/20 Analysis			LOQ, ng/mL 3/26/20 Analysis	LOQ, ng/mL 3/27/20 Analysis	LOQ, ng/mL 3/31/20 Analysis	
	Dilution Factor			Dilution Factor	Dilution Factor	Dilution Factor	
	10-fold	100-fold	500-fold	2-fold	2-fold	10-fold	1000-fold
PFOA	0.0479	4.79	24.0	NA	0.0480	NA	NA
PFBS	0.200	2.00	10.0	NA	0.100	NA	NA
PFHS	0.200	2.00	10.0	NA	0.0250	NA	NA
PFOS	0.464	4.64	23.3	NA	0.0232	0.464	46.4
PFOSA	NA	NA	NA	0.0500	0.0250	NA	NA

NA = Not Applicable

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of $100\%\pm 25\%$.

3.5 Blanks

Two types of blanks were prepared and analyzed with the samples: method/solvent blanks and field/trip blanks. Each blank result was reviewed and used to evaluate method performance. The method blanks were used to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. LCSs were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. Surrogates [$^{13}\text{C}_4$]-PFOA and [$^{13}\text{C}_4$]-PFOS were added post dilution when analyzed by external standard. The target analyte LCSs were spiked at the following nominal concentrations:

- LCSs analyzed on 3/25/20 were prepared at 100 ng/mL, 5000 ng/mL, and 35000 ng/mL.
- LCSs analyzed on 3/26/20 were prepared at 0.20 ng/mL, 20.0 ng/mL, and 140 ng/mL.
- LCSs analyzed on 3/27/20 were prepared at 0.20 ng/mL, 20.0 ng/mL, and 140 ng/mL.
- LCSs analyzed on 3/31/20 were prepared at 200 ng/mL, 20000 ng/mL, and 70000 ng/mL.

Method ETS-8-044.3 states that the average recovery of LCSs at each spiking level must be within 80%-120% with a RSD $\leq 20\%$. All LCS samples met criteria with the following exceptions:

- 3/27/19: High-level LCSs for PFOSA were spiked above the resulting ULOQ with dilution factor of 2 applied.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report. Individual LCS results are included in the raw data and available upon request.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.5. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in **Table 7** below.

Table 7. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	14.4	±29%
PFBS	External	8.33	±17%
PFHS	External	10.6	±21%
PFOS	External	13.8	±28%
PFOA	Internal	9.59	±19%
PFBS	Internal	7.84	±16%
PFHS	Internal	9.24	±18%
PFOS	Internal	8.22	±16%
PFOSA	Internal	6.02	±12%

3.8 Laboratory Matrix Spikes (LMS)

Initial analysis of sample set for location 13 produced a non-compliant FMS recovery for PFOS. The sample set for this location was re-prepared along with a target analyte laboratory matrix spike to verify that an analytical method is applicable for the collected matrix. The target analyte laboratory matrix spike was generated by adding a measured volume of spiking solution to an aliquot of the well mixed field sample following sample collection for a spike concentration of 1.0 ng/mL. Target analyte laboratory matrix spikes must be at least 0.5 times the analyte concentration to be considered an appropriate spike level. Target analyte laboratory matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the extraction and analysis of the analytes of interest. Re-analysis of the sample set produced an acceptable FMS and LMS recovery. The laboratory matrix spike result is included in section 4 of this report.

3.9 Field Matrix Spikes (FMS)

Target analyte field matrix spike samples were collected for select sampling points to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions at 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOA and PFOS. The standards used for the preparation of the field matrix spiking solutions greater than 10 ng/mL (nominal) contained reference

materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, select sample bottles contained stable isotope surrogate recovery spikes of [¹³C₄]-PFOA and [¹³C₄]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles prior to sample collection. The [¹³C₄]-PFOA was selected to represent the C8 perfluorocarboxylic acid. The [¹³C₄]-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

The following calculation was used to generate data in section 4 of the report.

$$\text{FMS Recovery} = \frac{\text{Sample Concentration of FMS} - \text{Average Concentration (Field Sample & Field Sample Dup)}}{\text{Spike Concentration}} \times 100\%$$

Table 8. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
3M vijver, L22	FMS	4.79	5.00	5.00	4.64	5.00
13, Collector Put	FMS	260	260	260	259	9.99
PP04, PP11	FMS	1048	1050	1050	1046	50.0
Trip Blank	Low	4.79	5.00	5.00	4.64	5.00
	High	1048	1050	1050	1046	50.0

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of ±30%, demonstrate that the method is appropriate for the given matrix.

The method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. At times, the spike level exceeded the recommended upper limit of 10 times the analyte concentration. In these instances, the FMS recovery was reported and flagged as above 10 times the sample concentration.

For PFOA, PFBS, PFHS, and PFOS, when the field matrix spike level was not appropriate as compared to the endogenous sample concentration, the surrogate recovery standards were used to assess method accuracy. All field matrix spike and surrogate recovery standard recoveries met acceptance criteria with the following exception:

Sample Location 13: The initial analysis of PFOS had an FMS recovery of 67.7%. The sample was re-prepared with an LMS to verify the non-compliant FMS recovery. The LMS met acceptance criteria of 100±30%, demonstrating that the method is appropriate for the sample matrix. The re-prepared FMS also met acceptance criteria; therefore, the data uncertainty will not be increased for the sample location.

Table 9. Location ID: 3M vijver

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-04-002	3M vijver; Sample	0.616	NA	0.362	NA
ISO19-14-04-002-DUP	3M vijver; Sample Duplicate	0.544	NA	0.368	NA
ISO19-14-04-002-FMS	3M vijver; Sample FMS	6.00	113	5.60	105 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		0.580 ng/mL ± 12%		0.365 ng/mL ± 1.6%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-04-002	3M vijver; Sample	1.77	NA	<0.0250	NA
ISO19-14-04-002-DUP	3M vijver; Sample Duplicate	1.58	NA	<0.0250	NA
ISO19-14-04-002-FMS	3M vijver; Sample FMS	6.90	113	4.90	98.0 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		1.68 ng/mL ± 11%		<0.0250 ng/mL	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 10. Location ID: L22

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-04-007	L22; Sample	0.304	NA	0.284	NA
ISO19-14-04-007-DUP	L22; Sample Duplicate	0.404	NA	0.340	NA
ISO19-14-04-007-FMS	L22; Sample FMS	5.50	107 ⁽¹⁾	5.74	109 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		0.354 ng/mL ± 28% ⁽²⁾		0.312 ng/mL ± 18%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-04-007	L22; Sample	3.34	NA	0.120	NA
ISO19-14-04-007-DUP	L22; Sample Duplicate	2.60	NA	0.172	NA
ISO19-14-04-007-FMS	L22; Sample FMS	8.06	110	5.18	101 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		2.97 ng/mL ± 25% ⁽²⁾		0.146 ng/mL ± 36% ⁽²⁾	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 11. Location ID: 13

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-04-025	13; Sample	36.9	NA	13.0	NA
ISO19-14-04-025-DUP	13; Sample Duplicate	37.1	NA	13.1	NA
ISO19-14-04-025-FMS	13; Sample FMS	228	73.6	218	78.8
Average Concentration (ng/mL) ± %RPD		37.0 ng/mL ± 0.54%		13.1 ng/mL ± 0.77%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-04-025	13; Sample	167	NA	0.204	NA
ISO19-14-04-025-DUP	13; Sample Duplicate	166	NA	0.214	NA
ISO19-14-04-025-FMS	13; Sample FMS	377	81.2	10.1	99.0 ⁽¹⁾
ISO19-14-04-025-LMS	13; Sample LMS	391	86.6	NA	NA
Average Concentration (ng/mL) ± %RPD		167 ng/mL ± 0.60%		0.209 ng/mL ± 4.8%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 12. Location ID: Collector put

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-04-028	Collector put; Sample	34.9	NA	17.2	NA
ISO19-14-04-028-DUP	Collector put; Sample Duplicate	34.5	NA	17.4	NA
ISO19-14-04-028-FMS	Collector put; Sample FMS	226	73.7	221	78.3
Average Concentration (ng/mL) ± %RPD		0.34.7 ng/mL ± 1.2%		17.3 ng/mL ± 1.2%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-04-028	Collector put; Sample	127	NA	12.7	NA
ISO19-14-04-028-DUP	Collector put; Sample Duplicate	123	NA	14.7	NA
ISO19-14-04-028-FMS	Collector put; Sample FMS	311	71.7	21.2	75.1
Average Concentration (ng/mL) ± %RPD		127 ng/mL ± 3.2%		13.7 ng/mL ± 15%	

NA = Not Applicable

Table 13. Location ID: PP04

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-04-015	PP04; Sample	1140	NA	986	NA	397	NA
ISO19-14-04-015-DUP	PP04; Sample Duplicate	1160	NA	992	NA	400	NA
ISO19-14-04-015-FMS	PP04; Sample FMS	2000	81.1	1830	80.1	1290	84.9
Average Concentration (ng/mL) ± %RPD		1150 ng/mL ± 1.7%		989 ng/mL ± 0.61%		399 ng/mL ± 0.75%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-04-015	PP04; Sample	2530	NA	113	NA
ISO19-14-04-015-DUP	PP04; Sample Duplicate	2560	NA	106	NA
ISO19-14-04-015-FMS	PP04; Sample FMS	3290	NC	152	NC
Average Concentration (ng/mL) ± %RPD		2550 ng/mL ± 1.2%		110 ng/mL ± 6.4%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 14. Location ID: PP11

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-04-021	PP11; Sample	312	NA	137	NA	127	NA
ISO19-14-04-021-DUP	PP11; Sample Duplicate	316	NA	140	NA	130	NA
ISO19-14-04-021-FMS	PP11; Sample FMS	1140	78.8	957	78.0	966	79.8
Average Concentration (ng/mL) ± %RPD		314 ng/mL ± 1.3%		139 ng/mL ± 2.2%		129 ng/mL ± 2.3%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-04-021	PP11; Sample	1780	NA	33.4	33.4
ISO19-14-04-021-DUP	PP11; Sample Duplicate	1680	NA	30.2	30.2
ISO19-14-04-021-FMS	PP11; Sample FMS	2530	76.5	82.6	82.6
Average Concentration (ng/mL) ± %RPD		1730 ng/mL ± 5.8%		31.8 ng/mL ± 10%	

NA = Not Applicable

Table 15. Location ID: Travel Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-04-030	Travel Blank	<0.0480	NA	<0.100	NA	<0.0250	NA
ISO19-14-04-030-FMS-LOW	Travel Blank FMS Low	4.94	103	5.20	104	5.32	106
ISO19-14-04-030-FMS-HIGH	Travel Blank FMS High	836	79.8	850	81.0	905	86.2

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO19-14-04-030	Travel Blank	<0.0232	NA	<0.0250	NA
ISO19-14-04-030-FMS-LOW	Travel Blank FMS Low	5.00	108	4.94	98.8
ISO19-14-04-030-FMS-HIGH	Travel Blank FMS High	880	84.1	52.8	106

NA = Not Applicable

Table 16. Surrogate Recovery Standard Results

3M LIMS ID	Sample Description	3/25/20 Analysis Percent Recovery (%) ⁽¹⁾	
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS
ISO19-14-04-001	D16; Sample	82.9	91.0
ISO19-14-04-001-DUP	D16; Sample Duplicate	80.8	92.3
ISO19-14-04-005	P321; Sample	86.3	90.8
ISO19-14-04-005-DUP	P321; Sample Duplicate	85.8	92.6
ISO19-14-04-014	PP02; Sample	86.0	92.5
ISO19-14-04-014-DUP	PP02; Sample Duplicate	86.3	92.3
ISO19-14-04-015	PP04; Sample	83.0	93.8
ISO19-14-04-015-DUP	PP04; Sample Duplicate	86.6	95.3
ISO19-14-04-015-FMS	PP04; Sample FMS	85.2	95.0
ISO19-14-04-016	PP05; Sample	85.1	91.8
ISO19-14-04-016-DUP	PP05; Sample Duplicate	82.3	88.8
ISO19-14-04-017	PP06; Sample	88.2	96.5
ISO19-14-04-017-DUP	PP06; Sample Duplicate	88.2	98.4
ISO19-14-04-018	PP07; Sample	92.5	101.9
ISO19-14-04-018-DUP	PP07; Sample Duplicate	96.4	105.8
ISO19-14-04-019	PP08; Sample	91.1	100.7
ISO19-14-04-019-DUP	PP08; Sample Duplicate	89.3	92.7
ISO19-14-04-020	PP10; Sample	89.9	99.8
ISO19-14-04-020-DUP	PP10; Sample Duplicate	87.3	97.7
ISO19-14-04-021	PP11; Sample	88.2	100.1
ISO19-14-04-021-DUP	PP11; Sample Duplicate	92.2	96.3
ISO19-14-04-021-FMS	PP11; FMS	89.5	98.8
ISO19-14-04-024	12; Sample ⁽²⁾	NA	91.3
ISO19-14-04-024-DUP	12; Sample Duplicate ⁽²⁾	NA	91.7
ISO19-14-04-025	13; Sample ⁽²⁾	82.4	89.9
ISO19-14-04-025-DUP	13; Sample Duplicate ⁽²⁾	83.4	92.3
ISO19-14-04-025-FMS	13; Sample FMS ⁽²⁾	80.4	90.2
ISO19-14-04-028	Collector put; Sample ⁽²⁾	78.9	89.3
ISO19-14-04-028-DUP	Collector put; Sample Duplicate ⁽²⁾	78.5	88.6
ISO19-14-04-028-FMS	Collector put; Sample FMS ⁽²⁾	80.3	92.1
ISO19-14-04-029	P21B; Sample	82.1	90.8
ISO19-14-04-029-DUP	P21B; Sample Duplicate	82.8	93.3
ISO19-14-04-030-FMS-HIGH	Travel Blank FMS High	84.1	94.2

NA = Not Applicable

- (1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.
- (2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.
- (3) Surrogate recovery did not meet method acceptance criteria of 100±30%.

Table 16 continued. Surrogate Recovery Standard Results

3M LIMS ID	Sample Description	3/27/20 Analysis Percent Recovery (%)	
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS
ISO19-14-04-002	3M vijver; Sample	117	97.6
ISO19-14-04-002-DUP	3M vijver; Sample Duplicate	96.0	95.7
ISO19-14-04-002-FMS	3M vijver; Sample FMS	111	97.2
ISO19-14-04-003	Blokkersdijkvijver Noord; Sample	104	102
ISO19-14-04-003-DUP	Blokkersdijkvijver Noord; Sample Duplicate	110	105
ISO19-14-04-004	Blokkersdijkvijver standaard; Sample	110	110
ISO19-14-04-004-DUP	Blokkersdijkvijver standaard; Sample Duplicate	104	110
ISO19-14-04-006	L21; Sample	101	97.8
ISO19-14-04-006-DUP	L21; Sample Duplicate	105	94.7
ISO19-14-04-007	L22; Sample	106	110
ISO19-14-04-007-DUP	L22; Sample Duplicate	108	104
ISO19-14-04-007-FMS	L22; Sample FMS	134 ⁽³⁾	95.5
ISO19-14-04-008	L31; Sample	110	99.9
ISO19-14-04-008-DUP	L31; Sample Duplicate	112	97.6
ISO19-14-04-009	L4; Sample	103	100
ISO19-14-04-009-DUP	L4; Sample Duplicate	89.6	108
ISO19-14-04-010	P114bis; Sample	94.0	101
ISO19-14-04-010-DUP	P114bis; Sample Duplicate	122	96.1
ISO19-14-04-011	P115; Sample	114	90.7
ISO19-14-04-011-DUP	P115; Sample Duplicate	102	101
ISO19-14-04-012	P116; Sample	104	84.4
ISO19-14-04-012-DUP	P116; Sample Duplicate	102	101
ISO19-14-04-013	Effluent WWTP; Sample	93.8	96.1
ISO19-14-04-013-DUP	Effluent WWTP; Sample Duplicate	107	85.2
ISO19-14-04-024	12; Sample	96.8	103
ISO19-14-04-024-DUP	12; Sample Duplicate	98.6	94.0
ISO19-14-04-026	5; Sample	106	94.5
ISO19-14-04-026-DUP	5; Sample Duplicate	112	107
ISO19-14-04-027	Bemalingstation; Sample	108	108
ISO19-14-04-027-DUP	Bemalingstation; Sample Duplicate	99.2	108
ISO19-14-04-030	Travel Blank	120	106
ISO19-14-04-030-FMS-LOW	Travel Blank FMS Low	102	108

NA = Not Applicable

- (1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.
- (2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.
- (3) Surrogate recovery did not meet method acceptance criteria of 100±30%.

Table 16 continued. Surrogate Recovery Standard Results

3M LIMS ID	Sample Description	3/31/20 Analysis Percent Recovery (%)
ISO19-14-04-025	13; Sample	105
ISO19-14-04-025-DUP	13; Sample Duplicate	104
ISO19-14-04-025-FMS	13; Sample FMS	97.4
ISO19-14-04-025-LMS-595085	13; Sample LMS	95.1
ISO19-14-04-029	P21B; Sample	104
ISO19-14-04-029-DUP	P21B; Sample Duplicate	97.5

NA = Not Applicable

- (1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.
- (2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.
- (3) Surrogate recovery did not meet method acceptance criteria of 100±30%.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 9-16 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M EHS Laboratory standard operating procedures.

7 Attachments

Chain of Custody Form

8 Signatures

Scott Porcher, 3M Report Author

Susan T. Wolf, 3M Principal Analytical Investigator

Brian T. Mader, Ph.D., 3M EHS Laboratory Manager

The 3M EHS Laboratory's Quality Assurance Unit has audited the data and report for this project.

Quality Assurance Representative

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ANALYTICAL REPORT

IAC19-09670_revision

The present document voids and replaces the previously issued report with the same reference.

3M Lab Request Number: E19-0772

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

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1. Introduction - Summary.

At the request (Lab Request Number: E19-0772) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected in January 23th and 24th 2020 from 3 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOA ($C_6F_{13}^{13}CF_2^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOS ($C_7F_{15}^{13}CF_2SO_3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Sven Herremans. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles.

Table 1 summarizes the sample results. All PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
Effluent WWTP	1.82	3.17	6.03	0.299	
Bemalingsstation	24.6	28.0	38.4	<0.246	
Collector Put	6.05	13.4	45.2	3.47	

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
Effluent WWTP	100
Bemalingsstation	100
Collector Put	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefloreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
Effluent WWTP	1.0	0.05	Solution A	1.0	-
Bemalingsstation	1.0	0.05	Solution A	1.0	-
Collector Put	1.0	0.05	Solution A	1.0	-

(*): MeOH:LCMS-water (60:40)
Solution A = 1000 ng/mL (nominal) ^{13}C -PFC Surrogate Standards

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetri C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
PFBS	2.08 - 2.50	298.93/79.82	40	28	45
		298.93/98.78	40	29	45
PFHS	2.45 - 3.05	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.80 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
¹³ C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of $\pm 25\%$ ($\pm 30\%$ for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.0730 to 0.120 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.150 to 0.246 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within $\pm 25\%$, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by diluting 1 mL water with 10.0 mL MeOH:LCMS-water (60:40). Method blanks were prepared to evaluate the levels of background contamination in the overall dilution process (glassware, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carryover.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of LCMS-water, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCS)

One lab control spike was prepared and analyzed.

LCS was prepared by spiking known amounts of the analytes into 1.0 mL of LCMS-water to produce the desired concentration.

The spiked water sample was diluted with 10.0 mL MeOH:LCMS-water (60:40) and analyzed in the same manner as the samples.

The LCS produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy, except for ^{13}C -PFOA and ^{13}C -PFOS.

Table 7 summarizes the LCS recovery results

Table 7. Lab Control Spike Results.

Dilution date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
20 February 2020	LCS1	80		93.8	78.4	72.3	119	102	130

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing PFBS, PFHS, PFOA, ^{13}C -PFOA, PFOS, FOSA and ^{13}C -PFOS (QC Lab Low and QC Lab High).

The QCs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 8 summarizes the QCs recovery results.

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C -PFOA	PFOS	FOSA	^{13}C -PFOS
7 Nov 2019	QC Lab Low inj1	10		94.7	98.4	124	116	99.1	114
	QC Lab High inj1	100		79.3	99.3	78.7	115	77.7	121
	QC Lab Low inj2	10		94.5	101	78.5	118	102	118
	QC Lab High inj2	100		82.7	102	76.4	119	80.4	120

3.7. Equations.Recovery.

$$\text{Recovery(%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

The following tables summarize the results for the sample locations.

Table 9. Sample Results PFBS, PFHS, PFOA, PFOS and FOSA

	PFBS		PFHS		PFOA	PFOS	FOSA
	ng/ml		ng/ml		ng/ml	ng/ml	ng/ml
Effluent WWTP			1.82		3.17	6.03	0.299
Bemalingsstation			24.6		28.0	38.4	<0.246
Collector Put			6.05		13.4	45.2	3.47
Field Trip Blank			<1.03		<1.18	<0.810	<1.33

Table 10. Sample Results ^{13}C -PFOA and ^{13}C -PFOS

	^{13}C -PFOA		^{13}C -PFOS	
	ng/ml	% Rec	ng/ml	% Rec
Effluent WWTP	50.5	86.3	62.3	99.4
Bemalingsstation	52.9	90.4	59.7	95.2
Collector Put	53.2	90.9	59.2	94.4
Field Trip Blank	82.7	70.6	156	125

5. Conclusion.

- The recovery of ¹³C-PFOA and ¹³C-PFOS fell within the method acceptance criterion of 70-130%.
- Lab control spike recoveries fell within the method acceptance criteria for all compounds, indicating that the analysis method is in control (except for ¹³C-PFOA and ¹³C-PFOS).
- QC Lab Standards recoveries fell within the method acceptance criteria for all compounds.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Julie Fichefet (ERM Belgium).

8. Signatures.

Sven Herremans,
Technical Manager

Date April 22nd, 2020



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date April 22nd, 2020

I.A.C.
A Division of SGS Belgium NV

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The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.

Final Report

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

January 2020 Sampling

Laboratory Request Number: ISO20-14-01

Report Date – Date of Last Signature

Testing Laboratory

3M EHS Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Charlotte Tack
3M Belgium
3M Belgium; ZW019/01/01
Phone: [REDACTED]



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2017 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M EHS Laboratory

3M EHS Laboratory Manager: Brian T. Mader
3M Principal Analytical Investigator: Susan Wolf
Report Author: Chelsie Grochow

Analytical Report ISO20-14-01

Water Sample Analysis at 3M Antwerp, Belgium
January 2020 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M EHS Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. All samples were collected between January 27 - 29, 2020, and returned to the 3M EHS Laboratory on February 3, 2020 or February 4, 2020, at ambient temperature. The results in this report apply to the samples as received from ERM. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS) (select locations only), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO20-14-01.

The 3M EHS Laboratory prepared sample containers for sixty-three sampling locations. Each sample set consisted of a field sample and field sample duplicate. Fourteen locations also included a target analyte field matrix spike. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with a mass-labeled internal standard mix and select sample bottles were fortified with surrogate recovery standards [$^{13}\text{C}_4$]-PFOA and [$^{13}\text{C}_4$]-PFOS prior to being sent to the field for sample collection. During sample collection, sample locations D09bis, P56, and PP13 were not sampled and sample location PP12 was changed to PP11 as indicated on the chain of custody.

Samples were prepared and analyzed using method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Select internal standards were used to aid in the data quality objectives.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2017 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary ⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: 2nd Aquifer					
ISO20-14-01-001	BD24-3; Sample	17.3	18.2	692	3.12
ISO20-14-01-001-DUP	BD24-3; Sample Dup	18.1	18.8	662	3.26
		Average	17.7 ⁽²⁾	18.5 ⁽²⁾	677
		%RPD Sample/Sample Dup	4.5	3.2	4.4
ISO20-14-01-002	BD24-4; Sample	146	54.0	1.31	0.268
ISO20-14-01-002-DUP	BD24-4; Sample Dup	145	53.5	1.30	0.276
		Average	146	53.8	1.31
		%RPD Sample/Sample Dup	0.69	0.93	0.77
ISO20-14-01-004	D10; Sample	165	57.1	25.8	0.133
ISO20-14-01-004-DUP	D10; Sample Dup	163	57.1	25.6	0.140
		Average	164	57.1	25.7
		%RPD Sample/Sample Dup	1.2	0.0	0.78
ISO20-14-01-005	D11; Sample	317	128	1160	21.6
ISO20-14-01-005-DUP	D11; Sample Dup	323	129	1260	23.8
		Average	320	129	1210
		%RPD Sample/Sample Dup	1.9	0.78	8.3
ISO20-14-01-006	D14; Sample	2.10	1.17	0.444	0.0326
ISO20-14-01-006-DUP	D14; Sample Dup	2.06	1.12	0.438	<0.0250
		Average	2.08 ⁽²⁾	1.15 ⁽²⁾	0.441 ⁽²⁾
		%RPD Sample/Sample Dup	1.9	4.4	1.4
ISO20-14-01-007	D16; Sample	801	320	2560	19.2
ISO20-14-01-007-DUP	D16; Sample Dup	776	317	2510	19.5
		Average	789	319	2540
		%RPD Sample/Sample Dup	3.2	0.94	2.0
ISO20-14-01-008	D17; Sample	383	68.0	399	0.278
ISO20-14-01-008-DUP	D17; Sample Dup	377	67.6	390	0.250
		Average	380	67.8	395
		%RPD Sample/Sample Dup	1.6	0.59	2.3
ISO20-14-01-009	D18; Sample	118	86.3	73.7	0.564
ISO20-14-01-009-DUP	D18; Sample Dup	116	86.5	73.0	0.548
		Average	117	86.4	73.4
		%RPD Sample/Sample Dup	1.7	0.23	0.95

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 21%, PFBS \pm 16%, PFHS \pm 18%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 17%, PFHS \pm 12%, PFOS \pm 17%, and PFOSA \pm 15%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: 2nd Aquifer					
ISO20-14-01-010	D5; Sample	150	50.1	232	2.40
ISO20-14-01-010-DUP	D5; Sample Dup	155	52.6	238	2.30
		Average	153	51.4	235
		%RPD Sample/Sample Dup	3.3	4.9	2.6
ISO20-14-01-011	ND7; Sample	195	36.7	145	4.90
ISO20-14-01-011-DUP	ND7; Sample Dup	192	36.2	145	5.08
		Average	194	36.5	145
		%RPD Sample/Sample Dup	1.6	1.4	0.0
ISO20-14-01-012	P118A; Sample	1280	1680	72.3	0.592
ISO20-14-01-012-DUP	P118A; Sample Dup	1290	1680	73.6	0.598
		Average	1290	1680	73.0
		%RPD Sample/Sample Dup	0.78	0.0	1.8
ISO20-14-01-013	P118B; Sample	1560	713	6330	2.44
ISO20-14-01-013-DUP	P118B; Sample Dup	1570	708	6380	2.48
		Average	1570	711	6360
		%RPD Sample/Sample Dup	0.64	0.70	0.79
ISO20-14-01-014	P119A; Sample	96.4	30.9	127	7.52
ISO20-14-01-014-DUP	P119A; Sample Dup	95.7	30.8	128	6.78
		Average	96.1	30.9	128
		%RPD Sample/Sample Dup	0.73	0.32	0.78
ISO20-14-01-015	P119B; Sample	678	262	217	0.216
ISO20-14-01-015-DUP	P119B; Sample Dup	672	255	215	0.208
		Average	675	259	216
		%RPD Sample/Sample Dup	0.89	2.7	0.93
ISO20-14-01-016	P121; Sample	0.794	0.542	0.372	0.0310
ISO20-14-01-016-DUP	P121; Sample Dup	0.706	0.486	0.336	0.0308
		Average	0.750 ⁽²⁾	0.514 ⁽²⁾	0.354 ⁽²⁾
		%RPD Sample/Sample Dup	12	11	10
ISO20-14-01-017	3M vijver; Sample	0.690	0.456	2.46	<0.0250
ISO20-14-01-017-DUP	3M vijver; Sample Dup	0.674	0.414	2.38	<0.0250
		Average	0.682 ⁽²⁾	0.435 ⁽²⁾	2.42 ⁽²⁾
		%RPD Sample/Sample Dup	2.3	9.7	3.3
ISO20-14-01-018	Blokkersdijkvijver Noord; Sample	0.924	0.554	2.12	0.0386
ISO20-14-01-018-DUP	Blokkersdijkvijver Noord; Sample Dup	0.942	0.554	2.08	0.0368
		Average	0.933 ⁽²⁾	0.554 ⁽²⁾	2.10 ⁽²⁾
		%RPD Sample/Sample Dup	1.9	0.0	1.9
					4.8

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 21%, PFBS \pm 16%, PFHS \pm 18%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 17%, PFHS \pm 12%, PFOS \pm 17%, and PFOSA \pm 15%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Blokkersdijk Nature Reserve					
ISO20-14-01-019	Blokkersdijkvijver standard; Sample	0.778	0.480	0.512	<0.0250
ISO20-14-01-019-DUP	Blokkersdijkvijver standard; Sample Dup	0.748	0.480	0.468	<0.0250
		Average %RPD Sample/Sample Dup	0.763 ⁽²⁾ 3.9	0.480 ⁽²⁾ 0.0	0.490 ⁽²⁾ 9.0
ISO20-14-01-020	P321; Sample	1430	360	4500	0.240
ISO20-14-01-020-DUP	P321; Sample Dup	1420	359	4470	0.240
		Average %RPD Sample/Sample Dup	1430 0.70	360 0.28	4490 0.67
ISO20-14-01-021	L21; Sample	0.148	0.0772	2.12	0.0362
ISO20-14-01-021-DUP	L21; Sample Dup	0.143	0.0852	2.14	0.0392
		Average %RPD Sample/Sample Dup	0.146 ⁽²⁾ 3.4	0.0812 ⁽²⁾ 9.9	2.13 ⁽²⁾ 0.94
ISO20-14-01-022	L22; Sample	0.324	0.296	2.38	0.0368
ISO20-14-01-022-DUP	L22; Sample Dup	0.342	0.302	2.42	0.0370
		Average %RPD Sample/Sample Dup	0.333 ⁽²⁾ 5.4	0.299 ⁽²⁾ 2.0	2.40 ⁽²⁾ 1.7
ISO20-14-01-023	L31; Sample	0.256	0.606	4.74	0.173
ISO20-14-01-023-DUP	L31; Sample Dup	0.276	0.616	4.40	0.125
		Average %RPD Sample/Sample Dup	0.266 ⁽²⁾ 7.5	0.611 ⁽²⁾ 1.6	4.57 ⁽²⁾ 7.4
ISO20-14-01-024	L4; Sample	2.96	1.97	17.3	0.348
ISO20-14-01-024-DUP	L4; Sample Dup	2.89	1.99	17.7	0.318
		Average %RPD Sample/Sample Dup	2.93 2.4	1.98 1.0	17.5 2.3
ISO20-14-01-025	P114bis; Sample	3.44	1.35	5.06	<0.0250
ISO20-14-01-025-DUP	P114bis; Sample Dup	3.44	1.34	5.00	<0.0250
		Average %RPD Sample/Sample Dup	3.44 0.0	1.35 0.74	5.03 1.2
ISO20-14-01-026	P115; Sample	3.31	1.70	1.88	<0.0250
ISO20-14-01-026-DUP	P115; Sample Dup	3.31	1.71	1.96	<0.0250
		Average %RPD Sample/Sample Dup	3.31 0.0	1.71 0.59	1.92 4.2
ISO20-14-01-027	P116; Sample	0.712	0.428	8.48	0.0566
ISO20-14-01-027-DUP	P116; Sample Dup	0.720	0.452	8.68	0.0536
		Average %RPD Sample/Sample Dup	0.716 ⁽²⁾ 1.1	0.440 ⁽²⁾ 5.5	8.58 ⁽²⁾ 2.3
					0.0551 5.4

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 21%, PFBS \pm 16%, PFHS \pm 18%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 17%, PFHS \pm 12%, PFOS \pm 17%, and PFOSA \pm 15%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Effluent WWTP					
ISO20-14-01-028	Effluent WWTP; Sample	2.68	1.52	6.80	0.266
ISO20-14-01-028-DUP	Effluent WWTP; Sample Dup	2.66	1.52	6.87	0.256
Average		2.67	1.52	6.84	0.261
%RPD Sample/Sample Dup		0.75	0.0	1.0	3.8

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Extraction Wells						
ISO20-14-01-029	PP02; Sample	622	27.3	4690	22600	19.9
ISO20-14-01-029-DUP	PP02; Sample Dup	587	27.2	4790	21200	20.8
Average		605	27.3	4740	21900	20.4
%RPD Sample/Sample Dup		5.8	0.37	2.1	6.4	4.4
ISO20-14-01-030	PP04; Sample	415	804	164	4230	44.4
ISO20-14-01-030-DUP	PP04; Sample Dup	416	803	162	4290	44.6
Average		416	804	163	4260	44.5
%RPD Sample/Sample Dup		0.24	0.12	1.2	1.4	0.45
ISO20-14-01-031	PP05; Sample	954	3690	2920	191	18.0
ISO20-14-01-031-DUP	PP05; Sample Dup	999	3920	3010	171	16.1
Average		977	3810	2970	181	17.1
%RPD Sample/Sample Dup		4.6	6.0	3.0	11	11
ISO20-14-01-032	PP06; Sample	191	19.9	51.3	987	48.2
ISO20-14-01-032-DUP	PP06; Sample Dup	194	20.1	50.9	977	53.0
Average		193	20.0	51.1	982	50.6
%RPD Sample/Sample Dup		1.6	1.0	0.78	1.0	9.5
ISO20-14-01-033	PP07; Sample	404	403	139	2200	63.6
ISO20-14-01-033-DUP	PP07; Sample Dup	398	388	132	2100	62.6
Average		401	396	136	2150	63.1
%RPD Sample/Sample Dup		1.5	3.8	5.2	4.7	1.6
ISO20-14-01-034	PP08; Sample	542	29.9	268	5350	38.4
ISO20-14-01-034-DUP	PP08; Sample Dup	543	31.0	272	5550	39.0
Average		543	30.5	270	5450	38.7
%RPD Sample/Sample Dup		0.18	3.6	1.5	3.7	1.6
ISO20-14-01-035	PP10; Sample	505	11.8	183	3230	20.2
ISO20-14-01-035-DUP	PP10; Sample Dup	503	13.0	186	3260	19.5
Average		504	12.4	185	3250	19.9
%RPD Sample/Sample Dup		0.40	9.7	1.6	0.92	3.5

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 21%, PFBS \pm 16%, PFHS \pm 18%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 17%, PFHS \pm 12%, PFOS \pm 17%, and PFOSA \pm 15%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary ⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Palingbeek & Tophatgracht					
ISO20-14-01-036	12; Sample	45.2	13.3	192	0.218
ISO20-14-01-036-DUP	12; Sample Dup	44.9	13.6	188	0.180
		Average	45.1	13.5	190
		%RPD Sample/Sample Dup	0.67	2.2	2.1
ISO20-14-01-037	13; Sample	43.6	14.3	176	0.169
ISO20-14-01-037-DUP	13; Sample Dup	42.9	13.7	172	0.176
		Average	43.3	14.0	174
		%RPD Sample/Sample Dup	1.6	4.3	2.3
ISO20-14-01-038	5; Sample	31.1	26.6	51.0	0.133
ISO20-14-01-038-DUP	5; Sample Dup	30.6	26.3	49.8	0.136
		Average	30.9	26.5	50.4
		%RPD Sample/Sample Dup	1.6	1.1	2.4
ISO20-14-01-039	Bemalingsstation; Sample	28.3	24.2	43.6	0.120
ISO20-14-01-039-DUP	Bemalingsstation; Sample Dup	27.9	23.4	44.6	0.126
		Average	28.1	23.8	44.1
		%RPD Sample/Sample Dup	1.4	3.4	2.3
Zone: Sewer					
ISO20-14-01-040	Collector put; Sample	13.6	6.33	50.8	5.14
ISO20-14-01-040-DUP	Collector put; Sample Dup	13.3	6.03	48.4	4.88
		Average	13.5	6.18	49.6
		%RPD Sample/Sample Dup	2.2	4.9	4.8

		Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - Building 16						
ISO20-14-01-041	P27; Sample	193	1460	14.1	1400	62.0
ISO20-14-01-041-DUP	P27; Sample Dup	195	1520	14.1	1410	55.6
		Average	194	1490	14.1	1410
		%RPD Sample/Sample Dup	1.0	4.0	0.0	0.71
		11				
ISO20-14-01-042	P21B; Sample	8790	6630	10900	67600	15.0
ISO20-14-01-042-DUP	P21B; Sample Dup	8680	6580	10900	70200	16.3
		Average	8740	6610	10900	68900
		%RPD Sample/Sample Dup	1.3	0.76	0.0	3.8
		8.3				
ISO20-14-01-043	P304; Sample	395	303	115	1030	13.7
ISO20-14-01-043-DUP	P304; Sample Dup	387	306	115	1010	13.5
		Average	391	305	115	1020
		%RPD Sample/Sample Dup	2.0	0.99	0.0	2.0
		1.5				

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 21%, PFBS \pm 16%, PFHS \pm 18%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 17%, PFHS \pm 12%, PFOS \pm 17%, and PFOSA \pm 15%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary ⁽¹⁾

		Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - Building 16						
ISO20-14-01-044	P305; Sample	101	169	111	318	22.0
ISO20-14-01-044-DUP	P305; Sample Dup	100	174	111	298	19.3
		Average	101	172	111	308
		%RPD Sample/Sample Dup	1.0	2.9	0.0	6.5
ISO20-14-01-045	P42; Sample	210	30.1	308	2660	16.0
ISO20-14-01-045-DUP	P42; Sample Dup	213	30.8	315	2870	16.0
		Average	212	30.5	312	2770
		%RPD Sample/Sample Dup	1.4	2.3	2.2	7.6
						0.0
		Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾	
Zone: Source Area – WWTP						
ISO20-14-01-047	L19; Sample	266	149	2620	50.4	
ISO20-14-01-047-DUP	L19; Sample Dup	267	147	2580	45.0	
		Average	267	148	2600	47.7
		%RPD Sample/Sample Dup	0.38	1.4	1.5	11
ISO20-14-01-048	M4; Sample	210	82.8	2140	98.6	
ISO20-14-01-048-DUP	M4; Sample Dup	206	81.3	2200	90.8	
		Average	208	82.1	2170	94.7
		%RPD Sample/Sample Dup	1.9	1.8	2.8	8.2
ISO20-14-01-049	P118C; Sample	232	119	1390	52.2	
ISO20-14-01-049-DUP	P118C; Sample Dup	239	124	1470	48.2	
		Average	236	122	1430	50.2
		%RPD Sample/Sample Dup	3.0	4.1	5.6	8.0
ISO20-14-01-050	P119C; Sample	803	232	6020	82.4	
ISO20-14-01-050-DUP	P119C; Sample Dup	783	230	5920	81.0	
		Average	793	231	5970	81.7
		%RPD Sample/Sample Dup	2.5	0.87	1.7	1.7
ISO20-14-01-051	P262bis; Sample	570	316	4680	8.36	
ISO20-14-01-051-DUP	P262bis; Sample Dup	573	318	4760	7.86	
		Average	572	317	4720	8.11
		%RPD Sample/Sample Dup	0.52	0.63	1.7	6.2
ISO20-14-01-052	P263; Sample	863	266	7080	15.3	
ISO20-14-01-052-DUP	P263; Sample Dup	843	267	6680	14.6	
		Average	853	267	6880	15.0
		%RPD Sample/Sample Dup	2.3	0.38	5.8	4.7

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 21%, PFBS \pm 16%, PFHS \pm 18%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 17%, PFHS \pm 12%, PFOS \pm 17%, and PFOSA \pm 15%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary ⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Source Area - WWTP					
ISO20-14-01-053	P264; Sample	413	260	1340	59.6
ISO20-14-01-053-DUP	P264; Sample Dup	401	262	1300	54.2
		Average	261	1320	56.9
		%RPD Sample/Sample Dup	2.9	3.0	9.5
ISO20-14-01-054	P265B; Sample	82.2	28.2	553	58.4
ISO20-14-01-054-DUP	P265B; Sample Dup	79.2	26.7	540	63.8
		Average	27.5	547	61.1
		%RPD Sample/Sample Dup	3.7	2.4	8.8
ISO20-14-01-055	P340; Sample	222	108	3420	90.8
ISO20-14-01-055-DUP	P340; Sample Dup	221	108	3410	101
		Average	108	3420	95.9
		%RPD Sample/Sample Dup	0.45	0.0	0.29
					11
ISO20-14-01-056	P341; Sample	222	124	2230	81.8
ISO20-14-01-056-DUP	P341; Sample Dup	225	122	2260	79.0
		Average	123	2250	80.4
		%RPD Sample/Sample Dup	1.3	1.6	1.3
					3.5
ISO20-14-01-057	P371; Sample	861	1060	2490	33.0
ISO20-14-01-057-DUP	P371; Sample Dup	848	1050	2500	31.4
		Average	1060	2500	32.2
		%RPD Sample/Sample Dup	1.5	0.95	0.40
					5.0
ISO20-14-01-058	P374; Sample	122	139	1010	58.4
ISO20-14-01-058-DUP	P374; Sample Dup	122	134	975	58.4
		Average	137	993	58.4
		%RPD Sample/Sample Dup	0.0	3.7	3.5
					0.0
ISO20-14-01-059	P379; Sample	48.4	11.2	501	68.0
ISO20-14-01-059-DUP	P379; Sample Dup	46.3	10.3	499	68.2
		Average	10.8	500	68.1
		%RPD Sample/Sample Dup	4.4	8.4	0.40
					0.29
ISO20-14-01-060	P380; Sample	1040	1050	2110	31.8
ISO20-14-01-060-DUP	P380; Sample Dup	1040	1060	2110	32.0
		Average	1060	2110	31.9
		%RPD Sample/Sample Dup	0.0	0.95	0.0
					0.63

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 21%, PFBS \pm 16%, PFHS \pm 18%, and PFOS \pm 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 17%, PFHS \pm 12%, PFOS \pm 17%, and PFOSA \pm 15%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFBS	PFHS	PFOS & PFOSA ⁽²⁾
Zone: Southern Site Boundary					
ISO20-14-01-061	P372; Sample	87.2	61.6	1150	28.8
ISO20-14-01-061-DUP	P372; Sample Dup	84.8	61.5	1130	25.4
Average		86.0	61.6	1140	27.1
%RPD Sample/Sample Dup		2.8	0.16	1.8	13
3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFBS	PFHS	PFOS & PFOSA ⁽²⁾
Zone: Extraction Wells					
ISO20-14-01-062	PP11; Sample	492	446	326	1450
ISO20-14-01-062-DUP	PP11; Sample Dup	502	448	319	1450
Average		497	447	323	1450
%RPD Sample/Sample Dup		2.0	0.45	2.2	0.0
Concentration (ng/mL)					
3M LIMS ID	Sample Description	PFOA	PFBS	PFHS	PFOS & PFOSA
ISO20-14-01-064	Travel Blank	<0.0240 ⁽²⁾	<1.00	<0.0250 ⁽²⁾	<0.0232 ⁽²⁾
					<0.0250 ⁽²⁾

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 21%, PFBS ± 16%, PFHS ± 18%, and PFOS ± 15%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 17%, PFHS ± 12%, PFOS ± 17%, and PFOSA ± 15%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis".

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluoroctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected on January 27 - 29, 2020, in Nalgene™ (high-density polyethylene) bottles prepared at the 3M EHS Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. All sample locations were spiked with a mass-labeled internal standard mix at a nominal concentration of 1 ng/mL and select samples were fortified with surrogate recovery standards (SRSs) at a nominal concentration of 0.1 ng/mL prior to sample collection. Samples were received on February 3, 2020 or February 4, 2020.

2.3 Sample Preparation

All sample locations were analyzed for FOSA with select locations analyzed for PFOA, PFHS, and PFOS by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples along with the method blanks.

Samples requiring dilutions were prepared for PFOA, PFHS, PFOS and PFBS (select locations only) using a direct injection by methanol dilution method. Samples that required a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples that required a 1:100 dilution were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol. Samples that required a 1:500 dilution were prepared by diluting 0.02 mL of a well-mixed sample with 10.0 mL of methanol. Samples that required a 1:1000 dilution were prepared by first diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol, followed by a second dilution prepared by diluting 1 mL of the diluted sample into 9 mL of methanol. Samples were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

2.4 Analysis

All samples and quality control samples were analyzed for the target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). SRSs were added and included in the analysis of these samples. Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.2. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

The following analytical runs were used to report the results herein:

5/1/20 (ETS Athena) External Standard Calibration Analysis:

- All sample locations were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported **except** for the following sample locations: BD24-3 (PFOA and PFHS), D14, P121, 3M vijver, Blokkersdijkvijver Noord, Blokkersdijkvijver standard, L21, L22, L31, and P116 (all analytes), and PP02 (PFBS and PFHS).

5/1/20 (ETS Kirk) External Standard Calibration Analysis:

- All sample locations were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported **except** for the following sample locations: P27 (PFHS) and P21B (PFOS).

5/5/20 (ETS Athena) External Standard Calibration Analysis:

- Sample locations were re-analyzed and reported for select analytes: P27 (PFHS), P21B (PFOS), and PP02 (PFBS and PFHS).

5/5/20 (ETS Hermes) Internal Standard Calibration Analysis:

- Sample locations were analyzed and reported for select analytes: BD24-3 (PFOA, PFHS, and PFOSA), D14, P121, 3M vijver, Blokkersdijkvijver Noord, Blokkersdijkvijver standard, L21, L22, L31, and P116 (all analytes).

5/6/20 (ETS Hermes) Internal Standard Calibration Analysis:

- Sample locations not previously analyzed for PFOSA were analyzed and reported for PFOSA.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk	ETS Athena	ETS Hermes
Liquid Chromatograph	Agilent 1260	Agilent 1260	Agilent 1100
Analysis Method	ETS-8-044.3	ETS-8-044.3	ETS-8-044.3
Analysis Date	5/1/20	5/1/20, 5/5/20	5/5/20, 5/6/20
Guard column	Betasil C8 (2.1 mm X 50 mm), 5 μ	Betasil C8 (2.1 mm X 50 mm), 5 μ	Betasil C8 (2.1 mm X 50 mm), 5 μ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ
Injection Volume	2 μ L	5 μ L	2 or 10 μ L
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 5500
Ion Source	Turbo Spray	Turbo Spray	Turbo Spray
Polarity	Negative	Negative	Negative
Software	Analyst 1.7.1	Analyst 1.7.1	Analyst 1.7.1

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis				
Step Number	Total Time (min)	Flow Rate (μ L/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0.00	300	90.0	10.0
1	0.50	300	90.0	10.0
2	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard	Mass Transition Q1/Q3
PFOA	413/369	$[^{13}\text{C}_8]\text{-PFOA}$	421/376
	413/219		
	413/169		
PFBS	299/99	$[^{18}\text{O}_2]\text{-PFBS}$	303/84
	299/80		
PFHS	399/99	$[^{13}\text{C}_3]\text{-PFHS}$	402/80
	399/80		
PFOS	499/99	$[^{13}\text{C}_8]\text{-PFOS}$	507/80
	499/80		
	499/130		
PFOSA	498/78	$[^{13}\text{C}_8]\text{-PFOSA}$	506/78
$[^{13}\text{C}_4]\text{-PFOA}$	417/372	$[^{13}\text{C}_8]\text{-PFOA}$	421/376
$[^{13}\text{C}_4]\text{-PFOS}$	503/80	$[^{13}\text{C}_8]\text{-PFOS}$	507/80

The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

3 Data Analysis

3.1 Calibration

5/5/20 and 5/6/20 Analysis (Internal Standard Calibration): Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of fourteen calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed. The calibration standards also contained surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

5/1/20 and 5/5/20 Analysis (External Standard Calibration): Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL to 25 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±25% (100±30% for the lowest curve point) were met for all analytes. The correlation coefficient (*r*) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes for each analysis.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.3 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the **Table 6** below.

Table 6. LOQ

Analyte	LOQ, ng/mL ⁽¹⁾ 5/1/20 [Athena] Analysis	LOQ, ng/mL ⁽¹⁾ 5/1/20 [Kirk] Analysis	LOQ, ng/mL ⁽¹⁾ 5/5/20 [Athena] Analysis	LOQ, ng/mL ⁽²⁾ 5/5/20 [Hermes] Analysis	LOQ, ng/mL ⁽²⁾ 5/6/20 [Hermes] Analysis
PFOA	0.240	0.0479	NA	0.0240	NA
PFBS	0.100	0.100	0.0200	NA	NA
PFHS	0.100	0.0500	0.0200	0.0250	NA
PFOS	0.0927	0.0464	0.0464	0.0232	NA
PFOSA	NA	NA	NA	0.0250	0.0250

NA = Not Applicable

(1) A dilution factor was not applied to the LOQ.

(2) A dilution factor of 2 was applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of $100\% \pm 25\%$.

3.5 Blanks

Two types of blanks were prepared and analyzed with the samples: method/solvent blanks and field/trip blanks. Each blank result was reviewed and used to evaluate method performance. The method blanks were used to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. LCSs were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. Surrogates [$^{13}\text{C}_4$]-PFOA and [$^{13}\text{C}_4$]-PFOS were added post dilution when analyzed by external standard.

- Target analyte LCSs analyzed on 5/1/20 were prepared at nominal concentrations of 100 ng/mL, 5000 ng/mL, and 35000 ng/mL.
- Target analyte LCSs analyzed on 5/5/20 using external calibration were prepared at nominal concentrations of 200 ng/mL, 20000 ng/mL, and 70000 ng/mL.
- Target analyte LCSs analyzed on 5/5/20 and 5/6/20 using internal standard were prepared at nominal concentrations of 0.2 ng/mL, 20 ng/mL, and 140 ng/mL.

Method ETS-8-044.3 states that the average recovery of LCSs at each spiking level must be within 80%-120% with an RSD $\leq 20\%$. All LCS samples met criteria with the following exceptions:

- 5/1/20 [ETS Athena]: Low-level LCSs had an average recovery of 79.3% for PFOA.
- 5/1/20 [ETS Kirk]: Low-level LCSs had an RSD of 22% for PFBS.
- 5/5/20 [ETS Hermes]: High-level LCSs for PFOSA were spiked above the resulting ULOQ. The low and mid-level LCSs were spiked at a more appropriate level compared to the reported sample concentrations.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report. Individual LCS results are included in the raw data and available upon request.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.5. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in **Table 7** below.

- The data uncertainty for PFOA using external calibration was calculated at $\pm 19\%$ following ETS-12-012.5; however, the data uncertainty was expanded to $\pm 21\%$ based on the PFOA recovery of the low-level LCSs from the analysis on 5/1/20.

Table 7. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	NA	$\pm 21\%$
PFBS	External	8.07	$\pm 16\%$
PFHS	External	9.17	$\pm 18\%$
PFOS	External	7.51	$\pm 15\%$
PFOA	Internal	8.38	$\pm 17\%$
PFHS	Internal	5.92	$\pm 12\%$
PFOS	Internal	8.35	$\pm 17\%$
PFOSA	Internal	7.43	$\pm 15\%$

NA = Not Applicable

3.8 Field Matrix Spikes (FMS)

Target analyte field matrix spike samples were collected for select sampling points to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of $100\pm 30\%$ confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions at 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOA and PFOS. The standards used for the preparation of the field matrix spiking solutions greater than 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, select sample bottles contained stable isotope surrogate recovery spikes of [$^{13}\text{C}_4$]-PFOA and [$^{13}\text{C}_4$]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles prior to sample collection. The [$^{13}\text{C}_4$]-PFOA was selected to represent the C8 perfluorocarboxylic acid. The [$^{13}\text{C}_4$]-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids and perfluorooctanesulfonamide. Surrogate matrix spike recoveries within method acceptance criteria of $100\pm 30\%$ confirm that “unknown” components in the sample matrix do not significantly

interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

The following calculation was used to generate data in section 4 of the report.

$$FMS\ Recovery = \frac{\text{Sample Concentration of FMS} - \text{Average Concentration (Field Sample & Field Sample Dup)}}{\text{Spike Concentration}} \times 100\%$$

Table 8. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
D14, Blokkersdijkvijver-standard, P116	FMS	4.79	5.00	5.00	4.64	5.00
Bemalingsstation ⁽¹⁾	FMS	92.7	92.9	92.9	92.6	4.42
D5	FMS	260	260	260	259	10.0
P118B, P304, P263	FMS	2010	2010	2010	2010	10.0
PP07, PP13, M4, P374, P380	FMS	1050	1050	1050	1050	50.0
Trip Blank	Low	4.79	5.00	5.00	4.64	5.00
	High	1050	1050	1050	1050	50.0

(1) Sample container for the FMS sample was overfilled by more than 10%. The FMS true values were adjusted accordingly.

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of $\pm 30\%$, demonstrate that the method is appropriate for the given matrix.

The method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. At times, the spike level exceeded the recommended upper limit of 10 times the analyte concentration. In these instances, the FMS recovery was reported and flagged as above 10 times the sample concentration.

For PFOA, PFBS, PFHS, and PFOS, when the field matrix spike level was not appropriate as compared to the endogenous sample concentration, the surrogate recovery standards were used to assess method accuracy. All field matrix spike and surrogate recovery standard recoveries met acceptance criteria.

Table 9. Location ID: D14

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-006	D14; Sample	2.10	NA	1.17	NA
ISO20-14-01-006-DUP	D14; Sample Duplicate	2.06	NA	1.12	NA
ISO20-14-01-006-FMS	D14; Sample FMS	6.46	91.4	6.00	97.1
Average Concentration (ng/mL) ± %RPD		2.08 ng/mL ± 1.9%		1.15 ng/mL ± 4.4%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-006	D14; Sample	0.444	NA	0.0326	NA
ISO20-14-01-006-DUP	D14; Sample Duplicate	0.438	NA	<0.0250	NA
ISO20-14-01-006-FMS	D14; Sample FMS	4.88	95.8 ⁽¹⁾	4.50	89.3 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		0.441 ng/mL ± 1.4%		0.0326 ng/mL	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 10. Location ID: D5

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-010	D5; Sample	150	NA	50.1	NA
ISO20-14-01-010-DUP	D5; Sample Duplicate	155	NA	52.6	NA
ISO20-14-01-010-FMS	D5; Sample FMS	378	86.9	273	85.3
Average Concentration (ng/mL) ± %RPD		153 ng/mL ± 3.3%		51.4 ng/mL ± 4.9%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-010	D5; Sample	232	NA	2.40	NA
ISO20-14-01-010-DUP	D5; Sample Duplicate	238	NA	2.30	NA
ISO20-14-01-010-FMS	D5; Sample FMS	446	81.4	11.7	93.5
Average Concentration (ng/mL) ± %RPD		235 ng/mL ± 2.6%		2.35 ng/mL ± 4.3%	

NA = Not Applicable

Table 11. Location ID: P118B

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-013	P118B; Sample	1560	NA	713	NA
ISO20-14-01-013-DUP	P118B; Sample Duplicate	1570	NA	708	NA
ISO20-14-01-013-FMS	P118B; Sample FMS	3350	88.8	2540	91.0
Average Concentration (ng/mL) ± %RPD		1570 ng/mL ± 0.64%		711 ng/mL ± 0.70%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-013	P118B; Sample	6330	NA	2.44	NA
ISO20-14-01-013-DUP	P118B; Sample Duplicate	6380	NA	2.48	NA
ISO20-14-01-013-FMS	P118B; Sample FMS	8080	NC	11.7	92.4
Average Concentration (ng/mL) ± %RPD		6360 ng/mL ± 0.79%		2.46 ng/mL ± 1.6%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5 times the endogenous sample concentration.

Table 12. Location ID: Blokkersdijkvijver standaard

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-019	Blokkersdijkvijver standaard; Sample	0.778	NA	0.480	NA
ISO20-14-01-019-DUP	Blokkersdijkvijver standaard; Sample Duplicate	0.748	NA	0.480	NA
ISO20-14-01-019-FMS	Blokkersdijkvijver standaard; Sample FMS	5.28	94.3	5.20	94.4 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		0.763 ng/mL ± 3.9%		0.480 ng/mL ± 0.0%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-019	Blokkersdijkvijver standaard; Sample	0.512	NA	<0.0250	NA
ISO20-14-01-019-DUP	Blokkersdijkvijver standaard; Sample Duplicate	0.468	NA	<0.0250	NA
ISO20-14-01-019-FMS	Blokkersdijkvijver standaard; Sample FMS	4.90	95.1	4.44	88.8 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		0.490 ng/mL ± 9.0%		<0.0250 ng/mL	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 13. Location ID: P116

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-027	P116; Sample	0.712	NA	0.428	NA
ISO20-14-01-027-DUP	P116; Sample Duplicate	0.720	NA	0.452	NA
ISO20-14-01-027-FMS	P116; Sample FMS	5.28	95.3	5.08	92.8 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		0.716 ng/mL ± 1.1%		0.440 ng/mL ± 5.5%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-027	P116; Sample	8.48	NA	0.0566	NA
ISO20-14-01-027-DUP	P116; Sample Duplicate	8.68	NA	0.0536	NA
ISO20-14-01-027-FMS	P116; Sample FMS	13.4	104	4.58	90.5 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		8.58 ng/mL ± 2.3%		0.0551 ng/mL ± 5.4%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 14. Location ID: PP07

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-033	PP07; Sample	404	NA	403	NA	139	NA
ISO20-14-01-033-DUP	PP07; Sample Duplicate	398	NA	388	NA	132	NA
ISO20-14-01-033-FMS	PP07; Sample FMS	1340	89.4	1310	87.1	1060	88.0
Average Concentration (ng/mL) ± %RPD		401 ng/mL ± 1.5%		396 ng/mL ± 3.8%		136 ng/mL ± 5.2%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-033	PP07; Sample	2200	NA	63.6	NA
ISO20-14-01-033-DUP	PP07; Sample Duplicate	2100	NA	62.6	NA
ISO20-14-01-033-FMS	PP07; Sample FMS	2950	NC	113	99.8
Average Concentration (ng/mL) ± %RPD		2150 ng/mL ± 4.7%		63.1 ng/mL ± 1.6%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5 times the endogenous sample concentration.

Table 15. Location ID: Bemalingstation

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-039	Bemalingstation; Sample	28.3	NA	24.2	NA
ISO20-14-01-039-DUP	Bemalingstation; Sample Duplicate	27.9	NA	23.4	NA
ISO20-14-01-039-FMS	Bemalingstation; Sample FMS	124	103	116	99.2
Average Concentration (ng/mL) ± %RPD		28.1 ng/mL ± 1.4%		23.8 ng/mL ± 3.4%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-039	Bemalingstation; Sample	43.6	NA	0.120	NA
ISO20-14-01-039-DUP	Bemalingstation; Sample Duplicate	44.6	NA	0.126	NA
ISO20-14-01-039-FMS	Bemalingstation; Sample FMS	130	92.8	4.42	97.1 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		44.1 ng/mL ± 2.3%		0.123 ng/mL ± 4.9%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 16. Location ID: P304

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-043	P304; Sample	395	NA	303	NA	115	NA
ISO20-14-01-043-DUP	P304; Sample Duplicate	387	NA	306	NA	115	NA
ISO20-14-01-043-FMS	P304; Sample FMS	2180	89.0	2060	87.3	1870	87.3 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		391 ng/mL ± 2.0%		305 ng/mL ± 0.99%		115 ng/mL ± 0.0%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-043	P304; Sample	1030	NA	13.7	NA
ISO20-14-01-043-DUP	P304; Sample Duplicate	1010	NA	13.5	NA
ISO20-14-01-043-FMS	P304; Sample FMS	2700	83.6	23.4	98.0
Average Concentration (ng/mL) ± %RPD		1020 ng/mL ± 2.0%		13.6 ng/mL ± 1.5%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 17. Location ID: M4

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-048	M4; Sample	210	NA	82.8	NA
ISO20-14-01-048-DUP	M4; Sample Duplicate	206	NA	81.3	NA
ISO20-14-01-048-FMS	M4; Sample FMS	1160	90.7	1020	89.3 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		208 ng/mL ± 1.9%		82.1 ng/mL ± 1.8%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-048	M4; Sample	2140	NA	98.6	NA
ISO20-14-01-048-DUP	M4; Sample Duplicate	2200	NA	90.8	NA
ISO20-14-01-048-FMS	M4; Sample FMS	2970	NC	148	107
Average Concentration (ng/mL) ± %RPD		2170 ng/mL ± 2.8%		94.7 ng/mL ± 8.2%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5 times the endogenous sample concentration.

(1) FMS concentration greater than 10 times the sample concentration.

Table 18. Location ID: P263

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-052	P263; Sample	863	NA	266	NA
ISO20-14-01-052-DUP	P263; Sample Duplicate	843	NA	267	NA
ISO20-14-01-052-FMS	P263; Sample FMS	2660	89.9	2010	86.7
Average Concentration (ng/mL) ± %RPD		853 ng/mL ± 2.3%		267 ng/mL ± 0.38%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-052	P263; Sample	7080	NA	15.3	NA
ISO20-14-01-052-DUP	P263; Sample Duplicate	6680	NA	14.6	NA
ISO20-14-01-052-FMS	P263; Sample FMS	8330	NC	24.4	94.5
Average Concentration (ng/mL) ± %RPD		6880 ng/mL ± 5.8%		15.0 ng/mL ± 4.7%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5 times the endogenous sample concentration.

Table 19. Location ID: P374

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-058	P374; Sample	122	NA	139	NA
ISO20-14-01-058-DUP	P374; Sample Duplicate	122	NA	134	NA
ISO20-14-01-058-FMS	P374; Sample FMS	1050	88.4	1040	86.0
Average Concentration (ng/mL) ± %RPD		122 ng/mL ± 0.0%		137 ng/mL ± 3.7%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-058	P374; Sample	1010	NA	58.4	NA
ISO20-14-01-058-DUP	P374; Sample Duplicate	975	NA	58.4	NA
ISO20-14-01-058-FMS	P374; Sample FMS	1840	80.7	106	95.2
Average Concentration (ng/mL) ± %RPD		993 ng/mL ± 3.5%		58.4 ng/mL ± 0.0%	

NA = Not Applicable

Table 20. Location ID: P280

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-060	P380; Sample	1040	NA	1050	NA
ISO20-14-01-060-DUP	P380; Sample Duplicate	1040	NA	1060	NA
ISO20-14-01-060-FMS	P380; Sample FMS	2010	92.4	2010	91.0
Average Concentration (ng/mL) ± %RPD		1040 ng/mL ± 0.0%		1060 ng/mL ± 0.95%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-060	P380; Sample	2110	NA	31.8	NA
ISO20-14-01-060-DUP	P380; Sample Duplicate	2110	NA	32.0	NA
ISO20-14-01-060-FMS	P380; Sample FMS	2850	NC	81.2	98.6
Average Concentration (ng/mL) ± %RPD		2100 ng/mL ± 0.0%		31.9 ng/mL ± 0.63%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5 times the endogenous sample concentration.

Table 21. Location ID: Travel Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-064	Travel Blank	<0.0240	NA	<1.00	NA	<0.0250	NA
ISO20-14-01-064-FMS-LOW	Travel Blank FMS Low	4.88	102	4.64	92.8	5.00	100
ISO20-14-01-064-FMS-HIGH	Travel Blank FMS High	991	94.4	946	90.1	938	89.3

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-01-064	Travel Blank	<0.0232	NA	<0.0250	NA
ISO20-14-01-064-FMS-LOW	Travel Blank FMS Low	4.76	103	4.74	94.8
ISO20-14-01-064-FMS-HIGH	Travel Blank FMS High	936	89.1	49.6	99.2

NA = Not Applicable

Table 22. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]PFOA	[¹³ C ₄]PFOS	[¹³ C ₄]PFOS Re-analysis
ISO20-14-01-001	BD24-3; Sample	88.4	117	86.1
ISO20-14-01-001-DUP	BD24-3; Sample Duplicate	89.2	120	86.5
ISO20-14-01-002	BD24-4; Sample	121	119	NA
ISO20-14-01-002-DUP	BD24-4; Sample Duplicate	114	112	NA
ISO20-14-01-004	D10; Sample	116	116	NA
ISO20-14-01-004-DUP	D10; Sample Duplicate	117	117	NA
ISO20-14-01-005	D11; Sample	120	119	NA
ISO20-14-01-005-DUP	D11; Sample Duplicate	121	121	NA
ISO20-14-01-006	D14; Sample	85.8 ⁽²⁾	87.1 ⁽²⁾	NA
ISO20-14-01-006-DUP	D14; Sample Duplicate	88.0 ⁽²⁾	93.2 ⁽²⁾	NA
ISO20-14-01-006-FMS	D14; Sample FMS	90.8 ⁽²⁾	87.3 ⁽²⁾	NA
ISO20-14-01-007	D16; Sample	117	116	NA
ISO20-14-01-007-DUP	D16; Sample Duplicate	116	117	NA
ISO20-14-01-008	D17; Sample	109	110	NA
ISO20-14-01-008-DUP	D17; Sample Duplicate	110	114	NA
ISO20-14-01-009	D18; Sample	121	121	NA
ISO20-14-01-009-DUP	D18; Sample Duplicate	116	118	NA
ISO20-14-01-010	D5; Sample	122	121	NA
ISO20-14-01-010-DUP	D5; Sample Duplicate	120	120	NA
ISO20-14-01-010-FMS	D5; Sample FMS	115	116	NA
ISO20-14-01-011	ND7; Sample	113	114	NA
ISO20-14-01-011-DUP	ND7; Sample Duplicate	111	113	NA
ISO20-14-01-012	P118A; Sample	119	119	NA
ISO20-14-01-012-DUP	P118A; Sample Duplicate	113	115	NA
ISO20-14-01-013	P118B; Sample	125	121	NA
ISO20-14-01-013-DUP	P118B; Sample Duplicate	120	122	NA
ISO20-14-01-013-FMS	P118B; Sample FMS	121	120	NA
ISO20-14-01-014	P119A; Sample	118	117	NA
ISO20-14-01-014-DUP	P119A; Sample Duplicate	117	117	NA
ISO20-14-01-015	P119B; Sample	122	120	NA
ISO20-14-01-015-DUP	P119B; Sample Duplicate	115	115	NA
ISO20-14-01-016	P121; Sample	90.2 ⁽²⁾	90.1 ⁽²⁾	NA
ISO20-14-01-016-DUP	P121; Sample Duplicate	88.2 ⁽²⁾	88.0 ⁽²⁾	NA
ISO20-14-01-017	3M vijver; Sample	88.8 ⁽²⁾	88.0 ⁽²⁾	NA
ISO20-14-01-017-DUP	3M vijver; Sample Duplicate	85.6 ⁽²⁾	86.1 ⁽²⁾	NA
ISO20-14-01-018	Blokkersdijkvijver Noord; Sample	88.4 ⁽²⁾	83.4 ⁽²⁾	NA
ISO20-14-01-018-DUP	Blokkersdijkvijver Noord; Sample Duplicate	83.2 ⁽²⁾	83.4 ⁽²⁾	NA
ISO20-14-01-019	Blokkersdijkvijver standaard; Sample	89.6 ⁽²⁾	87.3 ⁽²⁾	NA
ISO20-14-01-019-DUP	Blokkersdijkvijver standaard; Sample Duplicate	85.4 ⁽²⁾	90.1 ⁽²⁾	NA
ISO20-14-01-019-FMS	Blokkersdijkvijver standaard; Sample FMS	88.2 ⁽²⁾	90.7 ⁽²⁾	NA

NA = Not Applicable

(1) The surrogate recovery standards [¹³C₄]PFOA and [¹³C₄]PFOS were added to the samples during sample preparation unless noted otherwise.

(2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 22 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO20-14-01-020	P321; Sample	118	115	NA
ISO20-14-01-020-DUP	P321; Sample Duplicate	117	115	NA
ISO20-14-01-021	L21; Sample	87.2 ⁽²⁾	89.2 ⁽²⁾	NA
ISO20-14-01-021-DUP	L21; Sample Duplicate	91.8 ⁽²⁾	90.9 ⁽²⁾	NA
ISO20-14-01-022	L22; Sample	87.0 ⁽²⁾	94.7 ⁽²⁾	NA
ISO20-14-01-022-DUP	L22; Sample Duplicate	83.4 ⁽²⁾	86.3 ⁽²⁾	NA
ISO20-14-01-023	L31; Sample	91.2 ⁽²⁾	92.8 ⁽²⁾	NA
ISO20-14-01-023-DUP	L31; Sample Duplicate	90.8 ⁽²⁾	84.6 ⁽²⁾	NA
ISO20-14-01-024	L4; Sample	119	119	NA
ISO20-14-01-024-DUP	L4; Sample Duplicate	116	119	NA
ISO20-14-01-025	P114bis; Sample	116	116	NA
ISO20-14-01-025-DUP	P114bis; Sample Duplicate	120	119	NA
ISO20-14-01-026	P115; Sample	116	116	NA
ISO20-14-01-026-DUP	P115; Sample Duplicate	117	118	NA
ISO20-14-01-027	P116; Sample	83.8 ⁽²⁾	88.0 ⁽²⁾	NA
ISO20-14-01-027-DUP	P116; Sample Duplicate	91.6 ⁽²⁾	89.2 ⁽²⁾	NA
ISO20-14-01-027-FMS	P116; Sample FMS	88.4 ⁽²⁾	92.4 ⁽²⁾	NA
ISO20-14-01-028	Effluent WWTP; Sample	116	117	NA
ISO20-14-01-028-DUP	Effluent WWTP; Sample Duplicate	119	120	NA
ISO20-14-01-029	PP02; Sample	125	118	82.2
ISO20-14-01-029-DUP	PP02; Sample Duplicate	125	119	83.0
ISO20-14-01-030	PP04; Sample	118	113	NA
ISO20-14-01-030-DUP	PP04; Sample Duplicate	116	113	NA
ISO20-14-01-031	PP05; Sample	126	125	NA
ISO20-14-01-031-DUP	PP05; Sample Duplicate	122	120	NA
ISO20-14-01-032	PP06; Sample	120	118	NA
ISO20-14-01-032-DUP	PP06; Sample Duplicate	116	115	NA
ISO20-14-01-033	PP07; Sample	106	108	NA
ISO20-14-01-033-DUP	PP07; Sample Duplicate	105	105	NA
ISO20-14-01-033-FMS	PP07; Sample FMS	104	105	NA
ISO20-14-01-034	PP08; Sample	103	98.0	NA
ISO20-14-01-034-DUP	PP08; Sample Duplicate	105	106	NA
ISO20-14-01-035	PP10; Sample	106	107	NA
ISO20-14-01-035-DUP	PP10; Sample Duplicate	108	117	NA
ISO20-14-01-036	12; Sample	104	110	NA
ISO20-14-01-036-DUP	12; Sample Duplicate	102	103	NA
ISO20-14-01-037	13; Sample	106	107	NA
ISO20-14-01-037-DUP	13; Sample Duplicate	102	102	NA
ISO20-14-01-038	5; Sample	103	101	NA
ISO20-14-01-038-DUP	5; Sample Duplicate	106	106	NA
ISO20-14-01-039	Bemalingstation; Sample	107	107	NA
ISO20-14-01-039-DUP	Bemalingstation; Sample Duplicate	106	107	NA

NA = Not Applicable

(1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.

(2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 22 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO20-14-01-039-FMS	Bemalingstation; Sample FMS	105	110	NA
ISO20-14-01-040	Collector put; Sample	108	107	NA
ISO20-14-01-040-DUP	Collector put; Sample Duplicate	107	107	NA
ISO20-14-01-041	P27; Sample	107	109	88.4
ISO20-14-01-041-DUP	P27; Sample Duplicate	112	114	85.3
ISO20-14-01-042	P21B; Sample	104	105	103
ISO20-14-01-042-DUP	P21B; Sample Duplicate	105	107	98.1
ISO20-14-01-043	P304; Sample	106	113	NA
ISO20-14-01-043-DUP	P304; Sample Duplicate	107	110	NA
ISO20-14-01-043-FMS	P304; Sample FMS	102	106	NA
ISO20-14-01-044	P305; Sample	107	108	NA
ISO20-14-01-044-DUP	P305; Sample Duplicate	109	108	NA
ISO20-14-01-045	P42; Sample	107	106	NA
ISO20-14-01-045-DUP	P42; Sample Duplicate	107	108	NA
ISO20-14-01-047	L19; Sample	109	111	NA
ISO20-14-01-047-DUP	L19; Sample Duplicate	108	110	NA
ISO20-14-01-048	M4; Sample	108	110	NA
ISO20-14-01-048-DUP	M4; Sample Duplicate	108	111	NA
ISO20-14-01-048-FMS	M4; Sample FMS	99.9	106	NA
ISO20-14-01-049	P118C; Sample	106	106	NA
ISO20-14-01-049-DUP	P118C; Sample Duplicate	106	110	NA
ISO20-14-01-050	P119C; Sample	102	105	NA
ISO20-14-01-050-DUP	P119C; Sample Duplicate	110	109	NA
ISO20-14-01-051	P262bis; Sample	107	108	NA
ISO20-14-01-051-DUP	P262bis; Sample Duplicate	107	109	NA
ISO20-14-01-052	P263; Sample	105	109	NA
ISO20-14-01-052-DUP	P263; Sample Duplicate	93.6	99.0	NA
ISO20-14-01-052-FMS	P263; Sample FMS	109	107	NA
ISO20-14-01-053	P264; Sample	110	112	NA
ISO20-14-01-053-DUP	P264; Sample Duplicate	110	111	NA
ISO20-14-01-054	P265B; Sample	109	111	NA
ISO20-14-01-054-DUP	P265B; Sample Duplicate	108	111	NA
ISO20-14-01-055	P340; Sample	108	109	NA
ISO20-14-01-055-DUP	P340; Sample Duplicate	107	106	NA
ISO20-14-01-056	P341; Sample	107	109	NA
ISO20-14-01-056-DUP	P341; Sample Duplicate	108	107	NA
ISO20-14-01-057	P371; Sample	106	110	NA
ISO20-14-01-057-DUP	P371; Sample Duplicate	96.0	98.2	NA
ISO20-14-01-058	P374; Sample	109	110	NA
ISO20-14-01-058-DUP	P374; Sample Duplicate	111	107	NA
ISO20-14-01-058-FMS	P374; Sample FMS	108	108	NA

NA = Not Applicable

(1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.

(2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 22 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)	
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS
ISO20-14-01-059	P379; Sample	104	110
ISO20-14-01-059-DUP	P379; Sample Duplicate	106	108
ISO20-14-01-060	P380; Sample	101	102
ISO20-14-01-060-DUP	P380; Sample Duplicate	105	109
ISO20-14-01-060-FMS	P380; Sample FMS	101	103
ISO20-14-01-061	P372; Sample	94.1	99.4
ISO20-14-01-061-DUP	P372; Sample Duplicate	107	110
ISO20-14-01-062	PP11; Sample	106	110
ISO20-14-01-062-DUP	PP11; Sample Duplicate	104	104
ISO20-14-01-064	Travel Blank	92.2 ⁽²⁾	91.1 ⁽²⁾
ISO20-14-01-064-FMS-LOW	Travel Blank FMS Low	96.8 ⁽²⁾	90.3 ⁽²⁾
ISO20-14-01-064-FMS-HIGH	Travel Blank FMS High	111	113

NA = Not Applicable

(1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.

(2) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 9-22 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M EHS Laboratory standard operating procedures.

7 Attachment

Chain of Custody Form

8 Signatures

Chelsie Grochow, 3M Report Author

Susan T. Wolf, 3M Principal Analytical Investigator

Brian T. Mader, Ph.D., 3M EHS Laboratory Manager

The 3M EHS Laboratory's Quality Assurance Unit has audited the data and report for this project.

Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M EHS Laboratory.

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Project: ISO20-14-01

Phone: [REDACTED]
Alt. Phone: [REDACTED]
Fax: (651) 777-1111

Requester: Tack, Charlotte (ZWIJNDRECH-3MF)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/6/2020
Project Description: 3M Antwerp Water Sampling for PFCs
Comments:

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
✓ ISO20-14-01-001 ✗	BD24-3; Sample	28.01.2020 12:50 PM	WG	✓
✓ ISO20-14-01-001-DUP ✗	BD24-3; Sample Duplicate	28.01.2020 12:50 PM	WG	✓
✓ ISO20-14-01-002 ✗	BD24-4; Sample	28.01.2020 01:10 AM	WG	✓
✓ ISO20-14-01-002-DUP ✗	BD24-4; Sample Duplicate	28.01.2020 01:10 AM	WG	✓
✓ ISO20-14-01-003	D09bis; Sample	[REDACTED]		Not Sampled ✓
✓ ISO20-14-01-003-DUP	D09bis; Sample Duplicate			Not Sampled ✓
✓ ISO20-14-01-004 ✗	D10; Sample	28.01.2020 12:55 PM	WG	✓
✓ ISO20-14-01-004-DUP ✗	D10; Sample Duplicate	28.01.2020 12:55 PM	WG	✓
✓ ISO20-14-01-005 ✗	D11; Sample	28.01.2020 11:45 AM	WG	✓
✓ ISO20-14-01-005-DUP ✗	D11; Sample Duplicate	28.01.2020 11:45 AM	WG	✓
✓ ISO20-14-01-006	D14; Sample	29.01.2020 12:49 PM	WG	✓
✓ ISO20-14-01-006-DUP	D14; Sample Duplicate	29.01.2020 12:49 PM	WG	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print):	Collector's signature:						
Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
12	Julie Fichfjel	31/01/2020	09:00PM	FedEx	See pg 11		

3M EHS LABORATORY
Chain-of-Custody

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St. Paul, MN 55144

Phone: [REDACTED]
Alt. Pho: [REDACTED]
Fax: (651) 429-6389

Project: ISO20-14-01 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)

Department: 832202 Site Source: 01WJWT10

Project Number:

Date Created: 1/6/2020

Completion Date:

Project Lead: Susan T. Wolf

Phone Number: [REDACTED]

Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO20-14-01-006-FMS	D14; Sample FMS •	29.01.2020 18:19 PM	WQ	✓
ISO20-14-01-007	D16; Sample X	29/01/2020 - 11:15AM	WG	✓
ISO20-14-01-007-DUP	D16; Sample Duplicate X	29/01/2020 - 11:15AM	WG	✓
ISO20-14-01-008	D17; Sample •	29.01.2020 - 09:15AM	WG	✓
ISO20-14-01-008-DUP	D17; Sample Duplicate •	29.01.2020 - 09:15AM	WG	✓
ISO20-14-01-009	D18; Sample •	29.01.2020 09:45 AM	WG	✓
ISO20-14-01-009-DUP	D18; Sample Duplicate •	29.01.2020 09:45 AM	WG	✓
ISO20-14-01-010	D5; Sample •	29.01.2020 - 12:00 AM	WG	✓
ISO20-14-01-010-DUP	D5; Sample Duplicate •	29.01.2020 - 12:00 AM	WG	✓
ISO20-14-01-010-FMS	D5; Sample FMS •	29.01.2020 - 12:00 AM	WQ	✓
ISO20-14-01-011	ND7; Sample •	29.01.2020 - 08:15 AM	WG	✓
ISO20-14-01-011-DUP	ND7; Sample Duplicate •	29.01.2020 - 08:15 AM	WG	✓
ISO20-14-01-012	P118A; Sample •	29.01.2020 - 09:10 AM	WG	✓
ISO20-14-01-012-DUP	P118A; Sample Duplicate •	29.01.2020 - 09:10 AM	WG	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 40 Deg C Received on Ice Other:

Collected by (print): Julie Fichter

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Julie Fichter	31/01/2020	08:00 PM	FedEx	See pg 11		

3M EHS LABORATORY
Chain-of-Custody

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Phone [REDACTED]

Alt. Ph. [REDACTED]

Fax: ([REDACTED]

Project: ISO20-14-01 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3MF)

Department: 832202 Site Source: 01WJWT10

Project Number:

Date Created: 1/6/2020

Completion Date:

Project Lead: Susan T. Wolf

Phone Number [REDACTED]

Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
✓ ISO20-14-01-013	P118B; Sample •	28.01.2020 09:45 AM	WG	✓
✓ ISO20-14-01-013-DUP	P118B; Sample Duplicate •	28.01.2020 09:45 AM	WG	✓
✓ ISO20-14-01-013-FMS	P118B; Sample FMS •	28.01.2020 09:45 AM	WQ	✓
✓ ISO20-14-01-014	P119A; Sample X	28.01.2020 01:55 PM	WG	✓
✓ ISO20-14-01-014-DUP	P119A; Sample Duplicate X	28.01.2020 01:55 PM	WG	✓
✓ ISO20-14-01-015	P119B; Sample X	28.01.2020 02:15 PM	WG	✓
✓ ISO20-14-01-015-DUP	P119B; Sample Duplicate X	28.01.2020 02:15 PM	WG	✓
✓ ISO20-14-01-016	P121; Sample •	29.01.2020 11:25 AM	WG	✓
✓ ISO20-14-01-016-DUP	P121; Sample Duplicate •	29.01.2020 11:25 AM	WG	✓
✓ ISO20-14-01-017	3M vijver; Sample X	28.01.2020 09:30 AM	WG	✓
✓ ISO20-14-01-017-DUP	3M vijver; Sample Duplicate X	28.01.2020 09:30 AM	WG	✓
✓ ISO20-14-01-018	Blokkersdijkvijver Noord; Sample X	28.01.2020 10:00 AM	WG	✓
✓ ISO20-14-01-018-DUP	Blokkersdijkvijver Noord; Sample Duplicate X	28.01.2020 10:00 AM	WG	✓
✓ ISO20-14-01-019	Blokkersdijkvijver standaard; Sample X	28.01.2020 10:30 AM	WG	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Julie Fichyjt

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Julie Fichyjt	31/01/2020	01:00 PM	Fedex	See pg 11		

3M EHS LABORATORY
Chain-of-Custody

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Project: ISO20-14-01 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/6/2020

Completion Date:

Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO20-14-01-019-DUP	Blokkersdijkvijver standaard; Sample Duplicate ✓	28.01.2020 10:30 AM	WG	✓
ISO20-14-01-019-FMS	Blokkersdijkvijver standaard; Sample FMS ✗	28.01.2020 10:30 AM	WQ	✓
ISO20-14-01-020	P321; Sample ✗	29.01.2020 09:00 AM	WG	✓
ISO20-14-01-020-DUP	P321; Sample Duplicate ✗	29.01.2020 09:00 AM	WG	✓
ISO20-14-01-021	L21; Sample ✗	29.01.2020 09:10 AM	WG	✓
ISO20-14-01-021-DUP	L21; Sample Duplicate ✗	29.01.2020 09:10 AM	WG	✓
ISO20-14-01-022	L22; Sample ✗	29.01.2020 09:40 AM	WG	✓
ISO20-14-01-022-DUP	L22; Sample Duplicate ✗	29.01.2020 09:40 AM	WG	✓
ISO20-14-01-023	L31; Sample ✗	29.01.2020 08:30 AM	WG	✓
ISO20-14-01-023-DUP	L31; Sample Duplicate ✗	29.01.2020 08:30 AM	WG	✓
ISO20-14-01-024	L4; Sample ✗	28.01.2020 12:35 PM	WG	✓
ISO20-14-01-024-DUP	L4; Sample Duplicate ✗	28.01.2020 12:35 PM	WG	✓
ISO20-14-01-025	P114bis; Sample ✗	29.01.2020 11:05 AM	WG	✓
ISO20-14-01-025-DUP	P114bis; Sample Duplicate ✗	29.01.2020 11:05 AM	WG	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Juli Fichtl

Collector's signature: Juli Fichtl

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Juli Fichtl	31/01/2020	09:00 PM	Fedex	See pg 11		

3M EHS LABORATORY
Chain-of-Custody

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Project: ISO20-14-01 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/6/2020

Completion Date:

Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO20-14-01-026 •	P115; Sample •	29.01.2020 10:45 AM	WG	✓
ISO20-14-01-026-DUP •	P115; Sample Duplicate •	29.01.2020 10:45 AM	WG	
ISO20-14-01-027 •	P116; Sample •	29.01.2020 10:05 AM	WG	✓
ISO20-14-01-027-DUP •	P116; Sample Duplicate •	29.01.2020 10:05 AM	WG	✓
ISO20-14-01-027-FMS •	P116; Sample FMS •	29.01.2020 10:05 AM	WQ	✓
ISO20-14-01-028 ✗	Effluent WWTP; Sample ✗	27.01.2020 03:20 PM	WG	✓
ISO20-14-01-028-DUP ✗	Effluent WWTP; Sample Duplicate ✗	27.01.2020 03:20 PM	WG	✓
ISO20-14-01-029 ✗	PP02; Sample ✗	27.01.2020 02:15 PM	WG	✓
ISO20-14-01-029-DUP ✗	PP02; Sample Duplicate ✗	27.01.2020 02:15 PM	WG	✓
ISO20-14-01-030 ✗	PP04; Sample ✗	27.01.2020 02:45 PM	WG	✓
ISO20-14-01-030-DUP ✗	PP04; Sample Duplicate ✗	27.01.2020 02:45 PM	WG	✓
ISO20-14-01-031 ✗	PP05; Sample ✗	27.01.2020 02:50 PM	WG	✓
ISO20-14-01-031-DUP ✗	PP05; Sample Duplicate ✗	27.01.2020 02:50 PM	WG	✓
ISO20-14-01-032 ✗	PP06; Sample ✗	27.01.2020 02:30 PM	WG	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Juli Tichyjt

Collector's signature: Susan T. Wolf

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Juli Tichyjt	31/01/2020	02:00 PM	Fedex	See Pg 11		

3M EHS LABORATORY
Chain-of-Custody

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3M Center, Bldg 260-5N-17
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Phone: (651) [REDACTED]
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Project: ISO20-14-01 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/6/2020

Completion Date:

Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO20-14-01-032-DUP	PP06; Sample Duplicate X	27.01.2020 02:30 PM	WG	✓
ISO20-14-01-033	PP07; Sample X	27.01.2020 01:48 PM	WG	✓
ISO20-14-01-033-DUP	PP07; Sample Duplicate X	27.01.2020 01:48 PM	WG	✓
ISO20-14-01-033-FMS	PP07; Sample FMS X	27.01.2020 01:48 PM	WQ	✓
ISO20-14-01-034	PP08; Sample X	27.01.2020 01:55 PM	WG	✓
ISO20-14-01-034-DUP	PP08; Sample Duplicate X	27.01.2020 01:55 PM	WG	✓
ISO20-14-01-035	PP10; Sample X	27.01.2020 02:05 PM	WG	✓
ISO20-14-01-035-DUP	PP10; Sample Duplicate X	27.01.2020 02:05 PM	WG	✓
ISO20-14-01-036	12; Sample X	28.01.2020 12:15 PM	WG	✓
ISO20-14-01-036-DUP	12; Sample Duplicate X	28.01.2020 12:15 PM	WG	✓
ISO20-14-01-037	13; Sample X	28.01.2020 12:00 PM	WG	✓
ISO20-14-01-037-DUP	13; Sample Duplicate X	28.01.2020 12:00 PM	WG	✓
ISO20-14-01-038	5; Sample X	28.01.2020 11:30 AM	WG	N
ISO20-14-01-038-DUP	5; Sample Duplicate X	28.01.2020 11:30 AM	WG	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Julie Fichter

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Julie Fichter	31/01/2020	02:00 PM	Fedex	See pg 11		

3M EHS LABORATORY
Chain-of-Custody

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Project: ISO20-14-01 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)

Department: 832202 Site Source: 01WJWT10

Project Number:

Date Created: 1/6/2020

Project Description: 3M Antwerp Water Sampling for PFCs

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
✓ ISO20-14-01-039	Bemalingstation; Sample X	28.01.2020 11:00 AM	WQ	✓
✓ ISO20-14-01-039-DUP	Bemalingstation; Sample Duplicate X	28.01.2020 11:00 AM	WQ	✓
✓ ISO20-14-01-039-FMS	Bemalingstation; Sample FMS X	28.01.2020 11:00 AM	WQ	✓
✓ ISO20-14-01-040	Collector put; Sample X	28.01.2020 03:20 PM	WQ	✓
✓ ISO20-14-01-040-DUP	Collector.put; Sample Duplicate X	28.01.2020 03:20 PM	WQ	✓
✓ ISO20-14-01-041 *	P27; Sample	28.01.2020 08:45 AM	WQ	✓
✓ ISO20-14-01-041-DUP *	P27; Sample Duplicate	28.01.2020 08:45 AM	WQ	✓
✓ ISO20-14-01-042 *	P21B; Sample	28.01.2020 10:15 AM	WQ	✓
✓ ISO20-14-01-042-DUP *	P21B; Sample Duplicate	28.01.2020 10:15 AM	WQ	✓
✓ ISO20-14-01-043 *	P304; Sample	28.01.2020 09:45 AM	WQ	✓
✓ ISO20-14-01-043-DUP *	P304; Sample Duplicate	28.01.2020 09:45 AM	WQ	✓
✓ ISO20-14-01-043-FMS *	P304; Sample FMS	28.01.2020 09:45 AM	WQ	✓
✓ ISO20-14-01-044 *	P305; Sample	28/01/2020- 02:55 PM	WQ	✓
✓ ISO20-14-01-044-DUP *	P305; Sample Duplicate	28/01/2020- 02:55 PM	WQ	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Julie Fichfert

Collector's signature: Susan T. Wolf

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Julie Fichfert	31/01/2020	09:00 PM	FedEx	See pg 11		

3M EHS LABORATORY
Chain-of-Custody

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Fax: (651) 429-6380

Project: ISO20-14-01 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/6/2020

Completion Date:

Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO20-14-01-045	P42; Sample	29/01/2020 11:00 AM	WG	✓
ISO20-14-01-045-DUP	P42; Sample Duplicate	29/01/2020 11:00 AM	WG	✓
ISO20-14-01-046	P56; Sample			Not Sampled ✓
ISO20-14-01-046-DUP	P56; Sample Duplicate			Not Sampled ✓
ISO20-14-01-047	L19; Sample	29/01/2020 - 10:00 AM	WG	✓
ISO20-14-01-047-DUP	L19; Sample Duplicate	29/01/2020 - 10:00 AM	WG	✓
ISO20-14-01-048	M4; Sample	28/01/2020 - 03:10 PM	WG	✓
ISO20-14-01-048-DUP	M4; Sample Duplicate	28/01/2020 - 03:10 PM	WG	✓
ISO20-14-01-048-FMS	M4; Sample FMS	28/01/2020 - 03:10 PM	WQ	✓
ISO20-14-01-049	P118C; Sample	28/01/2020 10:10 AM	WG	✓
ISO20-14-01-049-DUP	P118C; Sample Duplicate	28/01/2020 10:10 AM	WG	✓
ISO20-14-01-050	P119C; Sample	28/01/2020 02:35 PM	WG	✓
ISO20-14-01-050-DUP	P119C; Sample Duplicate	28/01/2020 02:35 PM	WG	✓
ISO20-14-01-051	P262bis; Sample	29/01/2020 - 11:30 AM	WG	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Julie Tichijt

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
11	Julie Tichijt	31/01/2020	02:00 PM	Fedex	See pg 11		

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [REDACTED]
Alt. Pho: [REDACTED]
Fax: (651) 429-6400

Project: ISO20-14-01 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)

Department: 832202 Site Source: 01WJWT10

Project Number:

Date Created: 1/6/2020

Project Description: 3M Antwerp Water Sampling for PFCs

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO20-14-01-051-DUP ✓	P262bis; Sample Duplicate	29/01/2020 - 11:30 AM	WG	✓
ISO20-14-01-052 ✓	P263; Sample	29/01/2020 - 10:40 AM	WG	✓
ISO20-14-01-052-DUP ✓	P263; Sample Duplicate	29/01/2020 - 10:40 AM	WG	✓
ISO20-14-01-052-FMS ✓	P263; Sample FMS	29/01/2020 - 10:40 AM	WQ	✓
ISO20-14-01-053 ✓	P264; Sample	28.01.2020 09:50 AM	WG	✓
ISO20-14-01-053-DUP ✓	P264; Sample Duplicate	28.01.2020 09:50 AM	WG	✓
ISO20-14-01-054 ✓	P265B; Sample	28.01.2020 09:00 AM	WG	✓
ISO20-14-01-054-DUP ✓	P265B; Sample Duplicate	28.01.2020 09:00 AM	WG	✓
ISO20-14-01-055 X	P340; Sample	28/01/2020 - 02:00 PM	WG	✓
ISO20-14-01-055-DUP X	P340; Sample Duplicate	28/01/2020 - 02:00 PM	WG	✓
ISO20-14-01-056 ✓	P341; Sample	28/01/2020 - 02:20 AM	WG	✓
ISO20-14-01-056-DUP ✓	P341; Sample Duplicate	28/01/2020 - 02:20 AM	WG	✓
ISO20-14-01-057 X	P371; Sample	28/01/2020 - 12:00 AM	WG	✓
ISO20-14-01-057-DUP X	P371; Sample Duplicate	28/01/2020 - 11:00 AM	WG	✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Julie Fichfyt

Collector's signature: J. Fichfyt

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Julie Fichfyt	31/01/2020	02:00 PM	Fedex	See pg 11		

3M EHS LABORATORY
Chain-of-Custody

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3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [REDACTED]
Alt. Phone: [REDACTED]
Fax: (651) 733-1111

Project: ISO20-14-01 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3MF)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/6/2020

Completion Date:

Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
✓ ISO20-14-01-058	P374; Sample	28.01.2020 11:05 AM	WG	✓
✓ ISO20-14-01-058-DUP	P374; Sample Duplicate	28.01.2020 11:05 AM	WG	✓
✓ ISO20-14-01-058-FMS	P374; Sample FMS	28.01.2020 11:05 AM	WQ	✓
✓ ISO20-14-01-059 X	P379; Sample	28/01/2020 - 01:30 PM	WG	✓
✓ ISO20-14-01-059-DUP X	P379; Sample Duplicate	28/01/2020 - 01:30 PM	WG	✓
✓ ISO20-14-01-060	P380; Sample	28.01.2020 11:00 AM	WG	✓
✓ ISO20-14-01-060-DUP	P380; Sample Duplicate	28.01.2020 11:00 AM	WG	✓
✓ ISO20-14-01-060-FMS	P380; Sample FMS	28.01.2020 11:00 AM	WQ	✓
✓ ISO20-14-01-061	P372; Sample	28.01.2020 11:35 AM	WG	✓
✓ ISO20-14-01-061-DUP	P372; Sample Duplicate	28.01.2020 11:35 AM	WG	✓
✓ ISO20-14-01-062 X	PP13; Sample	27.01.2020 03:00 PM	WG	✓
✓ ISO20-14-01-062-DUP X	PP13; Sample Duplicate	27.01.2020 03:00 PM	WG	✓
✓ ISO20-14-01-063 X	PP13; Sample	[REDACTED]		Not Sampled ✓
✓ ISO20-14-01-063-DUP X	PP13; Sample Duplicate			not Sampled ✓

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Julie Tichyfit

Collector's signature: J. Tichyfit

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Julie Tichyfit	31/01/2020	02:00PM	FedEx	See pg 11		

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [REDACTED]
Alt. Phone: [REDACTED]
Fax: (651) 733-1111

Project: ISO20-14-01 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 1/6/2020

Project Description: 3M Antwerp Water Sampling for PFCs

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO20-14-01-063-FMS	PPI3; Sample FMS			not sampled ✓
ISO20-14-01-064	Travel Blank X	01/07/2020 1:07pm		① ✓
ISO20-14-01-064-FMS-LOW	Travel Blank FMS Low X	01/07/2020 1:07pm		① ✓
ISO20-14-01-064-FMS-HIGH	Travel Blank FMS High X	01/07/2020 1:07pm		① ✓

① Travel Blank set prepared by 3M EHS Laboratory. CSG 1/7/20

- Samples marked w/ "•" received 2-3-20 @ 1100.
- Samples marked w/ "X" received 2-4-20 @ 1130.
- "Sample condition" filled out by sender. Samples were all received as noted, Room Temp, acceptable.

Alex Ling 2-4-20

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 10 Deg C Received on Ice Other:

Collected by (print): Julie Fitchfet

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
4	Julie Fitchfet	3/09/2020	02:00PM	Fedex	See notes above		



3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC20-01194-002

3M Lab Request Number: E20-0388

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: June 5th, 2020

Requester

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Member of the SGS Group (Société Générale de Surveillance)

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1. Introduction - Summary.

At the request (Lab Request Number: E20-0388) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, one water sample was collected in April 2020 from 3 locations and analyzed for the following perfluorinated compounds:

- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$) (and its ^{13}C -labeled analogues)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$) (and its ^{13}C -labeled analogues)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$) (and its ^{13}C -labeled analogues)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$) (and its ^{13}C -labeled analogue)
- ^{13}C -labeled analogue of PFUdA ($C_4F_{21}^{13}C_6F_2^{13}COO^-$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared one sample container for each sampling location under the direction of Sven Herremans. Each empty container was marked with a "fill to here" line and was fortified with a surrogate recovery spike and an internal standard spike, prior to being sent to the field for sample collection.

Table 1 summarizes the sample results with their uncertainty. All PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$ unless noted otherwise. See Section 4 of the report for additional information on the method QC used to assess method uncertainty.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)			
	PFHS	PFOA	PFOS	FOSA
3M Vijver	0.521	1.41	3.57	<0.025
Blokkersdijkvijver Noord	0.423	1.01	0.970	0.056
Blokkersdijkvijver standaard (filter)	0.477	1.05	5.07	0.191
Blokkersdijkvijver standaard (nt filter)	0.449	1.07	4.45	0.171

(a): The recovery of FOSA for Blokkersdijkvijver Noord is $\pm 50\%$.

(b): The recovery of FOSA for Blokkersdijkvijver standard (not filter) is $\pm 230\%$.

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

The sample was collected in a polyethylene bottle prepared at the SGS Belgium NV, Division IAC Laboratory. Based on the concentrations as reported in previous reports, the bottle was spiked, prior to sample collection, in the laboratory with a known volume of a surrogate recovery solution ($^{13}\text{C}_2\text{-PFHS}$, $^{13}\text{C}_4\text{-PFOA}$, $^{13}\text{C}_4\text{-PFOS}$, $^{13}\text{C}_7\text{-PFUdA}$) and internal standard solution ($^{13}\text{C}_3\text{-PFHS}$, $^{13}\text{C}_8\text{-PFOA}$, $^{13}\text{C}_8\text{-PFOS}$, $^{13}\text{C}_8\text{-FOSA}$). Table 2 below details the sample collected and spikes added to the bottle.

Table 2. Sample Collection and Spike Information.

Sample Identification	Nominal Final Volume Collected (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Final Nominal Spike Concentration (ng/mL)	
				$^{13}\text{C-PFC-SS}$	$^{13}\text{C-PFC-IS}$
3M vijver	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
Blokkersdijkvijver Noord	400	0.1	Solution A	0.25	-
		0.25	Solution B	-	1.0
Blokkersdijkvijver standaard (filter)	400	0.1	Solution A	0.25	-
		0.25	Solution B	-	1.0
Blokkersdijkvijver standaard (nt filter)	400	0.1	Solution A	0.25	-
		0.25	Solution B	-	1.0

Solution A = 1000 ng/mL (nominal) $^{13}\text{C-PFC}$ Surrogate Recovery Standards

Solution B = 1500 ng/mL (nominal) $^{13}\text{C-PFC}$ Internal Standards

2.2. Extraction.

All samples, calibration standards, and associated quality control samples were extracted using a modified procedure of ECO/AV/IAC/064 “Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)”. Briefly, an amount of sample (see table 3) was loaded, onto a pre-conditioned Waters tC18 solid phase extraction (SPE) cartridge (Sep-Pak, 1g, 6cc) using a vacuum manifold. The loaded SPE cartridges were then eluted with 5 mL of methanol using vacuum.

Table 3. Sample amount used.

Sample	Amount of Sample used (mL)	Concentration Factor
3M Vijver	40	8
Blokkersdijkvijver Noord	40	8
Blokkersdijkvijver standard (filter)	40	8
Blokkersdijkvijver standard (nt filter)	40	8
Field Trip Blank	40	8

2.3. Determination of suspended solids in water.

To avoid the influence of algae on the analytical result, filtration is proposed prior to extraction at the sampling locations Blokkersdijkvijver - standard.

The filtration was done by using Whatman GF/C glass-fiber filters, with a pore size of 1.2 µm.

The total suspended solids of the samples is determined by pouring a measured volume of sample, typically 200 mL, through a pre-weighed filter then weighing the filter again after drying the filter overnight to remove all water. The gain in weight is a dry weight measure of the particulates present in the water sample. This is expressed in units calculated from the volume of water filtered, milligrams per liter. Table 4 summarizes the results.

Table 4. Suspended solids.

Sample Identification	Suspended solids/ volume (mg/L)
Blokkersdijkvijver - standard (filtered)	0.02

2.4. Analysis.

All solutions and extracts were analyzed for the PFCs (PFHS, PFOA, PFOS, FOSA) and the surrogate recovery standards ($^{13}\text{C}_2\text{-PFHS}$, $^{13}\text{C}_4\text{-PFOA}$, $^{13}\text{C}_4\text{-PFOS}$, $^{13}\text{C}_7\text{-PFUdA}$) and internal standards ($^{13}\text{C}_3\text{-PFHS}$, $^{13}\text{C}_8\text{-PFOA}$, $^{13}\text{C}_8\text{-PFOS}$, $^{13}\text{C}_8\text{-FOSA}$) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetrie C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C ₃ -PFHS	2.00 - 3.00	402.0 /80.0	60	38	51
¹³ C ₂ -PFHS	2.30 - 3.30	403.0/84.0	60	38	51
¹³ C ₈ -PFOS	3.00 - 4.00	507.0/80.0	60	48	56
¹³ C ₄ -PFOA	2.30 - 3.50	417.0 /372.0	100	11	14
¹³ C ₈ PFOA	2.30 - 3.50	421.0 /376.0	60	11	14
PFHS	2.00 - 3.00	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.30 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
¹³ C ₄ -PFOS	3.00 - 4.00	503.0 /80.0	60	48	56
¹³ C ₇ PFUD _A	4.30 - 5.60	570.0 /525.0	60	12	17
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
¹³ C ₈ FOSA	4.30 - 5.70	506.0/78.0	60	34	44

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Extracted Calibration Standard.

Extracted calibration standards were prepared by spiking known amounts of stock solutions containing PFHS, PFOA, PFOS, FOSA and ^{13}C -labeled analogues into 40 mL of HPLC water. Each spiked water standard was then extracted in the same manner as the collected samples. A total of 12 spiked standards ranging from 0.005 ng/mL to 100 ng/mL (nominal) were prepared. Each curve point contains the mixture of internal standards at a nominal concentration of 1 ng/mL. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. The calibration curve will be generated by taking the ratio of the standard peak area counts over the internal standard peak area counts to fit the data for each analyte. Each extracted calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of $\pm 25\%$ ($\pm 30\%$ for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The 0.025ng/mL (nominal) reporting limit is a practical quantitation limit (PQL) required by the requester and it is possible that the samples contain target analytes at quantifiable concentrations below the PQL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within $\pm 25\%$, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by loading 40 mL of HPLC water onto a Waters tC18 solid phase extraction (SPE) cartridge (SEP-Pak, 1g, 6cc) and eluting with 5 mL of methanol using the same extraction procedure as the samples. Method blanks were prepared to evaluate the levels of background contamination in the overall extraction process (glassware, SPE cartridges, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carry over.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of HPLC water, spiked with the surrogate recovery standards and internal standards, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCSs)

Low (0.125 ng/mL nominal concentration) and high (2.5 ng/mL nominal concentration) lab control spikes were prepared and analyzed in duplicate. LCSs were prepared by spiking known amounts of the analytes and surrogates into 40 mL of HPLC water to produce the desired concentration. The spiked water samples were extracted and analyzed in the same manner as the samples.

All LCSs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

All LCSs produced recoveries within the method acceptance criteria of $\pm 15\%$ RPD for precision, except for 13C4PFOS and PFOS.

Table 8 summarize the LCS recovery results.

Table 7. Lab Control Spike Results.

Extraction date	Description	Nominal Spike Level (ng/mL)	Percent Recovery							
			¹³ C ₄ -PFOA	¹³ C ₄ -PFOS	¹³ C ₂ -PFHS	¹³ C ₇ -PFuDA	PFHS	PFOA	PFOS	FOSA
20 May 2019	LCSL01	0.125	95.8	103	114	123	91.3	102	91.9	107
	LCSL02	0.125	94.3	102	105	119	81.0	89.8	90.0	91.2
	Average		95.1	102	109	121	86.2	96.0	91.0	99.2
	%RPD		1.6	1.6	8.2	2.9	12	13	2.1	16
	LCSH 01	2.5	119	121	116	118	118	86.0	124	119
	LCSH 02	2.5	102	98.8	121	110	114	76.6	97.6	106
	Average		110	110	118	114	116	81.3	111	113
	%RPD		15	20(a)	4.5	7.5	3.6	12	24(a)	12

(a) The recovery of the RPD fell outside the method acceptance criterion of $\pm 15\%$.

3.6. Surrogates.

Surrogate recovery standards were added to all samples to evaluate overall method performance.

3.7. Internal Standards.

Internal standards were added to all samples to calculate the concentration of PFCs in the samples by using internal standard calibration.

3.8. Equations.

Recovery.

$$\text{Recovery(%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

Tabel 8 and 9 summarizes the results for the sample locations.

Table 8. Sample Results PFHS and PFOA.

	PFHS ng/ml	¹³ C ₂ -PFHS %Rec	PFOA ng/ml	¹³ C ₄ -PFOA %Rec
Field Trip Blank	0.285	102	0.285	94.1
3M vijver	0.291	105	0.323	107
Blokkersdijkvijver Noord	0.301	108	0.338	112
Blokkersdijkvijver standaard (filter)	0.295	106	0.321	106
Blokkersdijkvijver standaard (nt filter)	0.299	107	0.315	104

Table 9. Sample Results PFOS and FOSA.

	PFOS ng/ml	¹³ C ₄ -PFOS %Rec	FOSA ng/ml	¹³ C ₇ -PFuDA %Rec
Field Trip Blank	0.264	96.2	0.453	153 (a)
3M vijver	0.255	93.1	0.363	122
Blokkersdijkvijver Noord	0.306	112	0.451	152 (a)
Blokkersdijkvijver standaard (filter)	0.242	88.2	0.308	104
Blokkersdijkvijver standaard (nt filter)	0.288	105	0.912	307 (a)

(a): The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130%.

All PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$, except for FOSA for the following sample locations:

- (a): The recovery of FOSA for Blokkersdijkvijver Noord is $\pm 50\%$.
- (b): The recovery of FOSA for Blokkersdijkvijver standard (nt filter) is $\pm 250\%$.

5. Conclusion.

- The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130% for two sample locations.
- Lab control spike recoveries fell within the method acceptance criteria of 25%.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Julie Fichefet (ERM Belgium).

8. Signatures.

Sven Herremans,
Lab Operations Manager

Date June 5th, 2020



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date June 5th, 2020

I.A.C.
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The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.



3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC20-01194-001

3M Lab Request Number: E20-0388

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: June 5th, 2020

Requester

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Member of the SGS Group (Société Générale de Surveillance)

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1. Introduction - Summary.

At the request (Lab Request Number: E20-0388) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected in April 2020 from 22 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOA ($C_6F_{13}^{13}CF_2^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOS ($C_7F_{15}^{13}CF_2SO_3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Sven Herremans. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles.

Table 1 summarizes the sample results. All PFBS, PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
L21		0.305	0.396	3.91	<0.215
L30		0.672	0.763	6.70	<0.215
L31		1.00	0.666	3.78	<0.215
L4		2.08	5.28	15.4	2.33
P114bis		1.61	5.20	4.30	<0.215
P115		2.01	4.32	2.14	<0.215
P116		0.771	1.32	8.89	<0.215
Effluent WWTP		145	15.9	32.4	0.605
PP02	49.9	3434	808	18511	26.3
PP04	740	148	368	3968	38.7
PP05	3448	1816	601	21.1	<12.8
PP06	19	71	612	1079	46
PP07	267.7	93.8	292.0	1694	51.9
PP08	32.4	210	537	4925	68.7
PP10	14.5	160	486	2845	20.0
PP11	226	112.0	295	1641	22.3
12		19.6	79.9	278	<0.641
13		21.3	74.4	225	<0.641
5		7.06	9.31	11.0	<0.215
Bemalingsstation		26.7	34.2	36.9	<0.215
Collector Put		7.57	25.4	43.4	4.94

2. Methods – Analytical and Preparatory.**2.1. Sample collection.**

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
L21	100
L30	100
L31	100
L4	100
P114bis	100
P115	100
P116	100
Effluent WWTP	100
PP02	100
PP04	100
PP05	100
PP06	100
PP07	100
PP08	100
PP10	100
PP11	100
12	100
13	100
5	100
Bemalingsstation	100
Collector Put	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
L21	1.0	0.05	Solution A	1.0	-
L30	1.0	0.05	Solution A	1.0	-
L31	1.0	0.05	Solution A	1.0	-
L4	1.0	0.05	Solution A	1.0	-
P114bis	1.0	0.05	Solution A	1.0	-
P115	1.0	0.05	Solution A	1.0	-
P116	1.0	0.05	Solution A	1.0	-
Effluent WWTP	1.0	0.05	Solution A	1.0	-
PP02	1.0	0.1	Solution A	10.0	0.5 mL extract + 10.0 mL MeOH (*)
PP04	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
PP05	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
PP06	1.0	0.1	Solution A	10.0	1.0 mL extract + 1.0 mL MeOH (*)
PP07	1.0	0.1	Solution A	10.0	1.0 mL extract + 5.0 mL MeOH (*)
PP08	1.0	0.1	Solution A	10.0	1.0 mL extract + 5.0 mL MeOH (*)
PP10	1.0	0.1	Solution A	10.0	1.0 mL extract + 5.0 mL MeOH (*)
PP11	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
12	1.0	0.1	Solution A	5.0	-
13	1.0	0.1	Solution A	5.0	-
5	1.0	0.05	Solution A	1.0	-
Bemalingsstation	1.0	0.05	Solution A	1.0	-
Collector Put	1.0	0.05	Solution A	1.0	-

(*): MeOH:LCMS-water (60:40)

Solution A = 1000 ng/mL (nominal) ¹³C-PFC Surrogate Standards

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetre C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate ($\mu\text{L}/\text{min}$)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
PFBS	2.08 - 2.50	298.93/79.82	40	28	45
		298.93/98.78	40	29	45
PFHS	2.45 - 3.05	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.80 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
¹³ C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of \pm 25% (\pm 30% for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.0770 to 0.120 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.158 to 28.0 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within \pm 25%, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by diluting 1 mL water with 10.0 mL MeOH:LCMS-water (60:40). Method blanks were prepared to evaluate the levels of background contamination in the overall dilution process (glassware, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carryover.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of LCMS-water, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCS)

One lab control spike was prepared and analyzed.

LCS was prepared by spiking known amounts of the analytes into 1.0 mL of LCMS-water to produce the desired concentration.

The spiked water sample was diluted with 10.0 mL MeOH:LCMS-water (60:40) and analyzed in the same manner as the samples.

The LCS produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 7 summarizes the LCS recovery results

Table 7. Lab Control Spike Results.

Dilution date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	¹³ C-PFOA	PFOS	FOSA	¹³ C-PFOS
20 May 2020	LCS1	80	89.6	86.9	86.9	94.1	97.3	94.5	106

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS (QC Lab Low and QC Lab High).

The QCs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy, except for one QC high lab (QC Lab FOSA High recovery is $>25\%$).

Table 8 summarizes the QCs recovery results.

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	¹³ C-PFOA	PFOS	FOSA	¹³ C-PFOS
20 May 2020	QC Lab Low inj1	10	94.3	83.1	102	124	102	95.7	102
	QC Lab High inj1	100	87.2	81.3	102	104	102	55.9*	96.3
	QC Lab Low inj2	10	95.6	83.8	98.9	95.3	103	96.4	105
	QC Lab High inj2	100	88.4	80.9	102	102	102	56.3*	98.2

- Outside the method acceptance criteria

3.7. Equations.Recovery.

$$\text{Recovery(%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

The following tables summarize the results for the sample locations.

Table 9. Sample Results PFBS, PFHS, PFOA, PFOS and FOSA

	PFBS ng/ml	PFHS ng/ml	PFOA ng/ml	PFOS ng/ml	FOSA ng/ml
L21		0.305	0.396	3.91	<0.215
L30		0.672	0.763	6.70	<0.215
L31		1.00	0.666	3.78	<0.215
L4		2.08	5.28	15.4	2.33
P114bis		1.61	5.20	4.30	<0.215
P115		2.01	4.32	2.14	<0.215
P116		0.771	1.32	8.89	<0.215
Effluent WWTP		145	15.9	32.4	0.605
PP02	49.9	3434	808	18511	26.3
PP04	740	148	368	3968	38.7
PP05	3448	1816	601	21.1	<12.8
PP06	19	71	612	1079	46
PP07	267.7	93.8	292.0	1694	51.9
PP08	32.4	210	537	4925	68.7
PP10	14.5	160	486	2845	20.0
PP11	226	112.0	295	1641	22.3
12		19.6	79.9	278	<0.641
13		21.3	74.4	225	<0.641
5		7.06	9.31	11.0	<0.215
Bemalingsstation		26.7	34.2	36.9	<0.215
Collector Put		7.57	25.4	43.4	4.94
Field Trip Blank	<1.21	<1.33	<0.855	<1.27	<1.17

Table 10. Sample Results ^{13}C -PFOA and ^{13}C -PFOS

	^{13}C -PFOA		^{13}C -PFOS	
	ng/ml	% Rec	ng/ml	% Rec
L21	72.3	126	46.9	76.2
L30	71.5	125	49.3	80.1
L31	71.8	125	49.1	79.8
L4	74.3	129	47.4	77.0
P114bis	68.2	119	51.7	84.0
P115	67.1	117	52.6	85.4
P116	72.9	127	48.4	78.5
Effluent WWTP	61.7	108	57.3	93.0
PP02	113.3	98.8	125.4	102
PP04	119	104	119	96.6
PP05	107	93.5	131	106
PP06	114	98.9	125	101
PP07	115.4	100	122.5	100
PP08	117	102	120	97.8
PP10	121	106	117	94.6
PP11	111.4	97.0	126.3	103
12	117	102	121	98.6
13	116	101	122	98.7
5	72.0	126	49.0	79.5
Bemalingsstation	68.9	120	51.2	83.2
Collector Put	71.0	124	49.7	80.7
Field Trip Blank	116	98.7	127	101

5. Conclusion.

- The recovery of ¹³C-PFOA and ¹³C-PFOS fell within the method acceptance criterion of 70-130%.
- Lab control spike recoveries fell within the method acceptance criteria for all compounds, indicating that the analysis method is in control,
- QC Lab Standards recoveries fell within the method acceptance criteria for all compounds, except for one QC lab high (QC Lab High FOSA is > 25%).

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Julie Fichefet (ERM Belgium).

8. Signatures.

Sven Herremans,
Lab Operations Manager

Date June 5th, 2020



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date June 5th, 2020

I.A.C.
A Division of SGS Belgium NV

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The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.

Final Report

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

April 2020 Sampling

Laboratory Request Number: ISO20-14-02

Report Date – Date of Last Signature

Testing Laboratory

3M EHS Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Charlotte Tack
3M Belgium
3M Belgium; ZW019/01/01
Phone: [REDACTED]



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2017 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M EHS Laboratory

3M EHS Laboratory Manager: Brian T. Mader
3M Principal Analytical Investigator: Susan Wolf
Report Author: Scott T. Porcher

Analytical Report ISO20-14-02

Water Sample Analysis at 3M Antwerp, Belgium
April 2020 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M EHS Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. All samples were collected between April 28 - 29, 2020, and returned to the 3M EHS Laboratory on May 4, 2020, at ambient temperature. The results in this report apply to the samples as received from ERM. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS) (select locations only), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO20-14-02.

The 3M EHS Laboratory prepared sample containers for twenty-nine sampling locations. Each sample set consisted of a field sample and field sample duplicate. Seven locations also included a target analyte field matrix spike. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with a mass-labeled internal standard mix and select sample bottles were fortified with surrogate recovery standards [$^{13}\text{C}_4$]-PFOA and [$^{13}\text{C}_4$]-PFOS prior to being sent to the field for sample collection. During sample collection, sample location L22 was replaced with location L30 as L22 was broken as indicated on the chain of custody. Sample locations PP12 and PP13 were not sampled, as they were not installed by the time of sampling.

Samples were prepared and analyzed using method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Select internal standards were used to aid in the data quality objectives.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2017 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary ⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: 2nd Aquifer					
ISO20-14-02-001	P121; Sample	1.37	2.05	7.02	<0.0990
ISO20-14-02-001-DUP	P121; Sample Dup	1.34	2.04	6.90	<0.0990
		Average	1.36	2.05	6.96
		%RPD Sample/Sample Dup	2.2	0.49	1.7
					<0.0990 ⁽¹⁾
ISO20-14-02-005	P321; Sample	2150	511	5560	0.734
ISO20-14-02-005-DUP	P321; Sample Dup	2320	558	5840	0.600
		Average	2240	535	5700
		%RPD Sample/Sample Dup	7.6	8.8	4.9
					0.667
Zone: Blokkersdijk Nature Reserve					
ISO20-14-02-002	3M vijver; Sample	0.706	0.529	3.65	<0.0990
ISO20-14-02-002-DUP	3M vijver; Sample Duplicate	0.643	0.510	3.41	<0.0990
		Average	0.675	0.520	3.53
		%RPD Sample/Sample Dup	9.3	3.7	6.8
					<0.0990
ISO20-14-02-003	Blokkersdijkvijver Noord; Sample	0.740	0.466	0.930	<0.0990
ISO20-14-02-003-DUP	Blokkersdijkvijver Noord; Sample Duplicate	0.740	0.464	0.910	<0.0990
		Average	0.740 ⁽²⁾	0.465 ⁽²⁾	0.920 ⁽²⁾
		%RPD Sample/Sample Dup	0.0	0.43	2.2
					<0.0990
ISO20-14-02-004	Blokkersdijkvijver standaard; Sample	0.597	0.444	4.70	0.194
ISO20-14-02-004-DUP	Blokkersdijkvijver standaard; Sample Duplicate	0.709	0.453	4.71	0.180
		Average	0.653	0.449	4.71
		%RPD Sample/Sample Dup	17	2.0	0.21
					0.187
ISO20-14-02-006	L21; Sample	0.308	0.198	6.38	0.0796
ISO20-14-02-006-DUP	L21; Sample Dup	0.304	0.200	6.08	0.0754
		Average	0.306 ⁽²⁾	0.199 ⁽²⁾	6.23 ⁽²⁾
		%RPD Sample/Sample Dup	1.3	1.0	4.8
					0.0775
ISO20-14-02-007	L30; Sample	0.392	0.492	8.46	0.0750
ISO20-14-02-007-DUP	L30; Sample Dup	0.398	0.496	8.46	0.0728
		Average	0.395 ⁽²⁾	0.494 ⁽²⁾	8.46 ⁽²⁾
		%RPD Sample/Sample Dup	1.5	0.81	0.0
					0.0739
ISO20-14-02-008	L31; Sample	0.296	0.782	3.50	<0.0990
ISO20-14-02-008-DUP	L31; Sample Dup	0.374	0.830	4.48	<0.0990
		Average	0.335 ⁽²⁾	0.806 ⁽²⁾	3.99 ⁽²⁾
		%RPD Sample/Sample Dup	23 ⁽³⁾	6.0	25 ⁽³⁾
					<0.0990
ISO20-14-02-009	L4; Sample	3.22	1.91	18.6	3.98
ISO20-14-02-009-DUP	L4; Sample Dup	3.21	2.04	19.2	4.24
		Average	3.22	1.98	18.9
		%RPD Sample/Sample Dup	0.31	6.6	3.2
					4.11
					6.3

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 27%, PFBS \pm 16%, PFHS \pm 24%, PFOS \pm 28% and PFOSA \pm 22%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 16%, PFHS \pm 11%, PFOS \pm 8.8%, and PFOSA \pm 15%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) Surrogate recoveries did not meet method acceptance criteria. See section 4 of the report for more information.

Table 1 continued. Sample Results Summary ⁽¹⁾

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Blokkersdijk Nature Reserve					
ISO20-14-02-010	P114bis; Sample	3.44	1.45	5.36	<0.0500
ISO20-14-02-010-DUP	P114bis; Sample Dup	3.33	1.53	5.22	<0.0500
		Average	3.39	1.49	5.29
		%RPD Sample/Sample Dup	3.2	5.4	<0.0500
ISO20-14-02-011	P115; Sample	2.91	1.73	2.94	<0.0500
ISO20-14-02-011-DUP	P115; Sample Dup	3.22	1.73	2.72	<0.0500
		Average	3.07	1.73	2.83
		%RPD Sample/Sample Dup	10	0.0	<0.0500
ISO20-14-02-012	P116; Sample	0.483	0.444	10.4	0.142
ISO20-14-02-012-DUP	P116; Sample Dup	0.524	0.368	10.3	0.133
		Average	0.504	0.406	10.4
		%RPD Sample/Sample Dup	8.1	19	0.138
					6.5
Zone: Effluent WWTP					
ISO20-14-02-013	Effluent WWTP; Sample	13.1	161	42.4	1.16
ISO20-14-02-013-DUP	Effluent WWTP; Sample Dup	13.8	164	44.4	1.00
		Average	13.5	163	43.4
		%RPD Sample/Sample Dup	5.2	1.8	1.08
					15
Zone: Palingbeek & Tophatgracht					
ISO20-14-02-021	12; Sample	89.1	25.8	387	0.646
ISO20-14-02-021-DUP	12; Sample Dup	89.2	25.8	387	0.670
		Average	89.2	25.8	387
		%RPD Sample/Sample Dup	0.11	0.0	0.658
					3.6
ISO20-14-02-022	13; Sample	86.3	27.2	313	0.558
ISO20-14-02-022-DUP	13; Sample Dup	85.1	26.8	316	0.560
		Average	85.7	27.0	315
		%RPD Sample/Sample Dup	1.4	1.5	0.559
					0.36
ISO20-14-02-023	5; Sample	6.70	6.43	14.9	0.108
ISO20-14-02-023-DUP	5; Sample Dup	6.77	6.32	13.1	0.0892
		Average	6.74	6.38	14.0
		%RPD Sample/Sample Dup	1.0	1.7	0.0986
					19
ISO20-14-02-024	Bemalingstation; Sample	27.5	24.3	44.3	0.170
ISO20-14-02-024-DUP	Bemalingstation; Sample Dup	27.4	24.3	45.7	0.176
		Average	27.5	24.3	45.0
		%RPD Sample/Sample Dup	0.36	0.0	0.173
					3.5

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 27%, PFBS \pm 16%, PFHS \pm 24%, PFOS \pm 28% and PFOSA \pm 22%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 16%, PFHS \pm 11%, PFOS \pm 8.8%, and PFOSA \pm 15%
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) Surrogate recoveries did not meet method acceptance criteria. See section 4 of the report for more information.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Sewer					
ISO20-14-02-025	Collector put; Sample	21.8	7.31	51.4	10.4
ISO20-14-02-025-DUP	Collector put; Sample Dup	21.6	7.22	51.0	10.6
Average		21.7	7.27	51.2	10.5
%RPD Sample/Sample Dup		0.92	1.2	0.78	1.9

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Extraction Wells						
ISO20-14-02-014	PP02; Sample	668	37.7	3780	19400	32.8
ISO20-14-02-014-DUP	PP02; Sample Dup	698	41.5	3960	20800	36.8
Average		683	39.6⁽⁴⁾	3870	20100	34.8
%RPD Sample/Sample Dup		4.4	9.6	4.7	7.0	11
ISO20-14-02-015	PP04; Sample	353	708	148	5110	64.6
ISO20-14-02-015-DUP	PP04; Sample Dup	354	706	148	5110	65.6
Average		354	707	148	5110	65.1
%RPD Sample/Sample Dup		0.28	0.28	0.0	0.0	1.5
ISO20-14-02-016	PP05; Sample	582	4170	2820	95.0	16.8
ISO20-14-02-016-DUP	PP05; Sample Dup	575	4170	2820	91.0	18.4
Average		579	4170	2820	93.0	17.6
%RPD Sample/Sample Dup		1.2	0.0	0.0	4.3	9.1
ISO20-14-02-017	PP06; Sample	659	14.9	90.9	1390	84.4
ISO20-14-02-017-DUP	PP06; Sample Dup	641	14.7	90.0	1320	74.6
Average		650	14.8	90.5	1360	79.5
%RPD Sample/Sample Dup		2.8	1.4	1.0	5.2	12
ISO20-14-02-018	PP07; Sample	313	299	124	2340	97.8
ISO20-14-02-018-DUP	PP07; Sample Dup	316	300	126	2270	91.0
Average		315	300	125	2310	94.4
%RPD Sample/Sample Dup		0.95	0.33	1.6	3.0	7.2
ISO20-14-02-019	PP08; Sample	518	31.2	259	6350	84.0
ISO20-14-02-019-DUP	PP08; Sample Dup	519	30.8	257	6280	95.4
Average		519	31.0	258	6320	89.7
%RPD Sample/Sample Dup		0.19	1.3	0.78	1.1	13
ISO20-14-02-020	PP10; Sample	478	10.4	195	3400	28.2
ISO20-14-02-020-DUP	PP10; Sample Dup	476	10.1	194	3400	26.0
Average		477	10.3	195	3400	27.1
%RPD Sample/Sample Dup		0.42	2.9	0.51	0.0	8.1

NA = Not Applicable

- (1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA \pm 27%, PFBS \pm 16%, PFHS \pm 24%, PFOS \pm 28% and PFOSA \pm 22%.
- (2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA \pm 16%, PFHS \pm 11%, PFOS \pm 8.8%, and PFOSA \pm 15%.
- (3) Sample/sample duplicate RPD did not meet acceptance criteria of \leq 20%.
- (4) Surrogate recoveries did not meet method acceptance criteria. See section 4 of the report for more information.

Table 1 continued. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA ⁽²⁾
Zone: Extraction Wells						
ISO20-14-02-028	PP11; Sample	262	197	126	1860	42.4
ISO20-14-02-028-DUP	PP11; Sample Dup	263	197	124	1720	32.2
Average		263	197	125	1790	37.3
%RPD Sample/Sample Dup		0.38	0.0	1.6	7.8	27⁽³⁾
Zone: Source Area - Building 16						
ISO20-14-02-026	P21B; Sample	9890	7880	13000	67100	6.42
ISO20-14-02-026-DUP	P21B; Sample Dup	8330	6680	10900	69500	6.02
Average		9110	7280	12000	68300	6.22
%RPD Sample/Sample Dup		17	16	18	3.5	6.4
ISO20-14-02-027	Travel Blank	<0.0480 ⁽²⁾	<0.500	<0.0250 ⁽²⁾	<0.0464 ⁽²⁾	<0.0500

NA = Not Applicable

(1) Unless noted otherwise, the sample set was reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 27%, PFBS ± 16%, PFHS ± 24%, PFOS ± 28% and PFOSA ± 22%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 16%, PFHS ± 11%, PFOS ± 8.8%, and PFOSA ± 15%.

(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(4) Surrogate recoveries did not meet method acceptance criteria. See section 4 of the report for more information.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis".

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected on April 28 - 29, 2020, in Nalgene™ (high-density polyethylene) bottles prepared at the 3M EHS Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. All sample locations were spiked with a mass-labeled internal standard mix at a nominal concentration of 1 ng/mL and select samples were fortified with surrogate recovery standards (SRSSs) at a nominal concentration of 0.1 ng/mL prior to sample collection. Samples were received on May 4, 2020.

2.3 Sample Preparation

All samples were initially prepared for PFOA, PFHS, PFOS and PFBS (select locations only) using a direct injection by methanol dilution method. Samples that required a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples that required a 1:100 dilution were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol. Samples that required a 1:500 dilution were prepared by diluting 0.02 mL of a well-mixed sample with 10.0 mL of methanol. Samples that required a 1:1000 dilution were prepared by first diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol, followed by a second dilution prepared by diluting 1 mL of the diluted sample into 9 mL of methanol. Samples were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

Samples requiring preparation with a lesser dilution factor were analyzed for FOSA with select locations analyzed for PFOA, PFHS, and PFOS by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples along with the method blanks.

2.4 Analysis

All samples and quality control samples were analyzed for the target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). SRSs were added and included in the analysis of these samples. Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.2. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

The following analytical runs were used to report the results herein:

6/29/20 (ETS Athena) External Standard Calibration Analysis:

- All sample locations were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported **except** for the following sample locations: Blokkersdijkvijer Noord, L21, L30 and L31(all analytes), Travel Blank (PFOA, PFHS and PFOS), PP02 (PFBS) and P21B (PFOS).

7/6/20 (ETS Kirk) Internal Standard Calibration Analysis:

- All sample locations were analyzed for PFOA, PFHS, and PFOS. All sample results were reported.

7/6/20 (ETS Ginger) Internal Standard Calibration Analysis:

- Sample locations were analyzed and reported for PFOSA: All sample results were reported, **except** for the following sample locations: P121, 3M vijver, Blokkersdijkvijer Noord, L31, PP04, PP06, PP07 and PP08.

7/9/20 (ETS Ginger) Internal Standard Calibration Analysis:

- Sample locations were analyzed and reported for PFOSA: All sample results were reported, **except** for the following sample locations: PP11 and Travel Blank. Sample P121 is reported using external standard calibration analysis.

7/10/20 (ETS Kirk) External Standard Calibration Analysis:

- Sample location PP02 is reported for PFBS and P21B for PFOS.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk	ETS Ginger	ETS Hermes
Liquid Chromatograph	Agilent 1260	Agilent 1100	Agilent 1100
Analysis Method	ETS-8-044.3	ETS-8-044.3	ETS-8-044.3
Analysis Date	7/6/20, 7/10/20	7/6/20, 7/9/20	6/29/20
Guard column	Betasil C8 (2.1 mm X 50 mm), 5 μ	NA	Betasil C8 (2.1 mm X 50 mm), 5 μ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ
Injection Volume	2 or 10 μ L	2 or 5 μ L	5 μ L
Mass Spectrometer	AB Sciex Triple Quad 5500	API 5000	AB Sciex Triple Quad 5500
Ion Source	Turbo Spray	Turbo Spray	Turbo Spray
Polarity	Negative	Negative	Negative
Software	Analyst 1.7.1	Analyst 1.7.1	Analyst 1.7.1

NA = Not Applicable

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis				
Step Number	Total Time (min)	Flow Rate (μ L/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0.00	300	90.0	10.0
1	0.50	300	90.0	10.0
2	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard	Mass Transition Q1/Q3
PFOA	413/369	$[^{13}\text{C}_8]\text{-PFOA}$	421/376
	413/219		
	413/169		
PFBS	299/99	$[^{18}\text{O}_2]\text{-PFBS}$	303/84
	299/80		
PFHS	399/99	$[^{13}\text{C}_3]\text{-PFHS}$	402/80
	399/80		
PFOS	499/99	$[^{13}\text{C}_8]\text{-PFOS}$	507/80
	499/80		
	499/130		
PFOSA	498/78	$[^{13}\text{C}_8]\text{-PFOSA}$	506/78
$[^{13}\text{C}_4]\text{-PFOA}$	417/372	$[^{13}\text{C}_8]\text{-PFOA}$	421/376
$[^{13}\text{C}_4]\text{-PFOS}$	503/80	$[^{13}\text{C}_8]\text{-PFOS}$	507/80

The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

3 Data Analysis

3.1 Calibration

7/6/20 Analysis (Internal Standard Calibration): Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of fourteen calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed. The calibration standards also contained surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

6/29/20 and 7/10/20 Analysis (External Standard Calibration): Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL to 25 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

7/9/20 Analysis of PFOSA (Internal Standard Calibration): Samples were analyzed for PFOSA against an internal standard matrix-matched calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into laboratory reagent water and diluted 1:1 with methanol in the same manner as the samples. Calibration standards ranging from 0.0125 ng/mL to 150 ng/mL (nominal) were analyzed. A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±25% (100±30% for the lowest curve point) were met for all analytes. The correlation coefficient (*r*) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes for each analysis, except for the following:

- Analysis 7/10/20: [¹³C₄]-PFOA area counts had a percent RSD of 11%. Other QC elements were used to determine the reportability of the sample results, including laboratory control samples and field matrix spikes. These other QC elements are discussed in sections 3.6 and 4 of the report.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.3 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the **Table 6** below.

Table 6. LOQ

Analyte	LOQ, ng/mL ⁽¹⁾ 6/29/20 [Kirk] Analysis	LOQ, ng/mL ⁽²⁾ 7/6/20 [Kirk] Analysis	LOQ, ng/mL ⁽²⁾ 7/6/20 [Ginger] Analysis	LOQ, ng/mL ⁽²⁾ 7/9/20 [Ginger] Analysis	LOQ, ng/mL ⁽¹⁾ 7/10/20 [Kirk] Analysis
PFOA	0.0479	0.0480	NA	NA	NA
PFBS	0.0500	0.0250	NA	NA	0.100
PFHS	0.0200	0.0250	NA	NA	NA
PFOS	0.0464	0.0464	NA	NA	0.232
PFOSA	NA	NA	0.0500	0.0990	NA

NA = Not Applicable

(1) A dilution factor was not applied to the LOQ.

(2) A dilution factor of 2 was applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of 100% \pm 25%.

3.5 Blanks

Two types of blanks were prepared and analyzed with the samples: method/solvent blanks and field/trip blanks. Each blank result was reviewed and used to evaluate method performance. The method blanks were used to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. LCSs were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. Surrogates [$^{13}\text{C}_4$]-PFOA and [$^{13}\text{C}_4$]-PFOS were added post dilution when analyzed by external standard.

- Target analyte LCSs analyzed on 6/29/20 using external calibration were prepared at nominal concentrations of 100 ng/mL, 5000 ng/mL, and 35000 ng/mL and diluted 502-fold.
- Target analyte LCSs analyzed on 7/6/20 using internal standard calibration were prepared at nominal concentrations of 0.2 ng/mL, 20 ng/mL, and 140 ng/mL and diluted 2-fold.
- Target analyte LCSs analyzed on 7/9/20 were prepared at nominal concentrations of 0.2 ng/mL, 20 ng/mL, and 210 ng/mL and diluted 2-fold.
- Target analyte LCSs analyzed on 7/10/20 using external calibration were prepared at nominal concentrations of 200 ng/mL, 20000 ng/mL, and 70000 ng/mL and diluted 1000-fold.

Method ETS-8-044.3 states that the average recovery of LCSs at each spiking level must be within 80%-120% with an RSD \leq 20%. All LCS samples met criteria with the following exceptions:

- 6/29/20 [ETS Hermes]: Low-level LCSs had an average recovery of 79.0% for PFOA. When control charted, the first sample replicate for the low-level LCSs had a recovery of 72.5%, which was below the lower control limit of 74.2%. The mid and high-level LCSs for PFOA were spiked at a more appropriate level for the samples that required the 502-fold dilution. A deviation is provided with the raw data.
- 6/29/20 [ETS Hermes]: High-level LCSs had an average recovery of 124% for PFHS with all three replicates not meeting acceptance criteria. The low and mid-level LCSs were spiked at a more appropriate level. A deviation is provided with the raw data.

- 7/6/20 [ETS Kirk]: High-level LCSs for PFOA, PFHS and PFOS were spiked above the resulting ULOQ. The low and mid-level LCSs were spiked at a more appropriate level compared to the reported sample concentrations.
- 7/6/20 [ETS Ginger]: High-level LCSs for PFOSA were spiked above the resulting ULOQ. The low and mid-level LCSs were spiked at a more appropriate level compared to the reported sample concentrations.
- 7/10/20 [ETS Kirk]: Low-level LCSs for PFOS were spiked below the resulting LOQ. The mid and high-level LCSs were spiked at a more appropriate level compared to the reported sample concentrations.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report. Individual LCS results are included in the raw data and available upon request.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.5. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in **Table 7** below.

- The data uncertainty for PFOA using external calibration was calculated at $\pm 17\%$ following ETS-12-012.5; however, the data uncertainty was expanded to $\pm 27\%$ based on the PFOA recovery of the first sample replicate for the low-level LCSs that had a recovery of 72.5% from the analysis on 6/29/20.
- The data uncertainty for PFHS using external calibration was calculated at $\pm 22\%$ following ETS-12-012.5; however, the data uncertainty was expanded to $\pm 24\%$ based on the PFHS recovery of the high-level LCSs from the analysis on 6/29/20.

Table 7. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	NA	$\pm 27\%$
PFBS	External	8.16	$\pm 16\%$
PFHS	External	NA	$\pm 24\%$
PFOS	External	13.8	$\pm 28\%$
PFOSA	External	10.9	$\pm 22\%$
PFOA	Internal	8.23	$\pm 16\%$
PFHS	Internal	5.48	$\pm 11\%$
PFOS	Internal	4.42	$\pm 8.8\%$
PFOSA	Internal	7.26	$\pm 15\%$

NA = Not Applicable

3.8 Field Matrix Spikes (FMS)

Target analyte field matrix spike samples were collected for select sampling points to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions at 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOA and PFOS. The standards used for the preparation of the field matrix spiking solutions greater than 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, select sample bottles contained stable isotope surrogate recovery spikes of [¹³C₄]-PFOA and [¹³C₄]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles prior to sample collection. The [¹³C₄]-PFOA was selected to represent the C8 perfluorocarboxylic acid. The [¹³C₄]-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids and perfluorooctanesulfonamide. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

The following calculation was used to generate data in section 4 of the report.

$$FMS\ Recovery = \frac{Sample\ Concentration\ of\ FMS - Average\ Concentration\ (Field\ Sample\ & Field\ Sample\ Dup)}{Spike\ Concentration} \times 100\%$$

Table 8. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
L31, P115	FMS	4.79	5.00	5.00	4.64	5.00
5	FMS	105	105	105	105	5.00
P321, P21B	FMS	2010	2010	2010	2009	10.0
PP06	FMS	1048	1050	1050	1046	50.0
Travel Blank	FMS	4.79	5.00	5.00	4.64	5.00

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of $\pm 30\%$, demonstrate that the method is appropriate for the given matrix.

The method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. At times, the spike level exceeded the recommended upper limit of 10 times the analyte concentration. In these instances, the FMS recovery was reported and flagged as above 10 times the sample concentration.

For PFOA, PFBS, PFHS, and PFOS, when the field matrix spike level was not appropriate as compared to the endogenous sample concentration, the surrogate recovery standards were used to assess method accuracy. All field matrix spike and surrogate recovery standard recoveries met acceptance criteria, except for the following:

PP02: The sample was initially prepared on 6/29/20 with the PFBS sample concentration below the resulting LOQ of 25 ng/mL. The corresponding surrogate [$^{13}\text{C}_4$]-PFOS met acceptance criteria. The sample was re-prepared on 7/8/20 with a lesser dilution factor of 10. This resulted in on-column concentration of PFOS that exceeded the calibration range, causing suppression of the corresponding surrogate [$^{13}\text{C}_4$]-PFOS response. This resulted in [$^{13}\text{C}_4$]-PFOS recoveries to be below 50%. The data uncertainty for the sample will not be increased and the PFBS results are reported without this sample QC element. A deviation is provided with the raw data.

Table 9. Location ID: P321

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-02-005	P321; Sample	2150	NA	511	NA
ISO20-14-02-005-DUP	P321; Sample Duplicate	2320	NA	558	NA
ISO20-14-02-005-FMS	P321; Sample FMS	4230	99.3	2570	101
Average Concentration (ng/mL) ± %RPD		2240 ng/mL ± 7.6%		535 ng/mL ± 8.8%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-02-005	P321; Sample	5560	NA	0.734	NA
ISO20-14-02-005-DUP	P321; Sample Duplicate	5840	NA	0.600	NA
ISO20-14-02-005-FMS	P321; Sample FMS	8260	NC	9.90	92.4 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		5700 ng/mL ± 4.9%		0.667 ng/mL ± 20%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5 times the endogenous sample concentration.

(1) FMS concentration greater than 10 times the sample concentration.

Table 10. Location ID: L31

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-02-008	L31; Sample	0.296	NA	0.782	NA
ISO20-14-02-008-DUP	L31; Sample Duplicate	0.374	NA	0.830	NA
ISO20-14-02-008-FMS	L31; Sample FMS	4.46	86.1 ⁽¹⁾	5.44	92.7
Average Concentration (ng/mL) ± %RPD		0.335 ng/mL ± 23% ⁽²⁾		0.806 ng/mL ± 6.0%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-02-008	L31; Sample	3.50	NA	<0.0990	NA
ISO20-14-02-008-DUP	L31; Sample Duplicate	4.48	NA	<0.0990	NA
ISO20-14-02-008-FMS	L31; Sample FMS	9.08	110	5.24	105 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		3.99 ng/mL ± 25% ⁽²⁾		<0.0990 ng/mL	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

(2) Sample/sample duplicate RPD did not meet method acceptance criteria of ≤20%.

Table 11. Location ID: P115

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-02-011	P115; Sample	2.91	NA	1.73	NA
ISO20-14-02-011-DUP	P115; Sample Duplicate	3.22	NA	1.73	NA
ISO20-14-02-011-FMS	P115; Sample FMS	6.89	79.9	6.34	92.2
Average Concentration (ng/mL) ± %RPD		3.07 ng/mL ± 10%		1.73 ng/mL ± 0.0%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-02-011	P115; Sample	2.94	NA	<0.0500	NA
ISO20-14-02-011-DUP	P115; Sample Duplicate	2.72	NA	<0.0500	NA
ISO20-14-02-011-FMS	P115; Sample FMS	6.78	85.2	4.60	92.1 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		2.83 ng/mL ± 7.8%		<0.0500 ng/mL	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 12. Location ID: PP06

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-02-017	PP06; Sample	659	NA	14.9	NA	90.9	NA
ISO20-14-02-017-DUP	PP06; Sample Duplicate	641	NA	14.7	NA	90.0	NA
ISO20-14-02-017-FMS	PP06; Sample FMS	1660	96.4	1040	97.6 ⁽¹⁾	1160	102 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		650 ng/mL ± 2.8%		14.8 ng/mL ± 1.4%		90.5 ng/mL ± 1.0%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-02-017	PP06; Sample	1390	NA	84.4	NA
ISO20-14-02-017-DUP	PP06; Sample Duplicate	1320	NA	74.6	NA
ISO20-14-02-017-FMS	PP06; Sample FMS	2350	95.1	118	77.1
Average Concentration (ng/mL) ± %RPD		1360 ng/mL ± 5.2%		79.5 ng/mL ± 12%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 13. Location ID: 5

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-02-023	5; Sample	6.70	NA	6.43	NA
ISO20-14-02-023-DUP	5; Sample Duplicate	6.77	NA	6.32	NA
ISO20-14-02-023-FMS	5; Sample FMS	107	95.7 ⁽¹⁾	110	98.7 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		6.74 ng/mL ± 1.0%		6.38 ng/mL ± 1.7%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-02-023	5; Sample	14.9	NA	0.108	NA
ISO20-14-02-023-DUP	5; Sample Duplicate	13.1	NA	0.0892	NA
ISO20-14-02-023-FMS	5; Sample FMS	108	89.8	4.74	92.9 ⁽¹⁾
Average Concentration (ng/mL) ± %RPD		14.0 ng/mL ± 13%		0.0986 ng/mL ± 19%	

NA = Not Applicable

(1) FMS concentration greater than 10 times the sample concentration.

Table 14. Location ID: P21B

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-02-026	P21B; Sample	9890	NA	7880	NA	13000	NA
ISO20-14-02-026-DUP	P21B; Sample Duplicate	8330	NA	6680	NA	10900	NA
ISO20-14-02-026-FMS	P21B; Sample FMS	10500	NC	8530	NC	13300	NC
Average Concentration (ng/mL) ± %RPD		9110 ng/mL ± 17%		7280 ng/mL ± 16%		12000 ng/mL ± 18%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-02-026	P21B; Sample	67100	NA	6.42	NA
ISO20-14-02-026-DUP	P21B; Sample Duplicate	69500	NA	6.02	NA
ISO20-14-02-026-FMS	P21B; Sample FMS	72500	NC	15.1	88.9
Average Concentration (ng/mL) ± %RPD		68300 ng/mL ± 3.5%		6.22 ng/mL ± 6.4%	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5 times the endogenous sample concentration.

Table 15. Location ID: Travel Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-02-027	Travel Blank	<0.0480	NA	<0.500	NA	<0.0250	NA
ISO20-14-02-027-FMS	Travel Blank FMS	3.88	81.0	4.25	85.0	4.54	90.8

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-02-027	Travel Blank	<0.0464	NA	<0.0500	NA
ISO20-14-02-027-FMS	Travel Blank FMS	3.96	85.4	4.62	92.4

NA = Not Applicable

Table 16. Surrogate Recovery Standard Results ⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO20-14-02-001	P121; Sample	112	115	NA
ISO20-14-02-001-DUP	P121; Sample Duplicate	106	107	NA
ISO20-14-02-002	3M vijver; Sample	107	111	NA
ISO20-14-02-002-DUP	3M vijver; Sample Duplicate	112	114	NA
ISO20-14-02-003	Blokkersdijkvijver Noord; Sample	80.2 ⁽²⁾	78.5 ⁽²⁾	NA
ISO20-14-02-003-DUP	Blokkersdijkvijver Noord; Sample Duplicate	83.0 ⁽²⁾	81.7 ⁽²⁾	NA
ISO20-14-02-004	Blokkersdijkvijver standaard; Sample	114	116	NA
ISO20-14-02-004-DUP	Blokkersdijkvijver standaard; Sample Duplicate	107	110	NA
ISO20-14-02-005	P321; Sample	98.4	98.2	NA
ISO20-14-02-005-DUP	P321; Sample Duplicate	103	101	NA
ISO20-14-02-005-FMS	P321; Sample FMS	97.3	97.2	NA
ISO20-14-02-006	L21; Sample	78.5 ⁽²⁾	79.3 ⁽²⁾	NA
ISO20-14-02-006-DUP	L21; Sample Duplicate	74.8 ⁽²⁾	79.4 ⁽²⁾	NA
ISO20-14-02-007	L30; Sample	79.8 ⁽²⁾	81.9 ⁽²⁾	NA
ISO20-14-02-007-DUP	L30; Sample Duplicate	80.8 ⁽²⁾	79.0 ⁽²⁾	NA
ISO20-14-02-008	L31; Sample	79.2 ⁽²⁾	80.8 ⁽²⁾	NA
ISO20-14-02-008-DUP	L31; Sample Duplicate	80.2 ⁽²⁾	80.0 ⁽²⁾	NA
ISO20-14-02-008-FMS	L31; Sample FMS	83.8 ⁽²⁾	83.8 ⁽²⁾	NA
ISO20-14-02-009	L4; Sample	105	110	NA
ISO20-14-02-009-DUP	L4; Sample Duplicate	108	111	NA
ISO20-14-02-010	P114bis; Sample	104	109	NA
ISO20-14-02-010-DUP	P114bis; Sample Duplicate	109	110	NA
ISO20-14-02-011	P115; Sample	108	112	NA
ISO20-14-02-011-DUP	P115; Sample Duplicate	104	112	NA
ISO20-14-02-011-FMS	P115; Sample FMS	110	113	NA
ISO20-14-02-012	P116; Sample	105	111	NA
ISO20-14-02-012-DUP	P116; Sample Duplicate	110	112	NA
ISO20-14-02-013	Effluent WWTP; Sample	101	106	NA
ISO20-14-02-013-DUP	Effluent WWTP; Sample Duplicate	113	116	NA
ISO20-14-02-014	PP02; Sample	107	96.5	48.2 ⁽³⁾
ISO20-14-02-014-DUP	PP02; Sample Duplicate	103	97.3	46.0 ⁽³⁾
ISO20-14-02-015	PP04; Sample	105	95.4	NA
ISO20-14-02-015-DUP	PP04; Sample Duplicate	108	101	NA
ISO20-14-02-016	PP05; Sample	96.8	101	NA
ISO20-14-02-016-DUP	PP05; Sample Duplicate	100	109	NA
ISO20-14-02-017	PP06; Sample	105	105	NA
ISO20-14-02-017-DUP	PP06; Sample Duplicate	107	109	NA
ISO20-14-02-017-FMS	PP06; Sample FMS	103	103	NA
ISO20-14-02-018	PP07; Sample	109	107	NA
ISO20-14-02-018-DUP	PP07; Sample Duplicate	105	102	NA

NA = Not Applicable

- (1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.
- (2) Sample was analyzed with the surrogate recovery standards added to the sample bottle prior to the sampling event.
- (3) Surrogate recovery did not meet method acceptance criteria of 100±30%. Data uncertainty is not increased. See section 4 of the report for more information.

Table 16 continued. Surrogate Recovery Standard Results⁽¹⁾

3M LIMS ID	Sample Description	Percent Recovery (%)		
		[¹³ C ₄]-PFOA	[¹³ C ₄]-PFOS	[¹³ C ₄]-PFOS Re-analysis
ISO20-14-02-019	PP08; Sample	108	97.4	NA
ISO20-14-02-019-DUP	PP08; Sample Duplicate	106	96.1	NA
ISO20-14-02-020	PP10; Sample	106	98.5	NA
ISO20-14-02-020-DUP	PP10; Sample Duplicate	107	102	NA
ISO20-14-02-021	12; Sample	103	100	NA
ISO20-14-02-021-DUP	12; Sample Duplicate	106	101	NA
ISO20-14-02-022	13; Sample	109	101	NA
ISO20-14-02-022-DUP	13; Sample Duplicate	102	98.7	NA
ISO20-14-02-023	5; Sample	110	112	NA
ISO20-14-02-023-DUP	5; Sample Duplicate	105	109	NA
ISO20-14-02-023-FMS	5; Sample FMS	99.0	105	NA
ISO20-14-02-024	Bemalingstation; Sample	109	109	NA
ISO20-14-02-024-DUP	Bemalingstation; Sample Duplicate	102	106	NA
ISO20-14-02-025	Collector put; Sample	114	115	NA
ISO20-14-02-025-DUP	Collector put; Sample Duplicate	105	108	NA
ISO20-14-02-026	P21B; Sample	100	97.5	103
ISO20-14-02-026-DUP	P21B; Sample Duplicate	98.7	92.4	101
ISO20-14-02-026-FMS	P21B; Sample FMS	99.9	98.5	99.3
ISO20-14-02-027	Travel Blank	81.2 ⁽²⁾	111	84.2 ⁽²⁾
ISO20-14-02-027-FMS	Travel Blank FMS	79.0 ⁽²⁾	110	80.6 ⁽²⁾
ISO20-14-02-028	PP11; Sample	101	102	NA
ISO20-14-02-028-DUP	PP11; Sample Duplicate	98.8	96.4	NA

NA = Not Applicable

- (1) The surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS were added to the samples during sample preparation unless noted otherwise.
- (2) Sample was analyzed with the surrogate recovery standards added to the sample bottle prior to the sampling event.
- (3) Surrogate recovery did not meet method acceptance criteria of 100±30%. Data uncertainty is not increased. See section 4 of the report for more information.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 9-16 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M EHS Laboratory standard operating procedures.

7 Attachment

Chain of Custody Form

8 Signatures

Susan T. Wolf, 3M Principal Analytical Investigator



Brian T. Mader, Ph.D., 3M EHS Laboratory Manager



The 3M EHS Laboratory's Quality Assurance Unit has audited the data and report for this project.



Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M EHS Laboratory.

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Project: ISO20-14-02

Phone: [REDACTED]
Alt. Pho: [REDACTED]
Fax: (651) 773-2000

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 4/6/2020

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs - April 2020

Comments:

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO20-14-02-001	P121; Sample	29/4 18:00		
ISO20-14-02-001-DUP	P121; Sample Duplicate	29/4 18:00		
ISO20-14-02-002	3M vijver; Sample	28/4 9:30	WS	
ISO20-14-02-002-DUP	3M vijver; Sample Duplicate	28/4 9:30	WS	
ISO20-14-02-003	Blokkersdijkvijver Noord; Sample	28/4 10:00	WS	
ISO20-14-02-003-DUP	Blokkersdijkvijver Noord; Sample Duplicate	28/4 10:00	WS	
ISO20-14-02-004	Blokkersdijkvijver standaard; Sample	28/4 10:30	WS	
ISO20-14-02-004-DUP	Blokkersdijkvijver standaard; Sample Duplicate	28/4 10:30	WS	
ISO20-14-02-005	P321; Sample	28/4 14:30	WG	
ISO20-14-02-005-DUP	P321; Sample Duplicate	28/4 14:30	WG	
ISO20-14-02-005-FMS	P321; Sample FMS	28/4 14:30	WQ	
ISO20-14-02-006	L21; Sample	29/4 8:30	WG	

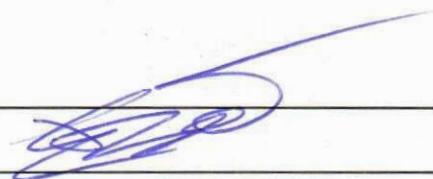
Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 15° Deg C Received on Ice Other:

Collected by (print):

E. h Boecker

Collector's signature:



Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
19	E. h Boecker	30-4-20		Fedex	by KENNS	5/4/20	09:27

3M EHS LABORATORY
Chain-of-Custody

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Project: ISO20-14-02 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 4/6/2020

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

Project Description: 3M Antwerp Water Sampling for PFCs - April 2020

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO20-14-02-006-DUP	L21; Sample Duplicate	29/4 8:30	WG	
ISO20-14-02-007	L22; Sample ~ L30	29/4 10:30	WG	L22 BROKE
ISO20-14-02-007-DUP	L22; Sample Duplicate ~ L30	29/4 10:30	WG	L22 BROKE
ISO20-14-02-008	L31; Sample	29/4 9:15	WG	
ISO20-14-02-008-DUP	L31; Sample Duplicate	29/4 9:15	WG	
ISO20-14-02-008-FMS	L31; Sample FMS	29/4 9:15	WQ	
ISO20-14-02-009	L4; Sample	29/4 14:00	WG	
ISO20-14-02-009-DUP	L4; Sample Duplicate	29/4 14:00	WG	
ISO20-14-02-010	P114bis; Sample	29/4 11:30	WG	
ISO20-14-02-010-DUP	P114bis; Sample Duplicate	29/4 11:30	WG	
ISO20-14-02-011	P115; Sample	29/4 11:05	WG	
ISO20-14-02-011-DUP	P115; Sample Duplicate	29/4 11:05	WG	
ISO20-14-02-011-FMS	P115; Sample FMS	29/4 11:05	WQ	
ISO20-14-02-012	P116; Sample	29/4 9:45	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 15° Deg C Received on Ice Other:

Collected by (print): Erik Boecher

Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Erik Boecher	304-20		FedEx	Say KEARNS	5/4/20	09:27

3M EHS LABORATORY
Chain-of-Custody

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Project: ISO20-14-02 (cont.)

Phone:
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Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 4/6/2020

Project Description: 3M Antwerp Water Sampling for PFCs - April 2020

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO20-14-02-012-DUP	P116; Sample Duplicate	28/4 09:45	WG	
ISO20-14-02-013	Effluent WWTP; Sample	28/4 13:00	WW	
ISO20-14-02-013-DUP	Effluent WWTP; Sample Duplicate	28/4 13:00	WW	
ISO20-14-02-014	PP02; Sample	29/4 13:30	WG	
ISO20-14-02-014-DUP	PP02; Sample Duplicate	29/4 13:30	WG	
ISO20-14-02-015	PP04; Sample	29/4 14:00	WG	
ISO20-14-02-015-DUP	PP04; Sample Duplicate	29/4 14:00	WG	
ISO20-14-02-016	PP05; Sample	29/4 14:30	WG	
ISO20-14-02-016-DUP	PP05; Sample Duplicate	29/4 14:30	WG	
ISO20-14-02-017	PP06; Sample	29/4 14:30	WG	
ISO20-14-02-017-DUP	PP06; Sample Duplicate	29/4 14:30	WG	
ISO20-14-02-017-FMS	PP06; Sample FMS	29/4 14:30	WQ	
ISO20-14-02-018	PP07; Sample	28/4 13:45	WG	
ISO20-14-02-018-DUP	PP07; Sample Duplicate	28/4 13:45	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 15 Deg C Received on Ice Other:

Collected by (print):

Erik Boeckx

Collector's signature: *[Signature]*

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Erik Boeckx	30-4-20		FedEx	<i>[Signature]</i>	5/4/20	09:27

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Project: ISO20-14-02 (cont.)

Phone: [REDACTED]
Alt. Pho: [REDACTED]
Fax: (651) 773-2100

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 4/6/2020

Project Description: 3M Antwerp Water Sampling for PFCs - April 2020

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO20-14-02-019	PP08; Sample	28/4 14:00	WG	
ISO20-14-02-019-DUP	PP08; Sample Duplicate	28/4 14:00	WG	
ISO20-14-02-020	PP10; Sample	28/4 14:15	WG	
ISO20-14-02-020-DUP	PP10; Sample Duplicate	28/4 14:15	WG	
ISO20-14-02-021	12; Sample	28/4 14:30	WS	
ISO20-14-02-021-DUP	12; Sample Duplicate	28/4 14:30	WS	
ISO20-14-02-022	13; Sample	28/4 14:45	WS	
ISO20-14-02-022-DUP	13; Sample Duplicate	28/4 14:45	WS	
ISO20-14-02-023	5; Sample	28/4 14:30	WS	
ISO20-14-02-023-DUP	5; Sample Duplicate	28/4 14:30	WS	
ISO20-14-02-023-FMS	5; Sample FMS	28/4 14:30	WS	
ISO20-14-02-024	Bemalingstation; Sample	28/4 14:45	WS	
ISO20-14-02-024-DUP	Bemalingstation; Sample Duplicate	28/4 14:45	WS	
ISO20-14-02-025	Collector put; Sample	28/4 14:45	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 150 Deg C Received on Ice Other:

Collected by (print): Erik Boeckx

Collector's signature: Jay Kewens

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	Erik Boeckx	30-4-20		Fedex	Jay Kewens	5/4/20	09:27

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
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3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone:
Alt. Phone:
Fax: (651) [REDACTED]

Project: ISO20-14-02 (cont.)

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 4/6/2020

Project Description: 3M Antwerp Water Sampling for PFCs - April 2020

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO20-14-02-025-DUP	Collector put; Sample Duplicate	28/4 13:15	WG	
ISO20-14-02-026	P21B; Sample	28/4 13:30	WG	
ISO20-14-02-026-DUP	P21B; Sample Duplicate	28/4 13:30	WG	
ISO20-14-02-026-FMS	P21B; Sample FMS	28/4 13:30	WQ	
ISO20-14-02-027	Travel Blank 200407	04/07/20 13:15	WQ	CLT
ISO20-14-02-027-FMS	Travel Blank FMS 200407	04/07/20 13:15	WQ	CLT
ISO20-14-02-028	PP11; Sample	29/4 15:00	WG	
ISO20-14-02-028-DUP	PP11; Sample Duplicate	29/4 15:00	WG	
ISO20-14-02-029	PP12; Sample	1	WG	Well
ISO20-14-02-029-DUP	PP12; Sample Duplicate		WG	
ISO20-14-02-029-FMS	PP12; Sample FMS		WQ	not labelled
ISO20-14-02-030	PP13; Sample		WG	Labeled
ISO20-14-02-030-DUP	PP13; Sample Duplicate		WG	ice

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 15 Deg C Received on Ice Other:

Collected by (print):

Erik Boecker

Collector's signature:

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
8	Erik Boecker	30-4-20		FedEx	Jay Kearns	5/4/20	09:27



3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC20-04009-002

3M Lab Request Number: E20-2077

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
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Author: Sandra Graré
Date issued: October 16th, 2020

Requester

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1. Introduction - Summary.

At the request (Lab Request Number: E20-2077) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, one water sample was collected in July 2020 from 3 locations and analyzed for the following perfluorinated compounds:

- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$) (and its ^{13}C -labeled analogues)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$) (and its ^{13}C -labeled analogues)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$) (and its ^{13}C -labeled analogues)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$) (and its ^{13}C -labeled analogue)
- ^{13}C -labeled analogue of PFUdA ($C_4F_{21}^{13}C_6F_2^{13}COO^-$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared one sample container for each sampling location under the direction of Sven Herremans. Each empty container was marked with a "fill to here" line and was fortified with a surrogate recovery spike and an internal standard spike, prior to being sent to the field for sample collection.

Table 1 summarizes the sample results with their uncertainty. All PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$ unless noted otherwise. See Section 4 of the report for additional information on the method QC used to assess method uncertainty.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)			
	PFHS	PFOA	PFOS	FOSA
3M Vijver	0.633	1.81	7.97	0.470
Blokkersdijkvijver Noord	0.643	1.39	2.38	0.190 (b)
Blokkersdijkvijver standaard (filter)	0.577	1.44	0.658	0.395
Blokkersdijkvijver standaard (nt filter)	0.564	1.16	0.650	0.069 (a)

(a): The recovery of FOSA for Blokkersdijkvijver standard (Nt Filter) is $\pm 20\%$.

(b): The recovery of FOSA for Blokkersdijkvijver Noord is $\pm 50\%$.

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

The sample was collected in a polyethylene bottle prepared at the SGS Belgium NV, Division IAC Laboratory. Based on the concentrations as reported in previous reports, the bottle was spiked, prior to sample collection, in the laboratory with a known volume of a surrogate recovery solution ($^{13}\text{C}_2\text{-PFHS}$, $^{13}\text{C}_4\text{-PFOA}$, $^{13}\text{C}_4\text{-PFOS}$, $^{13}\text{C}_7\text{-PFUdA}$) and internal standard solution ($^{13}\text{C}_3\text{-PFHS}$, $^{13}\text{C}_8\text{-PFOA}$, $^{13}\text{C}_8\text{-PFOS}$, $^{13}\text{C}_8\text{-FOSA}$). Table 2 below details the sample collected and spikes added to the bottle.

Table 2. Sample Collection and Spike Information.

Sample Identification	Nominal Final Volume Collected (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Final Nominal Spike Concentration (ng/mL)	
				$^{13}\text{C-PFC-SS}$	$^{13}\text{C-PFC-IS}$
3M vijver	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
Blokkersdijkvijver Noord	400	0.1	Solution A	0.25	-
		0.25	Solution B	-	1.0
Blokkersdijkvijver standaard (filter)	400	0.1	Solution A	0.25	-
		0.25	Solution B	-	1.0
Blokkersdijkvijver standaard (nt filter)	400	0.1	Solution A	0.25	-
		0.25	Solution B	-	1.0

Solution A = 1000 ng/mL (nominal) $^{13}\text{C-PFC}$ Surrogate Recovery Standards

Solution B = 1500 ng/mL (nominal) $^{13}\text{C-PFC}$ Internal Standards

2.2. Extraction.

All samples, calibration standards, and associated quality control samples were extracted using a modified procedure of ECO/AV/IAC/064 “Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)”. Briefly, an amount of sample (see table 3) was loaded, onto a pre-conditioned Waters tC18 solid phase extraction (SPE) cartridge (Sep-Pak, 1g, 6cc) using a vacuum manifold. The loaded SPE cartridges were then eluted with 5 mL of methanol using vacuum.

Table 3. Sample amount used.

Sample	Amount of Sample used (mL)	Concentration Factor
3M Vijver	40	8
Blokkersdijkvijver Noord	40	8
Blokkersdijkvijver standard (filter)	40	8
Blokkersdijkvijver standard (nt filter)	40	8
Field Trip Blank	40	8

2.3. Determination of suspended solids in water.

To avoid the influence of algae on the analytical result, filtration is proposed prior to extraction at the sampling locations Blokkersdijkvijver - standard.

The filtration was done by using Whatman GF/C glass-fiber filters, with a pore size of 1.2 µm. The total suspended solids of the samples is determined by pouring a measured volume of sample, typically 200 mL, through a pre-weighed filter then weighing the filter again after drying the filter overnight to remove all water. The gain in weight is a dry weight measure of the particulates present in the water sample. This is expressed in units calculated from the volume of water filtered, milligrams per liter. Table 4 summarizes the results.

Table 4. Suspended solids.

Sample Identification	Suspended solids/ volume (mg/L)
Blokkersdijkvijver - standard (filtered)	0.01

2.4. Analysis.

All solutions and extracts were analyzed for the PFCs (PFHS, PFOA, PFOS, FOSA) and the surrogate recovery standards ($^{13}\text{C}_2\text{-PFHS}$, $^{13}\text{C}_4\text{-PFOA}$, $^{13}\text{C}_4\text{-PFOS}$, $^{13}\text{C}_7\text{-PFUdA}$) and internal standards ($^{13}\text{C}_3\text{-PFHS}$, $^{13}\text{C}_8\text{-PFOA}$, $^{13}\text{C}_8\text{-PFOS}$, $^{13}\text{C}_8\text{-FOSA}$) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetrie C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C ₃ -PFHS	2.00 - 3.00	402.0 /80.0	60	38	51
¹³ C ₂ -PFHS	2.30 - 3.30	403.0/84.0	60	38	51
¹³ C ₈ -PFOS	3.00 - 4.00	507.0/80.0	60	48	56
¹³ C ₄ -PFOA	2.30 - 3.50	417.0 /372.0	100	11	14
¹³ C ₈ PFOA	2.30 - 3.50	421.0 /376.0	60	11	14
PFHS	2.00 - 3.00	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.30 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
¹³ C ₄ -PFOS	3.00 - 4.00	503.0 /80.0	60	48	56
¹³ C ₇ PFUD _A	4.30 - 5.60	570.0 /525.0	60	12	17
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
¹³ C ₈ FOSA	4.30 - 5.70	506.0/78.0	60	34	44

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Extracted Calibration Standard.

Extracted calibration standards were prepared by spiking known amounts of stock solutions containing PFHS, PFOA, PFOS, FOSA and ^{13}C -labeled analogues into 40 mL of HPLC water. Each spiked water standard was then extracted in the same manner as the collected samples. A total of 12 spiked standards ranging from 0.005 ng/mL to 100 ng/mL (nominal) were prepared. Each curve point contains the mixture of internal standards at a nominal concentration of 1 ng/mL. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. The calibration curve will be generated by taking the ratio of the standard peak area counts over the internal standard peak area counts to fit the data for each analyte. Each extracted calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of $\pm 25\%$ ($\pm 30\%$ for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The 0.025ng/mL (nominal) reporting limit is a practical quantitation limit (PQL) required by the requester and it is possible that the samples contain target analytes at quantifiable concentrations below the PQL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within $\pm 25\%$, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by loading 40 mL of HPLC water onto a Waters tC18 solid phase extraction (SPE) cartridge (SEP-Pak, 1g, 6cc) and eluting with 5 mL of methanol using the same extraction procedure as the samples. Method blanks were prepared to evaluate the levels of background contamination in the overall extraction process (glassware, SPE cartridges, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carry over.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of HPLC water, spiked with the surrogate recovery standards and internal standards, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCSs)

Low (0.125 ng/mL nominal concentration) and high (2.5 ng/mL nominal concentration) lab control spikes were prepared and analyzed in duplicate. LCSs were prepared by spiking known amounts of the analytes and surrogates into 40 mL of HPLC water to produce the desired concentration. The spiked water samples were extracted and analyzed in the same manner as the samples.

All LCSs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

All LCSs produced recoveries within the method acceptance criteria of $\pm 15\%$ RPD for precision, except for 13C4PFOS and PFOS.

Table 8 summarize the LCS recovery results.

Table 7. Lab Control Spike Results.

Extraction date	Description	Nominal Spike Level (ng/mL)	Percent Recovery							
			¹³ C ₄ -PFOA	¹³ C ₄ -PFOS	¹³ C ₂ -PFHS	¹³ C ₇ -PFuDA	PFHS	PFOA	PFOS	FOSA
1 Septembre 2020	LCSL01	0.125	99.1	112	120	113	91.6	89.8	93.8	98.0
	LCSL02	0.125	99.7	100	115	111	82.5	89.0	82.4	89.8
	Average		99.4	106	117	112	87.1	89.4	88.1	93.9
	%RPD		0.6	11	4.5	1.7	10	0.9	13	8.7
	LCSH 01	2.5	116	117	114	114	113	86.2	124	123
	LCSH 02	2.5	98.6	99.2	117	110	103	72.8	105	107
	Average		107	108	115	112	108	79.5	115	115
	%RPD		16 (a)	16(a)	2.3	3.0	9.3	17 (a)	16(a)	14

(a) The recovery of the RPD fell outside the method acceptance criterion of $\pm 15\%$.

3.6. Surrogates.

Surrogate recovery standards were added to all samples to evaluate overall method performance.

3.7. Internal Standards.

Internal standards were added to all samples to calculate the concentration of PFCs in the samples by using internal standard calibration.

3.8. Equations.

Recovery.

$$\text{Recovery(%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

Tabel 8 and 9 summarizes the results for the sample locations.

Table 8. Sample Results PFHS and PFOA.

	PFHS ng/ml	¹³ C ₂ -PFHS %Rec	PFOA ng/ml	¹³ C ₄ -PFOA %Rec
Field Trip Blank	<0.025	106	<0.025	99.3
3M vijver	0.633	96.8	1.81	103
Blokkersdijkvijver Noord	0.643	100	1.39	106
Blokkersdijkvijver standaard (filter)	0.577	96.5	1.44	114
Blokkersdijkvijver standaard (nt filter)	0.564	105	1.16	108

Table 9. Sample Results PFOS and FOSA.

	PFOS ng/ml	¹³ C ₄ -PFOS %Rec	FOSA ng/ml	¹³ C ₇ -PFuDA %Rec
Field Trip Blank	<0.025	101	<0.025	128
3M vijver	7.97	107	0.470	86.5 (a)
Blokkersdijkvijver Noord	2.38	105	0.190	187
Blokkersdijkvijver standaard (filter)	0.658	103	0.395	123
Blokkersdijkvijver standaard (nt filter)	0.650	108	0.069	151 (b)

(a): The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130%.

All PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$, except for FOSA for the following sample locations:

- (a): The recovery of FOSA for Blokkersdijkvijver Noord is $\pm 50\%$.
- (b): The recovery of FOSA for Blokkersdijkvijver standard (nt filter) is $\pm 20\%$.

5. Conclusion.

- The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130% for two sample locations.
- Lab control spike recoveries fell within the method acceptance criteria of 25%.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Julie Fichefet (ERM Belgium).

8. Signatures.

Sven Herremans,
Lab Operations Manager

Date October 16th, 2020



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date October 16th, 2020

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The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.



3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
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The present document voids and replaces the previously issued report(s) with same reference(s).

ANALYTICAL REPORT

IAC20-04009-001_revision

3M Lab Request Number: E20-2077

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
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Author: Sandra Graré
Date issued: December 1st, 2020

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1. Introduction - Summary.

At the request (Lab Request Number: E20-2077) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected in August 2020 from 60 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOA ($C_6F_{13}^{13}CF_2^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C -labeled analogue of PFOS ($C_7F_{15}^{13}CF_2SO_3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Sven Herremans. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles.

Table 1 summarizes the sample results. All PFBS, PFHS, PFOA, PFOS and FOSA results have an accuracy of $\pm 30\%$.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
BD24-3		17.2	20.1	608	5.47
BD24-4		33.6	99.8	3.31	<0.187
D10		87.9	278	24.9	<1.01
D11		123	205	793	57.6
D14		1.26	2.30	0.506	<0.187
D16		39.1	140	631	91.0
D17		27.0	162	317	<1.01
D18		16.1	40.5	42.5	<1.01
D2		0.203	0.195	6.70	0.400
D5		84.6	522	538	23.1
K3	225	9453	75.0	6839	16.2
L19		174	582	3728	77.3
L21		<0.178	<0.182	1.86	<0.187
L30		0.742	0.740	4.68	<0.187
L31		0.652	0.496	3.27	<0.187
L4		2.73	3.86	20.0	0.496
M4		42.6	142	1552	54.8
ND7		63.4	297	61.9	8.61
P114bis		1.26	3.70	3.75	<0.187
P115		1.87	4.02	2.86	<0.187
P116		0.469	1.04	5.69	<0.187
P118A		1375	1051	362	<3.03
P118B		929	1708	5855	<11.1
P118C		65.2	182	938	35.8
P119A		8.67	34.5	5.29	<1.01
P119B		119	354	81.0	<2.02
P119C		271	825	1315	71.4
P121		1.54	1.46	3.46	<0.187
P21B	6429	8683	8083	47591	<40.5

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
P262bis		311	642	3546	9.79
P263		210	706	5350	<11.1
P264		82.7	200	1012	73.9
P27	1660	39.9	174	1793	93.1
P304	375	109	398	978	19.9
P305	335	140	137	353	23.3
P321		219	928	2931	6.47
P340		54.3	168	3822	138
P341		186	287	2311	114
P371		189	237	2011	33.0
P372		156	218	1470	19.9
P374		240	246	1217	29.8
P379		13.9	49.4	328	73.5
P380		883	898	1360	27.8
P381		124	492	787	6.04
P382		83.6	307	677	45.8
P42		280	216	1500	14.3
PP02	33.3	3442	767	18391	21.7
PP04	562	62.9	242	2559	73.3
PP05	7782	5585	2937	93.2	14.2
PP06	17.3	32.1	199	903	61.5
PP07	289	97.8	287	1811	107
PP08	35.3	265	551	4837	51.6
PP10	21.7	181	533	2959	25.3
PP11	167	110	254	1222	18.2
Collector Put		5.39	22.7	40.7	5.05
12		24.1	101	295	<1.01
13		27.0	86.5	260	<1.01
5		32.5	39.6	38.5	<0.187
Bemalingsstation		38.5	48.1	58.2	<0.187
Effluent WWTP		<0.966	<0.988	<1.30	<1.01

2. Methods – Analytical and Preparatory.**2.1. Sample collection.**

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
BD24-3	100
BD24-4	100
D09bis	100
D10	100
D11	100
D14	100
D16	100
D17	100
D18	100
D2	100
D5	100
K3	100
L19	100
L21	100
L30	100
L31	100
L4	100
M4	100
ND7	100
P114bis	100
P115	100
P116	100
P118A	100
P118B	100
P118C	100
P119A	100
P119B	100
P119C	100
P121	100
P21B	100
P262bis	100
P263	100
P264	100
P265B	100
P27	100
P304	100
P305	100
P321	100
P340	100
P341	100
P371	100

Sample Identification	Nominal Final Volume Collected (ml)
P372	100
P374	100
P379	100
P380	100
P381	100
P382	100
P42	100
PP02	100
PP04	100
PP05	100
PP06	100
PP07	100
PP08	100
PP10	100
PP11	100
PP12	100
PP13	100
Collector Put	100
12	100
13	100
5	100
Bemalingsstation	100
Effluent WWTP	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
BD24-3	1.0	0.1	Solution A	10.0	-
BD24-4	1.0	0.05	Solution A	10.0	-
D10	1.0	0.1	Solution A	10.0	-
D11	1.0	0.1	Solution A	10.0	-
D14	1.0	0.05	Solution A	10.0	-
D16	1.0	0.1	Solution A	10.0	1.0 mL extract + 1.0 mL MeOH (*)
D17	1.0	0.1	Solution A	10.0	-
D18	1.0	0.1	Solution A	10.0	-
D2	1.0	0.05	Solution A	10.0	-
D5	1.0	0.1	Solution A	10.0	-
K3	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
L19	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
L21	1.0	0.05	Solution A	10.0	-
L30	1.0	0.05	Solution A	10.0	-
L31	1.0	0.05	Solution A	10.0	-
L4	1.0	0.05	Solution A	10.0	-
M4	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
ND7	1.0	0.1	Solution A	10.0	-
P114bis	1.0	0.05	Solution A	10.0	-
P115	1.0	0.05	Solution A	10.0	-
P116	1.0	0.05	Solution A	10.0	-
P118A	1.0	0.1	Solution A	10.0	1.0 mL extract + 2.0 mL MeOH (*)
P118B	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
P118C	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
P119A	1.0	0.1	Solution A	10.0	-
P119B	1.0	0.1	Solution A	10.0	1.0 mL extract + 1.0 mL MeOH (*)
P119C	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
P121	1.0	0.05	Solution A	10.0	-
P21B	0.5	0.1	Solution A	10.0	0.5 mL extract + 10.0 mL MeOH (*)
P262bis	1.0	0.1	Solution A	10.0	-
P263	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
P264	1.0	0.1	Solution A	10.0	1.0 mL extract + 5.0 mL MeOH (*)
P27	1.0	0.1	Solution A	10.0	1.0 mL extract + 1.0 mL MeOH (*)
P304	1.0	0.1	Solution A	10.0	1.0 mL extract + 1.0 mL MeOH (*)
P305	1.0	0.1	Solution A	10.0	-
P321	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
P340	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
P341	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
P371	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
P372	1.0	0.1	Solution A	10.0	1.0 mL extract + 1.0 mL MeOH (*)
P374	1.0	0.1	Solution A	10.0	1.0 mL extract + 5.0 mL MeOH (*)
P379	1.0	0.1	Solution A	10.0	1.0 mL extract + 1.0 mL MeOH (*)
P380	1.0	0.1	Solution A	10.0	1.0 mL extract + 1.0 mL MeOH (*)

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
P381	1.0	0.1	Solution A	10.0	1.0 mL extract + 1.0 mL MeOH (*)
P382	1.0	0.1	Solution A	10.0	1.0 mL extract + 1.0 mL MeOH (*)
P42	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
PP02	1.0	0.1	Solution A	10.0	0.5 mL extract + 10.0 mL MeOH (*)
PP04	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
PP05	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
PP06	1.0	0.1	Solution A	10.0	1.0 mL extract + 2.0 mL MeOH (*)
PP07	1.0	0.1	Solution A	10.0	1.0 mL extract + 5.0 mL MeOH (*)
PP08	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
PP10	1.0	0.1	Solution A	10.0	1.0 mL extract + 10.0 mL MeOH (*)
PP11	1.0	0.1	Solution A	10.0	1.0 mL extract + 5.0 mL MeOH (*)
Collector Put	1.0	0.05	Solution A	10.0	-
12	1.0	0.1	Solution A	10.0	-
13	1.0	0.1	Solution A	10.0	-
5	1.0	0.05	Solution A	10.0	-
Bemalingsstation	1.0	0.05	Solution A	10.0	-
Effluent WWTP	1.0	0.1	Solution A	10.0	-

(*): MeOH:LCMS-water (60:40)

Solution A = 1000 ng/mL (nominal) ¹³C-PFC Surrogate Standards

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetrie C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate ($\mu\text{L}/\text{min}$)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
¹³ C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
PFBS	2.08 - 2.50	298.93/79.82	40	28	45
		298.93/98.78	40	29	45
PFHS	2.45 - 3.05	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.80 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
¹³ C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of $\pm 25\%$ ($\pm 30\%$ for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.086 to 0.117 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.176 to 52.1 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within $\pm 25\%$, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by diluting 1 mL water with 10.0 mL MeOH:LCMS-water (60:40). Method blanks were prepared to evaluate the levels of background contamination in the overall dilution process (glassware, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carryover.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of LCMS-water, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCS)

One lab control spike was prepared and analyzed.

LCS was prepared by spiking known amounts of the analytes into 1.0 mL of LCMS-water to produce the desired concentration.

The spiked water sample was diluted with 10.0 mL MeOH:LCMS-water (60:40) and analyzed in the same manner as the samples.

The LCS produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy.

Table 7 summarizes the LCS recovery results

Table 7. Lab Control Spike Results.

Dilution date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	¹³ C-PFOA	PFOS	FOSA	¹³ C-PFOS
1 Septembre 2020	LCS1	80	84.0	81.8	82.6	93.4	88.3	94.4	107

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing PFBS, PFHS, PFOA, ¹³C-PFOA, PFOS, FOSA and ¹³C-PFOS (QC Lab Low and QC Lab High).

The QCs produced recoveries within the method acceptance criteria of $\pm 25\%$ for accuracy. One QC high lab (QC Lab FOSA) is excluded because the highest calibration point is excluded.

Table 8 summarizes the QCs recovery results.

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	¹³ C-PFOA	PFOS	FOSA	¹³ C-PFOS
20 May 2020	QC Lab Low inj1	10	97.4	89.2	103	124	105	87.6	98.6
	QC Lab High inj1	100	93.8	85.3	103	103	102	*	97.5
	QC Lab Low inj2	10	102	95.0	107	105	110	93.6	95.1
	QC Lab High inj2	100	92.0	85.4	103	104	103	*	96.1

- Excluded

3.7. Equations.Recovery.

$$\text{Recovery(%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

The following tables summarize the results for the sample locations.

Table 9. Sample Results PFBS, PFHS, PFOA, PFOS and FOSA

	PFBS	PFHS	PFOA	PFOS	FOSA
	ng/ml	ng/ml	ng/ml	ng/ml	ng/ml
BD24-3		17.2	20.1	608	5.47
BD24-4		33.6	99.8	3.31	<0.187
D10		87.9	278	24.9	<1.01
D11		123	205	793	57.6
D14		1.26	2.30	0.506	<0.187
D16		39.1	140	631	91.0
D17		27.0	162	317	<1.01
D18		16.1	40.5	42.5	<1.01
D2		0.203	0.195	6.70	0.400
D5		84.6	522	538	23.1
K3	225	9453	75.0	6839	16.2
L19		174	582	3728	77.3
L21		<0.178	<0.182	1.86	<0.187
L30		0.742	0.740	4.68	<0.187
L31		0.652	0.496	3.27	<0.187
L4		2.73	3.86	20.0	0.496
M4		42.6	142	1552	54.8
ND7		63.4	297	61.9	8.61
P114bis		1.26	3.70	3.75	<0.187
P115		1.87	4.02	2.86	<0.187
P116		0.469	1.04	5.69	<0.187
P118A		1375	1051	362	<3.03
P118B		929	1708	5855	<11.1
P118C		65.2	182	938	35.8
P119A		8.67	34.5	5.29	<1.01
P119B		119	354	81.0	<2.02
P119C		271	825	1315	71.4
P121		1.54	1.46	3.46	<0.187
P21B	6429	8683	8083	47591	<40.5
P262bis		311	642	3546	9.79
P263		210	706	5350	<11.1
P264		82.7	200	1012	73.9
P27	1660	39.9	174	1793	93.1
P304	375	109	398	978	19.9
P305	335	140	137	353	23.3
P321		219	928	2931	6.47
P340		54.3	168	3822	138
P341		186	287	2311	114
P371		189	237	2011	33.0
P372		156	218	1470	19.9
P374		240	246	1217	29.8

Table 9. Sample Results PFBS, PFHS, PFOA, PFOS and FOSA

	PFBS ng/ml	PFHS ng/ml	PFOA ng/ml	PFOS ng/ml	FOSA ng/ml
P379		13.9	49.4	328	73.5
P380		883	898	1360	27.8
P381		124	492	787	6.04
P382		83.6	307	677	45.8
P42		280	216	1500	14.3
PP02	33.3	3442	767	18391	21.7
PP04	562	62.9	242	2559	73.3
PP05	7782	5585	2937	93.2	14.2
PP06	17.3	32.1	199	903	61.5
PP07	289	97.8	287	1811	107
PP08	35.3	265	551	4837	51.6
PP10	21.7	181	533	2959	25.3
PP11	167	110	254	1222	18.2
Collector Put		5.39	22.7	40.7	5.05
12		24.1	101	295	<1.01
13		27.0	86.5	260	<1.01
5		32.5	39.6	38.5	<0.187
Bemalingsstation		38.5	48.1	58.2	<0.187
Effluent WWTP		<0.966	<0.988	<1.30	<1.01

Table 10. Sample Results ¹³C-PFOA and ¹³C-PFOS

	¹³ C-PFOA		¹³ C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
BD24-3	124	108	115	93.1
BD24-4	62.3	109	56.7	92.1
D10	112	97.7	126	103
D11	113	98.7	125	102
D14	58.6	102	60.4	98.0
D16	122	106	117	94.7
D17	112	97.2	127	103
D18	108	94.4	131	106
D2	59.7	104	59.2	96.1
D5	109	94.9	130	106
K3	120	105	119	96.2
L19	110	95.6	129	104
L21	61.0	106	58.0	94.1
L30	58.7	102	60.2	97.8
L31	59.9	104	59.1	95.9
L4	60.7	106	58.2	94.6
M4	114	99.0	124	101
ND7	110	95.7	129	105
P114bis	59.4	104	59.5	96.6
P115	59.5	104	59.4	96.5
P116	62.0	108	57.0	92.5
P118A	109	95.3	129	105
P118B	118	103	120	97.3
P118C	117	102	122	98.7
P119A	121	105	117	95.4
P119B	106	92.0	134	109
P119C	110	96.2	128	104
P121	58.8	102	60.2	97.7
P21B	196	85.2	282	115
P262bis	128	111	111	90.0
P263	104	90.5	135	110
P264	115	100	123	100
P27	123	107	116	93.9
P304	113	98.8	125	101
P305	119	104	119	96.8
P321	105	91.1	134	109
P340	116	101	122	98.9
P341	118	103	121	97.9
P371	113	98.0	126	102
P372	119	103	119	97.0
P374	111	96.9	127	103
P379	119	104	119	96.8
P380	115	100	124	100
P381	112	97.8	126	103
P382	125	109	113	91.8
P42	117	102	121	98.5
PP02	130	113	117	95.0
PP04	103	90.1	136	110

Table 10. Sample Results ^{13}C -PFOA and ^{13}C -PFOS

	^{13}C -PFOA		^{13}C -PFOS	
	ng/ml	% Rec	ng/ml	% Rec
PP05	105	91.2	134	109
PP06	114	99.4	124	101
PP07	120	105	118	95.8
PP08	109	94.7	130	105
PP10	127	110	113	91.5
PP11	106	92.6	133	108
Collector Put	60.1	105	58.8	95.6
5	112	97.7	126	103
Bemalingsstation	120	105	118	95.6
Effluent WWTP	62.8	109	56.3	91.4

5. Conclusion.

- The recovery of ¹³C-PFOA and ¹³C-PFOS fell within the method acceptance criterion of 70-130%.
- Lab control spike recoveries fell within the method acceptance criteria for all compounds, indicating that the analysis method is in control,
- QC Lab Standards recoveries fell within the method acceptance criteria for all compounds. One QC high lab (QC Lab FOSA) is excluded because the highest calibration point is excluded.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Julie Fichefet (ERM Belgium).

8. Signatures.

Sven Herremans,
Lab Operations Manager

Date December 1st, 2020



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date December 1st, 2020

I.A.C.
A Division of SGS Belgium NV

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Final Report

Water Sample Analysis for PFOA, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

August 2020 Sampling

Laboratory Request Number: ISO20-14-03

Report Date – Date of Last Signature

Testing Laboratory

3M EHS Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Charlotte Tack
3M Belgium
3M Belgium; ZW019/01/01
Phone: [REDACTED]



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2017 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M EHS Laboratory

3M EHS Laboratory Manager: Brian T. Mader, Ph.D.
3M Principal Analytical Investigator: Susan Wolf
Report Author: Devon Bulman, Ph.D.

Analytical Report ISO20-14-03

Water Sample Analysis at 3M Antwerp, Belgium
August 2020 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M EHS Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. All samples were collected between August 6 - 7, 2020, and returned to the 3M EHS Laboratory on August 13, 2020, at ambient temperature. The results in this report apply to the samples as received from ERM. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO20-14-03.

The 3M EHS Laboratory prepared sample containers for ten sampling locations. Each sample set consisted of a field sample. Two locations also included a sample duplicate and a target analyte field matrix spike. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 100 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with a mass-labeled internal standard mix and surrogate recovery standards [¹³C₄]-PFOA and [¹³C₄]-PFOS prior to being sent to the field for sample collection.

Samples were prepared and analyzed using method ETS-8-044.4 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Select internal standards were used to aid in the data quality objectives.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.



The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2017 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

Table 1. Sample Results Summary⁽¹⁾

3M LIMS ID	Sample Description	Concentration (ng/mL)			
		PFOA	PFHS	PFOS	FOSA
ISO20-14-03-001	3M vijver; Sample	1.27	0.586	7.28	0.274
ISO20-14-03-002	Blokkersdijkvijver Noord; Sample	0.984	0.602	3.76	0.193
ISO20-14-03-002-DUP	Blokkersdijkvijver Noord; Sample Duplicate	0.920	0.588	2.00	0.119
Average %RPD Sample/Sample Dup		0.952	0.595	2.88	0.156
6.7		6.7	2.4	61 ⁽²⁾	47 ⁽²⁾
ISO20-14-03-003	Blokkersdijkvijver standaard; Sample	1.65	0.960	3.74	0.334
ISO20-14-03-004	L21; Sample	0.370	0.246	2.28	0.0306
ISO20-14-03-005	L31; Sample	0.540	0.650	4.02	0.0616
ISO20-14-03-006	L4; Sample	3.06	2.26	23.0	0.458
ISO20-14-03-007	P114bis; Sample	3.28	1.28	5.38	0.0272
ISO20-14-03-007-DUP	P114bis; Sample Duplicate	3.28	1.28	5.56	0.0282
Average %RPD Sample/Sample Dup		3.28	1.28	5.47	0.0277
0.0		0.0	0.0	3.3	3.6
ISO20-14-03-008	P115; Sample	3.32	1.59	3.20	<0.0250
ISO20-14-03-009	P116; Sample	0.846	0.414	7.14	0.0778
ISO20-14-03-010	L30; Sample	0.536	0.624	5.62	0.0562
ISO20-14-03-011	Travel Blank	<0.192	<0.0250	<0.0232	<0.0250

NA = Not Applicable

- (1) The analytical data uncertainties for the reported results using internal standard calibration are as follows: PFOA ± 18%, PFHS ± 22%, PFOS ± 21% and PFOSA ± 20%.
- (2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M EHS Laboratory method ETS-8-044.4 “Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis”.

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected on August 6 - 7, 2020, in Nalgene™ (high-density polyethylene) bottles prepared at the 3M EHS Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. All sample locations were spiked with a mass-labeled internal standard mix at a nominal concentration of 1 ng/mL and select samples were fortified with surrogate recovery standards (SRSs) at a nominal concentration of 0.1 ng/mL prior to sample collection. Samples were received on August 13, 2020.

2.3 Sample Preparation

All samples were prepared by removing a 0.400 mL aliquot of the well mixed sample and diluting it with 0.400 mL of methanol (dilution factor of 2).

During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1.00 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1.00 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were prepared by diluting 5 mL of a well-mixed control sample with 5 mL of methanol.

2.4 Analysis

All samples and quality control samples were analyzed for the target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). SRSs were added and included in the analysis of these samples. Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.2. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

Table 3. Instrument Parameters.

Instrument Name	ETS Athena
Liquid Chromatograph	Agilent 1260
Analysis Method	ETS-8-044.4
Analysis Date(s)	11/6/20, 11/16/20, and 12/1/20
Guard column	Betasil C8 (2.1 mm X 50 mm), 5 μ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 μ
Injection Volume	10 μ L
Mass Spectrometer	AB Sciex Triple Quad 5500
Ion Source	Turbo Spray
Polarity	Negative
Software	Analyst 1.7.1

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.4 Analysis				
Step Number	Total Time (min)	Flow Rate (μ L/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
0	0.00	300	90.0	10.0
1	0.50	300	90.0	10.0
2	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard	Mass Transition Q1/Q3
PFOA	413/369	[¹³ C ₈]-PFOA	421/376
	413/219		
	413/169		
PFHS	399/99	[¹³ C ₃]-PFHS	402/80
	399/80		
PFOS	499/99	[¹³ C ₈]-PFOS	507/80
	499/80		
	499/130		
PFOSA	498/78	[¹³ C ₈]-PFOSA	506/78
[¹³ C ₄]-PFOA	417/372	[¹³ C ₈]-PFOA	421/376
[¹³ C ₄]-PFOS	503/80	[¹³ C ₈]-PFOS	507/80

The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

3 Data Analysis

3.1 Calibration

Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50 mL of 50:50 methanol: laboratory reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 0.500 ng/mL. A total of eleven or twelve spiked standards ranging from 0.0125 ng/mL up to 25.0 ng/mL (nominal) were analyzed. Ten calibration standards also contained the surrogates at concentrations ranging from 0.0125 ng/mL to 10.0 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for all analytes. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA, PFOS, and PFOSA used to prepare the calibration standards consisted of both linear and branched isomers.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±25% (100±30% for the lowest curve point) were met for all analytes. The correlation coefficient (*r*) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes for each analysis, except for the following:

- Analysis 11/6/20: PFOA area counts had a percent RSD of 5.7%. Other QC elements were used to determine the reportability of the sample results, including laboratory control samples and field matrix spikes. These other QC elements are discussed in sections 3.6 and 4 of the report.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.4 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the **Table 6** below.

Table 6. LOQ

Analyte	Analysis on 11/6/20 LOQ, ng/mL ⁽¹⁾	Analysis on 11/16/20 LOQ, ng/mL ⁽¹⁾	Analysis on 12/1/20 LOQ, ng/mL ⁽¹⁾
PFOA	0.192	NA	0.0240
PFHS	0.0250	0.0250	NA
PFOS	0.0232	NA	NA
PFOSA	0.0250	0.0250	0.0250

NA = Not Applicable

(1) A dilution factor of 2 was applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of $100\% \pm 25\%$.

3.5 Blanks

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks and field/trip blanks. Each blank result was reviewed and used to evaluate method performance. Method blank results were reviewed and used to evaluate method performance to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate, while only low and high lab control spikes were prepared for the surrogates. Target analytes were spiked at nominal concentrations of 0.2 ng/mL, 2.0 ng/mL, and 35 ng/mL for analysis on 11/6/20 and 0.2 ng/mL, 2.0 ng/mL, and 21 ng/mL for analysis on 11/16/20 and 12/1/20. LCSs were prepared by spiking known amounts of the analytes into 5 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted with 5 mL of methanol to create a dilution factor of 2. Method ETS-8-044.4 states that the average recovery of LCSs at each spiking level must be within 80%-120% with an RSD $\leq 20\%$. All LCS samples met method accuracy criteria with the following exceptions:

- Analysis on 11/6/20: The low-level LCSs (0.2 ng/mL) for PFOA were spiked below the resulting LOQ.
- Analysis on 11/6/20: The high-level LCSs (35 ng/mL) for PFHS were spiked above the upper limit of quantitation (ULOQ) when the dilution factor of 2 was applied.
- Analysis on 11/16/20: The mid-level LCSs (2.0 ng/mL) for PFHS had an average recovery of 122%, more than 67% of the LCSs passed and the data are reported.

The batch LCS recovery results were reviewed when evaluating the analytical method uncertainty in section 3.7 of the report. Individual LCS results are included in the raw data and available upon request.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.5. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.4, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in **Table 7** below.

Table 7. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	Internal	9.05	±18%
PFHS	Internal	11.2	±22%
PFOS	Internal	10.4	±21%
PFOSA	Internal	9.95	±20%

3.8 Field Matrix Spikes (FMS)

Target analyte field matrix spike samples were collected for select sampling points to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions at 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOA and PFOS. The standards used for the preparation of the field matrix spiking solutions greater than 10 ng/mL (nominal) contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, select sample bottles contained stable isotope surrogate recovery spikes of [¹³C₄]-PFOA and [¹³C₄]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles prior to sample collection. The [¹³C₄]-PFOA was selected to represent the C8 perfluorocarboxylic acid. The [¹³C₄]-labeled PFOS was selected to represent the C6 and C8 perfluorosulfonic acids and perfluorooctanesulfonamide. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that “unknown” components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

The following calculation was used to generate data in section 4 of the report.

$$FMS\ Recovery = \frac{Sample\ Concentration\ of\ FMS - Average\ Concentration\ (Field\ Sample\ & Field\ Sample\ Dup)}{Spike\ Concentration} \times 100\%$$

Table 8. Field Matrix Spike Concentrations

Sampling Location	Final Spike Concentration (ng/mL)			
	PFOA	PFHS	PFOS	PFOSA
Blokkersdijkvijver Noord	0.958	1.00	0.927	1.00
p114bis, Travel Blank	4.19	4.20	4.19	0.200

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of $\pm 30\%$, demonstrate that the method is appropriate for the given matrix. All FMS and surrogate recoveries met method acceptance criteria.

The method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. At times, the spike level exceeded the recommended upper limit of 10 times the analyte concentration. In these instances, the FMS recovery was reported and flagged as above 10 times the sample concentration.

Table 9. Location ID: Blokkersdijkvijer Noord

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-03-002	Blokkersdijkvijer Noord; Sample	0.984	NA	0.602	NA
ISO20-14-03-002-DUP	Blokkersdijkvijer Noord; Sample Duplicate	0.920	NA	0.588	NA
ISO20-14-03-002-FMS	Blokkersdijkvijer Noord; Sample FMS	1.79	87.5	1.45	85.5
Average Concentration (ng/mL) \pm %RPD		0.952 ng/mL \pm 6.7%		0.595 ng/mL \pm 2.4%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-03-002	Blokkersdijkvijer Noord; Sample	3.76	NA	0.193	NA
ISO20-14-03-002-DUP	Blokkersdijkvijer Noord; Sample Duplicate	2.00	NA	0.119	NA
ISO20-14-03-002-FMS	Blokkersdijkvijer Noord; Sample FMS	3.80	NC	1.04	88.4
Average Concentration (ng/mL) \pm %RPD		2.88 ng/mL \pm 61% ⁽¹⁾		0.156 ng/mL \pm 47% ⁽¹⁾	

NA = Not Applicable

NC = Not Calculated; Spike level was less than 0.5 times the endogenous sample concentration.

(1) Sample/sample duplicate RPD did not meet acceptance criteria of $\le 20\%$.

Table 10. Location ID: p114bis

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-03-007	P114bis; Sample	3.28	NA	1.28	NA
ISO20-14-03-007-DUP	P114bis; Sample Duplicate	3.28	NA	1.28	NA
ISO20-14-03-007-FMS	P114bis; Sample FMS	6.96	87.8	4.68	81.0
Average Concentration (ng/mL) ± %RPD		3.28 ng/mL ± 0.0%		1.28 ng/mL ± 0.0%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-03-007	P114bis; Sample	5.38	NA	0.272	NA
ISO20-14-03-007-DUP	P114bis; Sample Duplicate	5.56	NA	0.282	NA
ISO20-14-03-007-FMS	P114bis; Sample FMS	9.30	91.5	0.204	88.2
Average Concentration (ng/mL) ± %RPD		5.47 ng/mL ± 3.3%		0.0277 ng/mL ± 3.6%	

NA = Not Applicable

Table 11. Location ID: Travel Blank

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-03-011	Travel Blank	<0.192	NA	<0.0250	NA
ISO20-14-03-011-FMS	Travel Blank FMS	3.74	89.2	3.44	81.9

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO20-14-03-011	Travel Blank	<0.0232	NA	<0.0250	NA
ISO20-14-03-011-FMS	Travel Blank FMS	3.54	84.6	0.195	97.5

NA = Not Applicable

Table 12. Surrogate Recovery Standard Results

3M LIMS ID	Sample Description	Percent Recovery (%)				
		11/6/20 [¹³ C ₄]PFOA	11/6/20 [¹³ C ₄]PFOS	11/16/20 [¹³ C ₄]PFOS	12/1/20 [¹³ C ₄]PFOA	12/1/20 [¹³ C ₄]PFOS
ISO20-14-03-001	3M vijver; Sample	108	95.1	NA	NA	NA
ISO20-14-03-002	Blokkersdijkvijver Noord; Sample	NA	95.3	94.0	89.0	NA
ISO20-14-03-002-DUP	Blokkersdijkvijver Noord; Sample Duplicate	NA	96.8	92.8	91.4	NA
ISO20-14-03-002-FMS	Blokkersdijkvijver Noord; Sample FMS	NA	95.7	97.2	93.4	NA
ISO20-14-03-003	Blokkersdijkvijver standaard; Sample	96.4	95.3	NA	NA	NA
ISO20-14-03-004	L21; Sample	97.8	93.4	93.0	NA	NA
ISO20-14-03-005	L31; Sample	102	98.4	NA	NA	NA
ISO20-14-03-006	L4; Sample	105	98.2	NA	NA	NA
ISO20-14-03-007	P114bis; Sample	101	98.2	NA	NA	89.2
ISO20-14-03-007-DUP	P114bis; Sample Duplicate	105	98.2	NA	NA	89.0
ISO20-14-03-007-FMS	P114bis; Sample FMS	103	105	NA	NA	90.9
ISO20-14-03-008	P115; Sample	100	93.4	NA	NA	NA
ISO20-14-03-009	P116; Sample	104	97.2	NA	NA	NA
ISO20-14-03-010	L30; Sample	104	99.9	NA	NA	NA
ISO20-14-03-011	Travel Blank	108	103	NA	NA	NA
ISO20-14-03-011-FMS	Travel Blank FMS	104	99.3	NA	NA	NA

NA = Not Applicable

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M EHS Laboratory method ETS-8-044.4 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 9-12 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M EHS Laboratory standard operating procedures.

7 Attachment

Chain of Custody Form

8 Signatures

Devon Bulman, Ph.D., 3M Report Author

Susan T. Wolf, 3M Principal Analytical Investigator

Brian T. Mader, Ph.D., 3M EHS Laboratory Manager

The 3M EHS Laboratory's Quality Assurance Unit has audited the data and report for this project.

Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M EHS Laboratory.

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Project: ISO20-14-03

Phone: [REDACTED]
Alt. Pho: [REDACTED]
Fax: (651) 777-1111

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 7/17/2020
Project Description: 3M Antwerp Water Sampling for PFCs; August 2020 Sampling
Comments:

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
ISO20-14-03-001	3M vijver; Sample	06/08 - 9:00	WS	
ISO20-14-03-002	Blokkersdijkvijver Noord; Sample	06/08 - 9:15	WS	
ISO20-14-03-002-DUP	Blokkersdijkvijver Noord; Sample Duplicate	06/08 - 9:15	WS	
ISO20-14-03-002-FMS	Blokkersdijkvijver Noord; Sample FMS	06/08 - 9:15	WQ	
ISO20-14-03-003	Blokkersdijkvijver standaard; Sample	06/08 - 9:30	WS	
ISO20-14-03-004	L21; Sample	06/08 - 10:24	WG	
ISO20-14-03-005	L31; Sample	06/08 - 9:40	WG	
ISO20-14-03-006	L4; Sample	06/08 - 9:38	WG	
ISO20-14-03-007	P114bis; Sample	06/08 - 13:20	WG	
ISO20-14-03-007-DUP	P114bis; Sample Duplicate	06/08 - 13:20	WG	
ISO20-14-03-007-FMS	P114bis; Sample FMS	06/08 - 13:20	WQ	
ISO20-14-03-008	P115; Sample	06/08 - 13:04	WG	

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 30 Deg C Received on Ice Other: RT

Collected by (print): Julie FICHEFET (JFI) Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
12	Julie FICHEFET	06/08/20	10:00	FedEx	<u>JFK</u>	06/13/20	11:23

3M EHS LABORATORY
Chain-of-Custody

Shipping Address:
3M EHS Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Project: ISO20-14-03 (cont.)

Phone: [REDACTED]
Alt. Ph: [REDACTED]
Fax: (651) 753-2000

Requester: Tack, Charlotte (ZWIJNDRECH-3ME)
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 7/17/2020

Project Description: 3M Antwerp Water Sampling for PFCs; August 2020 Sampling

Completion Date:

Project Lead: Susan T. Wolf
Phone Number: [REDACTED]
Email Address: [REDACTED]

<u>3M Sample Number</u>	<u>Sample Description</u>	<u>Date/Time Sampled</u>	<u>Matrix</u>	<u>Comment</u>
X ISO20-14-03-009	P116; Sample	04/08/2020 12:30	WG	
X ISO20-14-03-010	L30; Sample	04/08/2020 12:02	WG	
X ISO20-14-03-011	Travel Blank	7/21/20 12:25	WQ	SNW
X ISO20-14-03-011-FMS	Travel Blank FMS	7/21/20 12:25	WQ	SNW

Sample Condition Upon Receipt: Acceptable All items accounted for

Temperature: 30 Deg C Received on Ice Other:
RT

Collected by (print): Julie FICHEFET Collector's signature: [Signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
--------	------------------	------	------	-------------	--------------	------	------

5	Julie FICHEFET	10/08/20	10:00	FedEx	[Signature]	8/13/20	11:23

BIJLAGE 6 OVERZICHT VELDMETINGEN EN ANALYSERESULTATEN

Analyseresultaten PFAS

Puntlocatie	12	12	12	12	12	12	12	12	12	12	12	12
Top filter (m)	0	0	0	0	0	0	0	0	0	0	0	0
Bodem filter (m)	0	0	0	0	0	0	0	0	0	0	0	0
Datum bemonstering	7/11/2017	22/01/2018	3/05/2018	9/07/2018	25/10/2018	31/01/2019	24/04/2019	30/07/2019	22/10/2019	28/01/2020	28/04/2020	4/08/2020
Eenheid												
Grondwaterstand	m-mv		0									
Redox	mV	84,10	96,40	106	76,80	46,30	96	248	184	-210	155	-83,40
pH		7,92	7,11	8,02	7,83	7,31	7,79	7,27	7,92	9,06	7,44	7,18
EC	µS/cm	877	891	887	856	582	786	897	377	617	890	705
Temperatuur	°C	7,13	8,09	12,66	16,07	12,46	2,47	16,31	30,20	15,81	7,18	16,92
PFOS	µg/L	243	143	205	449	355	173	296	224	167	190	387
PFOA	µg/L	65,50	32,10	52,10	121	109	44,20	62,90	56,70	53,50	45,10	89,20
PFHS	µg/L	16,70	9,85	16,50	34,60	29,90	14,30	18,20	17,70	17,40	13,50	25,80
PFOSA	µg/L	0,18	0,37	0,68	0,66	0,27	0,32	0,56	0,36	0,23	0,20	0,66
PFBS	µg/L											< 1,01

Puntlocatie	13	13	13	13	13	13	13	13	13	13	13	13
Top filter (m)	0	0	0	0	0	0	0	0	0	0	0	0
Bodem filter (m)	0	0	0	0	0	0	0	0	0	0	0	0
Datum bemonstering	7/11/2017	22/01/2018	3/05/2018	9/07/2018	25/10/2018	31/01/2019	24/04/2019	30/07/2019	22/10/2019	28/01/2020	28/04/2020	4/08/2020
Eenheid												
Grondwaterstand	m-mv		0									
Redox	mV	7	139	75,90	74,60	104	19,30	243	180	1,10	155	-69,30
pH		7,60	6,66	7,61	7,87	7,42	7,61	7,30	7,80	8,53	7,36	7,80
EC	µS/cm	809	960	685	950	540	796	897	619	593	885	697
Temperatuur	°C	7,58	8,08	11,83	15,75	12,36	2,38	16,38	27,50	16,90	7,22	16,99
PFOS	µg/L	198	138	179	369	281	155	274	226	167	174	315
PFOA	µg/L	63,20	34,30	52,70	108	90,80	43,80	69,80	60,50	37	43,30	85,70
PFHS	µg/L	21,30	11,60	18,10	36,60	33,80	15,60	22,40	23,40	13,10	14	27
PFOSA	µg/L	0,23	0,33	0,59	0,40	0,25	0,33	0,57	0,45	0,21	0,20	0,56
PFBS	µg/L											< 1,01

Puntlocatie	3M Vijver	3M Vijver	3M Vijver	3M Vijver	3M Vijver	3M Vijver	3M Vijver	3M Vijver	3M Vijver	3M Vijver	3M Vijver	3M Vijver
Top filter (m)												
Bodem filter (m)												
Datum bemonstering	7/11/2017	30/01/2018	3/05/2018	10/07/2018	25/10/2018	31/01/2019	24/04/2019	30/07/2019	22/10/2019	28/01/2020	28/04/2020	4/08/2020
Eenheid												
Grondwaterstand	m-mv		0									
Redox	mV	75,50	0	106	86,50	98,20	59,80	268	185	-28,70	128	-42,30
pH		8,31	184	7,69	7,88	6,90	7,58	7,09	8,11	9,06	7,39	7,88
EC	µS/cm	2425	7,26	621	881	944	777	1103	800	1250	1036	725
Temperatuur	°C	7,16	417	13,08	16,97	12,45	1,99	16,88	30,20	16,18	5,96	16,98
PFOS	µg/L	2,40	6,26	4,37	12,60	4,94	1,93	3,20	14,10	1,68	2,42	3,53
PFOA	µg/L	1,08	1,29	1,04	1,74	1,72	0,72	1,04	1,65	0,58	0,68	1,27
PFHS	µg/L	0,55	0,73	0,56	0,78	0,72	0,39	0,58	0,92	0,37	0,44	0,52
PFOSA	µg/L		< 0,0248	0,04	0,08	0,04	0,03	0,07	0,19	< 0,0250	< 0,0250	< 0,0990
PFBS	µg/L											0,27

Puntlocatie	5	5	5	5	5	5	5	5	5	5	5	5
Top filter (m)												
Bodem filter (m)												
Datum bemonstering	7/11/2017	22/01/2018	3/05/2018	9/07/2018	25/10/2018	31/01/2019	24/04/2019	30/07/2019	22/10/2019	28/01/2020	28/04/2020	4/08/2020
Eenheid												
Grondwaterstand	m-mv											
Redox	mV	47,20	67,40	664	83,20	27,10	37,10	238	187	22,90	167	-55,40
pH		8,05	7,64	7,66	7,67	7,46	7,68	7,36	7,80	8,01	7,26	7,71
EC	µS/cm	1567	943	743	1732	1277	1042	1180	1534	983	1330	1145
Temperatuur	°C	8,50	6,72	13,20	16,98	12,73	1,51	17,03	28	16,71	6,34	17
PFOS	µg/L	67,30	33,30	43	125	52,40	32,90	42,80	110	50,20	50,40	14
PFOA	µg/L	38,80	16,10	21,30	89,30	36,10	16,70	28	66,50	25,80	30,90	6,74
PFHS	µg/L	34,30	13,20	19,10	81,80	36,40	14,60	26,40	85,20	24,60	26,50	6,38
PFOSA	µg/L	0,32	0,10	0,18	0,29	0,14	0,21	0,18	0,32	0,14	0,14	0,10
PFBS	µg/L											< 0,187

Analyseresultaten PFAS

Puntlocatie		B3-BIS	B3-BIS	BD24-3	BD24-3	BD24-3	BD24-3	BD24-3	BD24-3	BD24-3	BD24-4	BD24-4	BD24-4
Top filter (m)	2,5	2,5	17	17	17	17	17	17	17	17	22	22	22
Bodem filter (m)	3,5	3,5	18	18	18	18	18	18	18	18	24	24	24
Datum bemonstering	1/11/2017	31/01/2018	7/11/2017	30/01/2018	10/07/2018	31/01/2019	30/07/2019	28/01/2020	4/08/2020	30/01/2018	10/07/2018	31/01/2019	10/07/2018
Eenheid													
Grondwaterstand	m-mv	2,25	1,85		2,98	3,83	3,22	3	2,88	2,82	3	4,11	3,21
Redox	mV	-182,70	93,40				-103						-81,20
pH		7,52	7,27		7,53	7,43	8,05	7,80	7,60	7,30	7,22	7,23	7,93
EC	µS/cm	238	437		698	938	882	860	756	669	1377	1409	1330
Temperatuur	°C	9,94	9,26		11,10	11,50	10,40	36	10,10	16	10,70	11,50	10,40
PFOS	µg/L		110	988	549	1030	977	842	677	608	6,50	4,52	3,12
PFOA	µg/L		11,10	31,60	54,50	35,30	28,10	24,40	17,70	20,10	10,60	15,30	21,80
PFHS	µg/L		3,27	29,30	40,20	35,80	28,40	26,60	18,50	17,20	3,79	5,22	8,57
PFOSA	µg/L		0,26	4,30	12,60	4,11	3,21	4,33	3,20	5,47	0,47	0,14	0,15
PFBS	µg/L												

Puntlocatie		BD24-4	BD24-4	BD24-4	Bemalingsstation								
Top filter (m)	22	22	24	0	0	0	0	0	0	0	0	0	0
Bodem filter (m)	24	24	24	24	0	0	0	0	0	0	0	0	0
Datum bemonstering	30/07/2019	28/01/2020	4/08/2020	7/11/2017	22/01/2018	3/05/2018	9/07/2018	25/10/2018	31/01/2019	24/04/2019	30/07/2019	22/10/2019	22/10/2019
Eenheid													
Grondwaterstand	m-mv		2,87	2,76		0							
Redox	mV				81,10	92,20	96,40	77,70	55,30	44,10	243	191	28,90
pH			7,41	7,14	7,77	7,46	7,58	7,85	7,05	7,66	7,26	7,86	7,98
EC	µS/cm		1290	1380	3065	850	995	2209	1187	1123	1152	1471	1075
Temperatuur	°C		10,60		9,12	7,60	12,70	17,32	13,12	1,51	16,95	27,10	15,96
PFOS	µg/L	0,70	1,31	3,31	56,60	35,40	39,40	94,70	49,60	29,90	38,20	87,50	31,60
PFOA	µg/L	82,20	146	99,80	39,80	18	18,30	67,40	35,90	16,70	24,40	60,80	28,60
PFHS	µg/L	45,30	53,80	33,60	35,10	14,50	16,20	62	36,50	12,40	23	73,80	22,70
PFOSA	µg/L	0,31	0,27	< 0,187	0,34	0,14	0,17	0,23	0,12	0,15	0,21	0,22	< 0,248
PFBS	µg/L												

Puntlocatie		Bemalingsstation	Bemalingsstation	Bemalingsstation	Ookkersdijkvijver Nootdorp								
Top filter (m)	0	0	0	0	0	0	0	0	0	0	0	0	0
Bodem filter (m)	0	0	0	0	0	0	0	0	0	0	0	0	0
Datum bemonstering	28/01/2020	28/04/2020	4/08/2020	7/11/2017	30/01/2018	10/07/2018	25/10/2018	31/01/2019	24/04/2019	30/07/2019	22/10/2019	22/10/2019	28/01/2020
Eenheid													
Grondwaterstand	m-mv				0								
Redox	mV	168	-42	91,20	70,40	170	80,60	86,60	62,20	250	187		131
pH		7,25	7,67	7,62	8,34	8,12	8,71	7,47	8,09	7,33	7,69		7,97
EC	µS/cm	1545	1232	738	1236	216	418	466	425	683	544		1300
Temperatuur	°C	6,27	17,03	29,80	6,47	6,84	19,44	12,70	0,40	17,21	29,30		5,77
PFOS	µg/L	44,10	45	58,20	0,95	1,97	0,73	1,04	5,20	2,98	1,69	2,61	2,10
PFOA	µg/L	28,10	27,50	48,10	1,14	1,12	0,88	1,10	0,88	0,97	1,27	1,12	0,93
PFHS	µg/L	23,80	24,30	38,50	0,61	0,63	0,54	0,64	0,48	0,56	0,78	0,74	0,55
PFOSA	µg/L	0,12	0,17	< 0,187	0,05	< 0,0248	< 0,25	0,03	0,17	0,06	0,08	0,09	0,10
PFBS	µg/L												

Puntlocatie		Ookkersdijkvijver Nootdorp												
Top filter (m)	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Bodem filter (m)	0	0	0	0	0	0	0	0	0	0	0	0	0	0
Datum bemonstering	28/04/2020	4/08/2020	7/11/2017	30/01/2018	3/05/2018	10/07/2018	25/10/2018	31/01/2019	24/04/2019	30/07/2019	22/10/2019	22/10/2019	28/01/2020	
Eenheid														
Grondwaterstand	m-mv				0									
Redox	mV	-51,30	45,20	92,60	183	70,90	80,60	93,80	75,80	241	175	-4,60	118	
pH			7,61	8,75	8,14	8,11	9,22	8,21	7,52	6,12	7,40	8,27	8,59	7,90
EC	µS/cm		237	385	1135	464	363	577	427	389	629	464	366	690
Temperatuur	°C	16,99	32	7,52	6,62	15,01	17,96	13,07	0,57	16,83	27,50	16,46	5,66	
PFOS	µg/L	0,92	2,88	14,10	0,68	0,74	5,87	1,13	1,05	0,85	1,08	3,06	0,50	
PFOA	µg/L	0,74	0,95	1,25	1,04	0,73	1,31	1,23	0,81	0,91	1,15	0,87	0,80	
PFHS	µg/L	0,47	0,60	1,11	0,62	0,44	0,71	0,77	0,47	0,60	0,77	0,50	0,50	
PFOSA	µg/L	< 0,0990	0,16	0,05	< 0,0248	0,03	0,17	0,06	0,04	0,04	0,12	0,17	< 0,0250	
PFBS	µg/L													

Analyseresultaten PFAS

Puntlocatie		Kkersdijkvijver Stand	Kkersdijkvijver Stand	Collector put	Collector put	Collector put	Collector put	Collector put	Collector put	Collector put	Collector put	Collector put	Collector put					
Top filter (m)	0	0	Bodem filter (m)	0	0	Datum bemonstering	28/04/2020	4/08/2020	6/11/2017	30/01/2018	3/05/2018	10/07/2018	25/10/2018	30/01/2019	24/04/2019	30/07/2019	22/10/2019	27/01/2020
Eenheid																		
Grondwaterstand	m-mv																	
Redox	mV	-83,40	54,20	-49,20	-59	-84,80	84,20	39,20	-31,90	77,40	144	-3,60	111					
pH		8,32	8,30	7,03	7,29	7,58	8,21	6,90	7,52	7,51	7,11	7,90	7,11					
EC	µS/cm	447	360	589	312	725	1059	620	595	759	760	430	1905					
Temperatuur	°C	17,22	35,40	12,96	10,27	13,60	18,66	15,86	6,34	20,40	19,69	15,77	11,61					
PFOS	µg/L	4,71	3,74	159	234	202	203	284	210	269	230	144	49,60					
PFOA	µg/L	0,65	1,65	43	75,30	75,20	60,30	71,90	50,80	85,60	67,10	65,30	13,50					
PFHS	µg/L	0,45	0,96	16,60	25,30	26,10	22,60	29,80	23,20	31,20	29,30	26,50	6,20					
PFOSA	µg/L	0,19	0,33	14,20	13,80	20,80	14,40	19,10	19,20	24,40	38,10	7,81	5					
PFBS	µg/L																	
Puntlocatie		Collector put	Collector put	D10	D10	D10	D10	D10	D10	D11	D11	D11	D11					
Top filter (m)	0	0	Bodem filter (m)	15	15	14	15	13	15	14,9	14,9	14,9	14,9					
Datum bemonstering	28/04/2020	4/08/2020	30/01/2018	10/07/2018	8/01/2019	30/01/2019	30/07/2019	28/01/2020	4/08/2020	4/08/2020	29/01/2018	12/07/2018	30/01/2019					
Eenheid																		
Grondwaterstand	m-mv			4,08	5,20	3,94	4,37		4	3,77	2,90	3,56	3,44					
Redox	mV	-42,40	109								-88,60	-146,10						
pH		7,82	7,85	6,98	7,19	7,53	7,23		7,07	6,45	7,02	7,38	7,49					
EC	µS/cm	509	899	2155	1837	477	1830		1820	2070	209	484	568					
Temperatuur	°C	16,81	20,30	11,50	11,90	12,20	10		10,70	13,50	11,11	13,25	10					
PFOS	µg/L	51,20	40,70	24,80	58,80		43	53,30	25,70	24,90	1500	816	712					
PFOA	µg/L	21,70	22,70	343	549		428	659	164	278	418,00	131	35,60					
PFHS	µg/L	7,27	5,39	141	229		177	282	57,10	87,90	155,00	83,60	50,30					
PFOSA	µg/L	10,50	5,05	0,15	< 0,25		0,07	< 0,100	0,10	< 1,01	13,80	61,10	96,50					
PFBS	µg/L																	
Puntlocatie		D11	D11	D11	D14	D14	D14	D14	D14	D16	D16	D16	D16					
Top filter (m)	14,9	14,9	14,9	15,5	15,5	15,5	15,5	15,5	15,5	14	14	14	14					
Bodem filter (m)	15,9	15,9	15,9	15,9	16,5	16,5	16,5	16,5	16,5	15,68	15,68	15,68	15,68					
Datum bemonstering	30/07/2019	28/01/2020	4/08/2020	29/01/2018	11/07/2018	30/01/2019	30/07/2019	29/01/2020	4/08/2020	3/05/2018	11/07/2018	31/01/2019						
Eenheid																		
Grondwaterstand	m-mv	3,65	3,38	2,63	4,82	5,02	4,90	5,17	4,22	3,96	3,07	3,61	3,48					
Redox	mV						175	-88,90			-34,90	-110,20	-105					
pH		7,44	7,36	6,78	6,89	6,92	6,80	6,86	7,09	7,01	7,05	6,91	7,20					
EC	µS/cm	940	812	868	3840	3750	3879	2615	2430	2950	600	872	705					
Temperatuur	°C	12,90	11,20	14,50	13,20	14	11,12	15,98	11,30	16,40	13,03	16,37	11,60					
PFOS	µg/L	1650	1210	793	0,39	1,08	1,83	0,81	0,44	0,51	2480	3920	1210					
PFOA	µg/L	685	320	205	2,10	1,93	2,10	2,37	2,08	2,30	386	661	189					
PFHS	µg/L	566	129	123	1,15	1,60	1,65	1,44	1,15	1,26	134	292	63,70					
PFOSA	µg/L	40,10	22,70	57,60	0,08	0,08	0,12	0,09	0,03	< 0,187	65,10	16	86,30					
PFBS	µg/L																	
Puntlocatie		D16	D16	D16	D16	D16	D17	D17	D17	D17	D17	D17	D18					
Top filter (m)	14	14	14	15,68	15,68	15,68	14	14	14	14	14	14	13					
Bodem filter (m)	15,68	15,68	15,68	15,68	15,68	15,68	15	15	15	15	15	15	15					
Datum bemonstering	24/04/2019	30/07/2019	21/10/2019	28/01/2020	4/08/2020	29/01/2018	11/07/2018	31/01/2019	30/07/2019	29/01/2020	4/08/2020	29/01/2018						
Eenheid																		
Grondwaterstand	m-mv	3,42	3,73			3,54	3,63	3,17	3,62	3,51	3,07	3,03	2,86					
Redox	mV				3,73	-51,10	16,40		-91	-89,40			2,35					
pH		7,07	7,40	7,21	7,19	7,17	7,07	7,02	7,71	7,09	7,33	7,78	7,45					
EC	µS/cm	940	930	726	1364	537	1143	1054	1075	567	682	652	978					
Temperatuur	°C	12,70	13,30	13	11,49	15,90	15,50	16,30	14,50	18,20	14,90	16,90	13,10					
PFOS	µg/L	3420	2640	2130	2540	631	202	273	234	310	395	317	56,50					
PFOA	µg/L	880	730	629	789	140	784	490	731	640	380	162	299					
PFHS	µg/L	393	338	261	319	39,10	304	171	238	152	67,80	27	180					
PFOSA	µg/L	4,22	7,52	12,50	19,40	91	0,13	1,50	0,10	0,27	0,26	< 1,01	3,47					
PFBS	µg/L																	

Analyseresultaten PFAS

Puntlocatie	D18	D18	D18	D18	D18	D2	D2	D2	D5	D5	D5	D5
Top filter (m)	13	13	13	13	13	23,3	23,3	23,3	15	15	15	15
Bodem filter (m)	15	15	15	15	15	24,3	24,3	24,3	16	16	16	16
Datum bemonstering	11/07/2018	30/01/2019	30/07/2019	29/01/2020	4/08/2020	10/07/2018	30/07/2019	4/08/2020	29/01/2018	10/07/2018	30/01/2019	30/07/2019
Eenheid												
Grondwaterstand	m-mv	2,84	2,69	3,02	2,36	2,21	6,32	5,42	4,47	3,20	3,62	3,45
Redox	mV	93,20	-86,70					-46,30			33	-93,60
pH		7,37	7,26	7,31	7,43	7,83	7,71	6,58	8,60	7,17	7,18	7,24
EC	µS/cm	1002	968	457	792	717	375	684	326	912	915	963
Temperatuur	°C	14,70	11,40	15,33	11,70	15,70	12,80	14,23	14,80	13,20	14,90	12,40
PFOS	µg/L	52,20	66,80	74,40	73,40	42,50	7,99	0,20	6,70	450	426	286
PFOA	µg/L	220	285	183	117	40,50	0,37	< 0,0240	0,20	495	433	311
PFHS	µg/L	152	205	156	86,40	16,10	0,17	< 0,0250	0,20	258	174	174
PFOSA	µg/L	2,86	1,90	1,98	0,56	< 1,01	0,42	0,09	0,40	7,24	8,77	2,44
PFBS	µg/L											11,80

Puntlocatie	D5	D5	D9	D9	D9	D9	Effluent WWTP					
Top filter (m)	15	15	13	13	13	15	0	0	0	0	0	0
Bodem filter (m)	16	16	15	15	15	15	0	0	0	0	0	0
Datum bemonstering	29/01/2020	4/08/2020	6/11/2017	29/01/2018	11/07/2018	31/01/2019	6/11/2017	30/01/2018	3/05/2018	10/07/2018	25/10/2018	30/01/2019
Eenheid												
Grondwaterstand	m-mv	2,70	2,60	3,30	3,15	3,57	3,43	0	0			
Redox	mV						86	-54,10	-54,90	-84,30	101	-48,90
pH		7,33	7,94	7,21	7,28	7,29	7,20	6,20	6,87	7,75	7,78	7,79
EC	µS/cm	604	615	1074	1039	1109	1158	382	1008	612	5289	1897
Temperatuur	°C	13,10	15,30	15,50	15,50	16,80	14,70	13,44	10,86	13,60	19,74	19,37
PFOS	µg/L	235	538	1090	1970	1410	3180	3,74	83,90	10,80	0,67	28,30
PFOA	µg/L	153	522	181	281	310	384	1,98	31,20	2,14	0,08	13
PFHS	µg/L	51,40	84,60	2470	5390	3790	14100	2,65	37,90	1,51	0,09	14
PFOSA	µg/L	2,40	23,10	17	22,10	15,50	22,70	0,06	3,24	0,89	0,06	0,46
PFBS	µg/L											1,04

Puntlocatie	Effluent WWTP	K3	K3	K3	L19	L19	L19					
Top filter (m)	0	0	0	0	0	0	3,6	3,6	3,6	2,8	2,8	2,8
Bodem filter (m)	0	0	0	0	0	0	5,6	5,6	5,6	4,8	4,8	4,8
Datum bemonstering	24/04/2019	30/07/2019	21/10/2019	27/01/2020	28/04/2020	4/08/2020	11/07/2018	15/10/2018	30/07/2019	4/08/2020	30/01/2018	11/07/2018
Eenheid												
Grondwaterstand	m-mv									2,35	2,90	1,68
Redox	mV	55,70	190		118	-56,40	132			-111		106
pH		7,12	8,02		6,82	8,06	7,87		7,33	7,30	8,04	7,26
EC	µS/cm	3877	3780		5118	2280	3053		647	479	516	737
Temperatuur	°C	21	26		11,60	16,80	19,43		16,70	18,43	18,20	10,22
PFOS	µg/L	29,60	0,05	7,09	6,80	43,40	< 1,30	4830	3303	6710	6839	6640
PFOA	µg/L	11,40	< 0,0240	7,83	2,70	13,50	< 0,988	78,10	70,10	81,10	75	281
PFHS	µg/L	10,60	< 0,0250	6,11	1,50	163	< 0,966	17100	11590	14900	9453	132
PFOSA	µg/L	0,58	< 0,0500	0,13	0,30	1,08	< 1,01	13,80	< 12,10	13	16,20	80,10
PFBS	µg/L							294	284	288	224,79	

Puntlocatie	L19	L19	L19	L19	L21	L21	L21	L21	L21	L21	L21	L21
Top filter (m)	2,8	2,8	2,8	2,8	3	3	3	3	3	3	0	0
Bodem filter (m)	4,8	4,8	4,8	4,8	5	5	5	5	5	5	0	0
Datum bemonstering	31/01/2019	30/07/2019	29/01/2020	4/08/2020	7/11/2017	31/01/2018	4/05/2018	10/07/2018	23/10/2018	31/01/2019	24/04/2019	22/10/2019
Eenheid												
Grondwaterstand	m-mv	3,73	3,53	3,57	3,67	1,92	1,66	1,67	2,09	0,02	1,76	2,07
Redox	mV	7		25,60	8,40			-157,10		-120	-175,60	-158
pH		8,15	7,31	7,08	7,20	7,63	7,54	7,42	7,55	7,25	7,74	7,71
EC	µS/cm	1035	1430	1000	601	505	528	583	501	365	319	403
Temperatuur	°C	9,10	17,20	9,72	18,82	9,60	9,40	11,79	16,20	15,96	8,43	14,22
PFOS	µg/L	3960	3320	2600	3728	3,85	3,43	4,53	5,01	5,92	4,18	4,14
PFOA	µg/L	257	242	267	582	0,19	0,22	0,22	0,28	0,18	< 0,192	0,15
PFHS	µg/L	176	159	148	174	0,09	0,10	0,12	0,14	0,09	0,13	0,10
PFOSA	µg/L	26,90	47,10	47,70	77,30	0,04	0,05	0,11	0,11	0,07	0,36	0,05
PFBS	µg/L											0,29

Analyseresultaten PFAS

Puntlocatie	L21	L21	L21	L21	L22	L22	L22	L22	L22	L22	L22	L22
Top filter (m)	3	3	3	3	2	2	2	2	2	2	2	2
Bodem filter (m)	5	5	5	5	4	4	4	4	4	4	4	4
Datum bemonstering	30/07/2019	29/01/2020	29/04/2020	4/08/2020	7/11/2017	31/01/2018	4/05/2018	10/07/2018	23/10/2018	30/01/2019	24/04/2019	30/07/2019
Eenheid												
Grondwaterstand	m-mv		1,32	2,15	1,48		2,21	1,93	2,28	0,03	2,59	2,61
Redox	mV	2,62		-161			-109,60		-111,40	-58,70	-102	-134,20
pH		-37,80	7,83	7,28	7,93		7,11	7,11	6,85	7,22	7,48	7,17
EC	µS/cm	7,30	435	3099	508		1371	1817	2290	1620	1412	1081
Temperatuur	°C	262	9,30	11,37	18,40		9,50	12,03	14,90	15,19	9,30	13,76
PFOS	µg/L	18,18	2,13	6,23	2,28	8,41	6,58	4,20	2,81	2,52	2,28	2,76
PFOA	µg/L	0,17	0,15	0,31	0,37	0,40	0,31	0,26	0,19	0,40	0,35	0,35
PFHS	µg/L	0,14	0,08	0,20	0,25	0,26	0,17	0,18	0,16	0,25	0,23	0,26
PFOSA	µg/L	0,06	0,04	0,08	0,03	0,03	0,07	0,07	0,03	0,06	0,09	0,06
PFBS	µg/L											0,11

Puntlocatie	L22	L22	L30	L30	L31	L31	L31	L31	L31	L31	L31	L31
Top filter (m)	2	2	1	1	1,2	1,2	1,2	1,2	1,2	1,2	1,2	1,2
Bodem filter (m)	4	4	3	3	3,2	3,2	3,2	3,2	3,2	3,2	3,2	3,2
Datum bemonstering	22/10/2019	29/01/2020	29/04/2020	4/08/2020	7/11/2017	31/01/2018	4/05/2018	10/07/2018	23/10/2018	31/01/2019	24/04/2019	30/07/2019
Eenheid												
Grondwaterstand	m-mv		1,72	2,75			2,05	2,11	2,45	2,64	2,12	2,42
Redox	mV	17,10						188		-92,10	-59	9,90
pH		7,78	7,46	8,14			7,43	7,18	7,22	7,45	7,55	7,21
EC	µS/cm	886	845	519			543	524	637	446	345	483
Temperatuur	°C	14,85	9,70	18			8,70	12,01	16,50	15,21	7,27	13,56
PFOS	µg/L	2,97	2,40	8,46	5,62	7,96	3,18	3,51	10,80	7,28	4,15	4,92
PFOA	µg/L	0,35	0,30	0,40	0,54	0,68	0,51	0,38	0,64	0,28	0,25	0,39
PFHS	µg/L	0,31	0,30	0,49	0,62	0,56	0,85	0,82	0,60	0,32	0,78	1,17
PFOSA	µg/L	0,15	0	0,07	0,06	0,08	< 0,0248	0,07	0,38	0,07	0,05	0,05
PFBS	µg/L											0,15

Puntlocatie	L31	L31	L31	L31	L4	L4	L4	L4	L4	L4	L4	L4
Top filter (m)	1,2	1,2	1,2	1,2	2	2	2	2	2	2	2	2
Bodem filter (m)	3,2	3,2	3,2	3,2	3,2	4	4	4	4	4	4	4
Datum bemonstering	22/10/2019	29/01/2020	29/04/2020	4/08/2020	7/11/2017	30/01/2018	3/05/2018	10/07/2018	24/10/2018	31/01/2019	24/04/2019	30/07/2019
Eenheid												
Grondwaterstand	m-mv		1,70	1,60	1,52	2,88	1,73	1,59		2,93	2,46	2,35
Redox	mV	2,84		-78,60		-159,30		-26,20		-172,90		
pH		6,63	7,60	7,90	7,41	7,14	7,18	7,14		6,93	7,41	7,11
EC	µS/cm	445	535	418	504	1541	607	480		621	706	940
Temperatuur	°C	14,64	8,50	10,74	18	13,07	9,30	11,06		14,26	7,80	11,20
PFOS	µg/L	7,83	4,57	3,99	4,02	27,70	25,10	20,10	28,20	28,20	26,80	24,80
PFOA	µg/L	0,39	0,27	0,34	0,54	1,87	2,41	2,15	2,19	2,39	2,42	2,10
PFHS	µg/L	0,66	0,61	0,81	0,65	1,94	1,89	1,76	2,01	2,03	1,76	1,87
PFOSA	µg/L	0,15	0,15	< 0,0990	0,06	0,22	5,85	3,88	0,70	0,21	1,52	1,88
PFBS	µg/L											0,46

Puntlocatie	L4	L4	L4	L4	M4	M4	M4	M4	M4	M4	ND7	ND7
Top filter (m)	2	2	2	2	1	1	1	1	1	1	15	15
Bodem filter (m)	4	4	4	4	3	3	3	3	3	3	16	16
Datum bemonstering	22/10/2019	28/01/2020	29/04/2020	4/08/2020	1/02/2018	9/07/2018	30/01/2019	30/07/2019	28/01/2020	4/08/2020	30/01/2018	11/07/2018
Eenheid												
Grondwaterstand	m-mv	1,83	60,50	60,15		1,67	1,84	2,14	2,12	2,11	2,35	2,65
Redox	mV	-22,30				139				116	-133,50	
pH		7,23	7,32	7,01		6,20	7,14	7,18	7,46	7,44	7,51	7,11
EC	µS/cm	1254	871	7,19		1210	1805	2136	1650	1180	1298	478
Temperatuur	°C	14,30	9,40	11,16		16,60	11,70	15	10,80	19,60	11,05	24,80
PFOS	µg/L	36,60	17,50	18,90		23,00	8940	9290	2120	1160	2170	1552
PFOA	µg/L	5,51	2,93	3,22		3,06	1790	576	194	134	208	142
PFHS	µg/L	4,14	1,98	1,98		2,26	277	145	78,80	56,20	82,10	42,60
PFOSA	µg/L	0,33	0,33	4,11		0,46	55,70	119	113	113	94,70	54,80
PFBS	µg/L											37,70

Analyseresultaten PFAS

Puntlocatie		ND7	ND7	ND7	ND7	P114BIS	P114BIS	P114BIS	P114BIS	P114BIS	P114BIS	P114BIS
Top filter (m)	15	15	15	15	3,6	3,6	3,6	3,6	3,6	3,6	3,6	3,6
Bodem filter (m)	16	16	16	16	4,6	4,6	4,6	4,6	4,6	4,6	4,6	4,6
Datum bemonstering	30/01/2019	30/07/2019	29/01/2020	4/08/2020	7/11/2017	31/01/2018	4/05/2018	10/07/2018	24/10/2018	1/02/2019	24/04/2019	30/07/2019
Eenheid												
Grondwaterstand	m-mv	2,72	3,23	2,34	2,24		1,46	1,53	2,15	2,31	1,72	1,91
Redox	mV	-56,30	-62,20					-152,30		-139,70	-142	-128,90
pH		7,58	6,94	7,33	7,73		7,16	7,14	7,16	6,73	7,44	7,03
EC	µS/cm	1530	619	857	721		1525	1917	1586	1773	3565	2480
Temperatuur	°C	11,90	17,25	12,50	14,40		10,60	12,37	14	14,87	10,60	12,40
PFOS	µg/L	47,30	103	145	61,90	14,40	13,30	13,90	11,90	9,73	3,92	6,50
PFOA	µg/L	154	182	194	297	1,87	2,14	2,14	1,33	3,23	3,38	3,40
PFHS	µg/L	75	43,30	36,50	63,40	1,23	1,28	1,26	1,41	1,45	1,44	1,54
PFOSA	µg/L	8,44	10,10	4,99	8,61	0,05	0,03	0,08	0,05	0,06	0,06	0,11
PFBS	µg/L											0,08

Puntlocatie		P114BIS	P114BIS	P114BIS	P114BIS	P115	P115	P115	P115	P115	P115	P115
Top filter (m)	3,6	3,6	3,6	3,6	3,3	3,3	3,3	3,3	3,3	3,3	3,3	3,3
Bodem filter (m)	4,6	4,6	4,6	4,6	4,3	4,3	4,3	4,3	4,3	4,3	4,3	4,3
Datum bemonstering	22/10/2019	29/01/2020	29/04/2020	4/08/2020	7/11/2017	31/01/2018	4/05/2018	10/07/2018	24/10/2018	1/02/2019	24/04/2019	30/07/2019
Eenheid												
Grondwaterstand	m-mv		1,43	1,42	1,86		1,78	1,75	2,23	2,42	2,03	2,20
Redox	mV	22,80		-166,30				-151,90		-131	-107	-125,10
pH		6,81	7,32	6,94	7,78		6,96	6,94	6,99	6,73	7,77	6,95
EC	µS/cm	1750	2150	1709	882		3260	2747	2970	2024	2392	2070
Temperatuur	°C	15,27	10,90	11,08	16,10		9,90	12,94	14,30	14,69	10,10	12,40
PFOS	µg/L	9,50	5	5,29	5,47	1,07	0,80	1,13	0,61	1,44	1,92	2,28
PFOA	µg/L	3,74	3,40	3,39	3,28	3,90	4,44	4,33	3,24	4,14	2,82	3,64
PFHS	µg/L	1,72	1,40	1,49	1,28	1,89	2,24	2,23	1,75	2,18	1,68	2,16
PFOSA	µg/L	0,06	< 0,0250	< 0,0500	0,03		0,07	< 0,01	< 0,025	0,04	< 0,0250	0,06
PFBS	µg/L											0,06

Puntlocatie		P115	P115	P115	P115	P116	P116	P116	P116	P116	P116	P116
Top filter (m)	3,3	3,3	3,3	3,3	2,5	2,5	2,5	2,5	2,5	2,5	2,5	2,5
Bodem filter (m)	4,3	4,3	4,3	4,3	3,5	3,5	3,5	3,5	3,5	3,5	3,5	3,5
Datum bemonstering	22/10/2019	29/01/2020	29/04/2020	4/08/2020	7/11/2017	31/01/2018	4/05/2018	10/07/2018	23/10/2018	1/02/2019	24/04/2019	30/07/2019
Eenheid												
Grondwaterstand	m-mv		8,03	8,01	1,77		2,09	2,42	2,25	2,86	2,50	2,72
Redox	mV	70,80		-166,70				-161,70		-140,50	-102,40	-168
pH		6,92	7,32	7,02	7,78		7,26	7,42	7,50	7,36	7,94	7,63
EC	µS/cm	1670	1770	1584	8,35		761	558	749	439	750	673
Temperatuur	°C	14,27	10,10	11,29	16,30		9,40	12,58	15,40	15,05	10,20	15,40
PFOS	µg/L	2,86	1,92	2,83	3,20	8,24	8,74	9,34	7,85	8,91	7,98	8,42
PFOA	µg/L	4,09	3,31	3,07	3,32	1,18	0,54	0,76	0,95	1	0,71	1,07
PFHS	µg/L	2,32	1,71	1,73	1,59	0,58	0,52	0,55	0,53	0,53	0,45	0,50
PFOSA	µg/L	0,05	< 0,0250	< 0,0500	< 0,0250	0,09	0,05	0,13	0,11	0,09	0,13	0,12
PFBS	µg/L											0,12

Puntlocatie		P116	P116	P116	P116	P117C	P118A	P118A	P118A	P118A	P118A	P118B
Top filter (m)	2,5	2,5	2,5	2,5	4	22,5	22,5	22,5	22,5	22,5	22,5	13
Bodem filter (m)	3,5	3,5	3,5	3,5	6	23,5	23,5	23,5	23,5	23,5	23,5	14
Datum bemonstering	22/10/2019	29/01/2020	29/04/2020	4/08/2020	15/10/2018	1/02/2018	11/07/2018	31/01/2019	30/07/2019	28/01/2020	4/08/2020	1/02/2018
Eenheid												
Grondwaterstand	m-mv		65,53		65,56		2,64	3,25	3,16	2,78	2,53	2,68
Redox	mV	52,30			-184,60			-67,70				2,66
pH		7,20	7,83		7,38		6,90	6,89	7,22	7,17	7,22	6,45
EC	µS/cm	615	681		570		1609	2082	1320	1800	1890	2000
Temperatuur	°C	14,77	10,20		10,79		10,90	17,23	9,10	12,50	10,60	13,20
PFOS	µg/L	9,12	8,58	10,40	7,14	288	4880	114	141	61,20	73	362
PFOA	µg/L	0,99	0,72	0,50	0,85	290	1100	1420	1460	1350	1290	1051
PFHS	µg/L	0,59	0,44	0,41	0,41	173	744	2040	2350	2180	1680	1375
PFOSA	µg/L	0,12	0,06	0,14	0,08	< 6,59	19,10	1,24	1,05	0,84	0,60	< 3,03
PFBS	µg/L						28,60					3,07

Analyseresultaten PFAS

Puntlocatie		P118B	P118B	P118B	P118B	P118B	P118C	P118C	P118C	P118C	P118C	P118C	P119A
Top filter (m)	13	13	13	13	13	2,5	2,5	2,5	2,5	2,5	2,5	2,5	23,5
Bodem filter (m)	14	14	14	14	14	4,5	4,5	4,5	4,5	4,5	4,5	3,5	24,5
Datum bemonstering	11/07/2018	31/01/2019	30/07/2019	28/01/2020	4/08/2020	1/02/2018	11/07/2018	31/01/2019	30/07/2019	28/01/2020	4/08/2020	4/08/2020	30/01/2018
Eenheid													
Grondwaterstand	m-mv	3,27	3,16	2,78	2,55	2,67	2,28	3,01	3,10	9,19	8,83	2,81	3,60
Redox	mV	-88,90					-130,80						
pH		7,13	7,41	7,31	7,42	6,78	7,03	7,06	7,17	7,29	7,13	6,64	6,99
EC	µS/cm	1525	1700	1390	1530	1610	2460	1769	2140	2120	2800	2780	1219
Temperatuur	°C	14,61	9,20	12,60	10,60	13,70	8	15,42	5,50	15,70	9,80	14,30	11,20
PFOS	µg/L	12000	14200	7310	6360	5855	2770	2980	1760	1690	1430	938	53,30
PFOA	µg/L	2100	1980	1620	1570	1708	579	214	189	166	236	182	127
PFHS	µg/L	3400	2340	756	711	929	249	87,20	84,10	84	122	65,20	37,70
PFOSA	µg/L	7,60	6,81	4,67	2,50	< 11,1	48,80	51,30	50,80	73,10	50,20	35,80	3,75
PFBS	µg/L												
Puntlocatie	P119A	P119A	P119A	P119A	P119A	P119B	P119B	P119B	P119B	P119B	P119B	P119C	
Top filter (m)	23,5	23,5	23,5	23,5	23,5	13	13	13	13	13	13	2,5	
Bodem filter (m)	24,5	24,5	24,5	24,5	24,5	14	14	14	14	14	14	4,5	
Datum bemonstering	11/07/2018	30/01/2019	30/07/2019	28/01/2020	4/08/2020	30/01/2018	11/07/2018	30/01/2019	30/07/2019	28/01/2020	4/08/2020	4/08/2020	30/01/2018
Eenheid													
Grondwaterstand	m-mv	4,01	3,91	3,54	3,75	3,41	3,64	0,04	3,91	3,58	3,63	3,42	7,02
Redox	mV	-95,40					-109,30						
pH		7,06	9,28	7,35	6,83	7,94	6,94	6,95	7,14	7,21	7,27	7,94	7,51
EC	µS/cm	1251	1300	1530	821	833	1826	1639	1520	1550	1330	807	702
Temperatuur	°C	12,78	10,20	12,50	11,10	13,60	10,90	9,50	13,10	10,90	14,30	9,60	
PFOS	µg/L	19,90	5,96	1,16	128	5,29	147	176	303	384	216	81	6830
PFOA	µg/L	62,20	35,20	12,60	96,10	34,50	1580	877	1420	2600	675	354	1050
PFHS	µg/L	18,60	10,70	4,71	30,90	8,67	948	424	672	1460	259	119	652
PFOSA	µg/L	2,19	0,92	0,41	7,15	< 1,01	0,56	2,08	0,53	0,46	0,21	< 2,02	70,00
PFBS	µg/L												
Puntlocatie	P119C	P119C	P119C	P119C	P119C	P121							
Top filter (m)	2,5	2,5	2,5	2,5	2,5	23,5	23,5	23,5	23,5	23,5	23,5	23,5	
Bodem filter (m)	4,5	4,5	4,5	4,5	4,5	24,5	24,5	24,5	24,5	24,5	24,5	24,5	
Datum bemonstering	11/07/2018	30/01/2019	30/07/2019	28/01/2020	4/08/2020	7/11/2017	31/01/2018	4/05/2018	10/07/2018	1/02/2019	24/04/2019	24/04/2019	30/07/2019
Eenheid													
Grondwaterstand	m-mv	2,22	2,02	2,41	2,05	2,04	4,78	4,38	4,49	4,58	4,61	4,93	4,87
Redox	mV	-159,40					-82,80		-150,30		-80		-82,30
pH		7,34	7,79	7,67	7,71	8,31	6,55	6,83	6,81	6,87	7,62	6,90	6,97
EC	µS/cm	398	642	654	673	525	2340	3020	3713	2950	2944	2450	657
Temperatuur	°C	13,19	8	13,20	10,40	14,90	12,30	11,60	13,78	12,70	11,40	13,70	14,40
PFOS	µg/L	3970	10500	8010	5970	1315	82	1,06	2,40	1,01	0,46	0,75	2,24
PFOA	µg/L	237	1760	1340	793	825	6,69	0,27	0,49	0,31	0,53	2,68	0,85
PFHS	µg/L	107	657	488	231	271	3,45	0,10	0,20	0,15	0,30	1,83	1,08
PFOSA	µg/L	68,80	130	95,50	81,70	71,40	2,18	0,07	0,13	0,08	0,07	0,10	0,33
PFBS	µg/L												
Puntlocatie	P121	P121	P121	P21B									
Top filter (m)	23,5	23,5	23,5	3,5	3,5	3,5	3,5	3,5	3,5	3,5	3,5	3,5	
Bodem filter (m)	24,5	24,5	24,5	24,5	5,5	5,5	5,5	5,5	5,5	5,5	5,5	5,5	
Datum bemonstering	29/01/2020	29/04/2020	4/08/2020	6/11/2017	29/01/2018	3/05/2018	12/07/2018	24/10/2018	30/01/2019	24/04/2019	30/07/2019	21/10/2019	
Eenheid													
Grondwaterstand	m-mv	3,47	4,62	4,11		1,05			1,42	1,23			1,52
Redox	mV		-126,30										
pH		7,18	6,68	7,41									
EC	µS/cm	2070	1215	1880									
Temperatuur	°C	11,10	11,28	15,30									
PFOS	µg/L	0,40	6,96	3,46	67600	35000	62200	62700	56500	65500	60600	71800	66900
PFOA	µg/L	0,80	1,36	1,46	9410	5200	10200	9970	9070	9540	9340	9190	6275
PFHS	µg/L	0,50	2,05	1,54	10500	6100	11900	12000	11300	12200	11900	13200	8610
PFOSA	µg/L	0	< 0,0990	< 0,187	3,99	2,86	7,07	6,42	5,03	3,80	6,56	14,40	5,67
PFBS	µg/L				6680	4270	7860	7830	7130	7910	7730	7930	5195

Analyseresultaten PFAS

Puntlocatie		P21B	P21B	P21B	P262	P262	P262BIS	P262BIS	P262BIS	P263	P263	P263	P263
Top filter (m)	3,5	3,5	3,5	6,2	6,2	6,5	6,5	6,5	7,4	7,4	7,4	7,4	7,4
Bodem filter (m)	5,5	5,5	5,5	7,2	7,2	7,5	7,5	7,5	8,4	8,4	8,4	8,4	8,4
Datum bemonstering	29/01/2020	28/04/2020	4/08/2020	1/02/2018	12/07/2018	30/07/2019	29/01/2020	4/08/2020	7/11/2017	30/01/2018	12/07/2018	31/01/2019	31/01/2019
Eenheid													
Grondwaterstand	m-mv	1,53		1,51	4,10	4,18	4,65	5	5,01	5,12	4,87	5,38	5,27
Redox	mV							-119,90	-154		-30,70		-149
pH					7,21	7,31	7,35	7,08	7,23	7,51	6,49	7,54	7,98
EC	µS/cm				232	766	1250	1354	684	1714	582	1491	1620
Temperatuur	°C				9,80	13	13,40	11,65	14,33	11,60	11,56	13,80	11,40
PFOS	µg/L	68900	68300	47591	217	5830	5470	4720	3546	7460	8630	9220	9020
PFOA	µg/L	8740	9110	8083	27,60	1470	532	572	642	1310	1380	1430	1190
PFHS	µg/L	10900	12000	8663	5,95	837	325	317	311	442	482	467	392
PFOSA	µg/L	15,70	6,22	< 40,5	109	64,50	8,95	8,11	9,79	23,30	10,10	22,20	9,09
PFBS	µg/L	6610	7280	6428,69									
Puntlocatie	P263	P263	P263	P264	P264	P264	P264	P264	P264	P265B	P265B	P265B	P265B
Top filter (m)	7,4	7,4	7,4	5	5	5	5	5	5	2,8	2,8	2,8	2,8
Bodem filter (m)	8,4	8,4	8,4	8,4	5,5	5,5	5,5	5,5	5,5	3,8	3,8	3,8	3,8
Datum bemonstering	30/07/2019	29/01/2020	4/08/2020	1/02/2018	11/07/2018	31/01/2019	30/07/2019	28/01/2020	4/08/2020	1/02/2018	31/01/2019	30/07/2019	30/07/2019
Eenheid													
Grondwaterstand	m-mv	5,54	5,46	5,45	2,43	3,10	2,98	2,84	2,67	2,74	2,21	2,62	2,32
Redox	mV		-131,80	-149,20		-163,40							
pH		7,67	7,27	7,45	7,29	7,28	7,55	7,39	7,46	6,82	7,37	7,45	7,67
EC	µS/cm	1400	1454	741	802	591	744	840	719	822	498	1400	1120
Temperatuur	°C	13,50	11,55	14,29	9,30	13,66	7,10	15,60	8,90	14,50	8,50	8,50	14,50
PFOS	µg/L	7280	6880	5350	1500	1490	1500	1350	1320	1012	703	582	454
PFOA	µg/L	927	853	706	298	259	336	392	407	200	73,10	125	110
PFHS	µg/L	342	267	210	194	153	221	269	261	82,70	26,80	44,20	43,10
PFOSA	µg/L	13,40	15	< 11,1	50,60	75,20	68,30	56,30	56,90	73,90	60,10	80,40	138
PFBS	µg/L												
Puntlocatie	P265B	P27	P27	P27	P27	P27	P27	P304	P304	P304	P304	P304	P304
Top filter (m)	2,8	1,8	1,8	1,9	1,8	1,9	1,8	4	4	4	4	4	4
Bodem filter (m)	3,8	3,8	3,8	2,9	3,8	2,9	3,8	6	6	6	6	6	6
Datum bemonstering	28/01/2020	30/01/2018	11/07/2018	30/01/2019	30/07/2019	29/01/2020	4/08/2020	29/01/2018	11/07/2018	31/01/2019	30/07/2019	29/01/2020	29/01/2020
Eenheid													
Grondwaterstand	m-mv	2,01	1,41	1,63	1,51	1,85	8,02	1,32	2,16	2,59	2,49	2,83	2,91
Redox	mV				-52,80	-52,50					-142	-145,60	
pH		7,32	7,43	7,21	8,09	7,27	7,63	7,93	7,15	7,14	7,73	7,05	7,23
EC	µS/cm	1780	582	1032	943	485	836	801	961	1030	965	624	775
Temperatuur	°C	9	8,40	20,40	8,70	19,25	8,80	18,80	14,10	16,50	12,30	17,45	13
PFOS	µg/L	547	708	1900	1710	1680	1410	1793	1310	1090	1080	1190	1020
PFOA	µg/L	80,70	166	231	241	189	194	174	520	413	402	467	391
PFHS	µg/L	27,50	18,60	85,40	42,10	35	14,10	39,90	169	130	120	150	115
PFOSA	µg/L	61,10	131	96,20	37,80	74,90	58,80	93,10	18,20	19,80	14,80	11	13,60
PFBS	µg/L		748	195	15000	4560	1490	1659,96	347	250	260	298	305
Puntlocatie	P304	P305	P305	P305	P305	P305	P305	P321	P321	P321	P321	P321	P321
Top filter (m)	4	3,5	3,5	3,5	3,5	3,5	3,5	15,5	15,5	15,5	15,5	15,5	15,5
Bodem filter (m)	6	5,5	5,5	5,5	5,5	5,5	5,5	16,5	16,5	16,5	16,5	16,5	16,5
Datum bemonstering	4/08/2020	29/01/2018	12/07/2018	30/01/2019	30/07/2019	28/01/2020	4/08/2020	7/11/2017	1/02/2018	3/05/2018	9/07/2018	24/10/2018	
Eenheid													
Grondwaterstand	m-mv	2,69	1,70	1,96	1,26	2,08	2,05	2,06	4,32	3,51	3,65	4,10	4,42
Redox	mV				55	-98,50	-93,50		-120,20		-108,30		-97,90
pH		7,79	7,07	7,07	7,48	6,94	7,03	7,66	7,10	7,15	7,12	7,21	6,86
EC	µS/cm	643	793	824	742	570	1810	520	3128	1581	1259	1482	1028
Temperatuur	°C	17,90	12,90	16,50	11,90	18	12,06	19	13,44	12,10	13,49	15	14,52
PFOS	µg/L	978	429	485	487	459	308	353	6160	8100	5310	4820	6020
PFOA	µg/L	398	144	177	145	168	101	137	2550	2730	2450	2370	2970
PFHS	µg/L	109	194	251	184	238	111	140	644	713	648	671	764
PFOSA	µg/L	19,90	18,10	18,80	10	9,80	20,70	23,30	0,18	0,61	0,36	1,99	0,20
PFBS	µg/L	374,63	107	155	132	230	172	335,18					

Analyseresultaten PFAS

Puntlocatie	P321	P340	P340	P340	P340	P340						
Top filter (m)	15,5	15,5	15,5	15,5	15,5	15,5	15,5	4	4	4	4	4
Bodem filter (m)	16,5	16,5	16,5	16,5	16,5	16,5	16,5	6	6	6	6	6
Datum bemonstering	30/01/2019	24/04/2019	30/07/2019	22/10/2019	29/01/2020	28/04/2020	4/08/2020	30/01/2018	9/07/2018	30/01/2019	30/07/2019	28/01/2020
Eenheid												
Grondwaterstand	m-mv	4,15	4,01	3,82		4,07	3,83	3,66	1,40	2,84	2,27	2,49
Redox	mV		-190,40		4,29	75,30	122					-36,20
pH		7,32	7,42	7,35	7,41	7,17	6,95	7	7,11	7,03	7,01	7,26
EC	µS/cm	1390	1390	1330	825	1660	903	1130	1018	1062	1930	1400
Temperatuur	°C	11,10	13,70	13,80	14,52	11,40	12,90	15,40	11,40	17,40	10,70	16,20
PFOS	µg/L	5900	5220	4700	2960	4490	5700	2931	2640	2480	2280	2560
PFOA	µg/L	2460	1930	2220	586	1430	2240	928	386	482	508	398
PFHS	µg/L	550	448	600	188	360	535	219	202	264	263	233
PFOSA	µg/L	0,26	0,21	0,43	14,60	0,24	0,67	6,47	37,70	52,30	22,40	53,40
PFBS	µg/L											95,90
Puntlocatie	P340	P341	P341	P341	P341	P341	P341	P343	P343	P343	P371	P371
Top filter (m)	4	3,5	3,5	3,5	3,5	3,5	3,5	4,8	4,8	4,8	1,5	1,5
Bodem filter (m)	6	5,5	5,5	5,5	5,5	5,5	5,5	6,8	6,8	6,8	3,5	3,5
Datum bemonstering	4/08/2020	9/07/2018	15/10/2018	30/01/2019	30/07/2019	29/01/2020	4/08/2020	30/01/2018	12/07/2018	31/01/2019	30/01/2018	12/07/2018
Eenheid												
Grondwaterstand	m-mv	2,55	2,81		2,76	2,94	2,84	2,83	3,21	3,70	3,75	2,01
Redox	mV					-52,70	-108,50	-108,50	-15		-140	39,30
pH		6,99	7,21		7,32	7,40	6,65	7,24	6,08	7,17	7,71	7,11
EC	µS/cm	830	1013		1080	886	1024	299	449	1227	1232	505
Temperatuur	°C	21,70	13		10,30	13,20	9,23	15,90	11,13	13	10,10	9,29
PFOS	µg/L	3822	5240	2758,60	5790	5430	2250	2311	7510	5740	6660	3100
PFOA	µg/L	168	651	457	795	702	224	287	835	516	637	208
PFHS	µg/L	54,30	223	162	248	216	123	186	735	366	457	97,90
PFOSA	µg/L	138	84,50	18,30	50,40	55	80,40	114	10,50	13,80	11,60	32,40
PFBS	µg/L			21,50								23,60
Puntlocatie	P371	P371	P371	P371	P372	P372	P372	P372	P372	P372	P374	P374
Top filter (m)	1,5	1,5	1,5	1,5	4,8	4,8	4,8	4,8	4,8	4,8	5	5
Bodem filter (m)	3,5	3,5	3,5	3,5	5,8	5,8	5,8	5,8	5,8	5,8	6	6
Datum bemonstering	30/01/2019	30/07/2019	28/01/2020	4/08/2020	30/01/2018	12/07/2018	30/01/2019	30/07/2019	28/01/2020	4/08/2020	30/01/2018	12/07/2018
Eenheid												
Grondwaterstand	m-mv	2,79	2,75	2,54	2,70	2,08	2,70	2,72	2,82	2,57	2,14	2,70
Redox	mV	38,20		29,80	-87,30	-113	-127,20	-128			-60,80	-95,90
pH		7,21	7,08	6,97	7,10	6,98	7,22	7,16	7,32	7,32	6,10	6,87
EC	µS/cm	992	1520	2175	469	393	347	375	530	450	656	1295
Temperatuur	°C	6,73	19,20	8,64	18,63	11,33	13,33	8,62	17	10,20	13,70	11,05
PFOS	µg/L	1720	3430	2500	2011	1350	1240	1220	1080	1140	1470	1970
PFOA	µg/L	605	614	855	237	70,60	51,80	52,10	85,20	86	218	701
PFHS	µg/L	489	397	1060	189	54,80	42,10	46,90	75,50	61,60	156	865
PFOSA	µg/L	10,30	18,40	32,20	33	37,70	50,90	26,50	40,10	27,10	19,90	28,30
PFBS	µg/L											12,70
Puntlocatie	P374	P374	P374	P374	P378	P378	P379	P379	P379	P379	P379	P380
Top filter (m)	5	5	5	5	3,5	3,5	3,5	3,5	3,5	3,5	3,5	3,5
Bodem filter (m)	6	6	6	6	6	5,5	5,5	5,5	5,5	5,5	5,5	5,5
Datum bemonstering	31/01/2019	30/07/2019	28/01/2020	4/08/2020	31/01/2018	11/07/2018	12/07/2018	31/01/2019	30/07/2019	28/01/2020	8/05/2020	12/07/2018
Eenheid												
Grondwaterstand	m-mv	3,17	3,33	3,01	2,68	1,90	2,58	2,52	2,52	2,70	2,39	2,59
Redox	mV					-84,40	-124,20				-104,50	-121,90
pH		7,03	6,91	6,85	6,43	6,73	6,42	7,28	7,65	7,26	7,36	7,49
EC	µS/cm	751	1160	884	1500	815	826	645	513	630	742	280
Temperatuur	°C	7,20	15	10,40	13,80	10,51	16,91	13,40	8,40	17,10	10,86	17,66
PFOS	µg/L	1460	1260	993	1217	1040	727	666	644	749	500	328
PFOA	µg/L	202	225	122	246	241	207	59,50	48,40	68	47,40	49,40
PFHS	µg/L	254	295	137	240	275	238	19,30	15,80	19,80	10,80	13,90
PFOSA	µg/L	48,60	42,40	58,40	29,80	1,70	1,47	60,60	66,90	59,40	68,10	73,50
PFBS	µg/L											97,30

Analyseresultaten PFAS

Puntlocatie		P380	P380	P380	P380	P381	P381	P382	P382	P382	P42	P42
Top filter (m)	3,5	3,5	3,5	3,5	5	5	2,5	2,5	2,5	3	3	
Bodem filter (m)	5,5	5,5	5,5	5,5	6	6	3,5	3,5	3,5	5	5	
Datum bemonstering	31/01/2019	30/07/2019	28/01/2020	4/08/2020	12/07/2018	30/07/2019	4/08/2020	11/07/2018	30/07/2019	4/08/2020	29/01/2018	11/07/2018
Eenheid												
Grondwaterstand	m-mv	2,58	2,90	2,48	2,56		3,32	2,84	0,03	3,29	2,94	2,48
Redox	mV								-121,40			
pH		7,08	7,13	6,93	6,50		7,01	6,29	7	7,22	6,33	7,12
EC	µS/cm	767	983	1350	1560		840	1410	938	1340	1410	917
Temperatuur	°C	9	15,70	9,50	13,40		16,30	17,30	14,46	16,60	17,60	11,80
PFOS	µg/L	2220	1930	2110	1360	1200	1110	787	781	939	677	6280
PFOA	µg/L	553	553	1040	898	535	508	492	470	663	307	400
PFHS	µg/L	495	566	1060	883	163	164	124	141	221	83,60	315
PFOSA	µg/L	37,60	37	31,90	27,80	9,51	11,60	6,04	73,40	101	45,80	15,40
PFBS	µg/L										36,40	39,60
Puntlocatie	P42	P42	P42	P42	P56	P56	PA109A	PA109A	PA111A	PA111A	PA112	PA112
Top filter (m)	3	3	3	3	1,2	1,2	5	5	4	4	3,5	3,5
Bodem filter (m)	5	5	5	5	3,2	3,2	6	6	6	6	5,5	5,5
Datum bemonstering	30/01/2019	30/07/2019	29/01/2020	4/08/2020	11/07/2018	15/10/2018	31/01/2018	11/07/2018	31/01/2018	11/07/2018	1/11/2017	31/01/2018
Eenheid												
Grondwaterstand	m-mv	2,95	3,42	2,67	2,48				2,79	4,03	2,18	3,82
Redox	mV	164	-89						-123,40	-173	-26,40	-41,60
pH		6,92	6,68	7,24	7,46				6,91	6,96	6,61	6,20
EC	µS/cm	1368	987	767	695				322	509	1119	1342
Temperatuur	°C	10,70	20,10	10,50	19				10,48	16,13	10,38	20,20
PFOS	µg/L	3690	2350	2770	1500	198	115	223	157		2070	10,99
PFOA	µg/L	276	231	212	216	24,20	52,50	114	40,20	753	742	506
PFHS	µg/L	268	365	312	280	14,20	20,40	114	42,40	1000	997	121
PFOSA	µg/L	12,10	11,60	16	14,30	32,90	5,54	1,50	2,35	9,11	10,20	46,80
PFBS	µg/L	32	44,20	30,50		7,59	12,30					
Puntlocatie	PA112	PB402	PP01	PP01	PP01	PP01	PP02	PP02	PP02	PP02	PP02	PP02
Top filter (m)	3,45	2,3	2,6	2,6	2,6	2,6	3,2	3,2	3,2	3,2	3,2	3,2
Bodem filter (m)	5,45	3,3	5,6	5,6	5,6	5,6	6,2	6,2	6,2	6,2	6,2	6,2
Datum bemonstering	11/07/2018	15/10/2018	6/11/2017	30/01/2018	3/05/2018	10/07/2018	15/10/2018	6/11/2017	30/01/2018	3/05/2018	10/07/2018	25/10/2018
Eenheid												
Grondwaterstand	m-mv	3,77		3,82	3,82	0			3,70	3,70	0	
Redox	mV	-174,10		-142,90	-116	-65,10	-26,60		-88,70	-66,30	-98,20	-24,70
pH		7,33		6,84	8,94	7,82	7,93		7,87	8,10	7,61	7,42
EC	µS/cm	595		603	685	604	881		999	502	1044	1327
Temperatuur	°C	15,87		11,74	10,20	13,70	17,77		12,86	8,92	13,72	19,24
PFOS	µg/L	350	89,50	2070	2310	2100	1790	4718	27200	24500	17100	19600
PFOA	µg/L	135	39,70	52,90	60,60	57,90	51,90	48,80	396	567	557	641
PFHS	µg/L	31,80	33,50	10600	8040	7720	8300	17285	4510	3930	2830	3630
PFOSA	µg/L	98,40	8,41	27,10	36,30	28,30	56,20	15,00	37,20	25,90	31,60	28
PFBS	µg/L		7,93	75,10	78,90	94,60	59,20	108	32,30	45,70	32,60	53,20
Puntlocatie	PP02	PP02	PP02	PP02	PP02	PP02	PP04	PP04	PP04	PP04	PP04	PP04
Top filter (m)	3,2	3,2	3,2	3,2	3,2	3,2	2	2	2	2	2	2
Bodem filter (m)	6,2	6,2	6,2	6,2	6,2	6,2	6	6	6	6	6	6
Datum bemonstering	30/01/2019	24/04/2019	30/07/2019	21/10/2019	27/01/2020	29/04/2020	4/08/2020	6/11/2017	30/01/2018	3/05/2018	10/07/2018	25/10/2018
Eenheid												
Grondwaterstand	m-mv								1,79	4	0	
Redox	mV	9,60	200	105	60,50	-55,70	-33	140	-82,60	-57,60	-70,20	-28
pH		7,39	6,98	7,06	7,15	6,96	7,41	7,32	6,53	7,72	7,55	7,65
EC	µS/cm	1030	1326	1199	1380	2604	944	1118	689	408	734	801
Temperatuur	°C	6,50	19,90	24,50	14,89	11,89	16,77	19,95	11,92	11,87	13,60	16,94
PFOS	µg/L	21300	21500	17300	17800	21900	20100	18391	6130	6640	5590	5570
PFOA	µg/L	502	562	648	552	605	683	767	716	1390	645	423
PFHS	µg/L	4000	4080	3770	4790	4740	3870	3442	552	285	227	298
PFOSA	µg/L	27,10	35,80	22,30	30,80	20,40	34,80	21,70	56,20	30,30	60,40	64,90
PFBS	µg/L	30,10	38,10	45,20	17,80	27,30	39,60	33,30	2840	2770	2040	1450

Analyseresultaten PFAS

Puntlocatie		PP04	PP04	PP04	PP04	PP04	PP04	PP05	PP05	PP05	PP05	PP05
Top filter (m)	2	2	2	2	2	2	1,76	1,76	1,76	1,76	1,76	1,76
Bodem filter (m)	6	6	6	6	6	6	5,76	5,76	5,76	5,76	5,76	5,76
Datum bemonstering	30/01/2019	24/04/2019	30/07/2019	21/10/2019	27/01/2020	29/04/2020	4/08/2020	6/11/2017	30/01/2018	3/05/2018	10/07/2018	25/10/2018
Eenheid												
Grondwaterstand	m-mv											
Redox	mV	7,60	84,30	-30,10	37,50	108	-32,20	121				
pH		7,36	7,11	7,58	7,89	7,07	7,45	7,65				
EC	µS/cm	618	738	493	812	1308	564	753				
Temperatuur	°C	7,90	16,45	18,83	15,21	11,79	14,64	20,60				
PFOS	µg/L	5430	5070	3950	2550	4260	5110	2559	776	653	229	167
PFOA	µg/L	682	680	234	1150	416	354	242	2260	1920	688	595
PFHS	µg/L	291	272	107	399	163	148	62,90	5470	4830	2360	3300
PFOSA	µg/L	58,10	58,40	73,60	110	44,50	65,10	73,30	39,80	98,70	76,10	17,20
PFBS	µg/L	1270	1080	383	989	804	707	562	20500	14900	4900	5960
Puntlocatie		PP05	PP05	PP05	PP05	PP05	PP05	PP06	PP06	PP06	PP06	PP06
Top filter (m)	1,76	1,76	1,76	1,76	1,76	1,76	1,76	1,3	1,3	1,3	1,3	1,3
Bodem filter (m)	5,76	5,76	5,76	5,76	5,76	5,76	5,76	5,3	5,3	5,3	5,3	5,3
Datum bemonstering	30/01/2019	24/04/2019	30/07/2019	21/10/2019	27/01/2020	29/04/2020	4/08/2020	6/11/2017	30/01/2018	3/05/2018	10/07/2018	25/10/2018
Eenheid												
Grondwaterstand	m-mv											
Redox	mV								2,92	1,58	0	
pH									-99,30	-101,60	-105,40	-74,70
EC	µS/cm								10,12	7,32	7,70	7,32
Temperatuur	°C								2985	655	391	745
PFOS	µg/L	427	456	87,70	110	181	93	93,20	1030	1920	1640	1290
PFOA	µg/L	476	855	2430	646	977	579	2937	99,90	190	294	269
PFHS	µg/L	2400	4350	9010	2660	2970	2820	5585	38,90	88,00	74,50	77,10
PFOSA	µg/L	39,30	102	18,90	9,20	17,10	17,60	14,20	70,70	58,20	226	59,80
PFBS	µg/L	3400	5850	15900	4575	3810	4170	7782	13,10	16,50	22,90	18,80
Puntlocatie		PP06	PP06	PP06	PP06	PP06	PP06	PP07	PP07	PP07	PP07	PP07
Top filter (m)	1,3	1,3	1,3	1,3	1,3	1,3	1,3	2	2	2	2	2
Bodem filter (m)	5,3	5,3	5,3	5,3	5,3	5,3	5,3	6	6	6	6	6
Datum bemonstering	30/01/2019	24/04/2019	30/07/2019	21/10/2019	27/01/2020	29/04/2020	4/08/2020	6/11/2017	30/01/2018	3/05/2018	10/07/2018	25/10/2018
Eenheid												
Grondwaterstand	m-mv											
Redox	mV	-14,60	86,20	5,20	28,40	118	-33,50	30,30	-140,80	-80,50	-95,90	-23,20
pH		7,23	7,04	7,20	7,42	7,19	7,25	7,45	6,47	7,34	7,94	7,26
EC	µS/cm	667	902	868	866	1671	712	595	691	449	885	1153
Temperatuur	°C	7,10	18,26	18,84	15,35	11,67	16,57	20,70	13,77	9,57	14,58	18,85
PFOS	µg/L	1690	1060	957	1650	982	1360	903	1660	2440	2920	2640
PFOA	µg/L	237	337	191	254	193	650	199	219	300	323	298
PFHS	µg/L	78	68	89,30	51,70	51,10	90,50	32,10	68,80	99,50	108	124
PFOSA	µg/L	62	87,40	52,70	65,40	50,60	79,50	61,50	68,90	68,70	104	74,20
PFBS	µg/L	21,50	35,70	25,60	46,30	20	14,80	17,30	75,50	143	215	208
Puntlocatie		PP07	PP07	PP07	PP07	PP07	PP07	PP08	PP08	PP08	PP08	PP08
Top filter (m)	2	2	2	2	2	2	3,2	3,2	3,2	3,2	3,2	3,2
Bodem filter (m)	6	6	6	6	6	6	6	7,2	7,2	7,2	7,2	7,2
Datum bemonstering	30/01/2019	24/04/2019	30/07/2019	21/10/2019	27/01/2020	28/04/2020	4/08/2020	6/11/2017	30/01/2018	3/05/2018	10/07/2018	25/10/2018
Eenheid												
Grondwaterstand	m-mv											
Redox	mV	-63,70	87,80	-68,70	34,40	-6,80	-91,70	44,40	-62	-69,10	-55,70	-19,70
pH		7,24	6,97	7,09	7,39	7,12	7,48	7,26	6,70	7,24	7,05	7,23
EC	µS/cm	894	1144	1592	986	3020	1238	1137	865	457	979	1047
Temperatuur	°C	6,50	19,87	22,70	15,59	12,13	17,04	19,25	12,85	10,19	13,50	19,48
PFOS	µg/L	2120	2280	1840	1390	2150	2310	1811	6870	5180	5520	5410
PFOA	µg/L	303	265	224	150	401	315	287	587	564	546	502
PFHS	µg/L	106	97,40	97,30	49,50	136	125	97,80	297	301	266	651
PFOSA	µg/L	58,60	79,60	59,10	64,40	63,10	94,40	107	51	34,00	51,60	64,90
PFBS	µg/L	177	251	656	79,30	396	300	289	42,20	23,40	43,50	89

Analyseresultaten PFAS

Puntlocatie	PP08	PP09	PP09	PP09	PP09	PP09						
Top filter (m)	3,2	3,2	3,2	3,2	3,2	3,2	3,2	3	3	3	3	3
Bodem filter (m)	7,2	7,2	7,2	7,2	7,2	7,2	7,2	6	6	6	6	6
Datum bemonstering	30/01/2019	24/04/2019	30/07/2019	21/10/2019	27/01/2020	28/04/2020	4/08/2020	6/11/2017	30/01/2018	3/05/2018	10/07/2018	25/10/2018
Eenheid												
Grondwaterstand	m-mv											
Redox	mV	-64,10	34,10	-62	-41	4	-14,90	53,60	-20,60	-48	22,70	-17,90
pH		7,24	6,84	7,28	8,89	7,03	7,18	7,15	7,10	7,37	6,76	7,30
EC	µS/cm	1015	1083	1056	1237	2029	237	1030	887	472	863	947
Temperatuur	°C	6,20	18,34	15,53	14,54	11,88	17,09	19,73	17,30	9,80	20	18,90
PFOS	µg/L	5920	5260	4820	4870	5450	6320	4837	2320	427	3120	2690
PFOA	µg/L	569	416	533	532	543	519	551	632	718	475	658
PFHS	µg/L	262	173	275	216	270	258	265	314	28,20	319	336
PFOSA	µg/L	38,10	46,60	64,00	58	38,70	89,70	51,60	15,20	2430	20,90	10
PFBS	µg/L	29,70	20,40	30,30	21,40	30,50	31	35,30	19,40	12,80	18	23,70
<hr/>												
Puntlocatie	PP09	PP09	PP09	PP10								
Top filter (m)	3	3	3	3	3	3	3	3	3	3	3	3
Bodem filter (m)	6	6	6	6	6	6	6	6	6	6	6	6
Datum bemonstering	30/01/2019	24/04/2019	25/04/2019	6/11/2017	30/01/2018	3/05/2018	10/07/2018	25/10/2018	30/01/2019	24/04/2019	30/07/2019	21/10/2019
Eenheid												
Grondwaterstand	m-mv				2,70	2,56	0					
Redox	mV	-43,20			-80,50	-52,40	-64,20	-11,50	-63,80	-33,20	83,50	-116,50
pH		7,31	82,50		6,65	7,09	7,75	7,23	7,10	7,27	7,11	7,31
EC	µS/cm	688	7,03		691	468	842	963	708	706	765	719
Temperatuur	°C	6,41	864		13,91	9,81	13,64	19,45	17,09	6,45	20,40	26,60
PFOS	µg/L	2360	20,10	2087,64	3260	4790	3410	3360	3750	3670	3430	3410
PFOA	µg/L	590	596	511,95	536	543	555	539	568	548	489	466
PFHS	µg/L	303	335	304,36	194	302	205	212	225	202	186	195
PFOSA	µg/L	14,40	13,60	10,19	16,30	34,00	22,30	16,20	15,60	17,40	24	23,10
PFBS	µg/L	18,30	20,80	21,05	15,80	37,80	16	19,10	16	15,80	14,60	14,70
<hr/>												
Puntlocatie	PP10	PP10	PP10	PP11								
Top filter (m)	3	3	3	3	3	3	3	3	3	3	3	3
Bodem filter (m)	6	6	6	6	6	6	6	6	6	6	6	6
Datum bemonstering	27/01/2020	28/04/2020	4/08/2020	30/01/2019	24/04/2019	30/07/2019	21/10/2019	27/01/2020	29/04/2020	4/08/2020		
Eenheid												
Grondwaterstand	m-mv											
Redox	mV	15,30	-23,40	44,70	28,60	65	-42	39,30	119	-47,90	47,50	
pH		7,13	7,25	7,31	7,59	7,12	7,58	7,44	7,21	7,47	7,34	
EC	µS/cm	1446	675	796	591	560	655	722	1391	608	697	
Temperatuur	°C	12,45	17,05	19,92	8,06	20,70	19,44	16,02	11,96	16,78	20,30	
PFOS	µg/L	3250	3400	2959	1270	672	2470	1730	1450	1790	1222	
PFOA	µg/L	504	477	533	167	104	324	314	497	263	254	
PFHS	µg/L	185	195	181	117	144	141	129	323	125	110	
PFOSA	µg/L	19,90	27,10	25,30	27,50	38,80	27,00	31,80	27,60	37,30	18,20	
PFBS	µg/L	12,40	10,30	21,70	96,10	49,40	172	139	447	197	167	

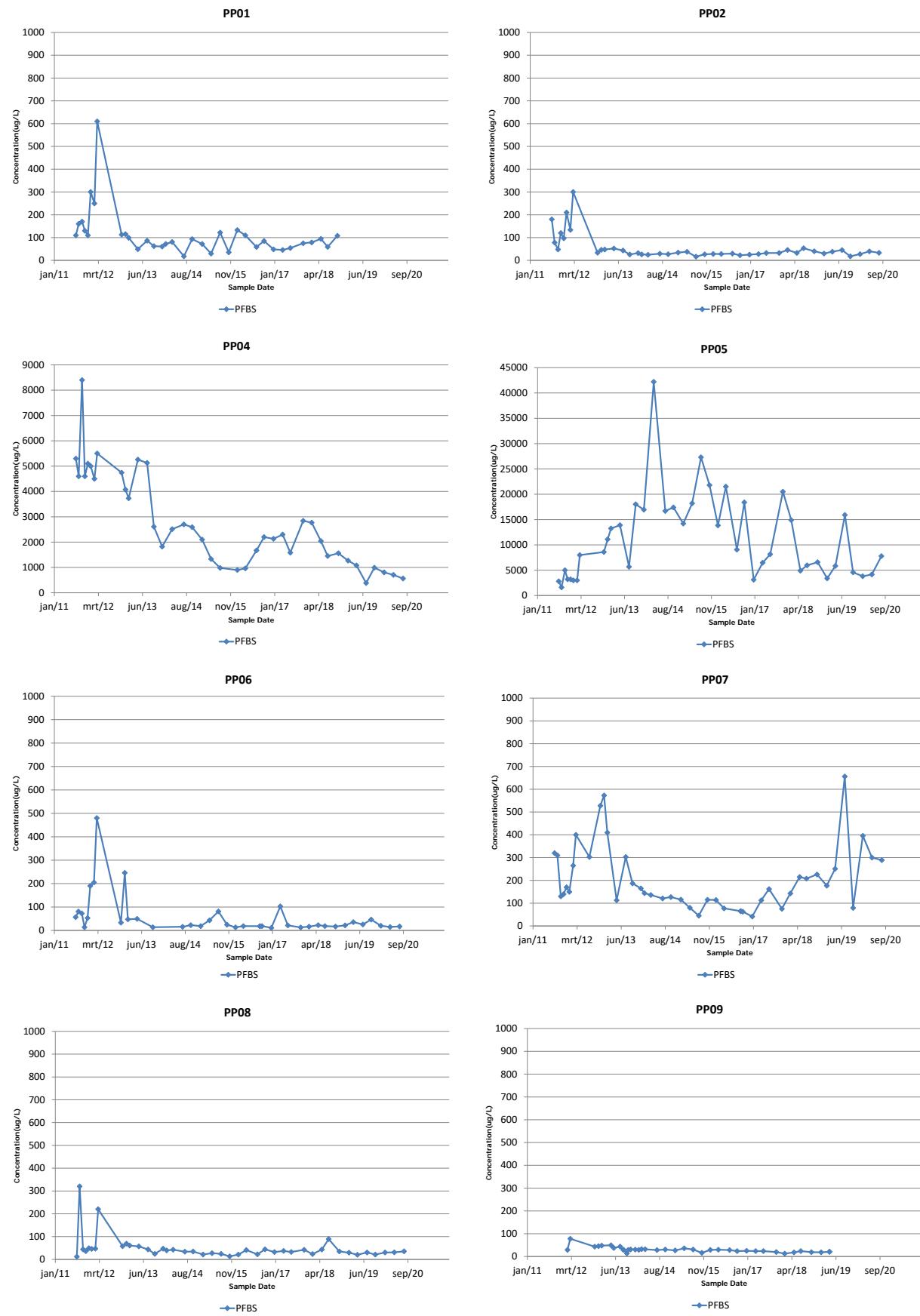
Analyseresultaten minerale olie

Puntlocatie	P18	P18	P18	P28
Top filter (m)	1,4	1,4	1,4	1,1
Bodem filter (m)	3,4	3,4	3,4	3,1
Datum bemonstering	29/07/2018	31/07/2019	4/08/2020	29/07/2018
Eenheid				
Grondwaterstand	m-mv	2,90		1,39
Redox	mV			2,21
pH		7,32		13,08
EC	µS/cm	863		36600
Temperatuur	°C	14,90		16,20
Minerale olie C10-C12	µg/L	81	140	420
minerale olie C12-C20	µg/L	680	1250	3100
Minerale olie C20-C30	µg/L	450	955	2300
Minerale olie C30-C40	µg/L	<25	85,5	430
Som minerale olie	µg/L	1200	2450	6300
				1400

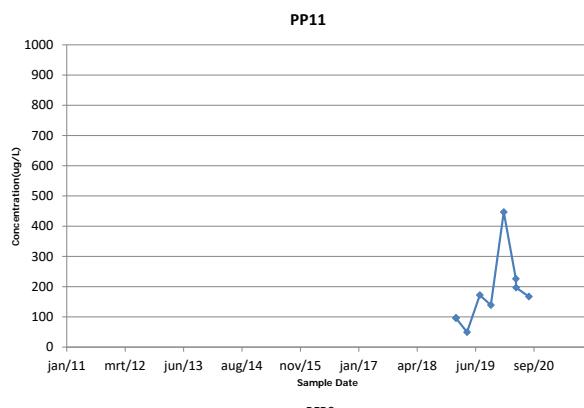
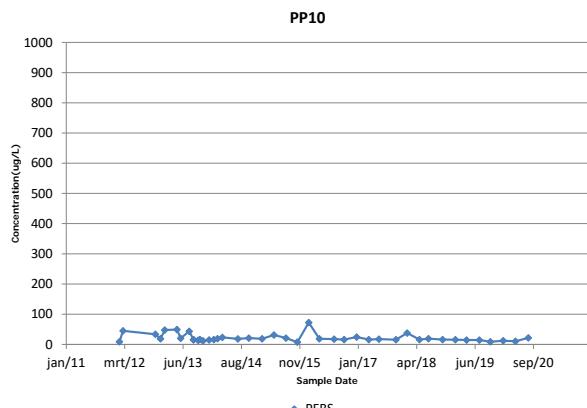
BIJLAGE 7

GRAFIEKEN

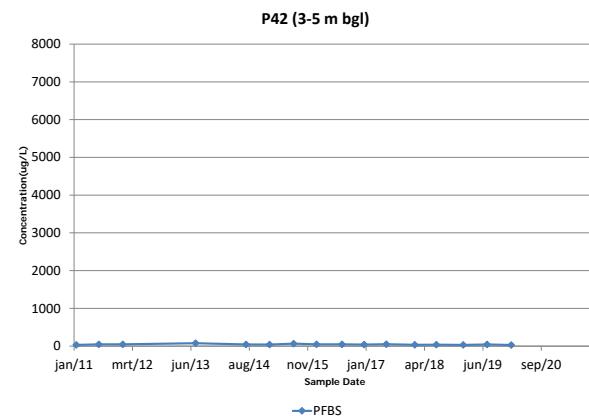
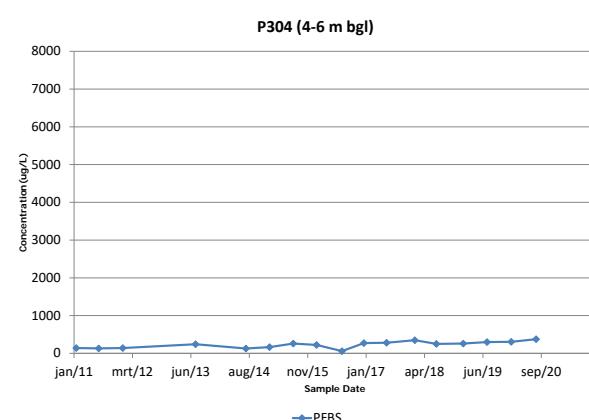
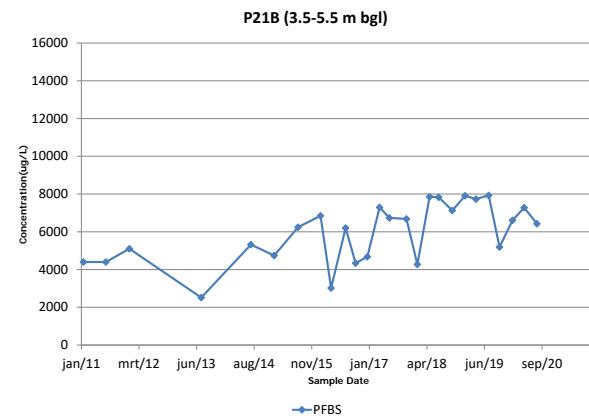
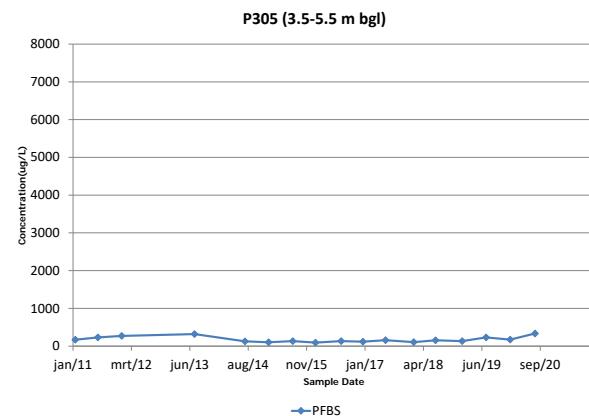
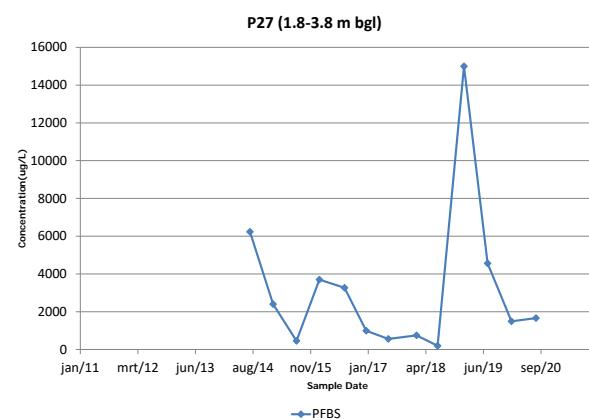
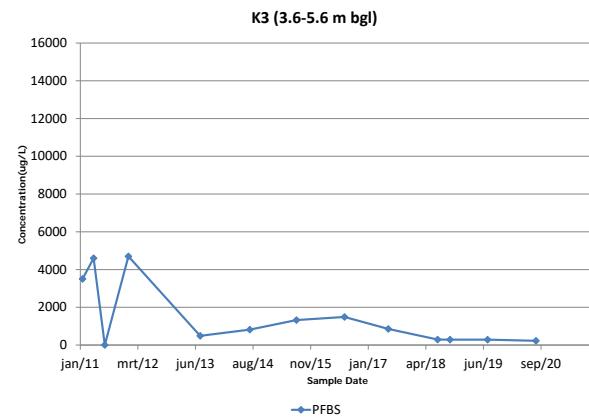
Pompputten



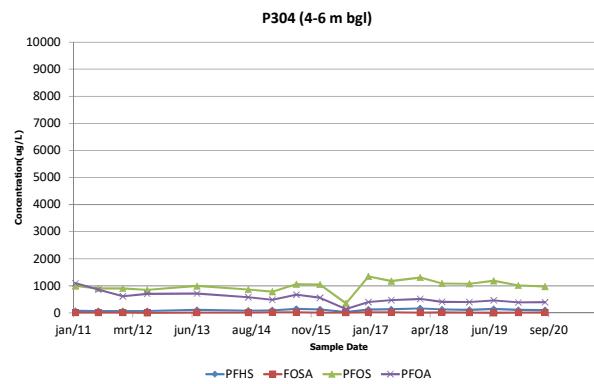
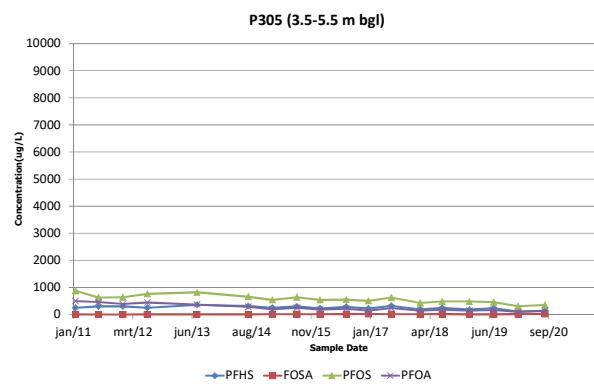
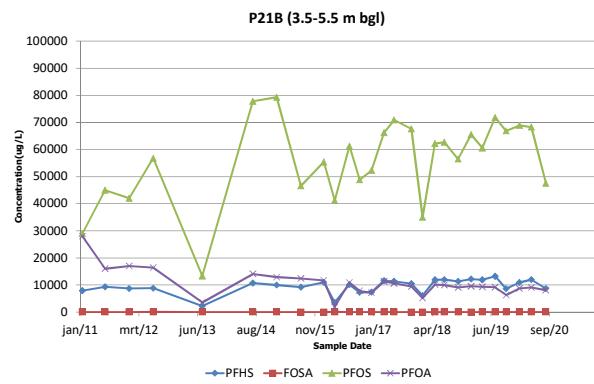
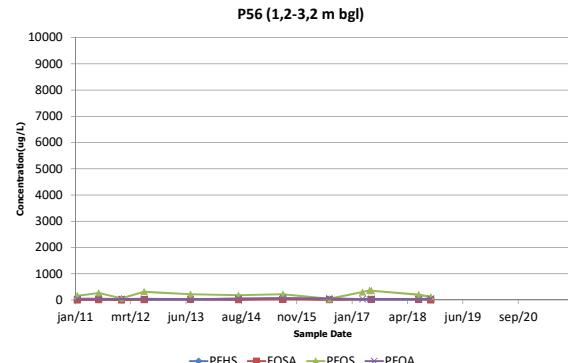
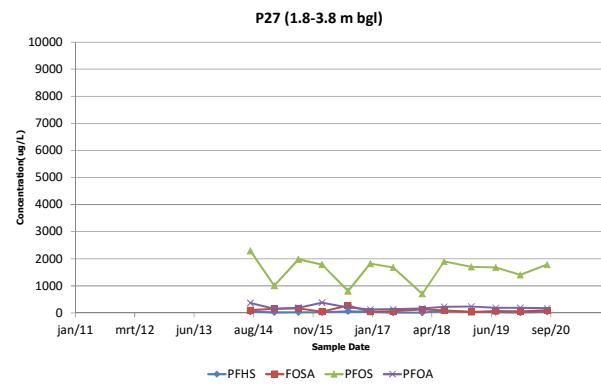
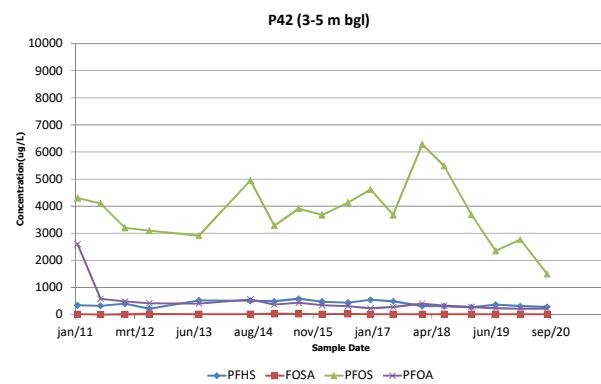
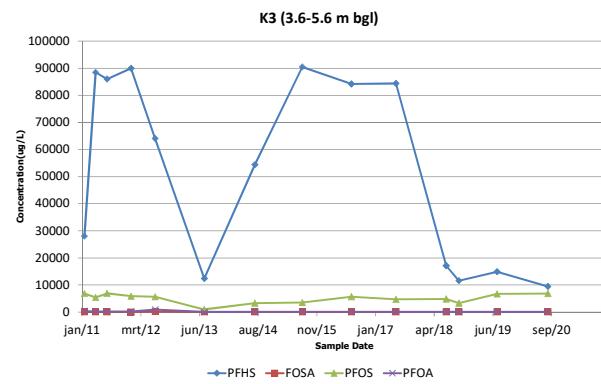
Pompputten



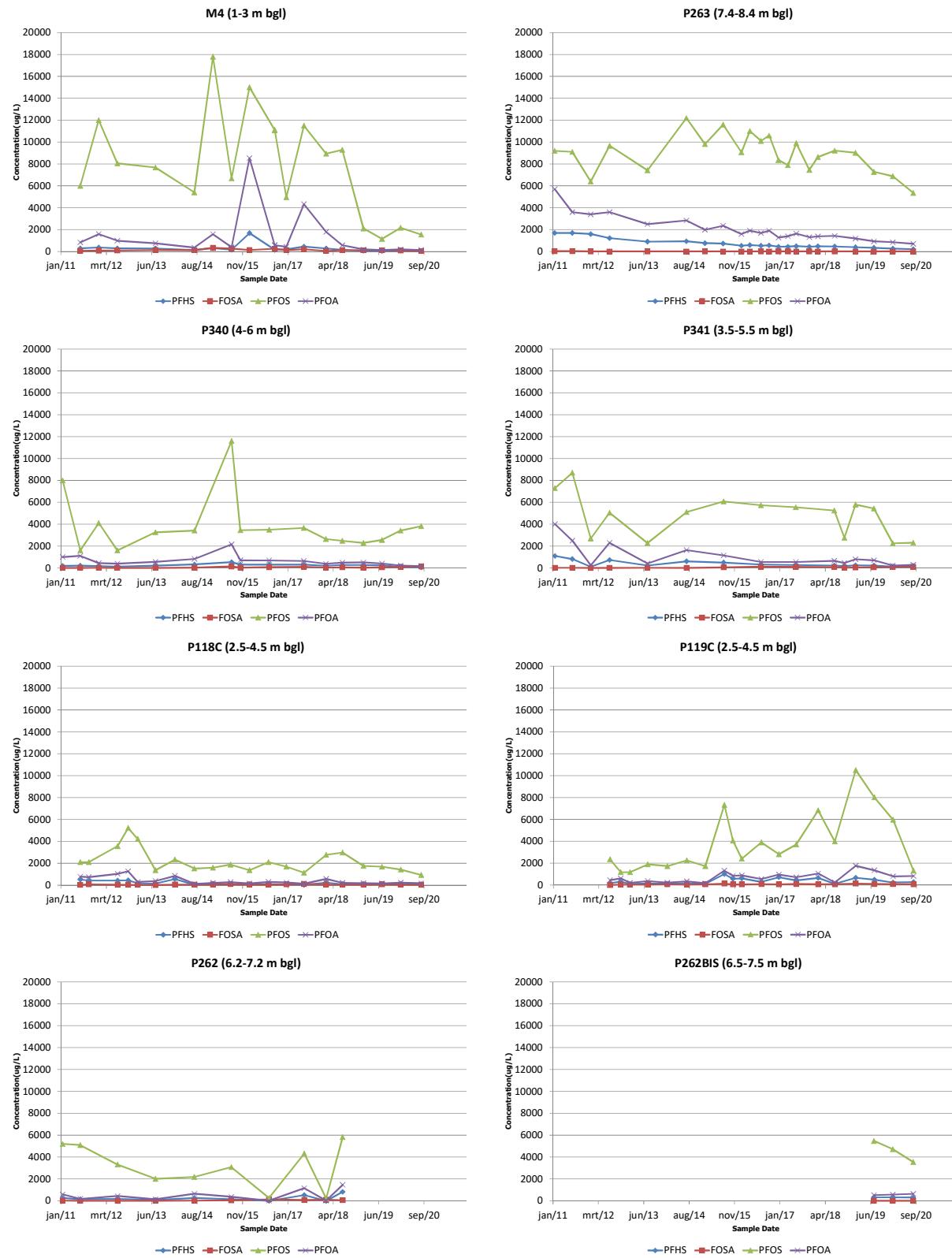
Bronzone - Gebouw 16



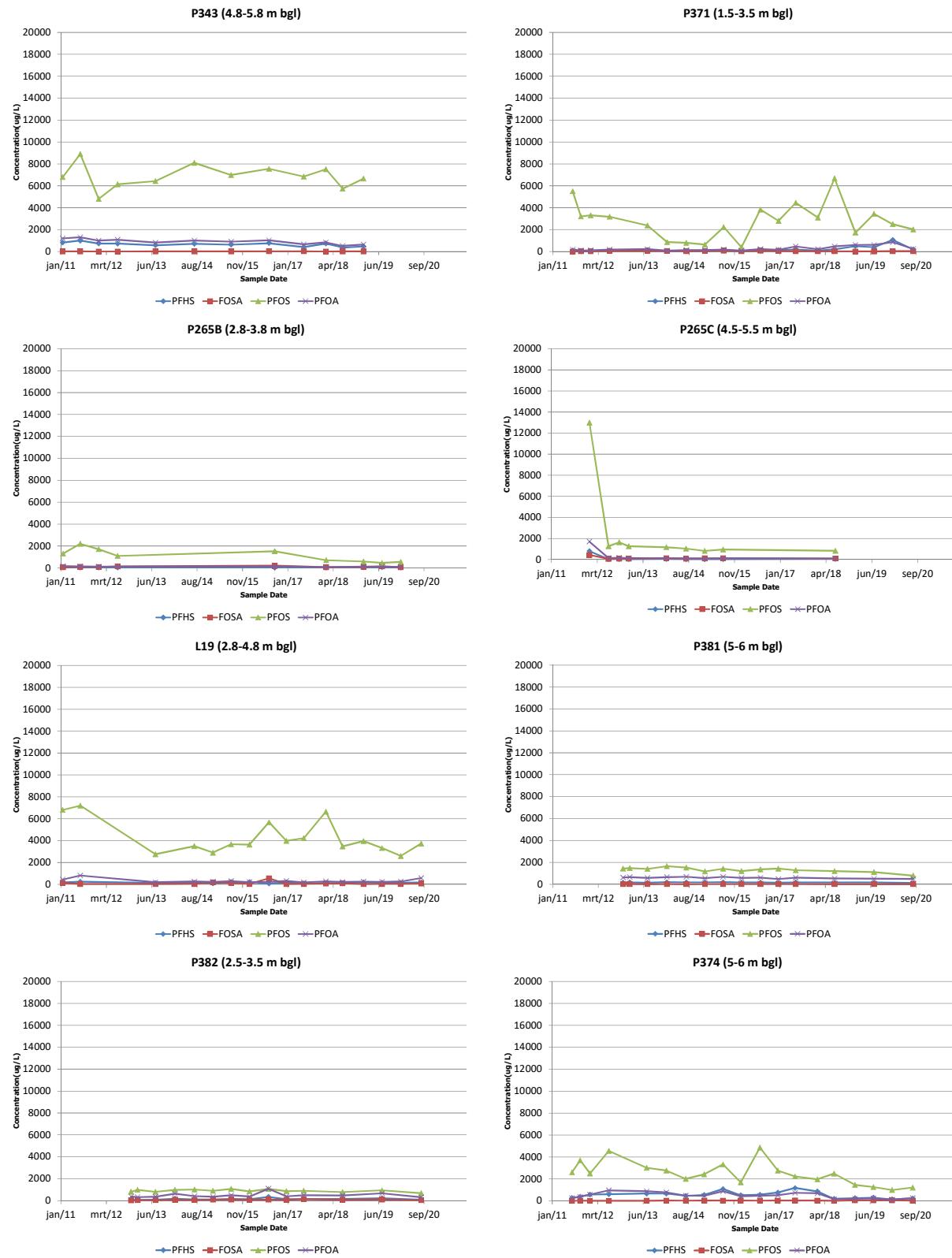
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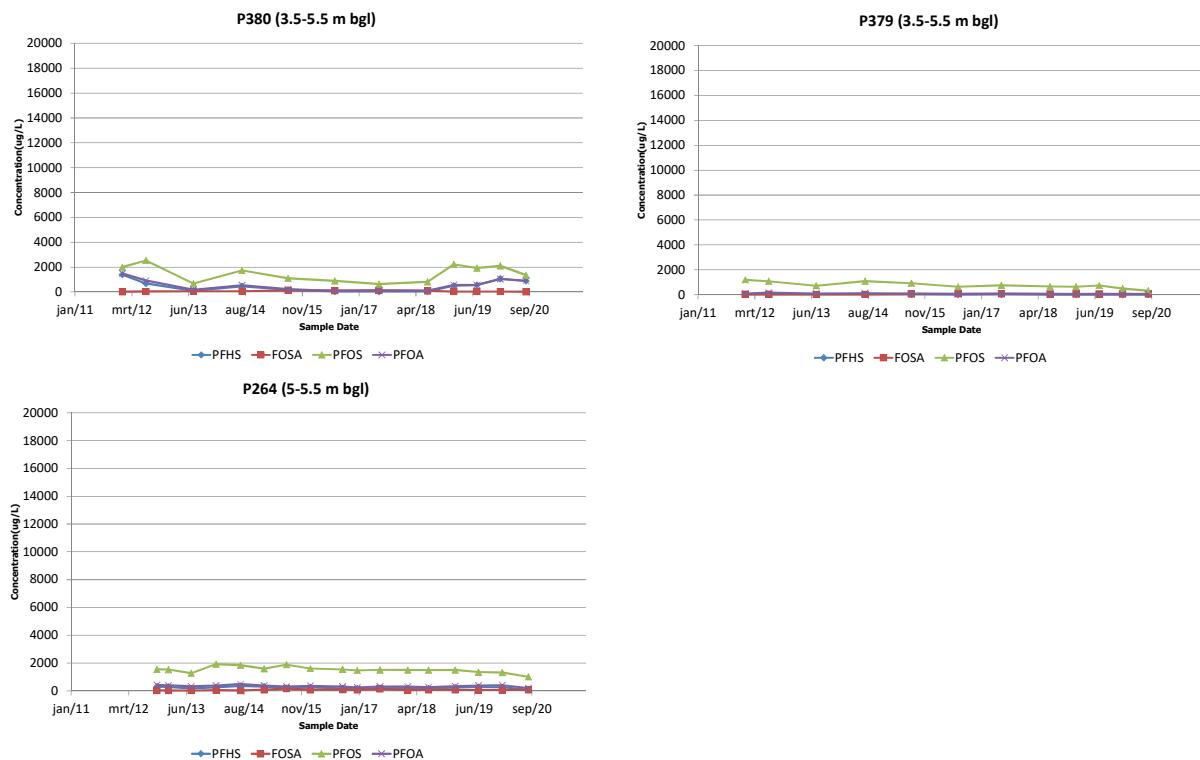
Bronzone - WWTP



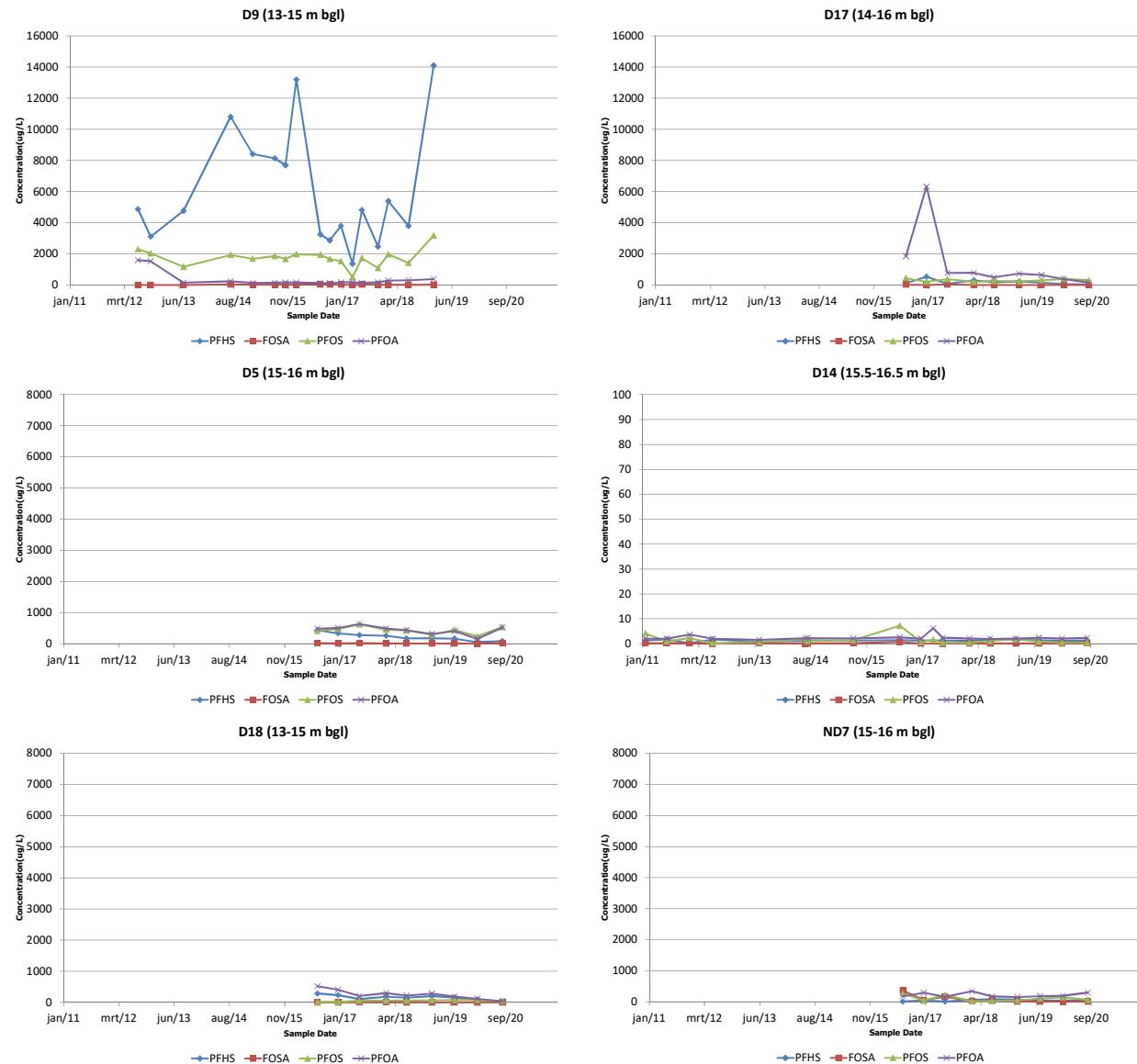
Bronzone - WWTP



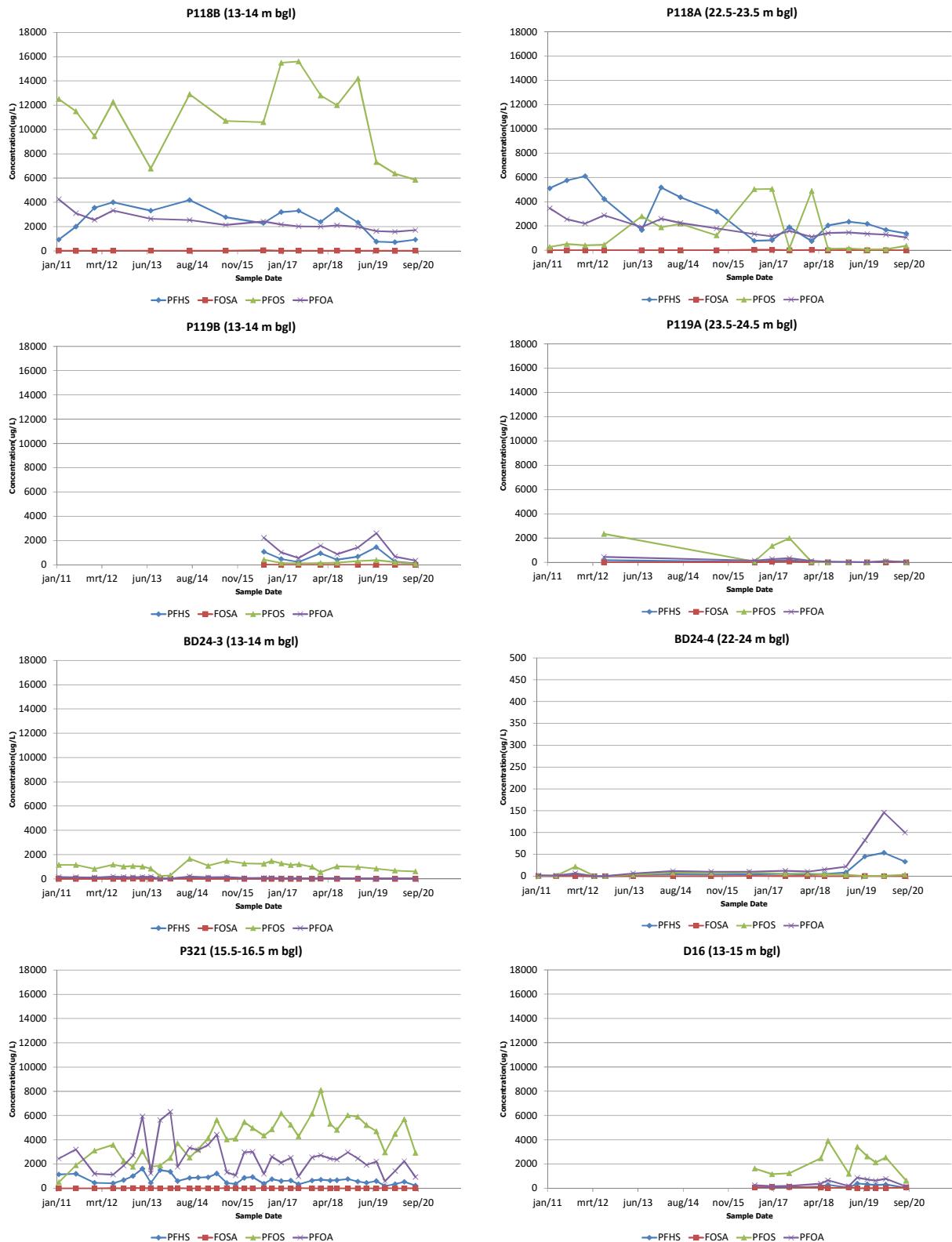
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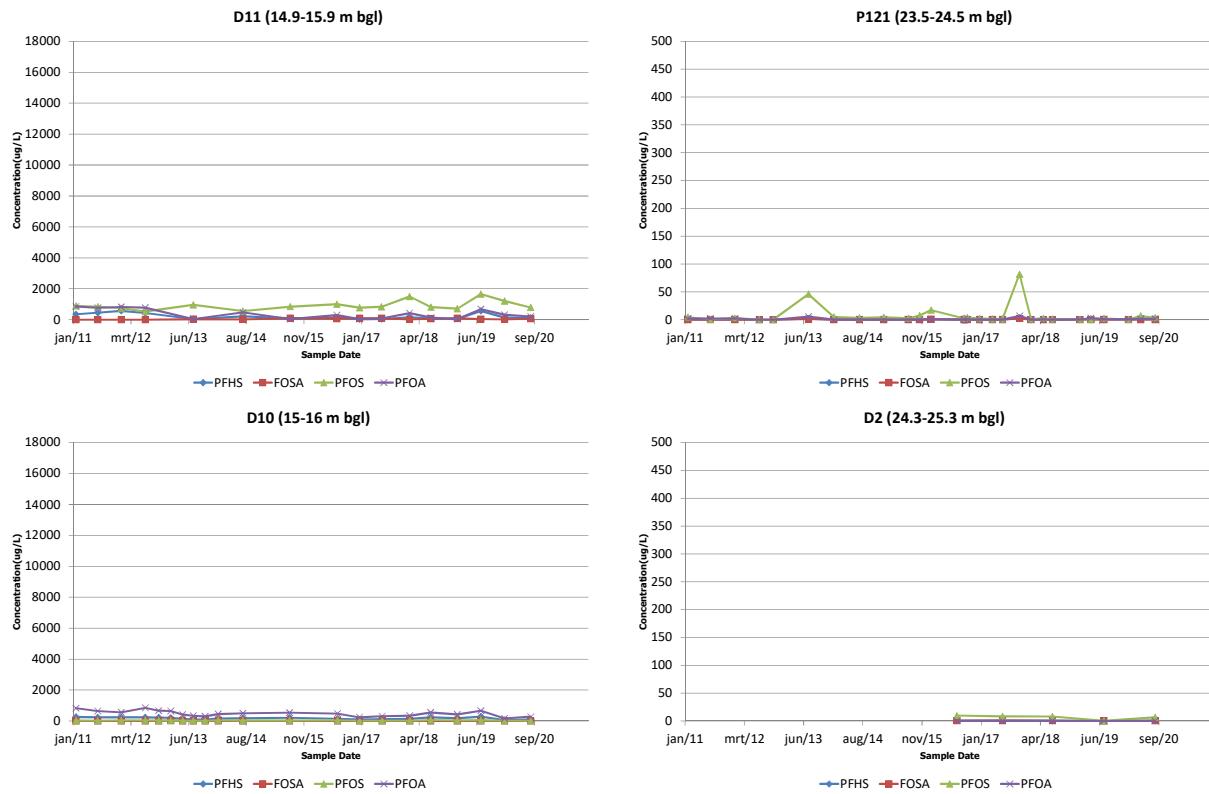
Tweede Aquifer - Gebouw 16



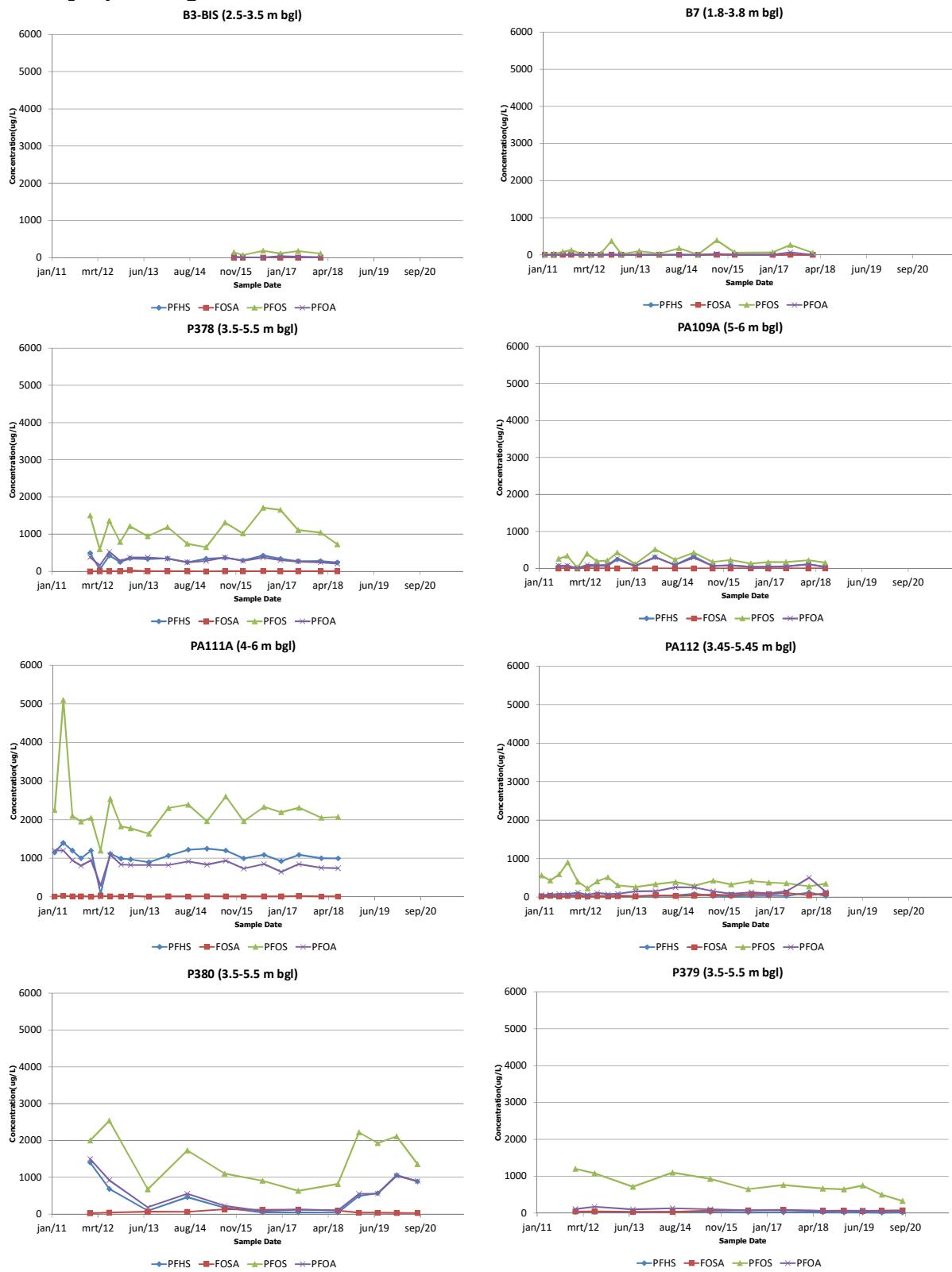
Tweede Aquifer - WWTP



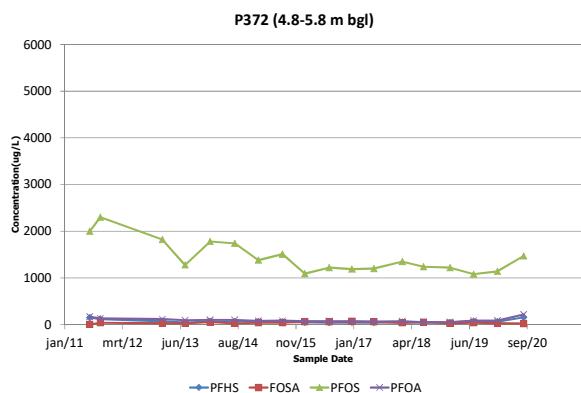
Tweede Aquifer - WWTP



Zuidelijke perceelsgrens



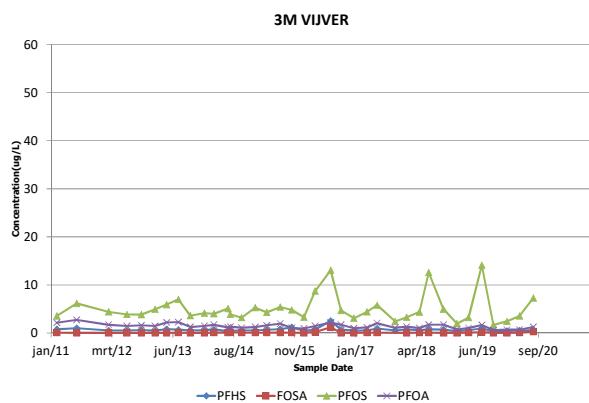
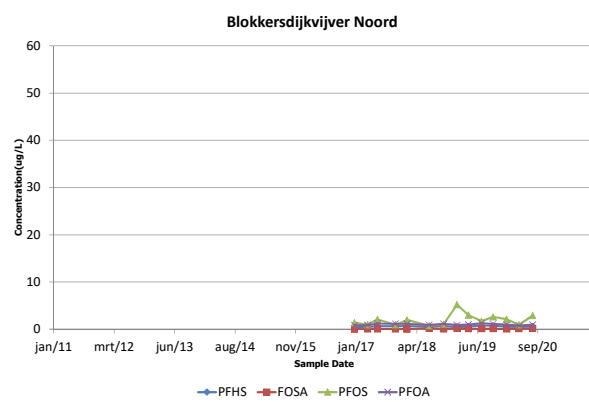
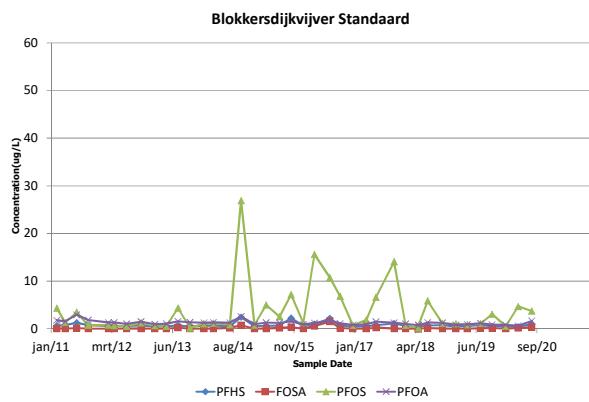
Zuidelijke perceelsgrens



Blokkersdijk natuurreserve

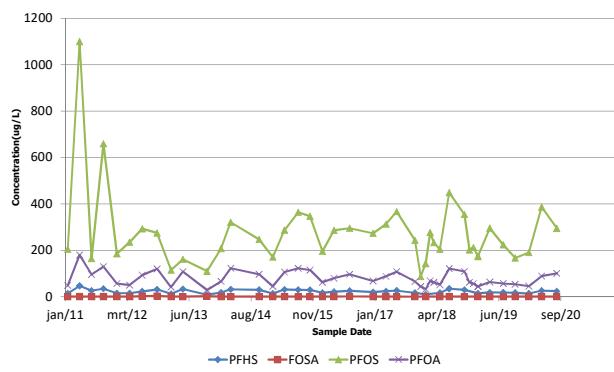


Blokkersdijk natuurreserve

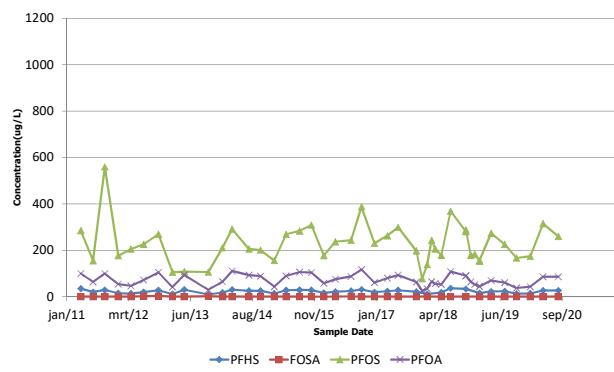


Palingbeek en tophatgracht

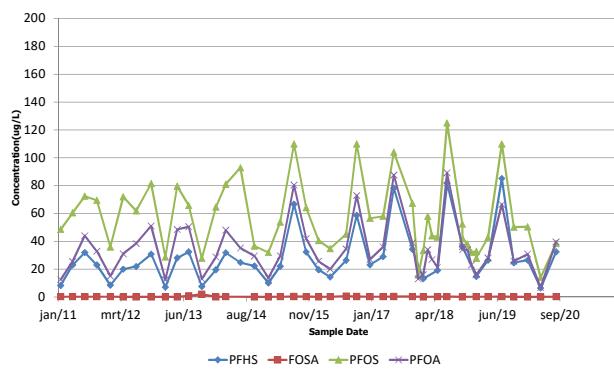
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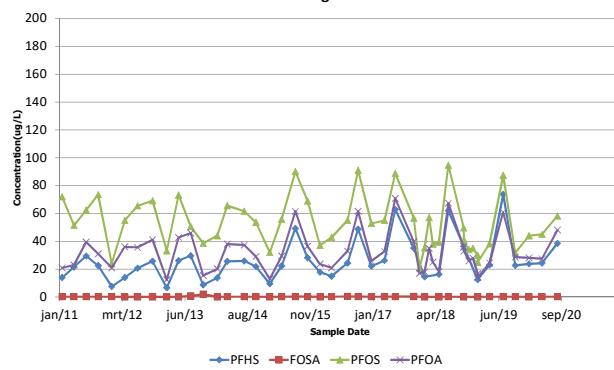
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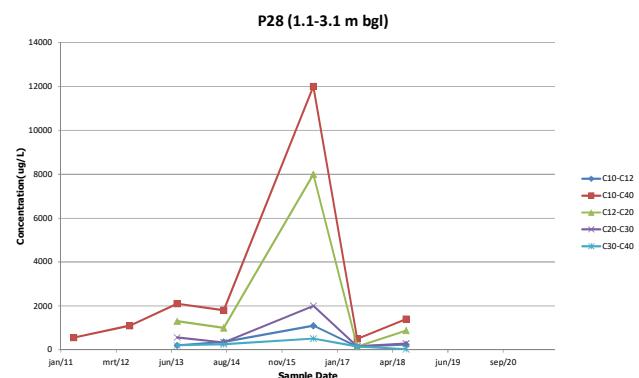
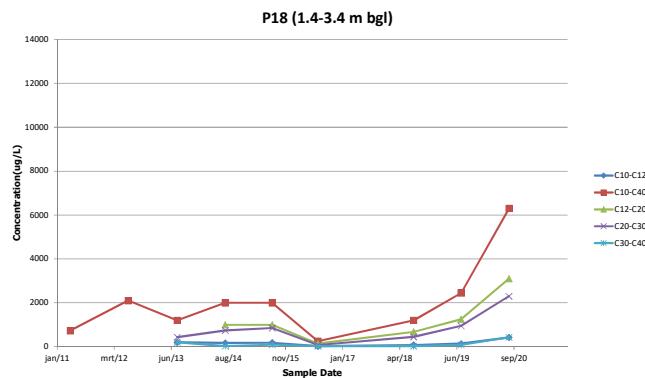
5



Bemalingsstation



Tankenpark



BIJLAGE 8

**STATISTISCHE EVALUATIE ANALYSERESULTATEN
BLOKKERSDIJK**

Memo

To 3M - Jim Kotsmith, Kristin Colberg, Charlotte Tack, Emma Tavernier

From ERM – Jenne Dierckx, Julie Fichefet and Lieselotte Sorgeloos

Date 15 Februari 2021

Project number 0550764

Subject Evaluation of the statistical trend analysis of the ground- and surface water concentrations collected in the Blokkersdijk nature reserve

Introduction

This memo presents an update to the statistical trend analysis of the perfluorooctane sulfonate (PFOS) concentrations in the Blokkersdijk nature reserve using data that are supplemental to the 29-November-2017 memo which presented an evaluation of the statistical trend of the PFOS concentrations. Performance of this PFOS trend analysis is part of the remedial action plan(1) that was drafted for the 3M site in Zwijndrecht.

Since 2009, periodic sampling has been conducted to collect water samples in the Blokkersdijk nature reserve pond, the 3M Pond and select wells located adjacent to these two ponds. Analytical results from these water samples have been used for the PFOS trend analysis. Since the start of the remediation program at the 3M site, different laboratories have been used to analyze the collected water samples. This change in analytical laboratories occurred, with OVAM's approval, because of concerns regarding the quality of the data generated by the laboratories in the early part of the data period. Since 2014 the analyses are being performed by the 3M Environmental Laboratory in St. Paul, Minnesota, USA. In addition, a local OVAM-certified laboratory (SGS nv) has been performing analyses on split samples. Based on an evaluation of the split sample data and the rigorous QA/QC program followed by the 3M Environmental Laboratory, it has been determined that the analytical data generated by the 3M Lab is the most representative data set. Given that using the data set with contributions from multiple laboratories for performing a trend analysis may not be meaningful, the 31-March-2016 memo argued that there were substantial differences between laboratories, and the 3M lab data should be used for the trend analysis. The current trend analysis includes sampling results collected from January 2014 through August 2020, and analyzed by the 3M lab.

The results of the revised evaluation will be included in the ninth annual interim report ("Eerste gefaseerd bodemsaneringsproject - Negende tussentijds verslag bodemsaneringswerken, Periode augustus 2017 - juli 2020", dd. 31 maart 2021) that was submitted to the Flemish environmental agency OVAM.

(1)"Eerste gefaseerd bodemsaneringsproject 3M Belgium NV, haven 1005 – Canadastraat 11, 2070 Zwijndrecht", opgesteld door Arcadis op oktober 2008

Background

The Blokkersdijk nature reserve lies east and adjacent to the 3M site (see Figure 1). During the evaluation of remedial options in 2008, monitoring of the water quality in the nature reserve was selected as the best remedial technique and 'a statistically increasing trend' was defined as trigger value. As PFOS is the main compound of concern in the remediation, the statistical evaluation was focused on this parameter alone.

Figure 1 - Location of 3M site and Blokkersdijk nature reserve



Since the start of the remediation in 2009 regular water samples have been taken in seven monitoring wells and two ponds that are located in the vicinity of the Blokkersdijk nature reserve. The location of these sample points is shown in Figure 2.

Figure 2 - Location of sampling points Blokkersdijk nature reserve



The presence of perfluorinated compounds have been quantified in pond surface water and monitoring wells installed near the Blokkersdijk pond. PFOS concentrations were subject to trend analyses to determine if concentrations were increasing, remaining stable or decreasing over time. The locations used in the trend analysis are wells L21, L22, L31, L4, P114bis, P115, P116, and the 3M vijver and Blokkersdijk vijver – standaardlocatie and Noord. Well L30 was sampled due to inaccessibility of well L22 but concerns a different location and cannot be compared to previous measurements from L22. As only 2 results are available for L30, no trend analysis can be done on this well.

Scope of Work

The PFOS trend analyses were conducted on only the 3M lab data for each location. The 3M lab data analysis started in January 2014 and continued through January 2021.

The trend analysis was conducted as follows:

1. Data distribution was determined using the Shapiro-Wilks and Lillifors tests for each location;
2. Q-Q plots were developed for each location to evaluate if data outliers were present in the data;
3. Time series figures were created for each location showing PFOS concentrations over time; and
4. Trend analyses were conducted using ordinary least-square (OLS) regression on either the original and log-transformed (natural log) data sets, as well as the non-parametric Mann-Kendall trend test as defined in the remedial action plan of 2008⁽¹⁾. The appropriate trend analysis used for each location and data set was based on the distribution of the data determined in step 1.

Annex 1 shows the data used in the trend analysis. PFOS was detected in all samples, except for the July 30, 2014 sampling in well P115, which was non-detect, and the May 3, 2018 sampling in well BD Noord, which was not reported.

All statistical analyses were conducted using appropriate statistical packages in R.

Results

The figures included as Annex 2 show time series graphs of PFOS concentrations for samples collected from each of the sampling locations. Annex 3 includes a visualization of the distribution goodness-of-fit testing for the datasets per sampling location, including Q-Q plots. The data was variously normally distributed, log-normally distributed, or distribution free (non-parametric). The results of the trend analyses are presented in Annex 3. These analyses show that wells L22, L31, L4, P114bis and P116 had significantly decreasing trends, and that well P115 had a significantly increasing trend.

Well L21

Annex 2 shows well L21 had no apparent trend in the data. The Q-Q plot for the 3M lab data set (Annex 3) does not show any potential outliers. The data set is normally distributed, and the OLS regression for this data did not show a significant trend in PFOS (see Annex 4).

Well L22

Annex 2 shows well L22 had an apparent downward trend in the data. Two intermediate values are substantially higher than the norm for this well (July and October 2015). The Q-Q plots show that these two values might be from a different population (i.e., are potential

outliers)(see Annex 3). To remove these data, and the previous, all data prior to the October 2015 event was excluded from analysis. The data set is normally distributed, and the OLS regression on long-transformed data shows a significant downward trend in PFOS (see Annex 4).

Well L31

Annex 2 shows well L31 had an apparent downward trend in the data. The Q-Q plots did not indicate any data outliers. The data is normally distributed and the OLS regression for this data showed a significant downward trend (see Annex 4).

Well L4

Annex 2 shows well L4 had an apparent downward trend in the data. The data set is normally distributed and the Q-Q plot indicated no outliers. The OLS regression for this data showed a significant downward trend (see Annex 3).

Well P114bis

Annex 2 shows well P114 had an apparent downward trend in the data. The Q-Q plots did not indicate any potential outliers. The data set is non-parametric, and the Mann-Kendall regression showed a significant downward trend (see Annex 3).

Well P115

Annex 2 shows well P115 had an apparent upward trend in the data. The Q-Q plots did not indicate outliers. The data was log-normally distributed, and there was a significant upward trend in PFOS (see Annex 3).

Well P116

Annex 2 shows well P116 had an apparent downward trend in the data. An outlier was observed in October 2015 and all data prior to and including this date was excluded from further analysis, the Q-Q plot of the resulting dataset does not indicate any further outliers. The data set is normally distributed, and the OLS regression showed a significant downward trend in PFOS (see Annex 3).

3M vijver

Annex 2 shows the 3M vijver PFOS concentrations with no apparent trend in the data, although there were several spikes in PFAS concentration during the 28-July-2016, 09-July-2018 and 01-August-2019 sampling events. The Q-Q plots (log-transformed) do not indicate data outliers. The data is log-normally distributed, and the OLS regression indicates no significant trend in PFOS (see Annex 3).

Blokkersdijk vijver standard

Annex 2 shows the Blokkersdijk vijver standard_PFOS concentrations with no apparent trend in the data. The Q-Q plot (log-transformed) did not indicate data outliers (see Annex 4). The data set was non-parametric, and the Mann-Kendall regression showed no significant trend in PFOS (see Annex 3).

Blokkersdijk vijver Noord

Annex 2 shows the Blokkersdijk vijver Noord_PFOS concentrations with no apparent trend in the data. The Q-Q plot (log-transformed) did not indicate data outliers (see Annex 4). The data set was normally distributed, and the OLS regression showed no significant trend in PFOS (see Annex 3).

Discussion and Conclusion

A statistical trend analysis was conducted on the PFOS data from selected locations, and consisted of a trend analysis of the data which was analyzed by 3M Environmental Lab for each location. Five of the locations showed statistically significant downward trends: L22, L31, L4, P114bis and P116. Well P115 showed a statistically significant increase in trend. None of the other wells evaluated showed a statistically significant trend in PFOS.

Annexes:

Annex 1 - Data used in the trend analysis

Annex 2 - Time series graphs of PFOS

Annex 3 - Distribution goodness-of-fit testing visualizations

Annex 4 - Results of the trend analyses

Annex 1

Table 1: Data used in the trend analysis

Date	Location	PFOS (µg/L)
21/01/2014	L21	4,16
30/07/2014	L21	3,58
27/01/2015	L21	4,36
28/07/2015	L21	7,23
16/01/2016	L21	5,65
6/04/2016	L21	8,37
26/07/2016	L21	6,17
11/10/2016	L21	5,22
12/01/2017	L21	5,41
14/04/2017	L21	5,01
3/07/2017	L21	6,13
6/11/2017	L21	3,85
29/01/2018	L21	3,43
3/05/2018	L21	4,53
9/07/2018	L21	5,01
25/10/2018	L21	5,92
30/01/2019	L21	4,18
25/04/2019	L21	4,05
1/08/2019	L21	4,43
29/01/2020	L21	2,13
29/04/2020	L21	3,91
4/08/2020	L21	2,28
21/01/2014	L22	12,1
30/07/2014	L22	11,9
27/01/2015	L22	11,4
23/07/2015	L22	30,9
21/10/2015	L22	38,6
16/01/2016	L22	5,39
6/04/2016	L22	6,00
26/07/2016	L22	4,94
11/10/2016	L22	4,94
12/01/2017	L22	4,29
14/04/2017	L22	4,43
3/07/2017	L22	4,97
6/11/2017	L22	8,41
29/01/2018	L22	6,58
3/05/2018	L22	4,2
9/07/2018	L22	2,81
25/10/2018	L22	2,52
30/01/2019	L22	2,28
25/04/2019	L22	2,50
1/08/2019	L22	3,12
29/01/2020	L22	2,40
29/04/2020	L30	6,70
4/08/2020	L30	5,62
21/01/2014	L31	8,07
14/07/2014	L31	6,30
30/07/2014	L31	8,03
27/01/2015	L31	5,72

28/07/2015	L31	10,7
16/01/2016	L31	7,61
6/04/2016	L31	4,32
26/07/2016	L31	9,94
11/10/2016	L31	9,2
12/01/2017	L31	5,22
14/04/2017	L31	5,39
3/07/2017	L31	7,53
6/11/2017	L31	7,96
29/01/2018	L31	3,18
3/05/2018	L31	3,51
9/07/2018	L31	10,8
25/10/2018	L31	7,28
30/01/2019	L31	4,15
25/04/2019	L31	4,57
1/08/2019	L31	7,59
29/01/2020	L31	4,57
29/04/2020	L31	3,78
4/08/2020	L31	4,02
21/01/2014	L4	21,9
2/04/2014	L4	23,2
14/07/2014	L4	41,0
30/07/2014	L4	27,5
20/10/2014	L4	29,9
27/01/2015	L4	38,6
21/04/2015	L4	19,0
23/07/2015	L4	30,0
21/10/2015	L4	46,2
16/01/2016	L4	37,3
6/04/2016	L4	24,5
19/09/2016	L4	30,2
11/10/2016	L4	31,8
11/01/2017	L4	29,2
14/04/2017	L4	28,8
3/07/2017	L4	32,8
6/11/2017	L4	27,7
29/01/2018	L4	25,1
3/05/2018	L4	20,1
9/07/2018	L4	28,2
25/10/2018	L4	28,2
30/01/2019	L4	26,8
25/04/2019	L4	25,3
1/08/2019	L4	29,5
28/01/2020	L4	17,5
29/04/2020	L4	15,4
4/08/2020	L4	23,0
2/04/2014	P114bis	16,3
14/07/2014	P114bis	11,1
30/07/2014	P114bis	11,5
20/10/2014	P114bis	13,4

27/01/2015	P114bis	14,2
21/04/2015	P114bis	16,2
23/07/2015	P114bis	14,0
21/10/2015	P114bis	14,0
16/01/2016	P114bis	4,29
6/04/2016	P114bis	13,3
26/07/2016	P114bis	15,8
12/10/2016	P114bis	13,4
12/01/2017	P114bis	9,21
14/04/2017	P114bis	12,5
3/07/2017	P114bis	11,7
6/11/2017	P114bis	14,4
29/01/2018	P114bis	13,3
3/05/2018	P114bis	13,9
9/07/2018	P114bis	11,9
25/10/2018	P114bis	9,73
30/01/2019	P114bis	3,92
25/04/2019	P114bis	6,73
1/08/2019	P114bis	5,34
29/01/2020	P114bis	5,00
29/04/2020	P114bis	4,30
4/08/2020	P114bis	5,47
21/01/2014	P115	0,52
14/07/2014	P115	0,91
30/07/2014	P115	<0,46
27/01/2015	P115	0,47
23/07/2015	P115	0,24
16/01/2016	P115	0,59
6/04/2016	P115	1,07
26/07/2016	P115	0,53
12/10/2016	P115	0,30
12/01/2017	P115	0,59
14/04/2017	P115	0,52
3/07/2017	P115	0,44
6/11/2017	P115	1,07
29/01/2018	P115	0,80
3/05/2018	P115	1,13
9/07/2018	P115	0,61
25/10/2018	P115	1,44
30/01/2019	P115	1,92
25/04/2019	P115	2,27
1/08/2019	P115	3,02
29/01/2020	P115	1,92
29/04/2020	P115	2,14
4/08/2020	P115	3,20
21/01/2014	P116	9,17
2/04/2014	P116	7,46
14/07/2014	P116	5,25
30/07/2014	P116	7,42
20/10/2014	P116	8,77

27/01/2015	P116	6,87
21/04/2015	P116	7,71
23/07/2015	P116	9,43
21/10/2015	P116	34,6
16/01/2016	P116	8,51
6/04/2016	P116	8,88
27/07/2016	P116	10,6
11/10/2016	P116	9,66
12/01/2017	P116	10,9
14/04/2017	P116	9,62
3/07/2017	P116	9,08
6/11/2017	P116	8,24
29/01/2018	P116	8,74
3/05/2018	P116	9,34
9/07/2018	P116	7,85
25/10/2018	P116	8,91
30/01/2019	P116	7,98
25/04/2019	P116	8,00
1/08/2019	P116	8,36
29/01/2020	P116	8,58
29/04/2020	P116	8,89
4/08/2020	P116	7,14
23/01/2014	3M vijver	4,13
2/04/2014	3M vijver	4,00
31/07/2014	3M vijver	3,85
21/10/2014	3M vijver	3,18
27/01/2015	3M vijver	5,27
21/04/2015	3M vijver	4,30
27/07/2015	3M vijver	5,39
20/10/2015	3M vijver	4,78
16/01/2016	3M vijver	3,29
7/04/2016	3M vijver	8,73
28/07/2016	3M vijver	13,1
11/10/2016	3M vijver	4,68
12/01/2017	3M vijver	3,08
18/04/2017	3M vijver	4,39
29/06/2017	3M vijver	5,82
6/11/2017	3M vijver	2,40
29/01/2018	3M vijver	3,27
3/05/2018	3M vijver	4,37
9/07/2018	3M vijver	12,6
25/10/2018	3M vijver	4,94
30/01/2019	3M vijver	1,93
25/04/2019	3M vijver	3,19
1/08/2019	3M vijver	14,1
28/01/2020	3M vijver	2,42
28/04/2020	3M vijver	3,57
6/08/2020	3M vijver	7,28
23/01/2014	BD standaard	1,06
2/04/2014	BD standaard	1,17

31/07/2014	BD standaard	0,86
21/10/2014	BD standaard	26,9
27/01/2015	BD standaard	0,79
21/04/2015	BD standaard	5,01
27/07/2015	BD standaard	2,54
20/10/2015	BD standaard	7,18
16/01/2016	BD standaard	1,23
7/04/2016	BD standaard	15,6
28/07/2016	BD standaard	10,8
11/10/2016	BD standaard	6,84
12/01/2017	BD standaard	0,74
18/04/2017	BD standaard	1,86
29/06/2017	BD standaard	6,62
6/11/2017	BD standaard	14,1
29/01/2018	BD standaard	0,68
3/05/2018	BD standaard	0,74
9/07/2018	BD standaard	5,87
25/10/2018	BD standaard	1,13
30/01/2019	BD standaard	1,05
25/04/2019	BD standaard	0,81
1/08/2019	BD standaard	1,08
28/01/2020	BD standaard	0,5
28/04/2020	BD standaard	4,45
6/08/2020	BD standaard	3,74
12/01/2017	BD Noord	1,36
18/04/2017	BD Noord	0,95
29/06/2017	BD Noord	2,08
6/11/2017	BD Noord	0,95
29/01/2018	BD Noord	1,97
3/05/2018	BD Noord	/
9/07/2018	BD Noord	0,73
25/10/2018	BD Noord	1,04
30/01/2019	BD Noord	5,2
25/04/2019	BD Noord	3,23
1/08/2019	BD Noord	1,69
28/01/2020	BD Noord	2,1
28/04/2020	BD Noord	0,97
6/08/2020	BD Noord	2,88

Annex 2

Time series graphs of PFOS

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Figure 1.1 Time Series L21

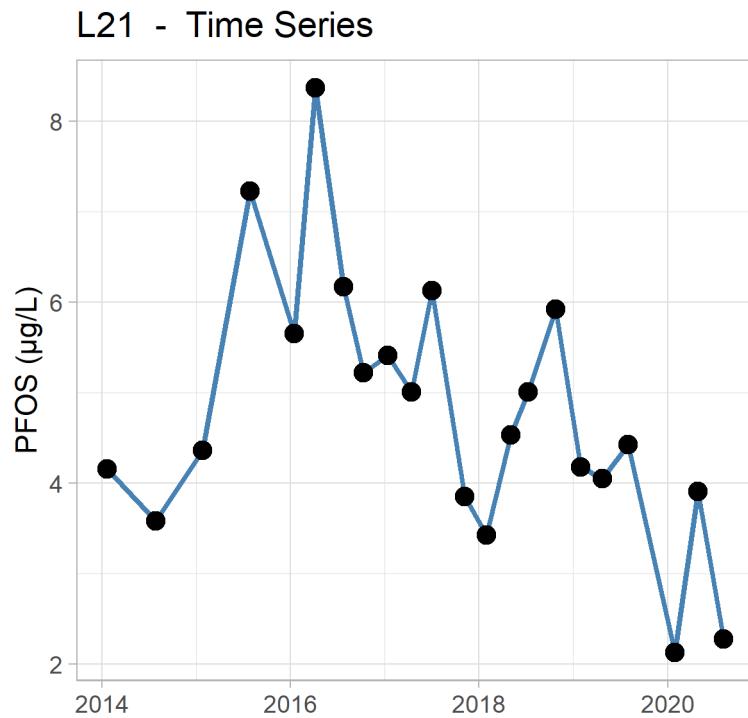


Figure 1.2 Time Series L22

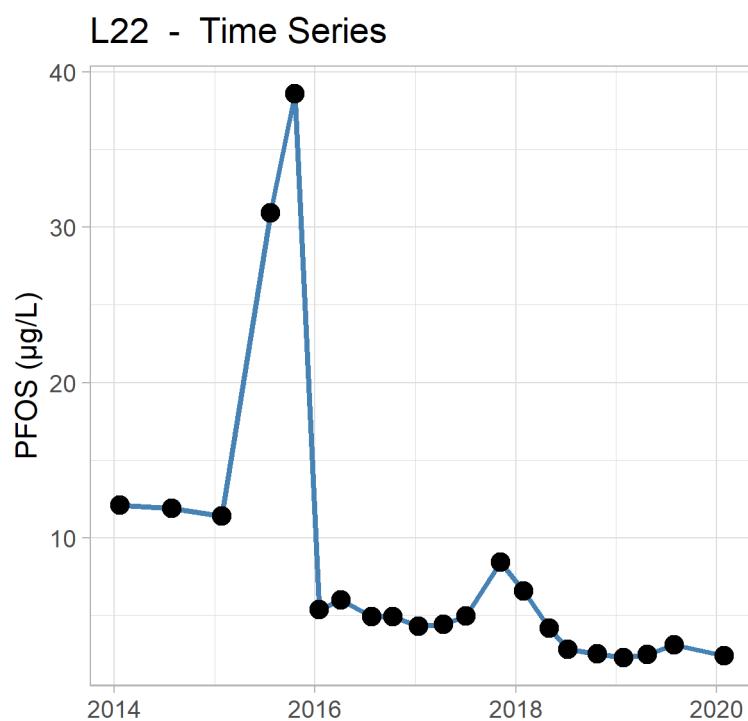


Figure 1.3 Times Series L30 (2020 data only)

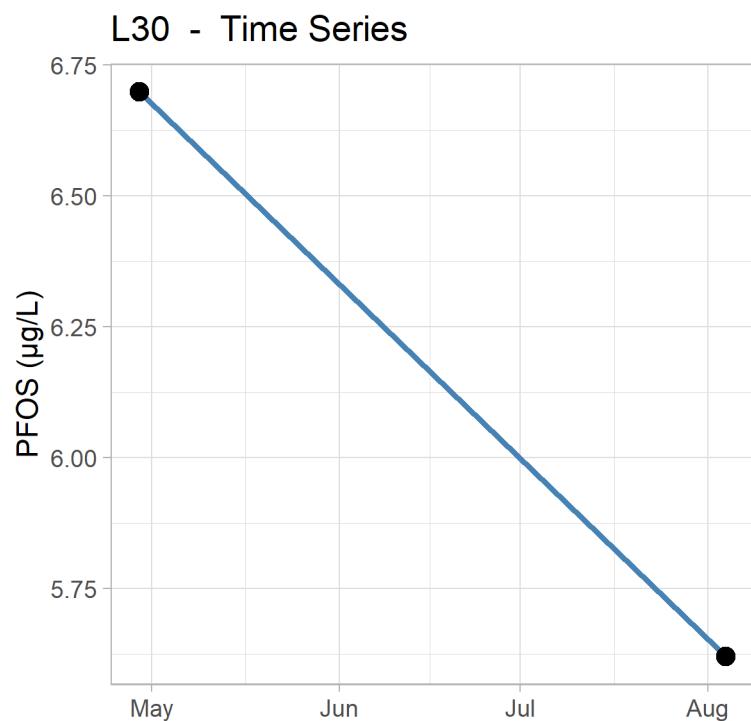


Figure 1.4 Time Series L31

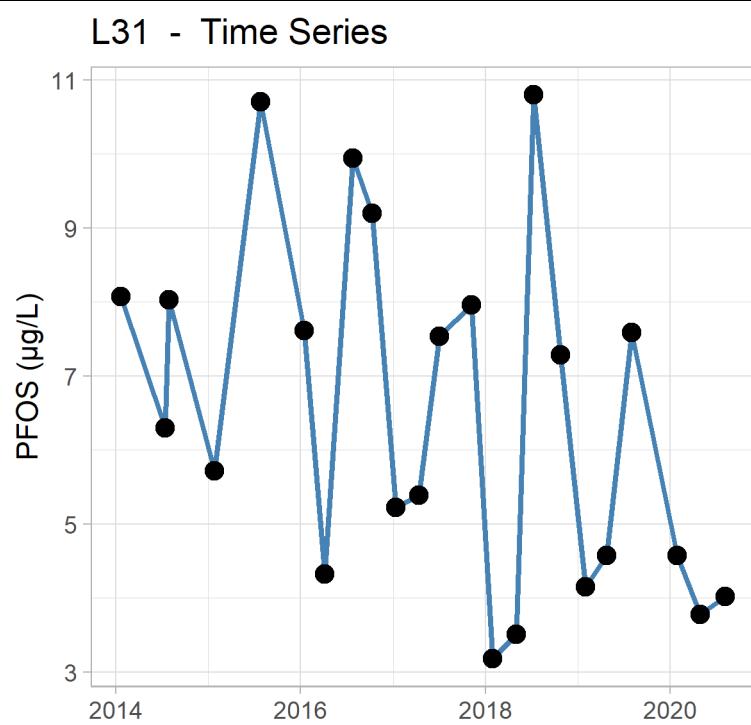


Figure 1.5 Time Series L4

L4 - Time Series

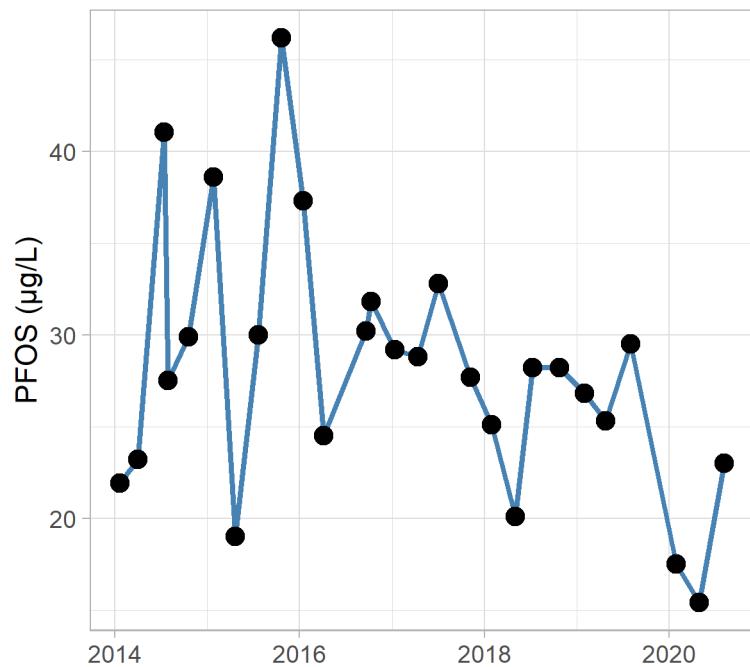


Figure 1.6 Time Series P114bis

P114bis - Time Series

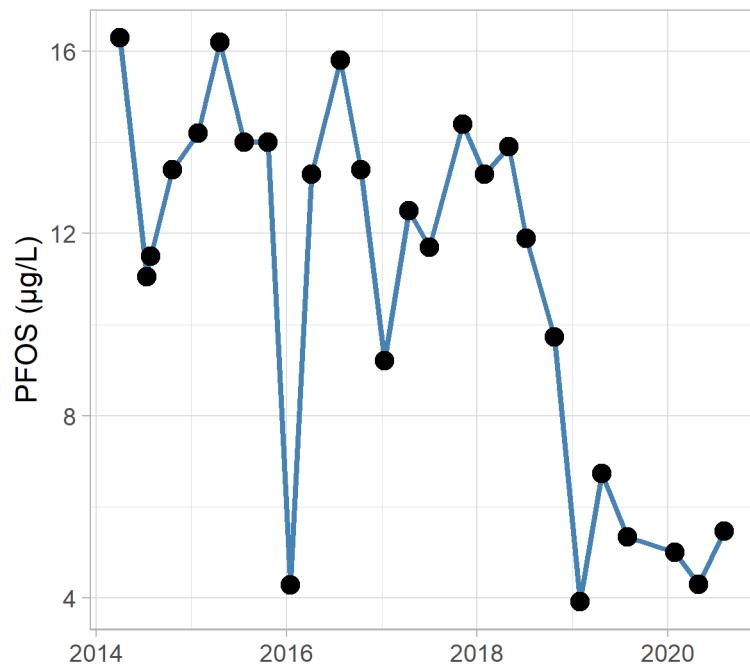


Figure 1.7 Time Series P115

P115 - Time Series

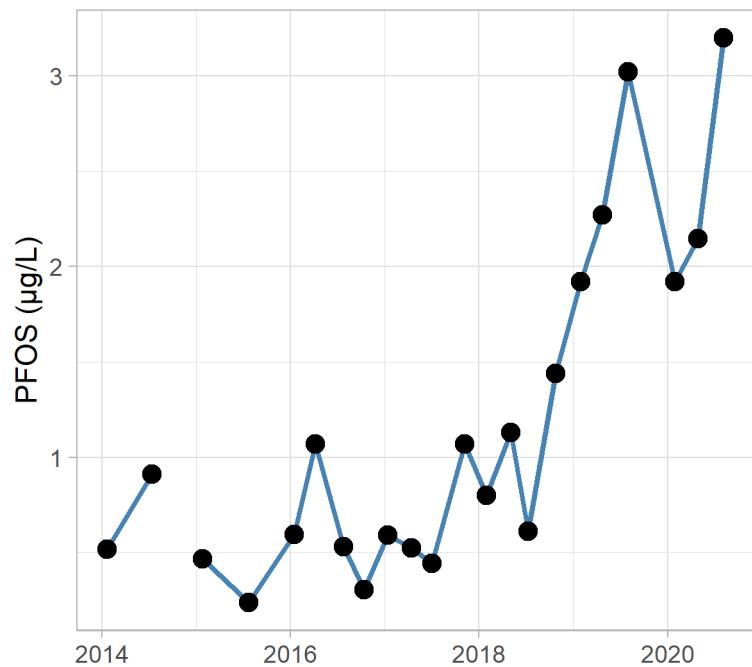


Figure 1.8 Time Series P116

P116 - Time Series

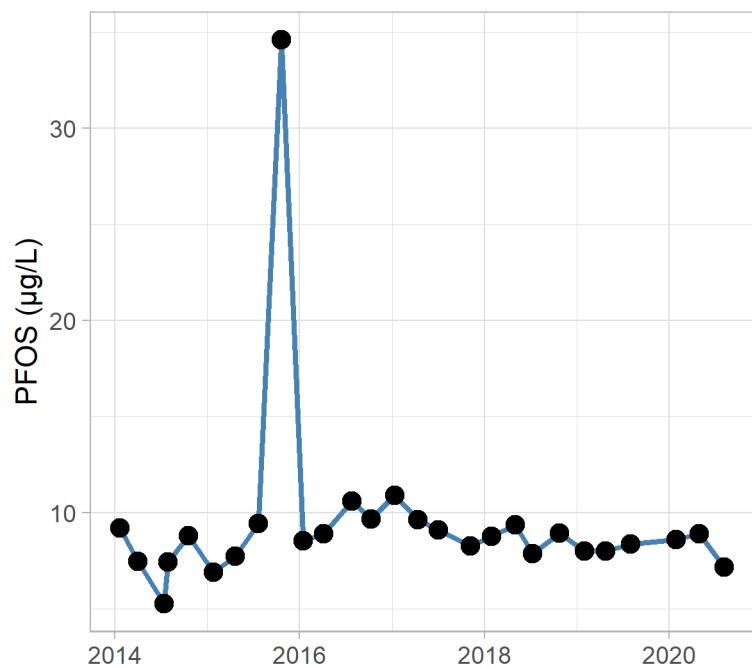


Figure 1.9 Time Series 3M Vijver

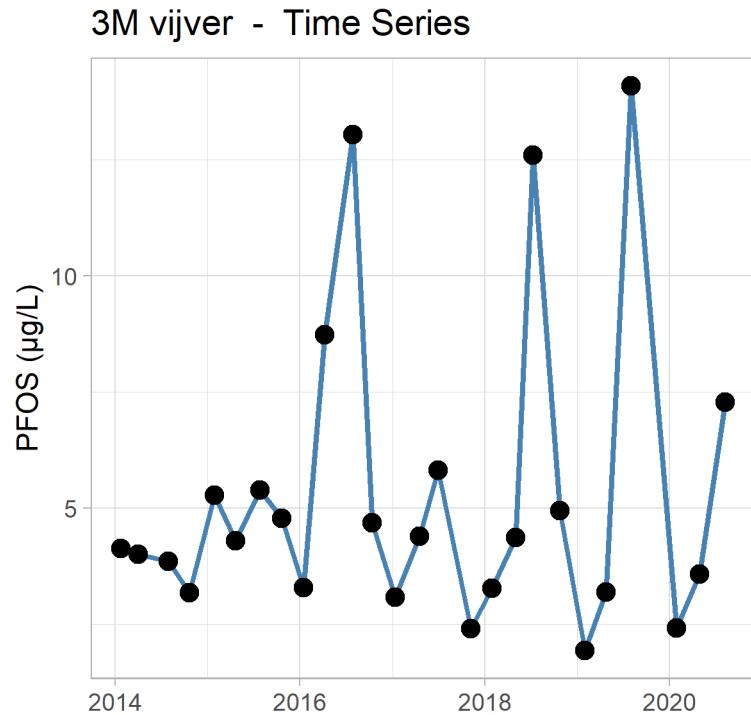


Figure 1.10 Time Series BD standaard

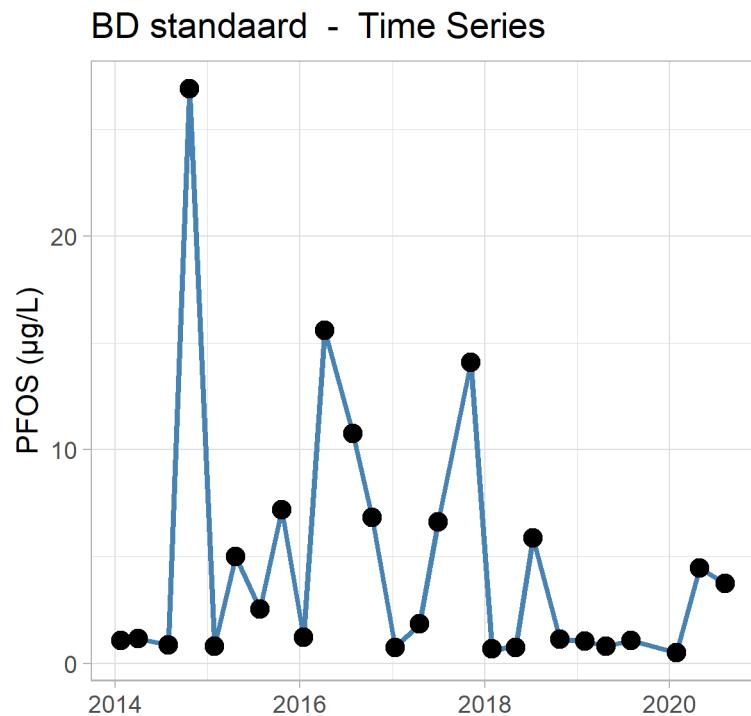
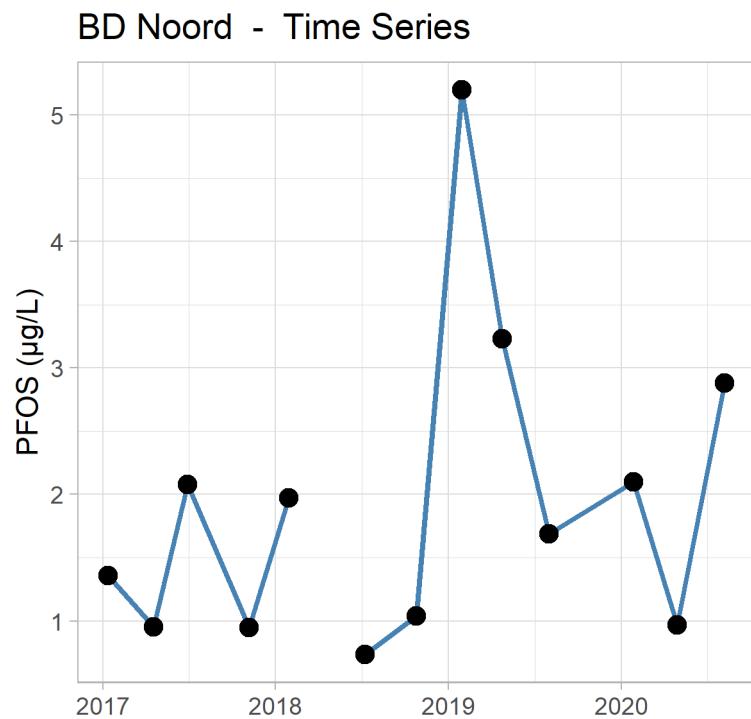


Figure 1.11 Time Series BD Noord



Annex 3

Distribution goodness-of-fit visualizations

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Figure 1.1 L21

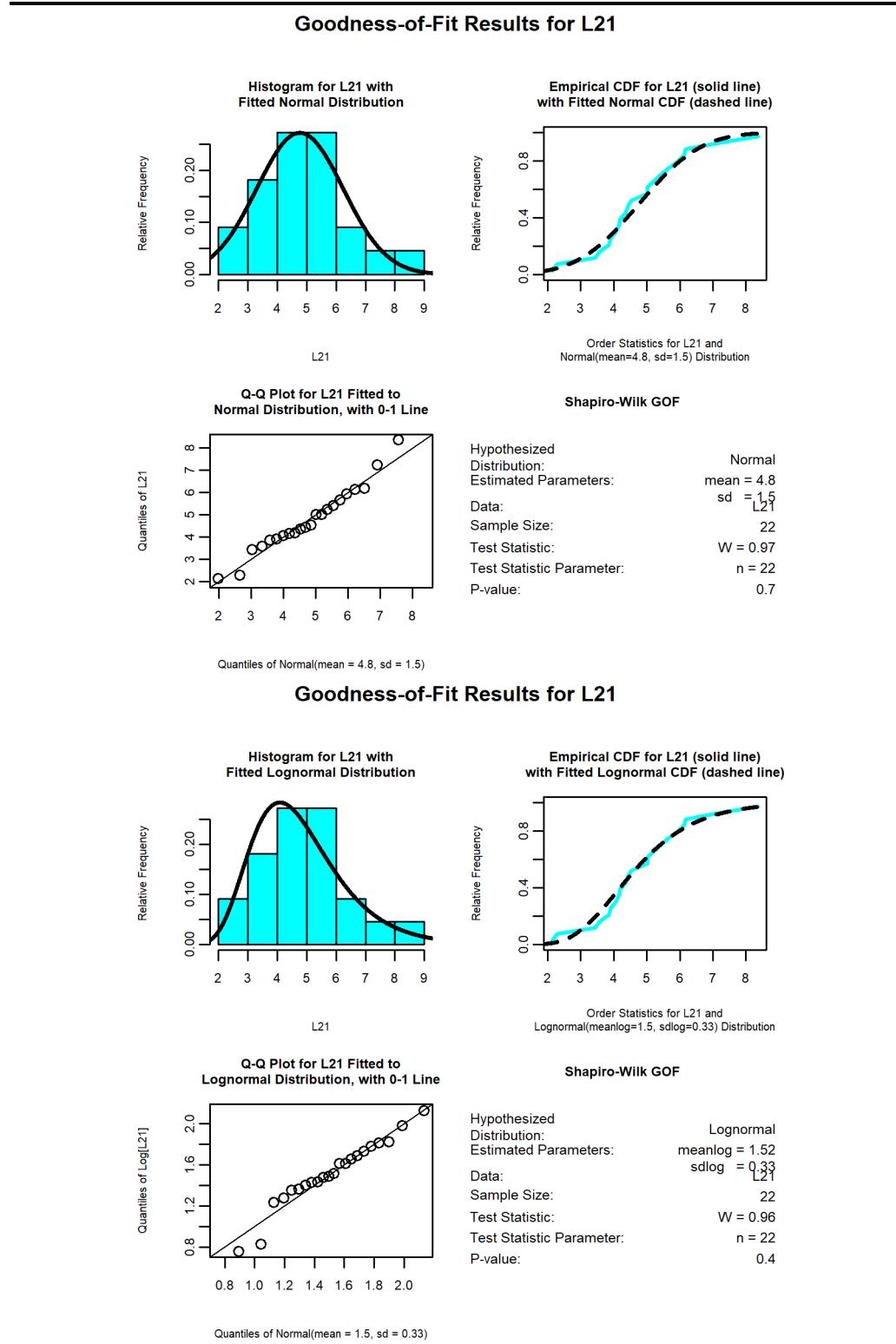


Figure 1.2 L22 (all data)

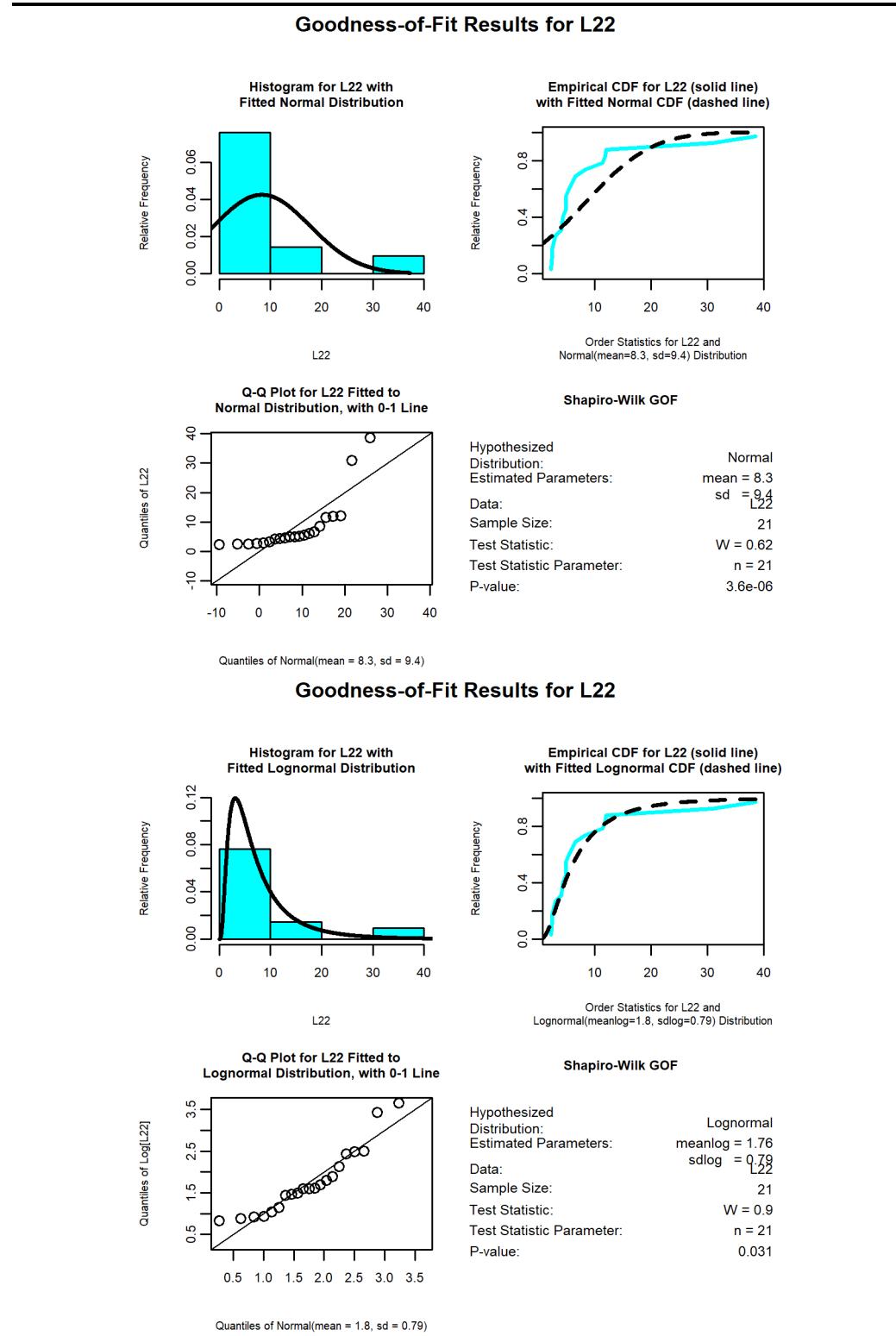


Figure 1.3 L22 (excl. outlying 2014-2015 data)

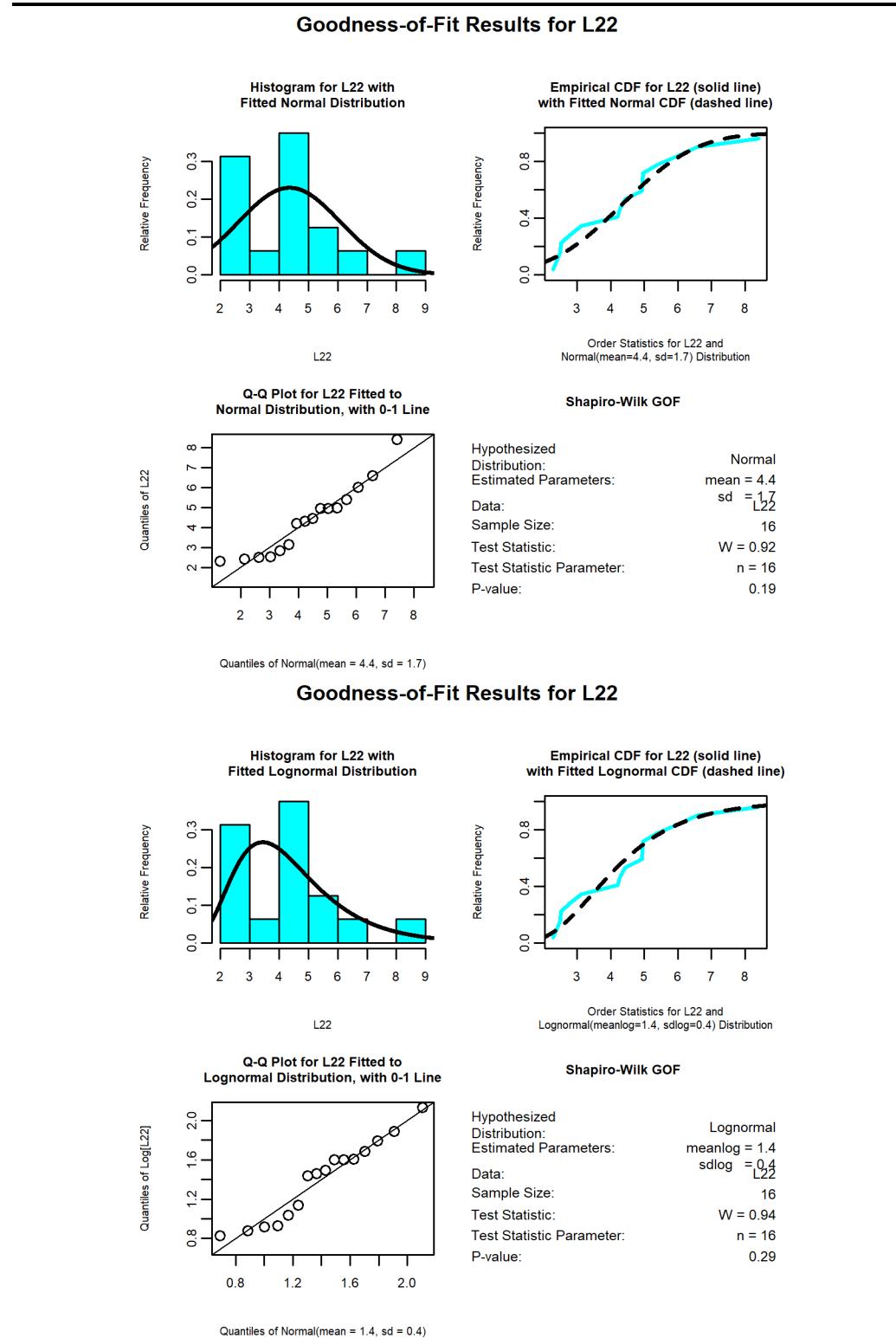
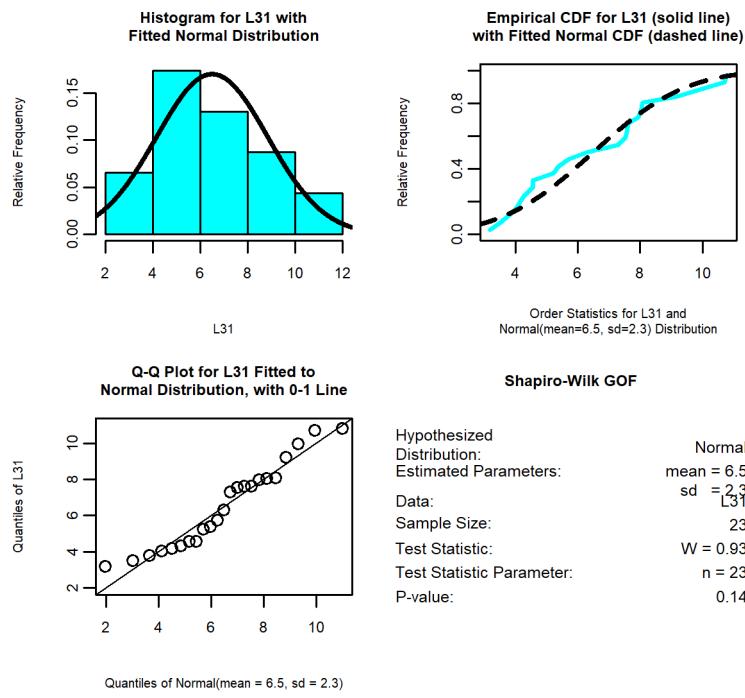


Figure 1.4 L31

Goodness-of-Fit Results for L31



Goodness-of-Fit Results for L31

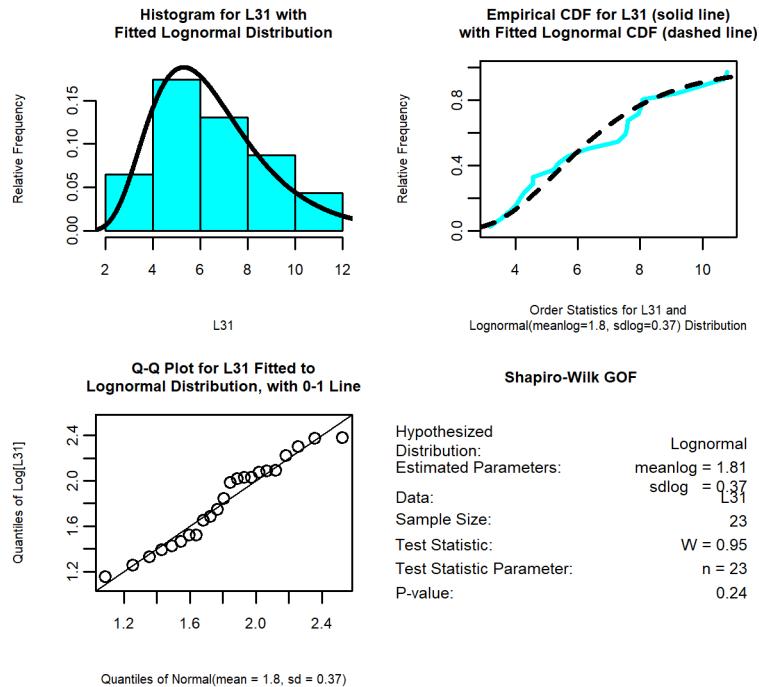


Figure 1.5 L4

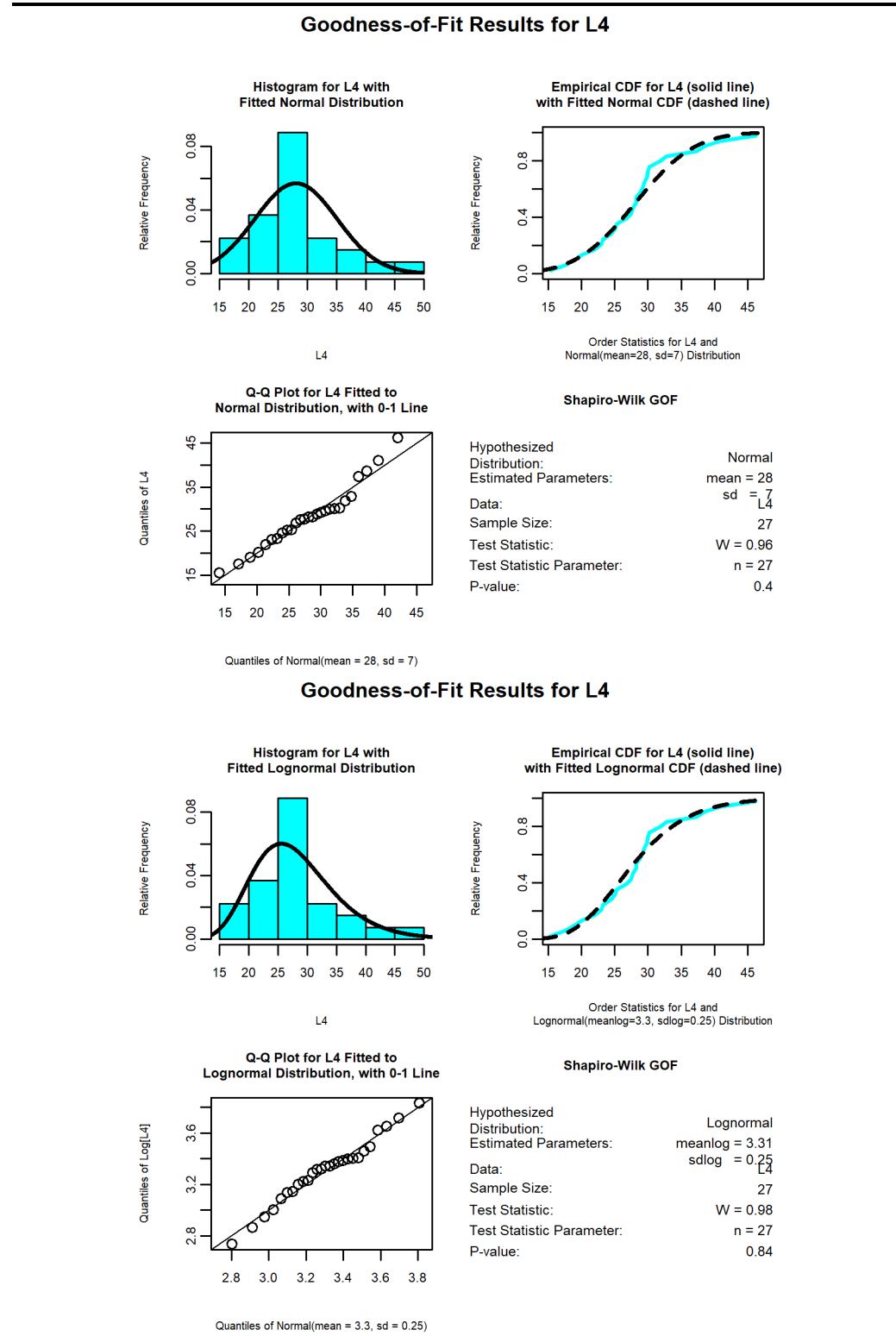


Figure 1.6 P114bis

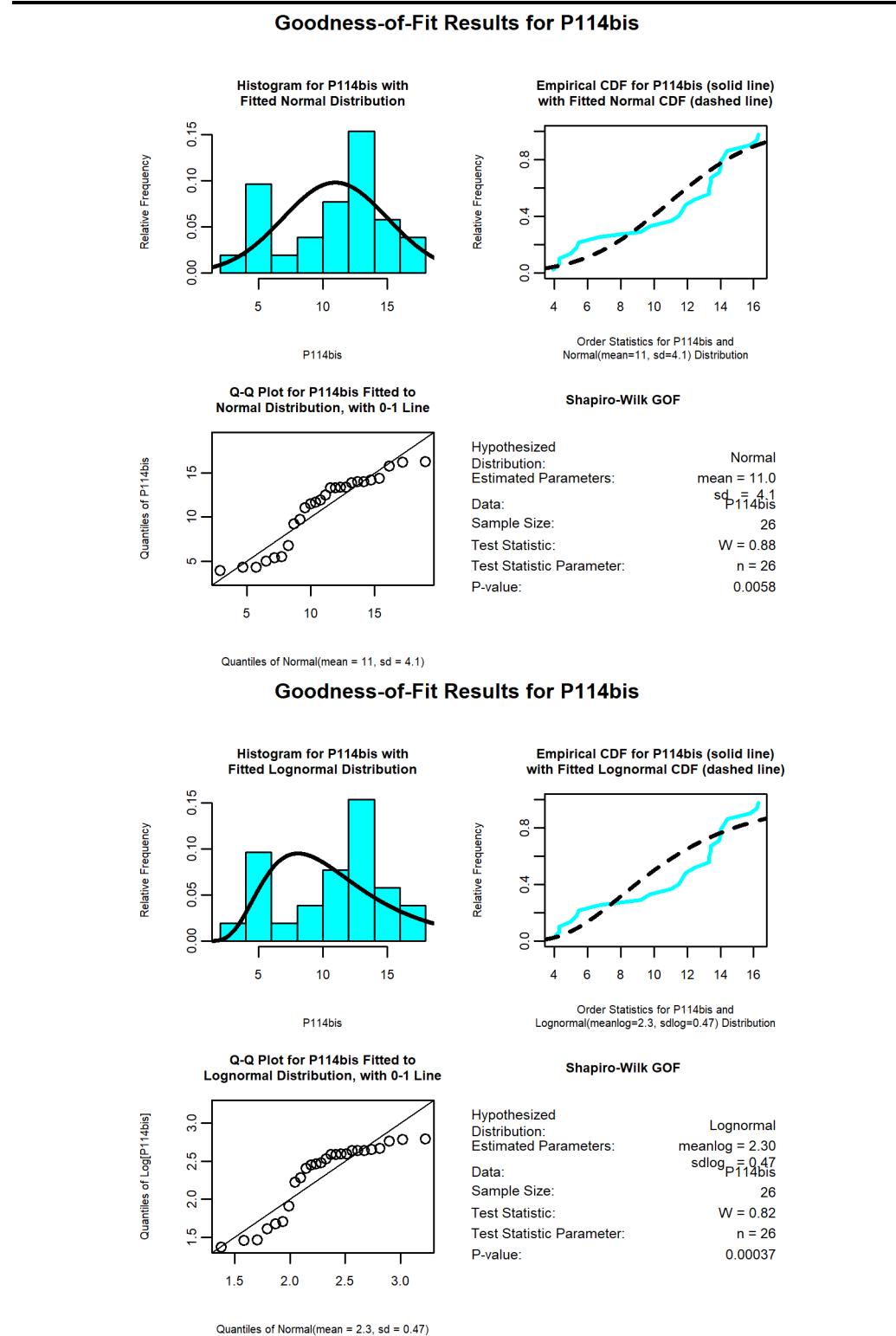


Figure 1.7 P115

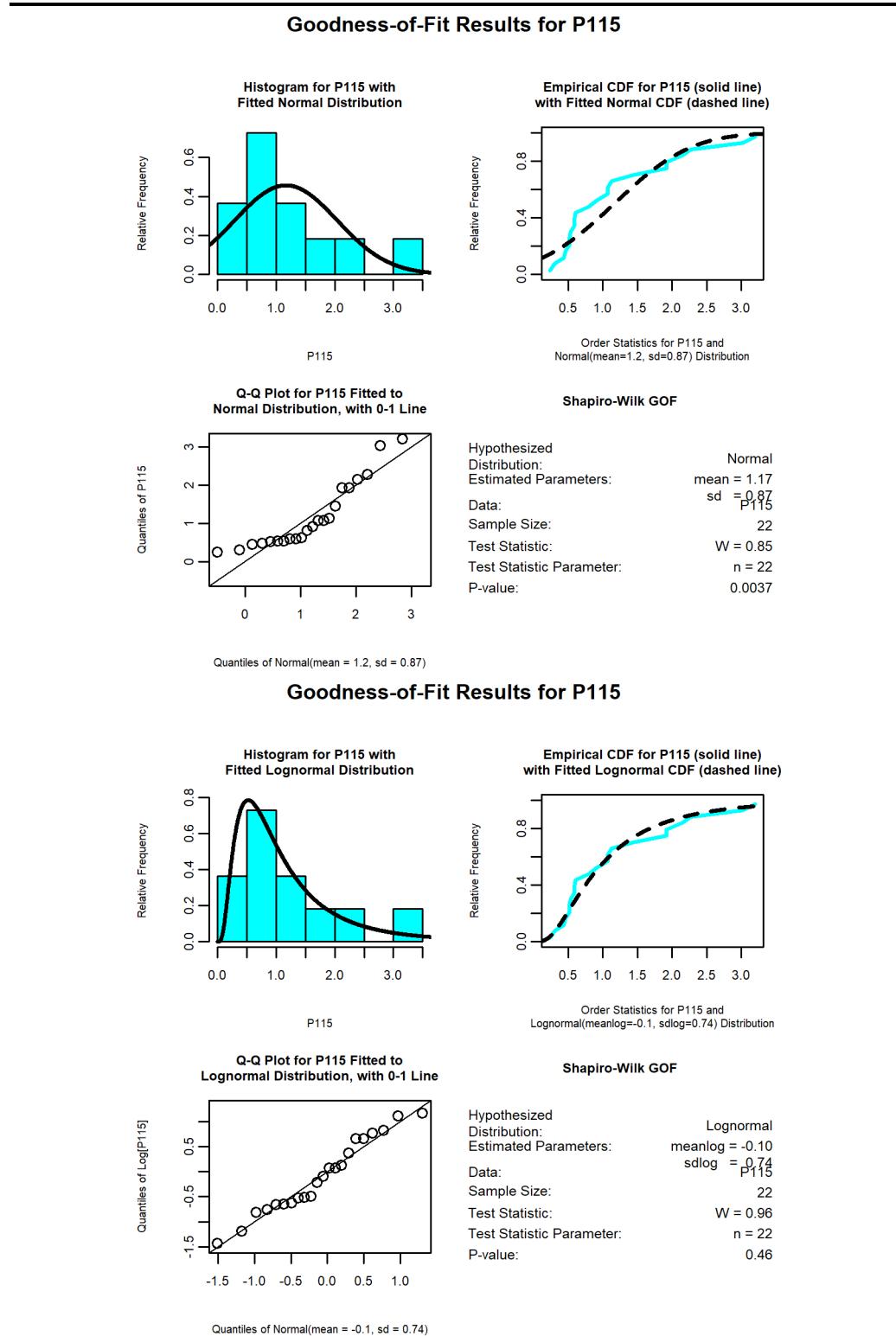


Figure 1.8 P116 (all data)

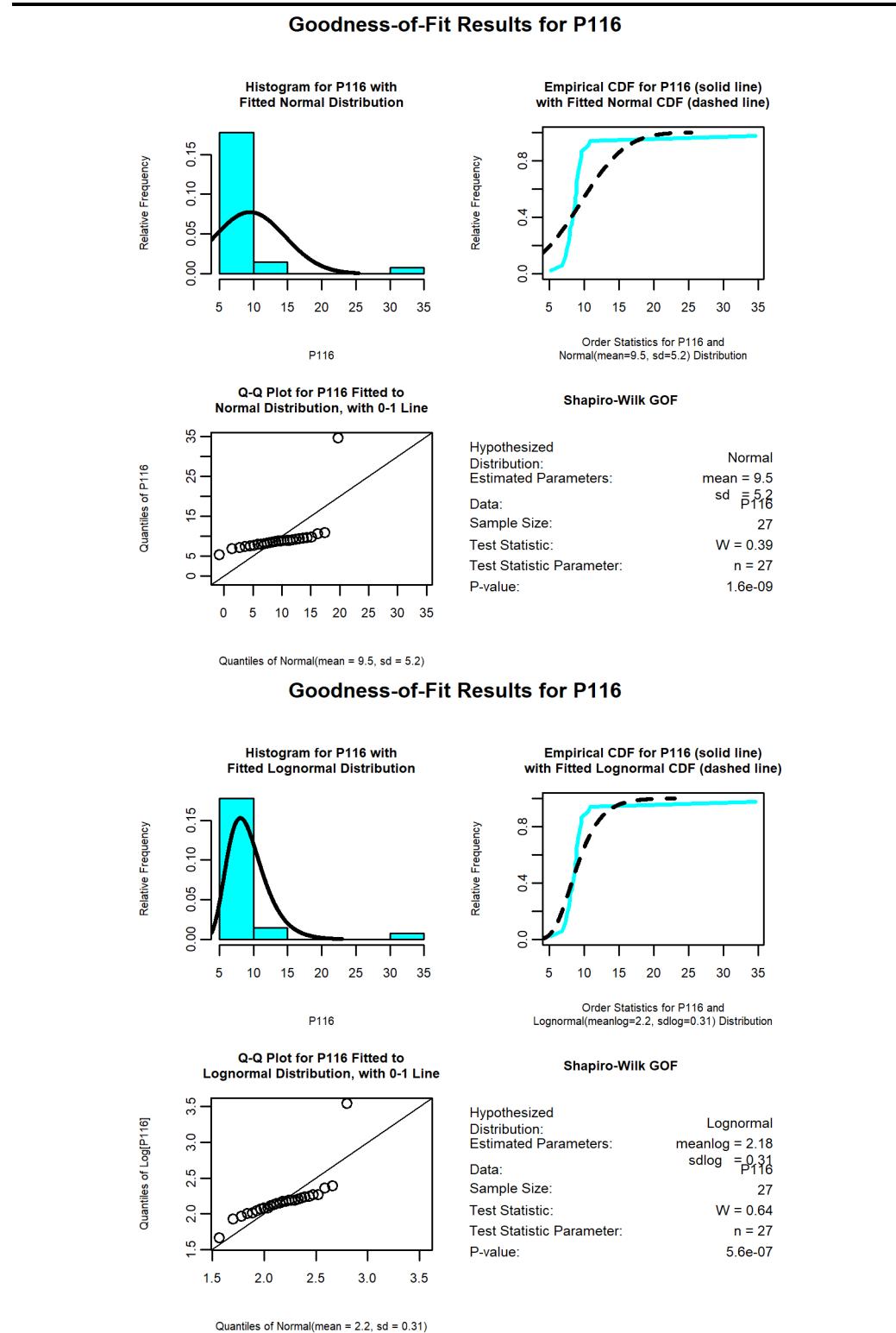


Figure 1.9 P116 (excl. outlying 2014-2015 data)

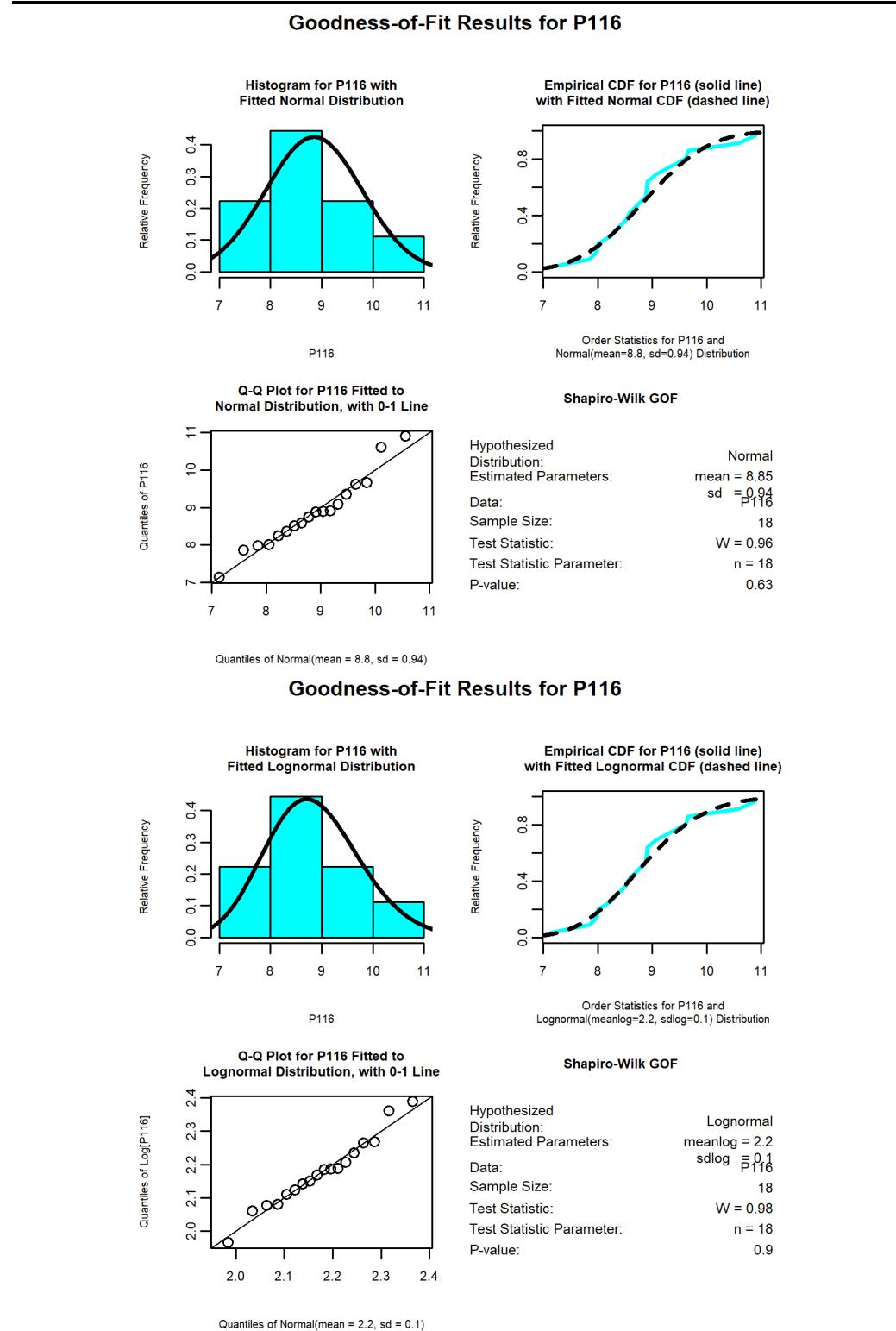


Figure 1.10 3M Vijver

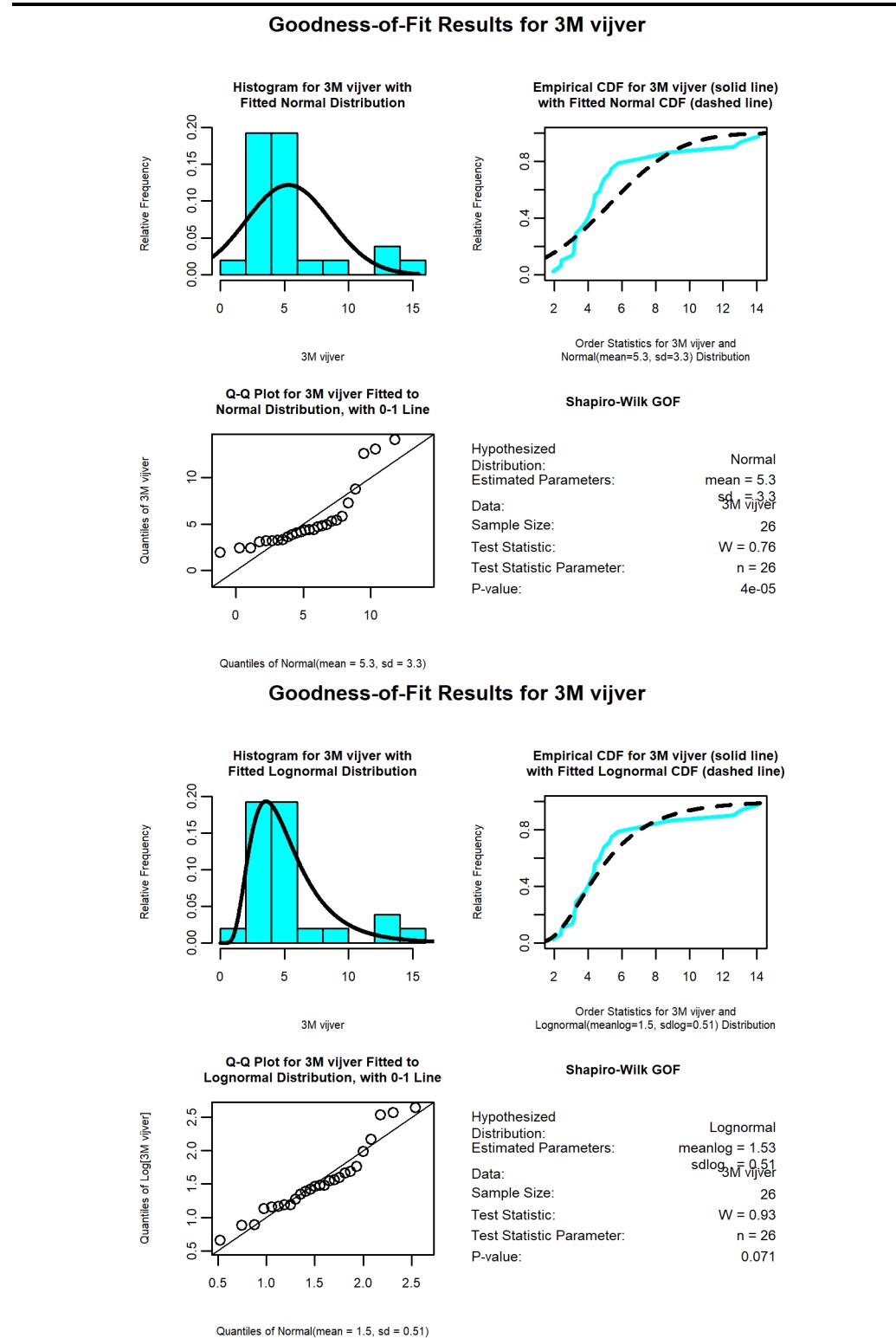


Figure 1.11 BD standaard

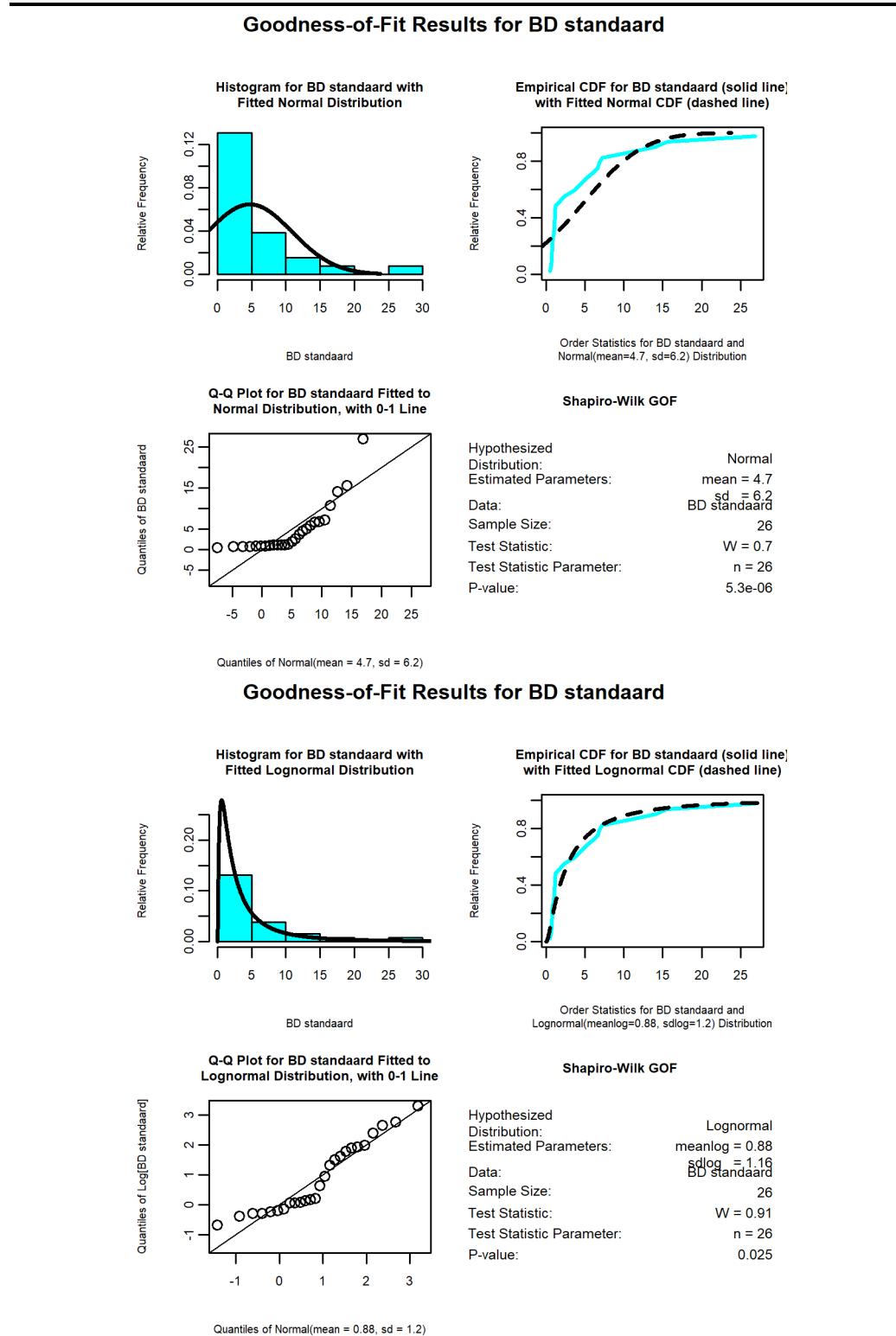
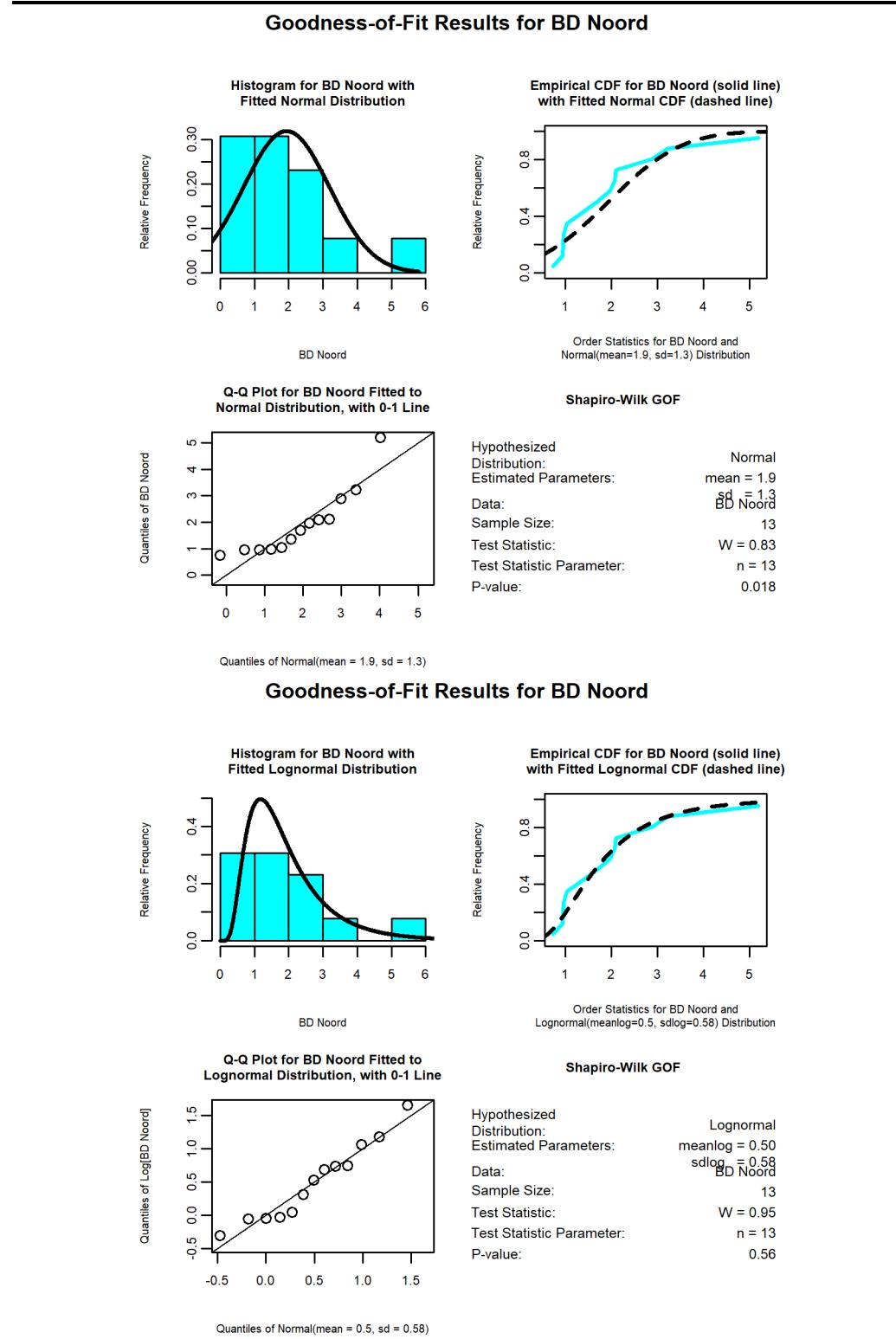


Figure 1.12 BD Noord



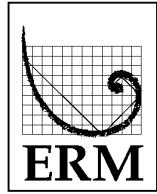
Annex 4

Results of the trend analyses

PFOS										
Well	N	Data Transformation	Normality			Distribution	Trend - OLS		Trend - Mann-Kendall	
			Test Result (SW) (p-value)	Test Result (LL) (p-value)	Correlation to linear (R)		R ²	P-value	Tau	P-value
L21	22	-	0,7018	0,6813	0,9818	Normal	0,1934	0,0405		None
L21	22	log	0,401	0,5364	0,9746					
L22	21	-	<u>0,0000</u>	<u>0,0001</u>	<u>0,7817</u>	Approximately Lognormal				None
L22	21	log	<u>0,0306</u>	0,2085	<u>0,9494</u>					
L22 - excl. outliers	16	-	0,1941	0,5739	0,9632	Normal	0,3964	<u>0,0090</u>		Down
L22 - excl. outliers	16	log	0,2938	0,3225	0,9735					
L30	2	-								
L30	2	log								
L31	23	-	0,1395	0,2649	0,9745	Normal	0,1814	<u>0,0427</u>		Down
L31	23	log	0,2399	0,1203	0,9801					
L4	27	-	0,3987	0,073	0,9785	Normal	0,1682	<u>0,0336</u>		Down
L4	27	log	0,8355	0,4049	0,9878					
P114bis	26	-	<u>0,0058</u>	<u>0,0310</u>	<u>0,9465</u>	<u>Non-parametric</u>			-0,4513	<u>0,0014</u>
P114bis	26	log	<u>0,0004</u>	<u>0,0006</u>	<u>0,9119</u>					
P115	22	-	<u>0,0037</u>	<u>0,0226</u>	<u>0,927</u>	Lognormal	0,6016	<u>0,0000</u>		Up
P115	22	log	0,4601	0,1699	0,9841					
P116	27	-	<u>0,0000</u>	<u>0,0000</u>	<u>0,6007</u>	<u>Non-parametric</u>				None
P116	27	log	<u>0,0000</u>	<u>0,0000</u>	<u>0,7744</u>					
P116 - excl. outliers	18	-	0,6278	0,4516	0,9781	Normal	0,3539	<u>0,0092</u>		Down
P116 - excl. outliers	18	log	0,9017	0,6964	0,9855					
3M vijver	26	-	<u>0,0000</u>	<u>0,0001</u>	<u>0,8713</u>	Lognormal	0,0177	0,5175		None
3M vijver	26	log	0,0706	0,1380	0,9647					
BD standaard	26	-	<u>0,0000</u>	<u>0,0003</u>	<u>0,8299</u>	<u>Non-parametric</u>			-0,1446	0,3106
BD standaard	26	log	<u>0,0245</u>	<u>0,0023</u>	<u>0,959</u>					
BD Noord	13	-	<u>0,0176</u>	0,0964	<u>0,9084</u>	Approximately Normal	0,0919	0,3139		None
BD Noord	13	log	0,5622	0,3861	0,9763					

BIJLAGE 9

GRONDHOPEN KWIK



**DAILY BOOK ENVIRONMENTAL SUPERVISION
DAGBOEK MILIEUKUNDIG TOEZICHT***

Date: 23/08/2018

Project: 0451640 – 3M Belgium

OVAM dossier: 732

Site Location	Weather
Canadastraat 11 2070 Zwijndrecht	Sun, +/- 25°C

Distribution list	
3M	Nynke De Schutter (██████████)
Weston Solutions	Dave Cairns (██████████)
ERM	Mattias Verbeeck (██████████), Erik Boeckx (██████████), Nanda Hermes (██████████), Lieselotte Sorgeloos (██████████)

Present				
<i>Name</i>	<i>Function</i>	<i>Firm</i>	<i>Hour in</i>	<i>Hour out</i>
Erik Boeckx	Project consultant	ERM	10:00	11:00

Storage and isolation soil containing Mercury:

As described in the RAP, 3M stored excavated soil containing Mercury on site near the WWTP. The excavated soil is isolated from the environment by means of a plastic liner at the bottom and over the top of the pile. This plastic liner over the top of the pile is visually checked every five year by ERM to guarantee that no polluted soil is in direct contact with the environment.

Site visit – Status cover

During the visit on August 23, 2018, photos were taken from different angles of the covered pile and the plastic liner was visually checked for damages. No damages were observed. Photos are listed below.

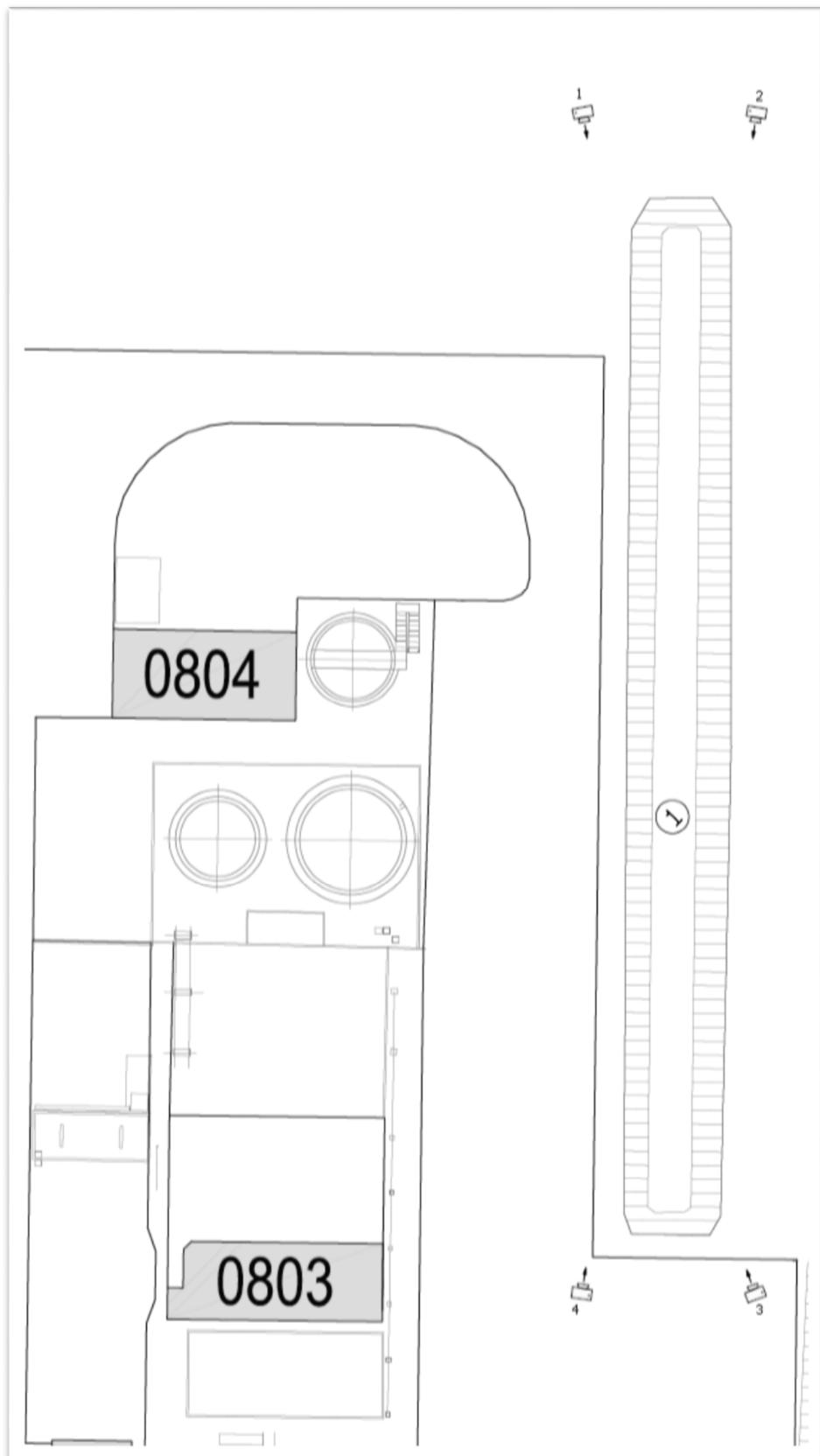
Health & Safety

Not applicable

* conform OVAM document "Bodemsaneringswerken en nazorg, standaardprocedure, september 2015"

Environmental Observation	
<u>Field observations</u>	
Not applicable	
<u>Sampling and Analyse</u>	
Not applicable	
<u>Photolog</u> Map with photo locations is added as Annex 1.	
1.	
2.	
3.	
4.	
<u>Additional comments</u>	
Not applicable	
For the Environmental Consultant <i>(bodemsaneringsdeskundige)</i> Name and Signature  Erik Boeckx	For the Client Name and Signature

Annex 1: Map with photo locations



BIJLAGE 10 SCHADEGEVAL



Evaluatierapport Schadegeval

Incident hydraulische olie – December
2018
Canadastraat 11 te 2070 Zwijndrecht

12 juni 2019

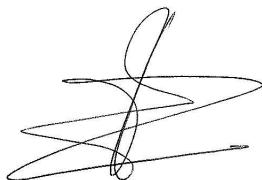
Project Nr.: R03-0458595-v1.0

Document details	De hieronder ingevoerde gegevens worden automatisch weergegeven op de omslag en op de voettekst van de hoofdpagina. LET OP: deze tabel mag NIET uit dit document worden verwijderd.
Document titel	Evaluatierapport Schadegeval
Document ondertitel	Incident hydraulische olie – December 2018 Canadastraat 11 te 2070 Zwijndrecht
Project Nr.	R03-0458595-v1.0
Datum	12 juni 2019
Versie	1.0
Geschreven door	Erik Boeckx, Kristof Bogaert
Klantnaam	3M Belgium bvba

Ondertekening

Evaluatierapport Schadegeval

Incident hydraulische olie – December 2018
Canadastraat 11 te 2070 Zwijndrecht



Dirk Nuyens
Principal Partner

ERM, Posthoflei 5 bus 6, 2600 Antwerpen-Berchem

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DEEL 1 ADMINISTRATIEVE GEGEVENS

ADMINISTRATIEVE GEGEVENS

In Tabel 0.1 zijn de administratieve gegevens van het rapport samengevat. De administratieve gegevens van het perceel waarop het incident is voorgevallen, zijn opgenomen in Tabel 0.2.

Tabel 0.1 Administratieve gegevens van het rapport

Titel:	EVALUATIERAPPORT SCHADEGEVAL Incident hydraulische olie – December 2018 Canadastraat 11 te 2070 Zwijndrecht
Referentie Erkende Bodemsaneringsdeskundige:	R03-0458595-v1.0
Rapportdatum:	12 juni 2019
Onderzoekslocatie:	3M Belgium bvba
- Risicogrond	Ja
- Dossiernummer OVAM gekend	732
- Straat + nummer of omschrijving:	Canadastraat 11
- Postcode:	2070
- Fusiegemeente:	Zwijndrecht
- Deelgemeente:	Zwijndrecht
Schadegeval:	<input type="checkbox"/> Lek in (leiding van) stookolietank, specifiek voor de verwarming van gebouw <input type="checkbox"/> Lek in (leiding van) tank, andere dan hierboven vermeld <input type="checkbox"/> Overvulling <input type="checkbox"/> Morsen <input type="checkbox"/> Ongeval <input type="checkbox"/> afspuiten hoge druk asbesthoudend dak <input type="checkbox"/> Brand met asbesthoudend materiaal <input type="checkbox"/> Vandalisme <input checked="" type="checkbox"/> Andere: Lek in hydraulische leiding ...
Naam opdrachtgever:	3M Belgium bvba
- Straat + nummer:	Hermeslaan 7
- Postcode:	1831
- Fusiegemeente:	Machelen
- Land:	België
- Telefoon:	-
- Fax:	-
- E-mail:	-
Hoedanigheid opdrachtgever:	<input checked="" type="checkbox"/> Eigenaar <input type="checkbox"/> Gebruiker <input checked="" type="checkbox"/> Exploitant <input type="checkbox"/> Optredend in opdracht van de eigenaar/gebruiker/exploitant <input type="checkbox"/> Andere:
Naam contactpersoon opdrachtgever:	Charlotte Tack
- Telefoon:	[REDACTED]

Titel:	EVALUATIERAPPORT SCHADEGEVAL Incident hydraulische olie – December 2018 Canadastraat 11 te 2070 Zwijndrecht
Referentie Erkende Bodemsaneringsdeskundige:	R03-0458595-v1.0
Rapportdatum:	12 juni 2019
- Fax:	[REDACTED]
- E-mail:	Charlotte Tack
Naam contactpersoon ter plaatse:	[REDACTED]
- Telefoon:	[REDACTED]
- Fax:	Charlotte Tack
- E-mail:	[REDACTED]
Naam bodemsaneringsdeskundige:	ERM nv
Naam contactpersoon:	Nanda Hermes
- Telefoon:	[REDACTED]
- Fax:	[REDACTED]
- E-mail:	[REDACTED]
Schadegeval ontstaan:	<input type="checkbox"/> Op particulier terrein <input type="checkbox"/> Op terrein in eigendom/beheer gemeente, autonoom gemeentebedrijf, intergemeentelijk samenwerkingsverband <input checked="" type="checkbox"/> Op een terrein waarop een inrichting gevestigd is die krachtens het decreet van 28 juni 1985 betreffende de milieuvergunning ingedeeld wordt in Klasse 1 <input type="checkbox"/> Op een grond waarop actueel een BBO of bodemsanering wordt uitgevoerd

Tabel 0.2 Identificatie van de betrokken kadastrale percelen

Toestand	Gemeentenummer	Sectie	Perceelnummer	Adres	Gemeente	Oppervlakte perceel (m ²)	Persoon (Eigenaar / gebruiker / exploitant)						Bron/Verspreiding/Onbeleid	Huidig	Toekomstig	Grondwaterkwetsbaarheid (2)				
							Periode		Type (3)	Naam	Adres	Letter(4)								
							Van	Tot												
07/06/2019	11056	A	467E	Canadastraat 11	Zwijndrecht	321 598			Ex	3M Belgium bvba	Canadastraat 11 2070 Zwijndrecht	A		V	V	Ca1/v				

(1) Bij de bestemmingstypes geeft u de code van I tot V. Als meerdere bestemmingstypes binnen het perceel vallen, geeft u alle codes.

(2) Bij de grondwaterkwetsbaarheid geeft u de correcte code. Als meerdere codes binnen het perceel vallen, geeft u de sterkste code.

(3) Bij het type voor de eigenaars en gebruikers geeft u aan of de betrokkenen eigenaar (E), gebruiker (G), beide (EG) of exploitant (Ex) is.

(4) Bij letter geeft u een letter aan de betrokken persoon. Deze letter is uniek

DEEL 2 NIET TECHNISCHE SAMENVATTING

NIET TECHNISCHE SAMENVATTING

In opdracht van 3M Belgium BVBA (3M) is door ERM nv een bodemonderzoek uitgevoerd naar aanleiding van een schadegeval op het terrein gelegen aan de Canadastraat 11 te Zwijndrecht.

Op 10 december 2018 is er een breuk geweest van een hydraulische leiding van een boorinstallatie ter hoogte van een onverhard deel van het terrein. Hierbij kwam een 10-tal liter (geraamd) hydraulische olie vrij. Het vrijgekomen product is deels opgevangen in een lekbak en deels afgegraven en opgeslagen in een cubitainer (1 m^3) na het strooien van absorberende korrels. Na de herstelling van de leiding kon de machine verplaatst worden. Op 12 december 2018 is de mogelijk geïmpacteerde grond verder afgegraven ($3 \times 3 \text{ m}$) tot een diepte van 20 cm. Er was geen zichtbare verkleuring of afwijkende geur. De vrijgekomen grond (+/- $1,5 \text{ m}^3$) is tijdelijk op een folie geplaatst. Om de mogelijke impact van de lekkage op de kwaliteit van de bodem na te gaan, zijn, na ontgraving, ter hoogte van de zone waar het schadegeval heeft plaatsgevonden ondiepe bodemstalen genomen en geanalyseerd op minerale olie. Van de putbodem is eveneens een dieper monster genomen. Er zijn eveneens reeds afperkende stalen genomen een meter buiten het ontgravingsvak om een eventuele verdere ontgraving uit te voeren indien er in de eerste serie controlemasters van de ontgraving nog verhoogde concentraties minerale olie vastgesteld worden.

Na de ontgraving van het vak van $3 \times 3 \text{ m}$ was er geen verkleuring of afwijkende geur vast te stellen.

Op 14 december 2018 heeft 3M melding gemaakt van het schadegeval aan de OVAM. In het schrijven van 20 december 2018 (ref. BB-LB2-KB-20180725215) ordeelt de OVAM dat maatregelen noodzakelijk zijn om de mogelijke bodemverontreiniging ten gevolge van het schadegeval te behandelen en dat deze maatregelen kunnen uitgevoerd worden binnen de 180 dagen.

De gemeten concentraties in het vaste deel van de aarde ter hoogte van de zone waar de hydraulische olie terechtkwam en in de afperkende boringen overschrijden de norm niet. Er zijn bijgevolg geen indicaties dat het schadegeval impact heeft gehad op de kwaliteit van de bodem.

Op basis hiervan is besloten dat het schadegeval door de beschadigde leiding de verontreinigingssituatie op de site niet significant heeft gewijzigd.

Na uitvoering van het onderzoek zoals beschreven in voorliggend evaluatierapport zijn voldoende gegevens beschikbaar om een eenduidige uitspraak te doen met betrekking tot het schadegeval in het kader van het Bodemdecreet.

De bodemsaneringsdeskundige catalogeert de voorliggende bodemverontreiniging niet als een geval van milieuschade.

Er zijn geen hiaten in het onderzoek.

Er is geen noodzaak tot het nemen van voorzogs- of veiligheidsmaatregelen.

Er is geen verdere aanpak vereist in het kader van het Bodemdecreet.

DEEL 3 RAPPORT

1. INLEIDING

Naar aanleiding van het incident dat zich heeft voorgedaan op het bedrijfsterrein van 3M Belgium bvba (3M) heeft 3M aan ERM nv (ERM) gevraagd de milieukundige begeleiding uit te voeren van de genomen maatregelen en een evaluatierapport op te maken. Het bedrijfsterrein van 3M is gelegen aan de Canadastraat 11 te 2070 Zwijndrecht en wordt ingedeeld als een klasse 1 inrichting.

Het incident werd vast gesteld en vond plaats op 10 december 2018 op een terrein ten noordoosten van gebouw 016. Daar heeft zich een lek voorgedaan in een leiding van een boorinstallatie. Hierbij is een tiental liter hydraulische olie vrijgekomen op een onverharde zone. Het vrijgekomen product is deels opgevangen in een lekbak en deels afgegraven en opgeslagen in een cubitainer (1m³) na het strooien van absorberende korrels. Na de herstelling van de leiding op 11 december 2018, kon de machine verplaatst worden. Op 12 december 2018 is de mogelijk geïmpacteerde grond verder afgegraven (3 x 3 m) tot een diepte van 20 cm.

Het incident is op 14 december 2018 gemeld aan de OVAM en de milieu-inspectie (melding aan OVAM dd. 14-12-2018; OVAM reactie dd. 20-12-2018 met referentie BB-LB2-KB-20180725215). De melding is opgenomen Bijlage 1. Het besluit van OVAM is opgenomen in Bijlage 9.

Het incident is behandeld in overeenstemming met Hoofdstuk VI, Afdeling VI van het Bodemdecreet. Tevens is bij de behandeling van het incident rekening gehouden met de "Richtlijn schadegevallen en evaluatierapport" (juni 2018) van OVAM.

Op de onderzoekslocatie bevindt zich één vergunde grondwaterwinning. Binnen een straal van 500 m rond de terreingrens bevinden zich vier vergunde grondwaterwinningen. Er bevinden zich geen waterwingebieden of beschermingszones binnen een straal van 2 km rond de terreingrens. Een overzicht van deze waterwinning is weergegeven in Bijlage 10. Het freatisch grondwater bevindt zich op een diepte van circa 2,5 m-mv. Door de beperkte omvang van het incident, een oppervlakkige lekkage van hydraulische olie en tijdig uitgevoerde maatregelen, wordt staalname van het grondwater niet nodig geacht.

Voorliggend evaluatierapport geeft een overzicht van de maatregelen die genomen zijn naar aanleiding van het incident en de resultaten van deze maatregelen.

2. BESCHRIJVING VAN HET INCIDENT EN DE UITGEVOERDE MAATREGELEN

2.1 Beschrijving Incident

Op 10 december 2018 heeft zich een incident voorgedaan op het terrein van 3M, gelegen aan de Canadastraat 11 te 2070 Zwijndrecht. De ligging van het 3M terrein is aangeduid op een topografische kaart opgenomen in Bijlage 2. Een plan van het terrein is opgenomen in Bijlage 3. Een detailplan van de zone van het incident is weergegeven in Bijlage 4.

Tijdens de installatie van een paalfundering is er een breuk geweest in een drukleiding van de boorinstallatie ten noordoosten van gebouw 016. Het gespilde product betrof hydraulische olie.

Door 3M is geraamd dat ongeveer een tiental liter hydraulische olie is vrijgekomen op een onverharde ondergrond.

Figuur 2.1 Locatie incident



Het vrijgekomen product is deels opgevangen in een lekbak en deels meteen afgegraven en opgeslagen in een cubitainer (1 m^3) na het strooien van absorberende korrels. Na de herstelling van de leiding op 11 december 2018 kon de machine verplaatst worden.

Figuur 2.2 Zone incident



Het incident is op 14 december 2018 (binnen de 30 dagen) gemeld aan OVAM. De melding is opgenomen in Bijlage 1. De product informatie en hydraulische olie is opgenomen in Bijlage 5.

2.2 Beschrijving genomen maatregelen

Na de herstelling van de leiding kon de machine verplaatst worden en is uit voorzorg op 12 december 2018 gestart met het selectief ontgraven van de zone waar het product is terechtgekomen (zie Figuur 2.3).

Figuur 2.3 Afgegraven zone



ERM heeft als erkend bodemdeskundige de graafwerken opgevolgd. De mogelijk geïmpacteerde zone is afgegraven (3 x 3m) tot een diepte van 30 cm. De afgegraven grond is tijdelijk op een waterdichte folie gelegd om verdere verspreiding van verontreiniging naar grond en grondwater te voorkomen (zie Figuur 2.4).

Figuur 2.4 Tijdelijke stockage grond



Er zijn stalen genomen na de ontgraving, van de bodem van de ontgravingsput (CS-B1), monsters van de wanden (CS-W) een een aantal preventieve ondiepe stalen van de bodem 1 meter buiten het ontgravingsvak. De boorwerkzaamheden en bemonsteringen zijn uitgevoerd door ERM conform de meest recente CMA-procedures. De stalen zijn genomen met gebruik van een edelmanboor. De samenstelling van het opgeboorde materiaal betreft schelpen houdend, grof, lichtbruin zand.

Het milieukundig dagboek dat opgemaakt is door ERM, is opgenomen in Bijlage 6.

De locatie van het incident, de vermoedelijke zone waar het product is terechtgekomen, de ontgravingszone en de locatie van de controlestalen, zijn aangeduid op het detailplan opgenomen in Bijlage 4. Een overzicht van de bijhorende Lambert coördinaten, lithologie en organoleptische waarnemingen is weergeven in **Error! Reference source not found.**

3. RESULTATEN VELD- EN LABORATORIUMONDERZOEK

Voor de start van de graafwerken en tijdens de graafwerken is organoleptisch geen verontreiniging vastgesteld.

De stalen van de bodem en wanden van de ontgravingsput ((CS-B1 (0-20), CS-B1 (50-70), CS-B1 (100-120), CS-W1A, CS-W2A)) zijn door het erkende laboratorium Al-West B.V. geanalyseerd op droge stof en minerale olie. Het originele analyseverslag is opgenomen in Bijlage 7, de resultaten zijn weergegeven in onderstaande tabel.

Tabel 3.1 Analyseresultaten Al-West B.V.

Staalaanduiding en diepte in cm-mv	Datum	Perceel	Minerale Olie (mg/kg.ds)
staal bodem van de ontgravingsput: CS-B1 (0-20)	12/12/2018	467E	71
staal bodem van de ontgravingsput: CS-B1 (50-70)	12/12/2018	467E	58
staal bodem van de ontgravingsput: CS-B1 (100-120)	12/12/2018	467E	<50
staal wand van de ontgravingsput: CS-W1A	12/12/2018	467E	<50
staal wand van de ontgravingsput: CS-W2A	12/12/2018	467E	<50

4. SANERINGSDOELSTELLING

Voor de bepaling van de saneringsdoelstelling is door ERM de richtwaarde als toetsingswaarde voor een standaardbodem en industrieel bodemgebruik gehanteerd: voor minerale olie wordt de richtwaarde van 300 mg/kg.ds gehanteerd.

De concentratie minerale olie gemeten in de stalen genomen van de bodem en wanden van de ontgravingsput overschrijden de richtwaarde niet (zie Tabel 4.1).

Tabel 4.1 Toetsing analyseresultaten

Staalaanduiding en diepte in cm-mv	Datum	Perceel	MO (mg/kg.ds)	Richtwaarde (300 mg/kg.ds)
bodem van de ontgravingsput CS-B1 (0-20):	12/12/2018	467E	71	-
bodem van de ontgravingsput: CS-B1 (50-70):	12/12/2018	467E	58	-
bodem van de ontgravingsput: CS-B1 (100-120):	12/12/2018	467E	<50	-
wand van de ontgravingsput: CS-W1A:	12/12/2018	467E	<50	-
wand van de ontgravingsput: CS-W2A :	12/12/2018	467E	<50	-

(1) -- : geen overschrijding

5. EVALUATIE RESULTATEN

Uit de analyseresultaten van de controlestalen van de bodem en de wanden van de ontgravingsput blijkt dat er na de schadebeperkende maatregelen geen concentraties minerale olie in de grond aanwezig zijn die hoger zijn dan de richtwaarde. In het diepe controlemonster onder de bodem van de ontgravingsput benadert de gemeten concentratie minerale olie de streefwaarde (50 mg/ kg ds). Gelet op het oppervlakkige karakter van de ontgraving en de gunstige resultaten in het diepe controlemonster is de uitvoering van grondwateronderzoek niet noodzakelijk geacht.

Aangezien in de controlestalen van de ontgravingsput geen overschrijdingen van de richtwaarde zijn gemeten, kan besloten worden dat de doelstelling van de schadebeperkende maatregelen gehaald is. Bijgevolg is geoordeeld dat er geen verdere maatregelen of een beschrijvend bodemonderzoek nodig zijn.

Er zijn voldoende gegevens aanwezig om een eenduidige uitspraak te doen in het kader van het Bodemdecreet. Er zijn geen hiaten in het onderzoek die aanleiding kunnen geven tot een ander besluit. Er zijn geen problemen opgetreden bij het nemen van de controlestalen. Er is geen noodzaak tot het nemen van voorzorgs- of veiligheidsmaatregelen.

5.1 Evaluatie van de verzamelde gegevens per kadastral perceel

Tabel 5.1 Samenvatting van de verontreinigingstoestand ter hoogte van het incident met hydraulische olie

Perceel	Identificatienummer verontreiniging ⁽¹⁾	Medium ⁽²⁾	Restverontreiniging	Beoordeling	Bron/Verspreiding
467E	niet van toepassing	vaste deel van de aarde	Nee	O ⁽³⁾	B

(1) Uniek nummer voor de aanduiding van de verontreiniging.

(2) Vaste deel van de aarde, Grondwater of Puur product (drijflaag of zaklaag).

(3) Voor geen enkele genormeerde parameter werd de richtwaarde voor het vaste deel van de aarde overschreden.

5.2 Verklaring van de erkende bodemsaneringsdeskundige

De afgegraven grond (5,22 ton) en absorberende korrels zijn afgevoerd naar de erkende verwerker Indaver. Het verwerkingscertificaat is bijgevoegd in Bijlage 11.

6. BESLUIT

Naar aanleiding van het incident dat zich op 10 december 2018 heeft voorgedaan op het bedrijfsterrein van 3M te Zwijndrecht, heeft ERM in opdracht van 3M de milieukundige begeleiding uitgevoerd van de genomen maatregelen en een evaluatierapport opgemaakt van het incident.

Samengevat kunnen we stellen dat:

- Op 10 december 2018 heeft zich een schadegeval voorgedaan;
- Er zijn schadebeperkende maatregelen getroffen waarbij verontreinigde grond is ontgraven;
- Nadien zijn controlestalen genomen die voldeden aan de richtwaarde;
- De schadebeperkende maatregelen zijn in voldoende mate uitgevoerd; en
- Er is geen aanleiding voor bijkomend grondwateronderzoek.

Uit onderhavig evaluatierapport volgt dat er geen bodemverontreiniging tot stand is gekomen als gevolg van het incident op 10 december 2018 en er geen verdere maatregelen noodzakelijk zijn.

Er zijn voldoende gegevens aanwezig om een eenduidige uitspraak te doen in het kader van het Bodemdecreet. Er zijn geen hiaten in het onderzoek die aanleiding kunnen geven tot een ander besluit.

6.1 Besluit kadastraal perceel 467 E

Na de uitvoering van de maatregelen ten gevolge van het schadegeval is er geen reden meer om aan te nemen dat er ten noordoosten van gebouw 016 nog een verontreiniging met hydraulische olie aanwezig is die de richtwaarde overschrijdt. Er is geen noodzaak tot een beschrijvend bodemonderzoek.

Uit dit evaluatierapport is gebleken dat na de maatregelen, genomen in het kader van de zorgvuldigheidsplicht, er geen bodemverontreiniging tot stand is gekomen en er geen verdere maatregelen noodzakelijk zijn.

O-zin:

Na analyses van de stalen is er geen reden om aan te nemen dat er als gevolg van het bewuste schadegeval nog een bodemverontreiniging aanwezig is op dit perceel.

Er dienen geen voorzogs- of veiligheidsmaatregelen getroffen te worden.

De bodemsaneringsdeskundige catalogeert de voorliggende bodemverontreiniging niet als een geval van milieuschade⁽¹⁾.

¹ Milieuschade is schade zoals gedefinieerd in artikel 15.1.1, 1° van titel XV van het Decreet Algemene Bepalingen Milieubeleid (DABM) van 5 april 1995, zijnde schade die:

1. veroorzaakt is door een emissie, een gebeurtenis of een incident die/dat heeft plaatsgevonden ná 30 april 2007;
2. door een inrichting of installatie die vermeld wordt in bijlage IV van het DABM; en
3. die de bodemsaneringsnorm overschrijdt of dreigt te overschrijden.

7. VERKLARING EN ONDERTEKENING

De bodemsaneringsdeskundige verklaart hierbij dat het voorliggende rapport representatief is voor de verontreinigingstoestand van de onderzoekslocatie (spill hydraulische olie, ten noordoosten van gebouw 16). Tevens verklaart de bodemsaneringsdeskundige dat de meegestuurde digitale gegevens overeenstemmen met de inhoud van het rapport.

Daarnaast verklaart de bodemsaneringsdeskundige dat alle analyses werden uitgevoerd door een daartoe erkend laboratorium, dat de resultaten van alle uitgevoerde analyses zijn opgenomen in het bodemonderzoek en dat analyseresultaten opgenomen in het bodemonderzoek identiek zijn aan de analyseresultaten die zijn aangeleverd door het erkend laboratorium.

De bodemsaneringsdeskundige verklaart dat hij voor het uitvoeren van deze opdracht niet verkeert in een van de gevallen van onverenigbaarheid zoals bepaald in artikel 53/5 van het VLAREL.

Tabel 7.1 Ondertekeningstabel

Hoedanigheid	Naam + functie	Handtekening
Naam van de persoon die beschikt over de individuele handtekeningsbevoegdheid (artikel 53/4 §1, eerste lid van het Vlarel):	Nanda Hermes	
Naam van de Kwaliteitsverantwoordelijke bij de bodemsaneringsdeskundige voor dit bodemonderzoekt:	Dirk Nuyens	
Naam van de persoon die de bodemsaneringsdeskundige rechtsgeldig kan vertegenwoordigen tegenover derden:	Dirk Nuyens	

DEEL 4 BIJLAGEN

BIJLAGE 1

**MELDINGSFORMULIER SCHADEGEVAL BIJ EEN KLASSE
1 INRICHTING**

Melding van de vaststelling van bodemverontreiniging

SAMEN MAKEN WE

MORGEN MOOIER

OVAM

Openbare Vlaamse Afvalstoffenmaatschappij

Schadegevallen

Stationsstraat 110, 2800 MECHELEN

T 015 284 488

www.ovam.be

schade@ovam.be

Waarvoor dient dit formulier?

Met dit formulier kunt u aan de OVAM melden dat er een geval van bodemverontreiniging werd vastgesteld.

Meer informatie vindt u op www.ovam.be/schadegevallen.

Het meldingsformulier wordt bij voorkeur via schade@ovam.be aan de OVAM overgemaakt. Opsturen met de post blijft uiteraard ook mogelijk (zie onze contactgegevens bovenaan dit formulier).

Identiteit van de melder of vaststeller

1 Vul hieronder uw persoonlijke gegevens in.

(bedrijfs)naam 3M Belgium

voor- en achternaam Nynke De Schutter

straat en nummer Canadastraat 11

postnummer en gemeente 2070 Zwijndrecht

telefoonnummer [REDACTED]

e-mailadres [REDACTED]

- | | | |
|---|--|------------------------------------|
| <input type="checkbox"/> eigenaar | <input type="checkbox"/> exploitant | <input type="checkbox"/> gebruiker |
| <input type="checkbox"/> schadelijder | <input type="checkbox"/> politie | <input type="checkbox"/> gemeente |
| <input type="checkbox"/> bodemsaneringsdeskundige | <input type="checkbox"/> verzekeraar of expert | |
| <input type="checkbox"/> milieucoördinator | <input checked="" type="checkbox"/> andere: | EHS Engineer 3M Zwijndrecht |

2 Wanneer is de bodemverontreiniging ontstaan?

datum dag 10 maand 12 jaar 2018

onbekend

3 Wanneer werd de bodemverontreiniging vastgesteld?

datum dag 10 maand 12 jaar 2018



Vlaamse
overheid

4 Kruis aan welk product er vrijgekomen is.

- | | | | |
|--|---|------------------------------------|---------------------------------------|
| <input type="checkbox"/> benzine | <input type="checkbox"/> diesel | <input type="checkbox"/> stookolie | <input type="checkbox"/> asbest |
| <input type="checkbox"/> solventen | <input type="checkbox"/> smeermiddelen | <input type="checkbox"/> zuren | <input type="checkbox"/> drugs(afval) |
| <input type="checkbox"/> pcb-houdende olie | <input checked="" type="checkbox"/> ander product, namelijk: Hydraulische olie | | |

5 Is de grond waar de bodemverontreiniging is ontstaan (het bronperceel), gekend?

Let op: hier wordt een onderscheid gemaakt tussen de situaties waar het bronperceel gekend is en de situaties waar het bronperceel niet gekend is.

Wanneer het **bronperceel gekend** is, vult u de **vragen 6 tot en met 11** in.

Wanneer het **bronperceel niet gekend** is, vult u de vragen **12 tot en met 18** in.

ja. *Ga naar vraag 6.*

nee. *Ga direct naar vraag 12.*

Identificatie van het bronperceel

A Het bronperceel is gekend: vragen 6 tot en met 11

6 Het terrein betreft:

- Een particulier terrein
 Een terrein waarop een inrichting gevestigd is, vallende onder klasse 1
 Een terrein waarop een inrichting gevestigd is, vallende onder klasse 2 of klasse 3
 Een terrein in eigendom of beheer van een gemeente, autonoom gemeentebedrijf of intergemeentelijk samenwerkingsverband

7 Vul de adresgegevens van het bronperceel in.

straat en nummer Canadastraat 11

postnummer en gemeente 2070 Zwijndrecht

kadastrale gegevens afdeling 1

sectie A

grondnummer 467E

8 Vul de contactgegevens van het bronperceel in.

naam van de **exploitant** 3M Belgium bvba

adres van de exploitant Hermeslaan 7

1831 Diegem

telefoonnummer:

naam van de **gebruiker**

adres van de gebruiker

telefoonnummer:

naam van de eigenaar 3M Belgium bvba

adres van de eigenaar Hermeslaan 7

1831 Diegem

telefoonnummer:

9 Veroorzaakt de bodemverontreiniging van het bronperceel ook verontreiniging op de buurtpercelen?

- Nee. Ga naar vraag 11.

10 Vul de gegevens van de verontreinigde buurtpercelen in.

Naam en adres van de eigenaar	Adres verspreiding bodemverontreiniging	Kadastrale gegevens		
		afdeling	sectie	grondnr.

11 Kruis de oorzaak van de bodemverontreiniging aan.

- | | | | |
|--|---|--|--------------------------------------|
| <input type="checkbox"/> lek in leiding tank voor verwarming gebouw | <input type="checkbox"/> lek in bovengr. tank dat gebruikt wordt/werd voor verwarming gebouw | <input type="checkbox"/> afspuiten hoge druk asbesthoudend dak | <input type="checkbox"/> overvulling |
| <input type="checkbox"/> lek in leiding andere dan hierboven vermeld | <input type="checkbox"/> lek in ondergr. tank dat gebruikt wordt/werd voor verwarming gebouw | <input type="checkbox"/> brand met asbesthoudend materiaal | <input type="checkbox"/> lek |
| <input type="checkbox"/> ongeval met voertuig | <input type="checkbox"/> lek in tank andere dan hierboven vermeld | <input type="checkbox"/> breuk in afvoer | <input type="checkbox"/> morsen |
| <input type="checkbox"/> vandalisme | <input checked="" type="checkbox"/> andere oorzaak, namelijk: Slang met hydraulische olie van boorinstallatie losgekomen. | | |

B Het bronperceel is niet gekend: vragen 12 tot en met 18

12 Vul de adresgegevens in van het perceel waar de verontreiniging het eerst werd vastgesteld.

straat en nummer

postnummer en gemeente

kadastrale gegevens afdeling

sectie

grondnummer

13 Vul de eigenaarsgegevens in van het perceel waar de verontreiniging het eerst werd vastgesteld.

naam van de **exploitant**

adres van de exploitant

telefoonnummer:

naam van de gebruiker

adres van de gebruiker

telefoonnummer:

naam van de eigenaar

adres van de eigenaar

telefoonnummer:

14 Is de bodemverontreiniging ook vastgesteld op buurtparcelen?

- Ja. Ga naar vraag 15.
 - nee. Ga naar vraag 16.

15 Vul de gegevens van de buurtpercelen in.

Naam en adres van de eigenaar	Adres bodemverontreiniging vastgesteld	Kadastrale gegevens		
		afdeling	sectie	grondnr.

16 Welke onderzoeken of inspecties werden op de percelen uitgevoerd?

- een buurtonderzoek
 - een rioolinspectie

17 Zijn er in de buurt potentiële bronnen voor de bodemverontreiniging?

- Ja. Ga naar vraag 18.
 - Nee.

18 Vul de gegevens in van de locaties waar zich potentiële bronnen voor de bodemverontreiniging bevinden.

Een voorbeeld van een potentiële bron is een boven- of ondergrondse tank op de locatie.

naam van de eigenaar	adres	Kadastrale gegevens		
		afdeling	sectie	grondnr.

Genomen maatregelen tegen de bodemverontreiniging

- | | |
|---|---|
| <input checked="" type="checkbox"/> de verontreinigde grond werd afgegraven | <input type="checkbox"/> er werden absorberende korrels gestrooid |
| <input checked="" type="checkbox"/> de bron of het lek werd gestopt | <input type="checkbox"/> de drijflaag werd afgezogen |
| <input type="checkbox"/> er was een interventie door de brandweer of de politie | <input checked="" type="checkbox"/> er werden stalen genomen en analyses uitgevoerd |
| <input type="checkbox"/> de verzekерingsmaatschappij werd ingelicht | <input type="checkbox"/> er werd puur product weggenomen |
| <input checked="" type="checkbox"/> er werden andere maatregelen genomen, namelijk:

 | Meteen na het vaststellen van de losgekomen leiding met hydraulische olie (op 10/12/2018), werd absorptiemateriaal en een opvangbak onder de lek aangebracht. Bovendien werd zo snel mogelijk de mogelijks geïmpacteerde grond ontgraven en opgevangen in een container. Op 12/12/2018 werden controlestalen genomen door de bodemsaneringsdeskundige en naar een door OVAM erkend laboratorium gestuurd voor analyse. In functie van deze resultaten zullen de eventueel verder te nemen acties met 3M en OVAM worden besproken. |
| <input checked="" type="checkbox"/> er werd een bodemdeskundige aangesteld, namelijk: | <input type="checkbox"/> Environmental Resources Management nv – ERM nv |

Ondertekening

20 Vul de onderstaande verklaring in.

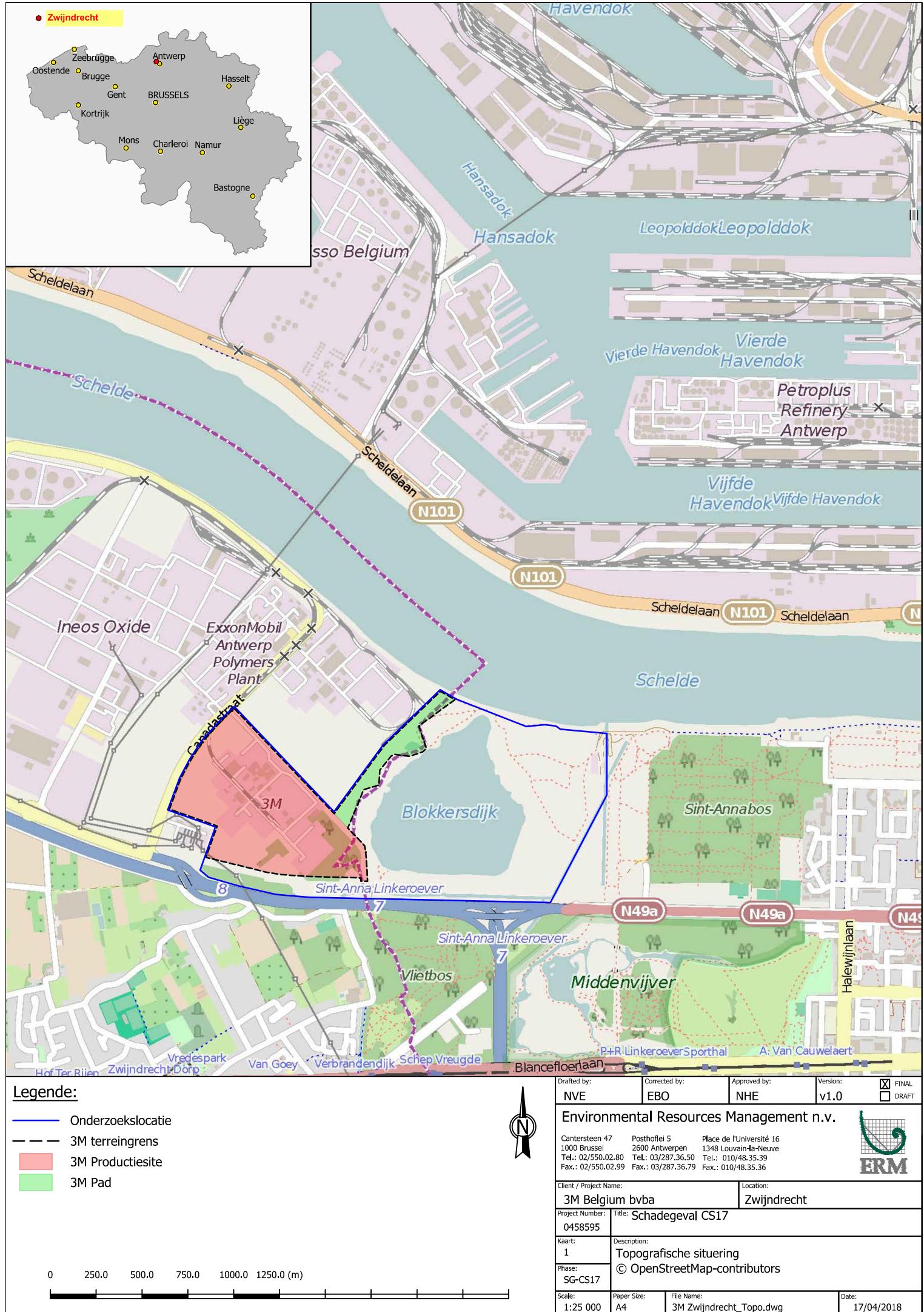
Ik verklaar dat alle gegevens in deze melding naar waarheid zijn ingevuld.

datum dag maand jaar

handtekening

BIJLAGE 2

SITUERING VAN DE ONDERZOEKSLOCATIE



BIJLAGE 3

OVERZICHT VAN DE ONDERZOEKSLOCATIE



Onderzoekslocatie

Legende:

3M terreingrens

Kadastrale grens

267v2 Kadastrale nummer

Drafted by: NVE Corrected by: EBO Approved by: NHE Version: v1.0 FINAL DRAFT

Environmental Resources Management n.v.

Cantersteen 47 Posthoflei 5 Place de l'Université 16
1000 Brussel 2600 Antwerpen 1348 Louvain-la-Neuve
Tel.: 02/550.02.80 Tel.: 03/287.36.59 Tel.: 010/48.35.39
Fax.: 02/550.02.99 Fax.: 03/287.36.79 Fax.: 010/48.35.36



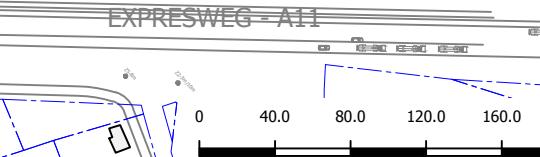
Client / Project Name: 3M Belgium bvba Location: Zwijndrecht

Project Number: 0458595 Title: Schadegeval CS17

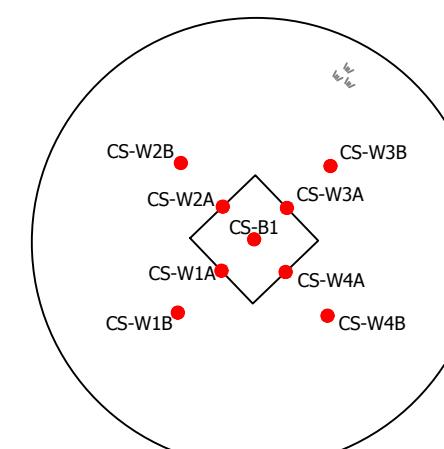
Kaart: 2 Description: Overzichtsplan

Phase: SG-CS17

Scale: 3" = 1'-0" Paper Size: KREF File Name: 3M Zwijndrecht.dwg Date: 15/02/2019



BIJLAGE 4 DETAILPLAN VAN DE ZONE VAN HET INCIDENT



CS-W2B
CS-W2A
CS-B1
CS-W1A
CS-W1B
CS-W3A
CS-W4A
CS-W4B
CS-W3B

Legende:

- 3M terreingrens
- Kadastrale grens
- 267v2 Kadastrale nummer

Drafted by: NVE Corrected by: EBO Approved by: NHE Version: v1.0 FINAL DRAFT

Environmental Resources Management n.v.

Cantersteen 47 Posthoflei 5 Place de l'Université 16
1000 Brussel 2600 Antwerpen 1348 Louvain-la-Neuve
Tel.: 02/550.02.80 Tel.: 03/287.36.50 Tel.: 010/48.35.39
Fax.: 02/550.02.99 Fax.: 03/287.36.79 Fax.: 010/48.35.36



Client / Project Name: 3M Belgium bvba Location: Zwijndrecht

Project Number: 0458595 Title: Schadegeval CS17

Kaart: 3 Description: Detailplan met aanduiding van het ontgravingsvak en Staalnamepunten

Phase: SG-CS17

Scale: 1:250 Paper Size: A3 File Name: 3M Zwijndrecht.dwg Date: 15/02/2019

1798-A-01
1798-A-02

0 2.5 5.0 7.5 10.0 12.5 (m)

BIJLAGE 5

PRODUCT INFORMATIE FICHE

HF-95 hydraulische olie

Veiligheidsinformatieblad

volgens Verordening (EG) nr. 453/2010

Datum van uitgifte: 02/03/2004 datum herziening: 10/02/2015 vervangt: 24/02/2012

versie: 4.0

RUBRIEK 1: Identificatie van de stof of het mengsel en van de vennootschap/onderneming

1.1. Productidentificatie

Soort product	:	Mengeling
Productnaam	:	HF-95 hydraulische olie
Productgroep	:	Mengsel

1.2. Relevant geïdentificeerd gebruik van de stof of het mengsel en ontraden gebruik

1.2.1. Relevant geïdentificeerd gebruik

Hoofdgebruikscategorie	:	Industrieel gebruik, beroepsmatig gebruik en consumentengebruik
Specificatie industrieel/beroepsmatig gebruik	:	Niet-dispersief gebruik, gebruik in gesloten systemen
Functie of gebruikscategorie	:	Smeermiddelen en additieven

1.2.2. Ontraden gebruik

Er is geen aanvullende informatie beschikbaar.

1.3. Details betreffende de verstrekker van het veiligheidsinformatieblad

Enerpac B.V.
Postbus 8097, 6710 AB Ede
NEDERLAND
Tel: +31(0)31-853 59 11

1.4. Telefoonnummer voor noodgevallen

Nummer voor noodgevallen	:	+32 70 245 245
--------------------------	---	----------------

RUBRIEK 2: Identificatie van de gevaren

2.1. Indeling van de stof of het mengsel

Indeling overeenkomstig Verordening (EG) nr. 1272/2008 (CLP)
Niet ingedeeld

Indeling overeenkomstig Richtlijn 67/548/EEG of 1999/45/EG
Niet ingedeeld

Nadelige fysisch-chemische effecten, gezondheids- en milieueffecten
Er is geen aanvullende informatie beschikbaar.

2.2. Etiketteringselementen

Etikettering overeenkomstig Verordening (EG) nr. 1272/2008 (CLP) EUH-zinnen	:	EUH210 - veiligheidsinformatieblad op verzoek verkrijgbaar
--	---	--

2.3. Andere gevaren

Er is geen aanvullende informatie beschikbaar.

RUBRIEK 3: Samenstelling en informatie over de bestanddelen

3.1. Stoffen

Niet van toepassing

3.2. Mengsels

Naam	Productidentificatie	%	Indeling overeenkomstig Richtlijn 67/548/EEG
Basisolie - niet ingedeeld	(CAS-nr.) 64742-55-8 (EG-nr.) 265-158-7	1 - 25	Niet ingedeeld
Zinkalkyldithiofosfaat	(CAS-nr.) 68649-42-3 (EG-nr.) 272-028-3 (REACH-nr.) 01-2119493635-27	0,1 - 0,5	XI R41 XI R38 N R51/53
2,6-di-tert-butylfenol	(CAS-nr.) 128-39-2 (EG-nr.) 204-884-0	0,1 - 0,25	XI R38 N R50/53

Naam	Productidentificatie	%	Indeling overeenkomstig Verordening (EG) nr. 1272/2008 (CLP)
Basisolie - niet ingedeeld	(CAS-nr.) 64742-55-8 (EG-nr.) 265-158-7	1 - 25	Asp. Tox. 1, H304
Zinkalkyldithiofosfaat	(CAS-nr.) 68649-42-3 (EG-nr.) 272-028-3 (REACH-nr.) 01-2119493635-27	0,1 - 0,5	Eye Dam. 1, H318 Aquatic Chronic 2, H411
2,6-di-tert-butylfenol	(CAS-nr.) 128-39-2 (EG-nr.) 204-884-0	0,1 - 0,25	Skin Irrit. 2, H315 Aquatic Acute 1, H400 Aquatic Chronic 1, H410

Zie rubriek 16 voor de volledige inhoud van de R-, H- en EUH-zinnen.

RUBRIEK 4: Eerstehulpmaatregelen

4.1. Beschrijving van de eerstehulpmaatregelen

Na het inademen	:	Speciale maatregelen worden niet noodzakelijk geacht.
Na contact met de huid	:	Huid wassen met water en milde zeep.
Na contact met de ogen	:	Onmiddellijk met stromend water spoelen gedurende 10 tot 15 minuten.
Na inslikken	:	Geen braken opwekken. Mond spoelen. Raadpleeg onmiddellijk een arts.

4.2. Belangrijkste acute en uitgestelde symptomen en effecten

Symptomen/letsel na het inademen	:	Wordt in normale gebruiksomstandigheden niet geacht een ernstig ademhalingsrisico in te houden.
Symptomen/letsel na contact met de huid	:	Wordt in normale gebruiksomstandigheden niet geacht een ernstig gevaar voor de huid in te houden.
Symptomen/letsel na contact met de ogen	:	Wordt in normale gebruiksomstandigheden niet geacht een groot oogcontactrisico in te houden.
Symptomen/letsel na inslikken	:	Wordt in normale gebruiksomstandigheden niet geacht een ernstig gevaar bij inslikken in te houden.

4.3. Vermelding van de vereiste onmiddellijke medische verzorging en speciale behandeling

Er is geen aanvullende informatie beschikbaar.

RUBRIEK 5: Brandbestrijdingsmaatregelen

5.1. Blusmiddelen

Geschikte blusmiddelen	:	Waternevel. Schuim. Poeder. Droog chemisch product.
Ongeschikte blusmiddelen	:	Krachtige waterstraal

5.2. Speciale gevaren die door de stof of het mengsel worden veroorzaakt

Er is geen aanvullende informatie beschikbaar.

5.3. Advies voor brandweerlieden

Voorzorgsmaatregelen brand	:	Wees uiterst voorzichtig bij de bestrijding van een chemische brand.
Blusinstructies	:	Koel blootgestelde vaten af door besproeiing met water of met waternevel.
Bescherming tijdens brandbestrijding	:	Brandzone niet betreden zonder aangepaste veiligheidsuitrusting, inclusief ademhalingsbescherming.

RUBRIEK 6: Maatregelen bij het accidenteel vrijkomen van de stof of het mengsel

6.1. Persoonlijke voorzorgsmaatregelen, beschermende uitrusting en noodprocedures

6.1.1. Voor andere personen dan de hulpdiensten	:	Draag speciale beschermende kleding en handschoenen.
Beschermende uitrusting	:	

6.1.2. Voor de hulpdiensten

Beschermende uitrusting	:	Draag speciale beschermende kleding en handschoenen.
-------------------------	---	--

6.2. Milieuvoorzorgsmaatregelen

Niet in riolering of openbare wateren laten wegstromen. Waarschuw de betreffende autoriteiten indien dit product een riolering of open water binnendringt.

6.3. Insluitings- en reinigingsmethoden en -materiaal

Insluiting	:	Grote gemorste hoeveelheid op grond indijken en opnemen door te mengen met inerte korrelige vaste stoffen.
Reinigingsmethoden	:	Detergent. Morsvloeistof absorberen in absorptiemiddel zand, zaagmeel, kiezelgoer.
Overige informatie	:	Het strooioppervlak kan glad zijn. Geschikte afvalvaten gebruiken.

6.4. Verwijzing naar andere rubrieken

Er is geen aanvullende informatie beschikbaar.

RUBRIEK 7: Hantering en opslag

7.1 Voorzorgsmaatregelen voor het veilig hanteren van de stof of het mengsel

Voorzorgsmaatregelen voor het veilig hanteren van de stof of het mengsel	:	Vermijd onnodige blootstelling. Zowel plaatselijke afvoer als algemene ventilatie van de ruimte zijn gewoonlijk vereist.
Hanteringstemperatuur	:	< 40 °C
Instructies voor algemene hygiëne	:	De handen en andere blootgestelde huidgedeelten wassen met zachte zeep en water alvorens te eten, drinken, roken of het werk te verlaten.

7.2. Voorwaarden voor een veilige opslag, met inbegrip van incompatibele producten

Opslagtemperatuur	:	< 40 °C
Opslagruimte	:	Opslaan in een droge, koele, goed geventileerde ruimte.

7.3. Specifiek eindgebruik

Er is geen aanvullende informatie beschikbaar.

RUBRIEK 8: Maatregelen ter beheersing van blootstelling/persoonlijke bescherming

8.1. Controleparameters

:	5 mg/m ³ olieniveau (TWA, 8-urige werkdag) aanbevolen, gebaseerd op ACGIH TLV (analyse volgens US NIOSH Method 5026, NIOSH Manual of Analytical Methods, 3e editie).
---	---

8.2. Maatregelen ter beheersing van blootstelling

Persoonlijke beschermingsmiddelen	:	Veiligheidsbril. Handschoenen.
-----------------------------------	---	--------------------------------



Bescherming van de handen	:	Geschikte handschoenen dragen die bestand zijn tegen relevante chemicaliën.
Bescherming van huid en lichaam	:	Geen speciale kleding/huidbescherming aanbevolen in normale gebruiksomstandigheden.
Bescherming van luchtwegen	:	In normale gebruiksomstandigheden met voldoende ventilatie wordt geen speciale ademhalingsbescherming aanbevolen.

RUBRIEK 9: Fysische en chemische eigenschappen

9.1. Informatie over fysische en chemische basiseigenschappen

Fysische toestand	:	Vloeibaar
Vorkomen	:	Olieachtige vloeistof
Kleur	:	Blauw
Geur	:	Kenmerkend
Geurdempelwaarde	:	Geen gegevens beschikbaar
pH	:	Geen gegevens beschikbaar
Relatieve verdampingssnelheid (butylacetaat=1)	:	Geen gegevens beschikbaar
Smelpunt	:	Geen gegevens beschikbaar
Vriespunt	:	Geen gegevens beschikbaar
Kookpunt	:	Geen gegevens beschikbaar
Vlampunt	:	> 180 °C @ ASTM D92
Zelfontbrandingstemperatuur	:	Geen gegevens beschikbaar
Ontbindingstemperatuur	:	Geen gegevens beschikbaar
Ontvlambaarheid (vast, gas)	:	Geen gegevens beschikbaar
Dampdruk	:	Geen gegevens beschikbaar
Relatieve dampdichtheid bij 20 °C	:	Geen gegevens beschikbaar
Relatieve dichtheid	:	Geen gegevens beschikbaar
Dichtheid	:	872 kg/m ³ @ 15°C
Oplosbaarheid	:	Product is licht oplosbaar en drijft op het wateroppervlak
Log Pow	:	Geen gegevens beschikbaar
Viscositeit, kinematisch	:	32 mm ² /s @ 40°C
Viscositeit, dynamisch	:	Geen gegevens beschikbaar
Ontploffingseigenschappen	:	Geen gegevens beschikbaar
Oxiderende eigenschappen	:	Geen gegevens beschikbaar
Explosiegrenswaarden	:	Geen gegevens beschikbaar

9.2. Overige informatie

Er is geen aanvullende informatie beschikbaar.

RUBRIEK 10: Stabiliteit en reactiviteit

10.1. Reactiviteit

Geen in normale omstandigheden.

10.2. Chemische stabiliteit

Stabiel onder normale omstandigheden.

10.3. Mogelijke gevaarlijke reacties

Geen in normale omstandigheden.

10.4. Te vermijden omstandigheden

Geen gegevens beschikbaar

10.5. Chemisch op elkaar inwerkende materialen

Sterk oxiderende stoffen, zuren. Basen.

10.6. Gevaarlijke ontledingsproducten

Geen in normale omstandigheden.

RUBRIEK 11: Toxicologische informatie

11.1. Informatie over toxicologische effecten

Acute toxiciteit	:	Niet ingedeeld
Huidcorrosie/huidirritatie	:	Niet ingedeeld
Ernstig oogletsel/oogirritatie	:	Niet ingedeeld
Sensibilisatie van de luchtwegen/de huid	:	Niet ingedeeld
Mutageniteit in geslachtscellen	:	Niet ingedeeld
Kankerverwekkendheid	:	Niet ingedeeld

RUBRIEK 12: Ecologische informatie

12.1. Toxiciteit

Er is geen aanvullende informatie beschikbaar.

12.2. Persistentie en afbreekbaarheid

HF-95 hydraulische olie

Persistentie en afbreekbaarheid Niet oplosbaar in water, dus zeer beperkt biologisch afbreekbaar.

12.3. Bioaccumulatie

Er is geen aanvullende informatie beschikbaar.

12.4. Mobiliteit in de bodem

Er is geen aanvullende informatie beschikbaar.

12.5. Resultaten van PBT- en zPzB-beoordeling

Er is geen aanvullende informatie beschikbaar.

12.6. Andere schadelijke effecten

Er is geen aanvullende informatie beschikbaar.

RUBRIEK 13: Instructies voor verwijdering**13.1. Afvalverwerkingsmethoden**

Aanvullende informatie	:	Op een veilige manier opruimen in overeenstemming met lokale/nationale voorschriften.
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RUBRIEK 14: Informatie met betrekking tot het vervoer

Overeenkomstig de eisen van ADR / RID / ADNR / IMDG / ICAO / IATA.

14.1. VN-nummer

Niet geklassificeerd als gevaarlijk volgens de transportwetgeving.

14.2. Juiste ladingnaam overeenkomstig de modelreglementen van de VN

Niet van toepassing

14.3. Transportgevarenklasse(n)

Niet van toepassing

14.4. Verpakkingsgroep

Niet van toepassing

14.5. Milieugevaren

Overige informatie	:	Er is geen aanvullende informatie beschikbaar.
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14.5. Milieugevaren

Overige informatie

14.6. Bijzondere voorzorgen voor de gebruiker**14.6.1. Landtransport**

Er is geen aanvullende informatie beschikbaar.

14.6.2. Transport op open zee

Er is geen aanvullende informatie beschikbaar.

14.6.3. Luchttransport

Er is geen aanvullende informatie beschikbaar.

14.6.4. Transport op binnenlandse wateren

Er is geen aanvullende informatie beschikbaar.

14.7. Vervoer in bulk overeenkomstig bijlage II bij MARPOL 73/78 en de IBC-code

Niet van toepassing

RUBRIEK 15: Regelgeving**15.1. Specifieke veiligheids-, gezondheids- en milieureglementen en -wetgeving voor de stof of het mengsel****15.1.1. EU-voorschriften**

Geen beperkingen overeenkomstig bijlage XVII van REACH.

Bevat geen REACH kandidaatstof

15.1.2. Nationale voorschriften

Er is geen aanvullende informatie beschikbaar.

15.2. Chemische veiligheidsbeoordeling

Er is geen aanvullende informatie beschikbaar.

RUBRIEK 16: Overige informatie

Overige informatie	:	De informatie in dit veiligheidsblad werd verkregen uit bronnen die, naar beste weten, betrouwbaar zijn. Niettemin wordt de informatie wat betreft de juistheid zonder enige garantie, hetzij uitdrukkelijk of stilzwijgend, ter beschikking gesteld. De omstandigheden of methodes van behandeling, opslag, gebruik of verwijdering van het product vallen buiten onze controle en kunnen niet tot onze bevoegdheden behoren. Om deze en andere redenen wijzen wij elke verantwoordelijkheid af in geval van verlies, schade of onkosten, die op welke wijze dan ook zijn ontstaan tijdens of zijn verbonden met de behandeling, de opslag, het gebruik of de verwijdering van het product. Dit veiligheidsblad is opgesteld voor dit product en dient uitsluitend hiervoor gebruikt te worden. Als het product wordt gebruikt als een component in een ander product, is het mogelijk dat de informatie in dit veiligheidsblad niet van toepassing is.
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Volledige inhoud van de R-, H-en EUH-zinnen:

Aquatic Acute 1	Acuut gevaar voor het aquatisch milieu - gevarencategorie 1
Aquatic Chronic 1	Chronisch gevaar voor het aquatisch milieu - gevarencategorie 1
Aquatic Chronic 2	Chronisch gevaar voor het aquatisch milieu - gevarencategorie 2
Asp. Tox. 1	Aspiratiegevaar, Categorie 1
Eye Dam. 1	Ernstig oogletsel/ernstige oogirritatie, Categorie 1
Skin Irrit. 2	Huidcorrosie/huidirritatie, Categorie 2
H304	Kan dodelijk zijn als de stof bij inslikken in de luchtwegen terechtkomt
H315	Veroorzaakt huidirritatie
H318	Veroorzaakt ernstig oogletsel
H400	Zeer giftig voor in het water levende organismen
H410	Zeer giftig voor in het water levende organismen, met langdurige gevolgen
H411	Giftig voor in het water levende organismen, met langdurige gevolgen
R38	Irriterend voor de huid
R41	Gevaar voor ernstig oogletsel
R50/53	Zeer giftig voor in het water levende organismen; kan in het aquatisch milieu op lange termijn schadelijke effecten veroorzaken.
R51/53	Giftig voor in het water levende organismen; kan in het aquatisch milieu op lange termijn schadelijke effecten veroorzaken.
N	Gevaarlijk voor het milieu
Xi	Irriterend

SDS EU (REACH bijlage II)

Deze informatie is gebaseerd op onze huidige kennis en is alleen bedoeld om de gezondheids-, veiligheids- en milieuspecten van het product te beschrijven. Het mag dus niet worden opgevat als garantie voor gelijk welke specifieke eigenschap van het product.

BIJLAGE 6 MILIEUKUNDIG DAGBOEK

**DAGBOEK MILIEUKUNDIG TOEZICHT***Volgnummer en terreinbezoek : 1Datum : 11/12/2018Projectidentificatie : 0458595OVAM dossier: 732

Locatie: 3M	Weersomstandigheden: Koud, 0°C
Straat en huisnummer: Canadastraat 11 Gemeente: Zwijndrecht	<i>Spill Hydraulische olie ten NO van Gebouw 16</i>

Aanwezigen en hun taak				
Naam	Functie	Firma	Uur in	Uur uit
Erik Boeckx	Milieukundige begeleiding	ERM	7:30	8:30

Beschrijving van de lopende bodemsaneringswerken op het ogenblik van de controle
Op 10 december 2018 is er een spill geweest op de site van 3M te Zwijndrecht ten gevolge van een breuk in een hydraulische leiding van een paalfunderingsmachine. Hierbij kwam een 10-tal liter (geraamd) hydraulische olie vrij. De MSDS sheet is als bijlage toegevoegd. De olie kwam op een onverharde ondergrond terecht. De geïmpacteerde zone situeert zich ten noordoosten van gebouw 16.
 Het vrijgekomen product is deels opgevangen in een lekbak en deels afgegraven en opgeslagen in een cubitainer na het strooien van absorberende korrels (1m³). Na de herstelling van de leiding kon de machine verplaatst worden. Op 12 december 2018 is de rest afgegraven (3x3m) tot een diepte van 20 cm. Er was geen zichtbare verkleuring of afwijkende geur. De

* conform OVAM document "Bodemsaneringswerken en nazorg, standaardprocedure, oktober 2014"

vrijgekomen grond (+/- 1m³) is tijdelijk op een folie geplaatst. Van de putbodem en de putwanden zijn stalen genomen. Van de putbodem is eveneens een dieper monster genomen. Er zijn eveneens reeds afperkende stalen genomen indien er overschrijdingen vastgesteld worden.

Na ontgraving was er geen verkleuring of afwijkende geur vast te stellen.

Health & Safety

Geen opmerkingen

Milieukundige vaststellingen

Visueel

Zie foto-reportage.

Monsternam(e)s en Analyse(s) – zie plan voor locatie

monsternamelpunten

- CSB1 (0-20)
- CSB1 (50-70)
- CSB1 (100-120)
- CSW1A en B
- CSW2A en B
- CSW3A en B
- CSW4A en B

Bindend advies en/of opmerkingen

1. Uit de analyseresultaten van de grondstalen blijkt dat er geen overschrijding gemeten is ten opzichte van de Richtwaarde.
2. Het incident dient gemeld te worden aan OVAM (lopende)

Voor de bodemsaneringsdeskundige

Naam

Erik Boeckx

Voor de opdrachtgever

Handtekening en naam

Foto-reportage

Foto 1 : Situering achter Gebouw 16



Foto 2: Beschadigde leiding



Foto 3: Ontgravingsvak

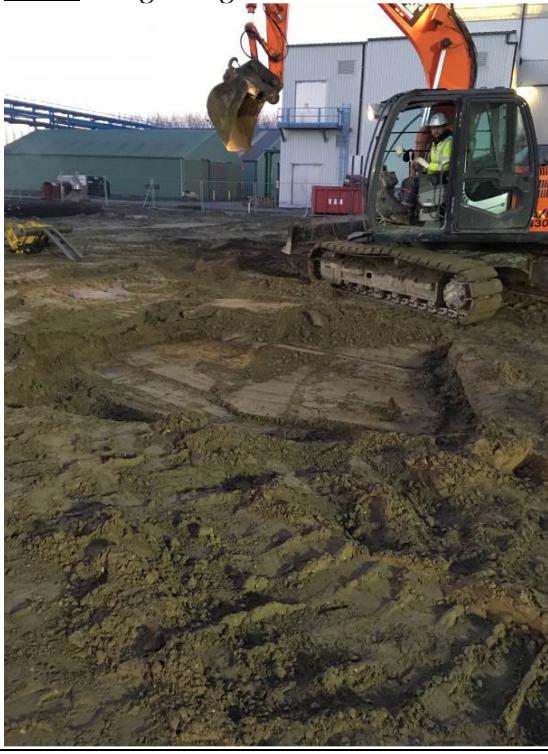


Foto 4: Tijdelijke depot



BIJLAGE 7 ANALYSERESULTATEN

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Environmental Resource Management N.V. Belgium
Sorgeloos Lieselotte
Cantersteen 47
1000 BRUSSEL
BELGIQUE

Datum 13.12.2018
Relatielnr. 35005802
Opdrachtnr. 816360

ANALYSERAPPORT

Opdracht 816360 Bodem / Eluaat

Opdrachtgever 35005802 Environmental Resource Management N.V. Belgium
Uw referentie 0458595 3M Additional 2018
Opdrachtacceptatie 12.12.18
Monsternemer Opdrachtgever

Geachte heer, mevrouw,

Hierbij zenden wij u de resultaten van het door u aangevraagde laboratoriumonderzoek.
De analyses zijn, tenzij anders vermeld, geaccrediteerd volgens NEN-EN-ISO/IEC 17025 en uitgevoerd
overeenkomenstig de onderzoeksmethoden die worden genoemd in de meest actuele versie van onze
verrichtingenlijst van de Raad voor Accreditatie, accreditatienummer L005.

AL-West is erkend volgens VLAREL als laboratorium voor het uitvoeren van analyses in bodem, grondwater en
afvalstoffen door de OVAM. In het rapport staat aangegeven welke analyses onder deze erkenning zijn uitgevoerd.

Indien u gegevens wenst over de meetonzekerheden van een methode, kunnen wij u deze op verzoek verstrekken.

Dit rapport mag alleen in zijn geheel worden gereproduceerd. Indien u nog vragen heeft of aanvullende informatie
wenst, verzoeken wij u om contact op te nemen met Klantenservice.

Wij vertrouwen erop u met de toegezonden informatie van dienst te zijn.

Met vriendelijke groet,

AL-West B.V. Dhr. Wouter Wanders, Tel. [REDACTED]
Klantenservice

De in dit rapport vermelde analyses zijn geaccrediteerd volgens ISO/IEC 17025:2005, tenzij bij de analyse het symbool " * " staat vermeld.

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Opdracht 816360 Bodem / Eluaat

Monsternr.	Monstername	Monsteromschrijving
816545	12.12.2018	CS-B1 (0-20)
816546	12.12.2018	CS-B1 (50-70)
816547	12.12.2018	CS-B1 (100-120)
816548	12.12.2018	CS-W1A
816549	12.12.2018	CS-W2A

Eenheid	816545 CS-B1 (0-20)	816546 CS-B1 (50-70)	816547 CS-B1 (100-120)	816548 CS-W1A	816549 CS-W2A
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Algemene monstervoorbehandeling

R3 Drobe stof	%	91,3	93,8	82,5	90,4	87,6
---------------	---	------	------	------	------	------

Minerale olie (CMA)

R3 Koolwaterstoffractie C10-C40	mg/kg Ds	71 *	58 *	<50 *	<50 *	<50 *
R3 Koolwaterstoffractie C10-C12	mg/kg Ds	<8 *	<8 *	<8 *	<8 *	<8 *
R3 Koolwaterstoffractie C12-C20	mg/kg Ds	<12 *	<12 *	<12 *	<12 *	<12 *
R3 Koolwaterstoffractie C20-C30	mg/kg Ds	47 *	36 *	<15 *	<15 *	<15 *
R3 Koolwaterstoffractie C30-C40	mg/kg Ds	<15 *	<15 *	<15 *	<15 *	<15 *

De in dit rapport vermelde analyses zijn geaccrediteerd volgens ISO/IEC 17025:2005, tenzij bij de analyse het symbool " * " staat vermeld.

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Opdracht 816360 Bodem / Eluaat

Monsternr.	Monstername	Monsteromschrijving
816550	12.12.2018	CS-W3A
816551	12.12.2018	CS-W4A

Eenheid **816550**
 CS-W3A **816551**
 CS-W4A

Algemene monstervoorbehandeling

R3 Droege stof	%	88,3	91,0
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Minerale olie (CMA)

R3 Koolwaterstoffractie C10-C40	mg/kg Ds	<50 *	<50 *
R3 Koolwaterstoffractie C10-C12	mg/kg Ds	<8 *	<8 *
R3 Koolwaterstoffractie C12-C20	mg/kg Ds	<12 *	<12 *
R3 Koolwaterstoffractie C20-C30	mg/kg Ds	<15 *	<15 *
R3 Koolwaterstoffractie C30-C40	mg/kg Ds	<15 *	<15 *

R3) Erkend volgens OVAM

Verklaring: "<" of n.a. betekent dat het gehalte van de component lager is dan de rapportagegrens.

Begin van de analyses: 12.12.2018

Einde van de analyses: 13.12.2018

De onderzoeksresultaten hebben alleen betrekking op het aangeleverde monstermateriaal. Monsters met onbekende herkomst kunnen slechts beperkt gecontroleerd worden op plausibiliteit.

AL-West B.V. Dhr. Wouter Wanders, Tel. [REDACTED]
Klantenservice

Toegepaste methoden

CMA/2/II/A.1: Droge stof

CMA/3/R1: Koolwaterstoffractie C10-C40 Koolwaterstoffractie C10-C12 Koolwaterstoffractie C12-C20
 Koolwaterstoffractie C20-C30 Koolwaterstoffractie C30-C40

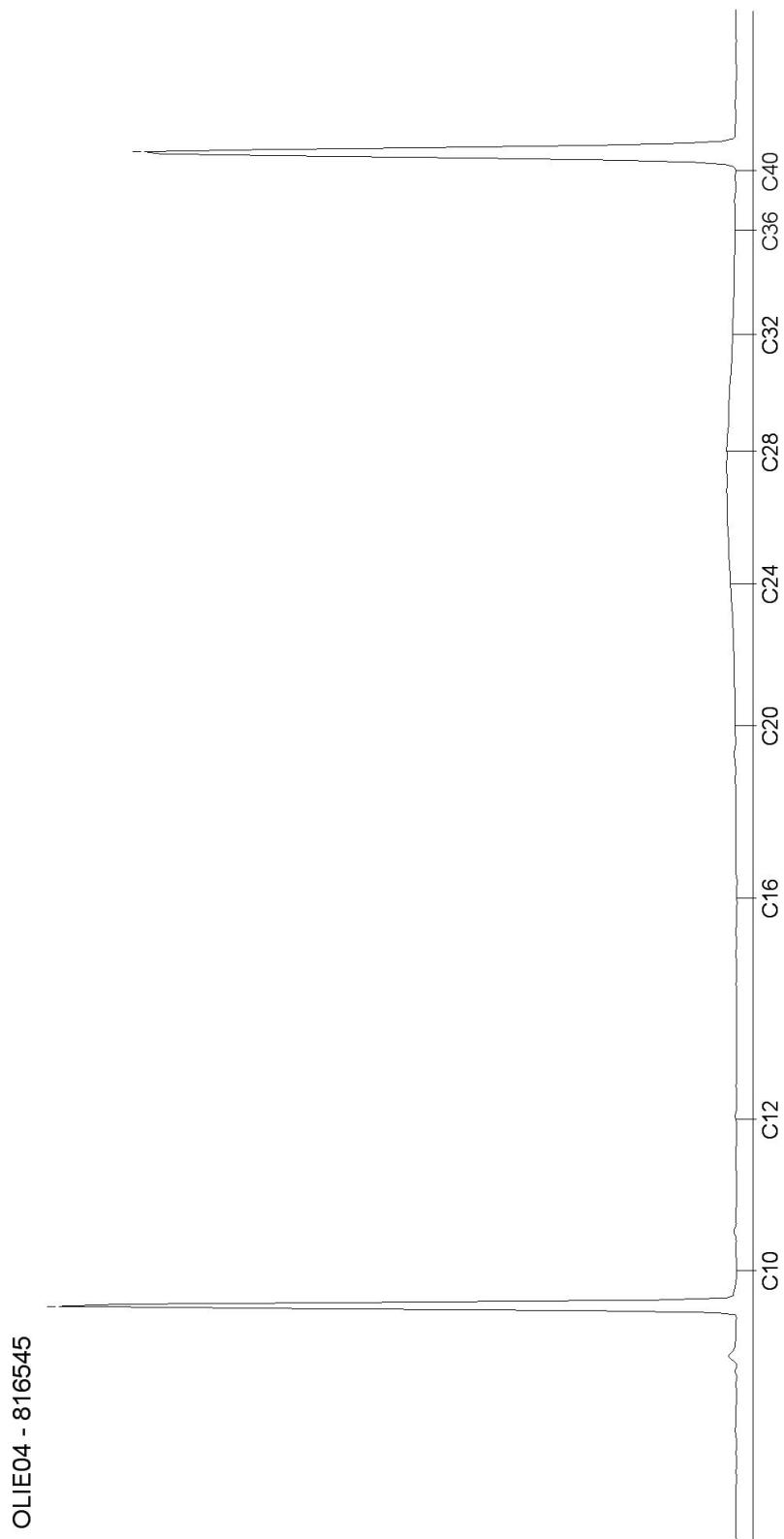
De in dit rapport vermelde analyses zijn geaccrediteerd volgens ISO/IEC 17025:2005, tenzij bij de analyse het symbool " * " staat vermeld.

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CHROMATOGRAM for Order No. 816360, Analysis No. 816545, created at 13.12.2018 12:50:26

Monsteromschrijving: CS-B1 (0-20)



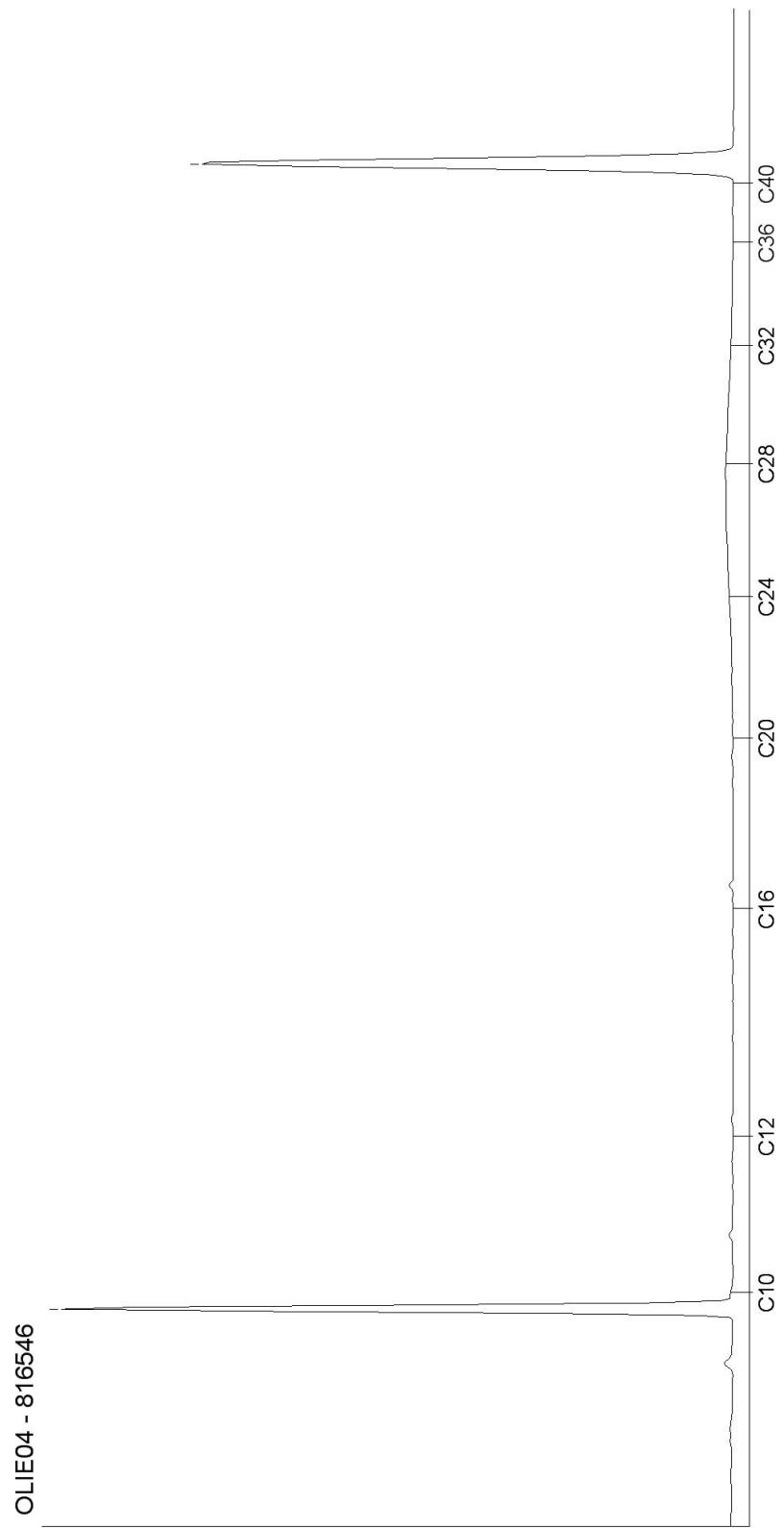
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CHROMATOGRAM for Order No. 816360, Analysis No. 816546, created at 13.12.2018 12:50:26

Monsteromschrijving: CS-B1 (50-70)



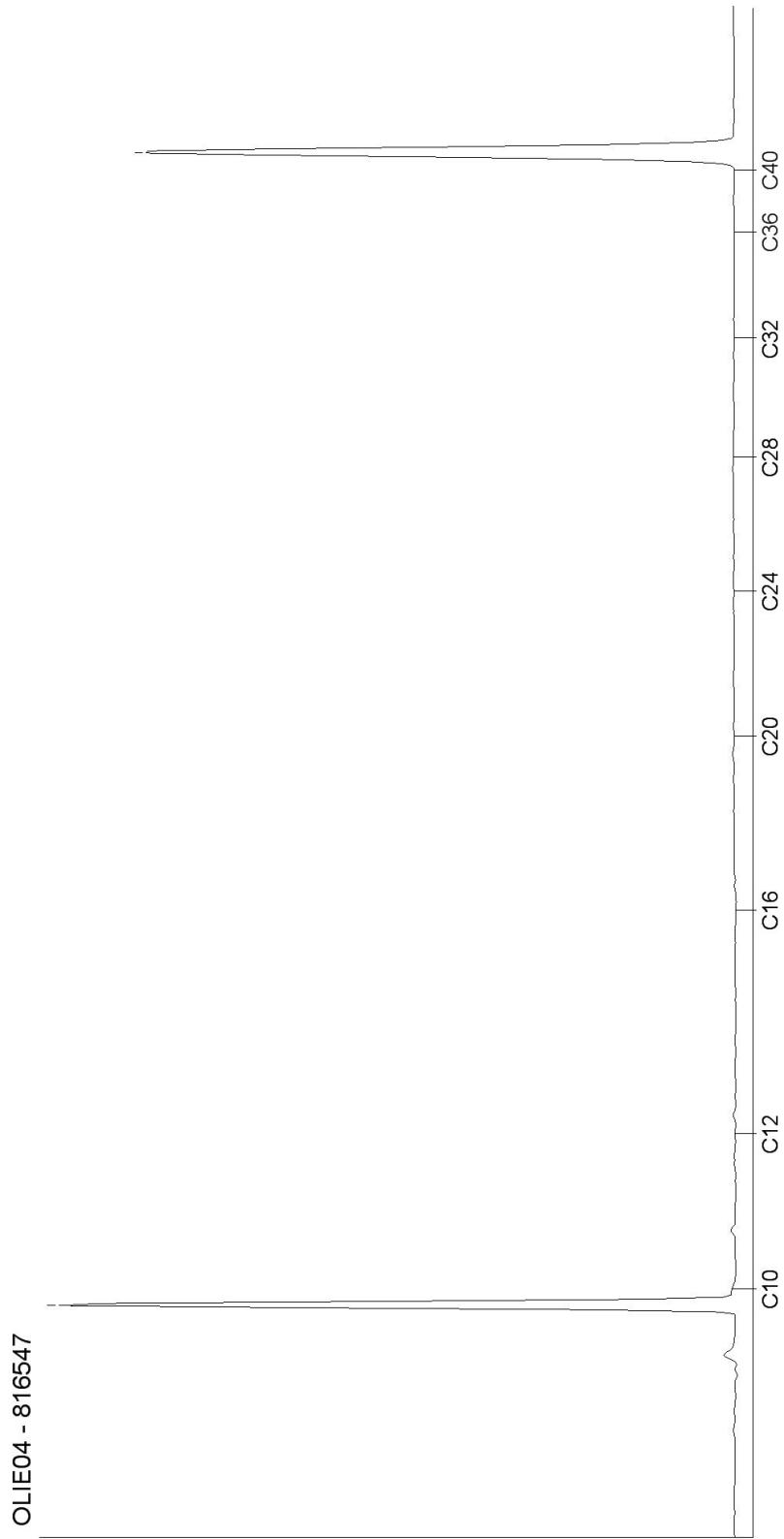
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CHROMATOGRAM for Order No. 816360, Analysis No. 816547, created at 13.12.2018 12:50:26

Monsteromschrijving: CS-B1 (100-120)



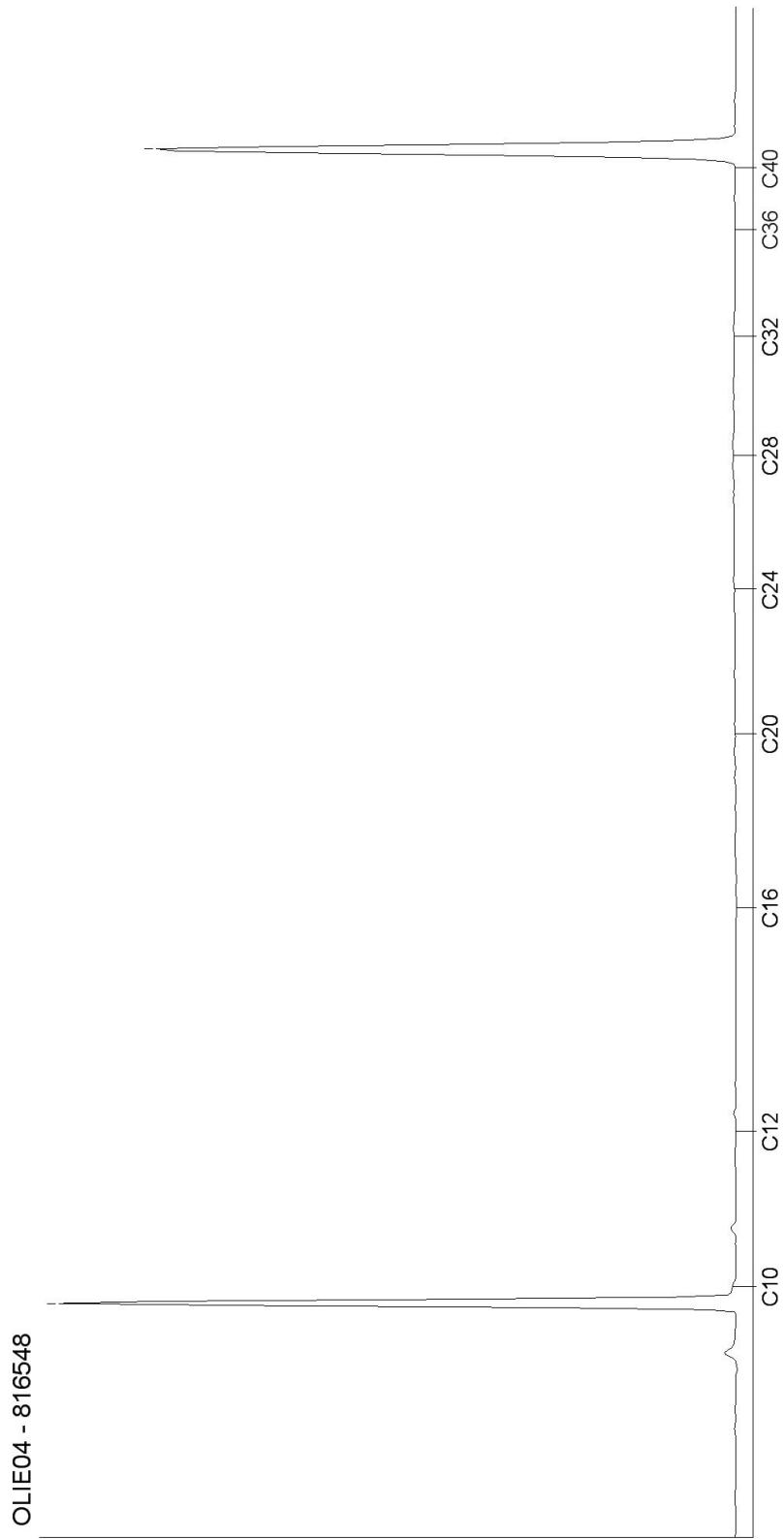
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CHROMATOGRAM for Order No. 816360, Analysis No. 816548, created at 13.12.2018 12:50:27

Monsteromschrijving: CS-W1A



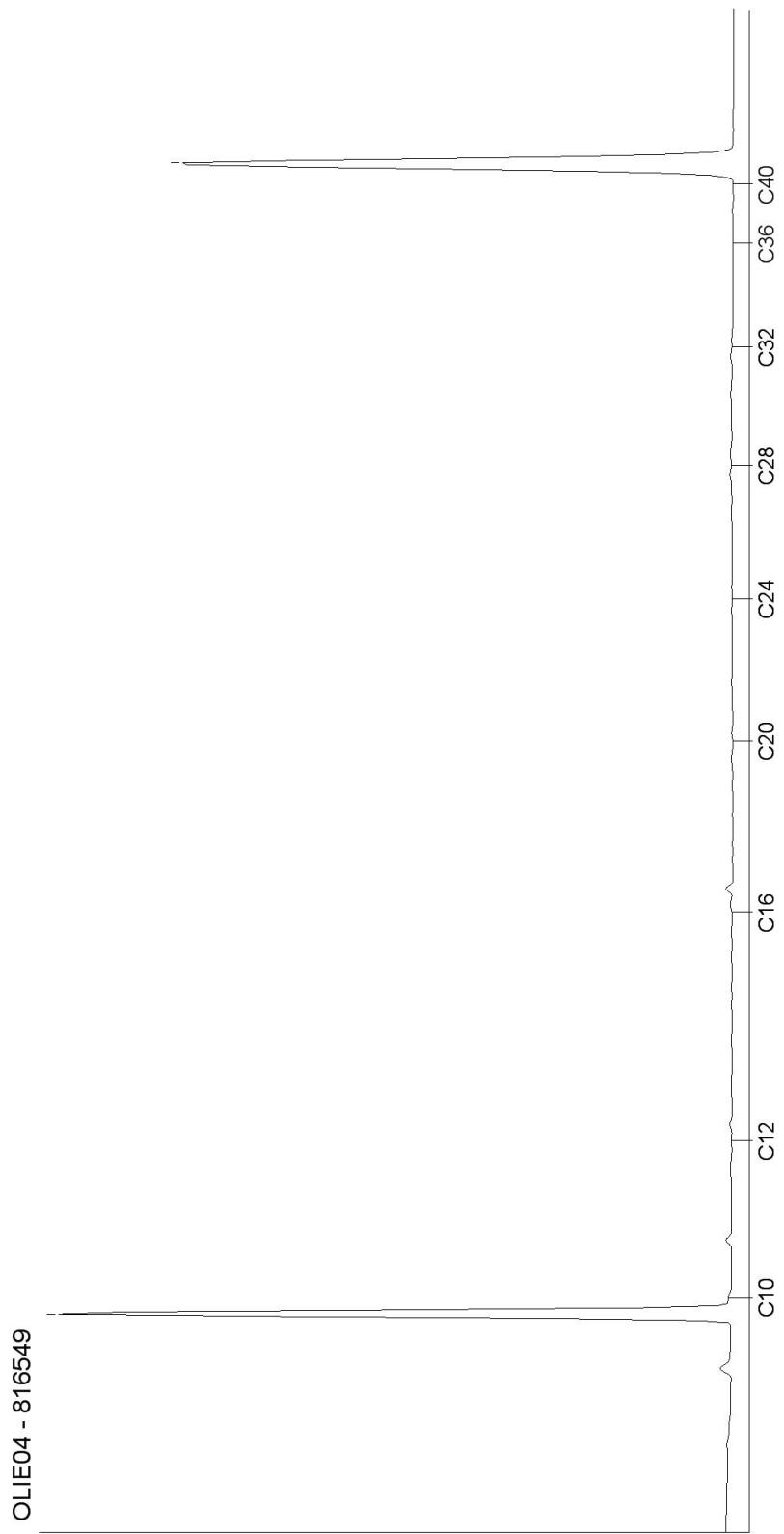
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CHROMATOGRAM for Order No. 816360, Analysis No. 816549, created at 13.12.2018 12:50:27

Monsteromschrijving: CS-W2A



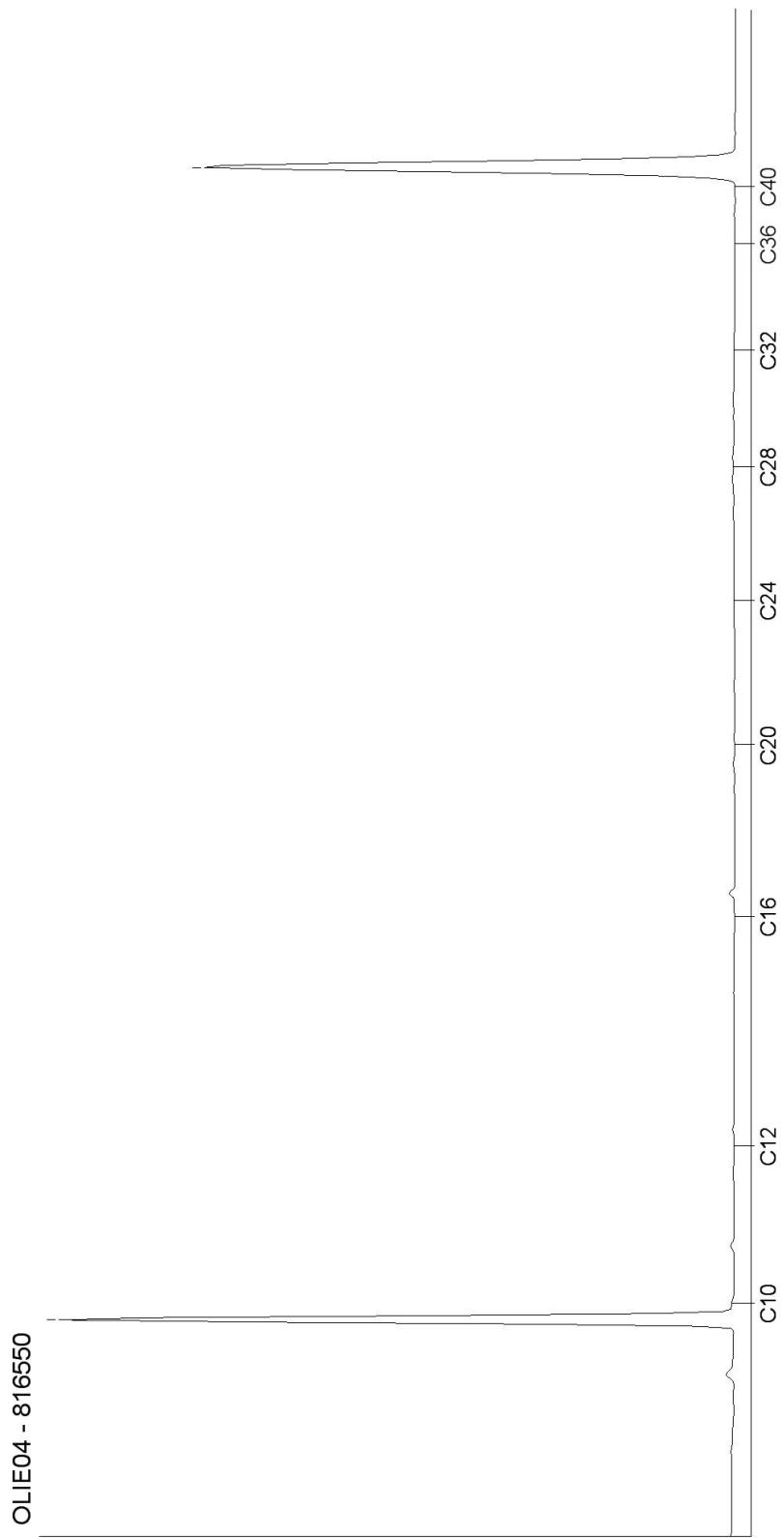
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CHROMATOGRAM for Order No. 816360, Analysis No. 816550, created at 13.12.2018 12:50:27

Monsteromschrijving: CS-W3A



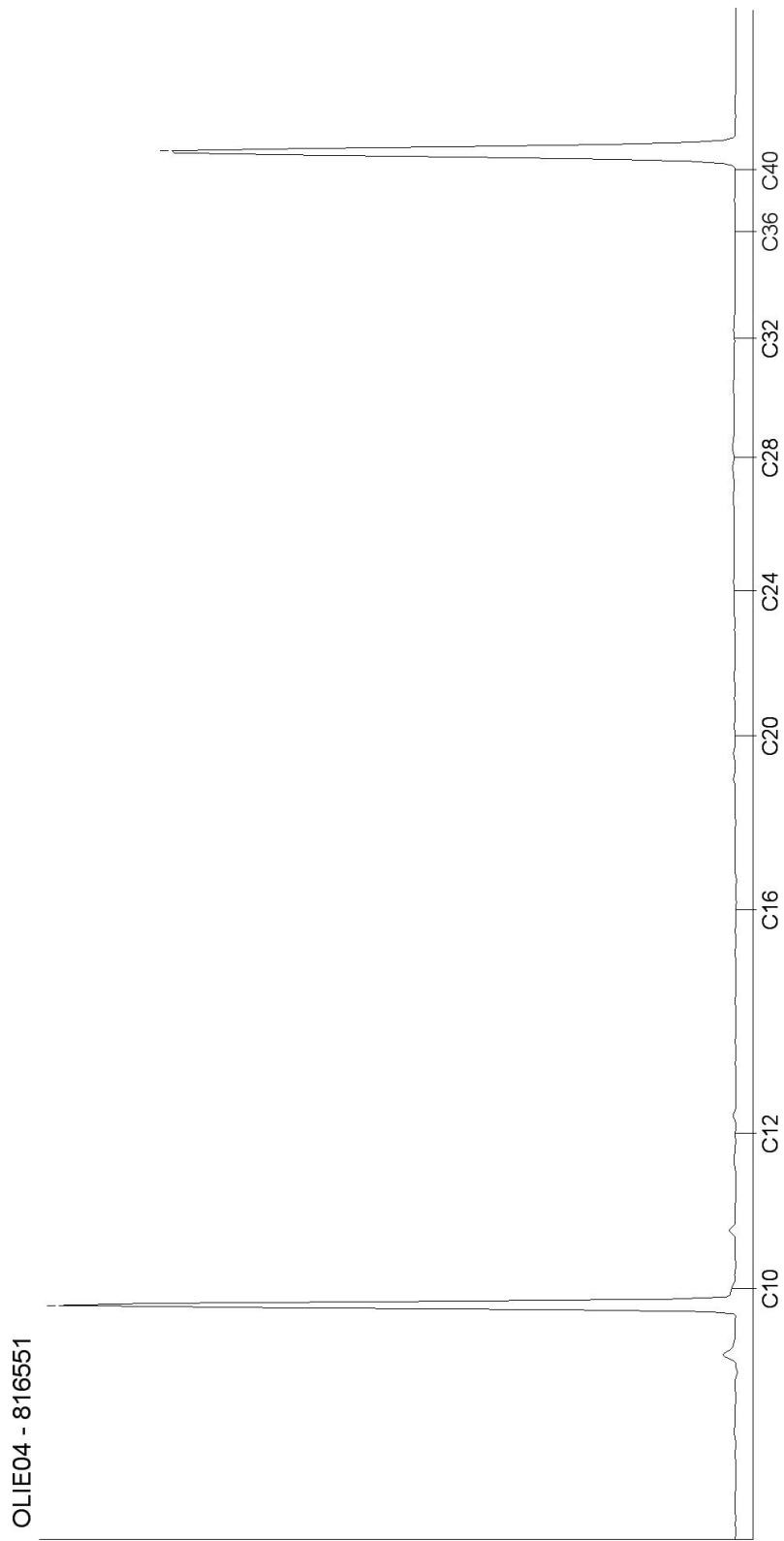
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CHROMATOGRAM for Order No. 816360, Analysis No. 816551, created at 13.12.2018 12:50:27

Monsteromschrijving: CS-W4A



Blad 7 van 7

BIJLAGE 8 OVERZICHT BOORPUNTEN

NR	X	Y	Diepte (cm)	Lithologie
CS-W3B	147731.6491	213629.8965	20	zand
CS-W2B	147726.7004	213629.9966	20	zand
CS-W1B	147726.6003	213625.0478	20	zand
CS-W4B	147731.5491	213624.9478	20	zand
CS-W4A	147730.1637	213626.3903	20	zand
CS-W1A	147728.0428	213626.4332	20	zand
CS-W2A	147728.0857	213628.5541	20	zand
CS-W3A	147730.2066	213628.5112	20	zand
CS-B1	147729.1247	213627.4722	120	zand

Organoleptische waarnemingen
-
-
-
-
-
-
-
-
-
-
-

BIJLAGE 9 BESLUIT OVAM

BIJLAGE 10 OVERZICHT VERGUNDE GRONDWATERWINNINGEN

BIJLAGE 11 VERWERKINGSCERTIFICAAT GROND

BB-LB2-KB-20180725215

3M Belgium bvba
Hermeslaan 7
1831 DIEGEM

Vlaamse overheid
Openbare Vlaamse
Afvalstoffenmaatschappij
Stationsstraat 110
2800 MECHELEN
T 015 284 284
F 015 203 275
www.ovam.be

uw bericht van		afdeling	Bodembeheer
uw kenmerk		dienst/team	Team Lokale en bovenlokale besturen
bijlagen	Besluit	contactpersoon	Kathleen Baets [REDACTED] schade@ovam.be
Mechelen	20.12.2018	ons kenmerk	BB-LB2-KB-20180725215 (Dossiernummer: 732)

melding schadegeval: Zwijndrecht - Canadalaan 11

Geachte heer,
Geachte mevrouw,

Op 14 december 2018 werd de OVAM via een melding schadegeval in kennis gesteld van een mogelijke bodemverontreiniging ter hoogte van Canadalaan 11 in Zwijndrecht. Dit bronperceel is kadastral bekend als:

Gemeentenr.	Afdeling	Sectie	Grondnr.	Bisnr.	Exp.1	Exp.2
11056	ZWIJDRECHT 1 AFD/ZWIJDRECHT/	A	467		E	

De verontreiniging is ontstaan naar aanleiding van een schadegeval waarbij een slang met hydraulische olie van een boorinstallatie is vrijgekomen met mogelijke bodemverontreiniging tot gevolg. Het schadegeval heeft zich voorgedaan op een grond:

- waarop een inrichting gevestigd is die krachtens titel V van het decreet van 5 april 1995 houdende algemene bepalingen inzake milieubeleid ingedeeld wordt in de eerste klasse

Op grond van het decreet van 27 oktober 2006 betreffende de bodemsanering en de bodembescherming (Bodemdecreet) heeft de OVAM de bevoegdheid om maatregelen op te leggen ter behandeling van de bodemverontreiniging die ontstaan is ingevolge dit schadegeval. Als bijlage vindt u het besluit waarbij de OVAM die maatregelen oplegt.

De verplichting om over te gaan tot die maatregelen ligt, conform artikel 80 van het Bodemdecreet, bij:

- 1° de exploitant, als op de grond in kwestie een milieuvergunningsplichtige inrichting wordt geëxploiteerd;
- 2° bij gebrek aan exploitant, de gebruiker van de grond waar de bodemverontreiniging tot stand kwam;
- 3° bij gebrek aan exploitant en gebruiker, de eigenaar van de grond waar de bodemverontreiniging tot stand kwam.

In uw hoedanigheid van exploitant van de grond waar het schadegeval zich heeft voorgedaan, bent u krachtens artikel 80 van het Bodemdecreet ertoe gehouden de maatregelen binnen de vastgestelde termijn uit te voeren onder leiding van een bodemsaneringsdeskundige. Deze maatregelen kunnen zowel door een type I als door een type II deskundige worden uitgevoerd. Een lijst met alle bodemsaneringsdeskundigen kan u terugvinden via www.ovam.be/lijstbsd. Gelieve uw bodemsaneringsdeskundige in te lichten over de inhoud van deze brief en het bijgevoegde besluit.

Als u de maatregelen niet of in onvoldoende mate uitvoert, kan de OVAM de maatregelen zelf uitvoeren en de kosten ervan op u verhalen. Met behoud van de toepassing van het Strafwetboek zijn, in voorkomend geval, de strafbepalingen van artikel 173 van het Bodemdecreet van toepassing.

Als de opgelegde termijn van 180 dagen overschreden wordt, zoals bepaald in het besluit, kan u als saneringsplichtige verplicht worden om een beschrijvend bodemonderzoek en eventuele bodemsanering uit te voeren.

Beroep

Binnen zestig dagen na ontvangst ervan kan u tegen dit besluit een verzoekschrift tot schorsing of nietigverklaring indienen bij de Raad van State, Wetenschapsstraat 33 in 1040 Brussel. Dat verzoekschrift moet aangetekend worden verzonden, moet gedagtekend en ondertekend worden door de partij of door een advocaat en moet enkele verplichte vermeldingen bevatten:

- het opschrift "verzoekschrift tot nietigverklaring" als het niet eveneens een vordering tot schorsing bevat;
- de naam, hoedanigheid, woonplaats of zetel van de verzoekende partij en de gekozen woonplaats;
- het voorwerp van het beroep en een uiteenzetting van de feiten en de middelen;
- de naam en het adres van de verwerende partij.

Meer weten?

Uw bodemsaneringsdeskundige bezorgt u graag alle praktische informatie.

Heeft u nog geen bodemsaneringsdeskundige om u te begeleiden bij de volgende stappen? Kies er dan een uit de lijst met deskundigen op www.ovam.be/lijstBsd.

Voor andere vragen kan u terecht:

- Op www.ovam.be/schadegevallen.
- Of mail naar schade@ovam.be.
- Bij het team klantenbeheer van de OVAM op 015/284458. Wij beantwoorden uw vragen elke werkdag van 8.30 u tot 12.30 u en van 13.30 u tot 16.30 u. Op maandag zijn we bereikbaar vanaf 10 u, op vrijdag tot 16 u.

Wij vragen u het dossiernummer **732** te vermelden als u contact opneemt met de OVAM.

Hoogachtend,


20180705215

Ann Cuyckens
Afdelingshoofd

Kopie:

3M Belgium NV
Canadastraat 11
2070 ZWIJNDRECHT

GEMEENTE ZWIJNDRECHT
Binnenplein 1
2070 ZWIJNDRECHT

**Besluit van de OVAM
Maatregelen tot behandeling
schadegeval**

Vlaamse overheid
Openbare Vlaamse
Afvalstoffenmaatschappij
Stationsstraat 110
2800 MECHELEN
T 015 284 284
F 015 203 275
www.ovam.be

Naam: Kathleen Baets
Dossier: 732
Datum: 20.12.2018

Zwijndrecht - Canadalaan 11

Gelet op het Decreet van 27 oktober 2006 betreffende de bodemsanering en de bodembescherming (Bodemdecreet), artikel 74 tot en met 82;

Overwegende dat op de grond gelegen aan de Canadalaan 11 te Zwijndrecht er zich een schadegeval heeft voorgedaan waarbij een slang met hydraulische olie van een boorinstallatie is vrijgekomen met mogelijke bodemverontreiniging tot gevolg. De grond is kadastral gekend als:

Gemeente-nummer	Afdeling	Sectie	Grondnr.	Bisnr.	Exp.1	Exp.2
11056	ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	467		E	

Deze grond is eigendom van:

Perceel	Gegevens eigenaar
467 E	3M Belgium BVBA, Hermeslaan 7, 1831 Machelen

De exploitant op deze grond is:

Perceel	Gegevens exploitant
467 E	3M Belgium BVBA, Hermeslaan 7, 1831 Machelen

Overeenkomstig artikel 75 van het Bodemdecreet is de OVAM de bevoegde overheid voor het betreffende schadegeval, omdat het schadegeval plaatsvond op een grond:

- waarop een inrichting of activiteit gevestigd is die krachtens titel V van het decreet van 5 april 1995 houdende algemene bepalingen inzake milieubeleid ingedeeld wordt in de eerste klasse

De OVAM is van oordeel dat maatregelen noodzakelijk zijn om de (mogelijke) bodemverontreiniging ingevolge het schadegeval te behandelen. De maatregelen tot behandeling van de schade gevallen



kunnen worden uitgevoerd binnen 180 dagen na de melding of vaststelling van het schadegeval. Het schadegeval valt dan ook onder het toepassingsgebied van de schadegevallenregeling, vermeld in artikel 74 tot en met artikel 82 van het Bodemdecreet.

Op basis van de beschikbare gegevens zijn volgende maatregelen al genomen:

- de bron of het lek werden gestopt;
- absorptiemateriaal en een opvangbak aangebracht;
- er werd een bodemsaneringsdeskundige aangesteld;
- de verontreinigde grond werd ontgraven;
- stalen en analyses werden uitgevoerd;

Op basis van de verkregen gegevens dienen volgende maatregelen genomen te worden om de bodemverontreiniging te behandelen vóór 17 juni 2019:

- de erkend bodemsaneringsdeskundige laten verifiëren of er een bodemverontreiniging ontstaan is die een verdere aanpak vereist conform het Bodemdecreet;
- afhankelijk van de bevindingen van de erkende bodemsaneringsdeskundige: overgaan tot de ontgraving van de verontreinigde grond, indien nodig een waterzuivering plaatsen, waarbij het afvalwater geloosd wordt op oppervlaktewater/bodem (herinfiltratie)/riolering en/of andere saneringsmaatregelen onder leiding van de erkende bodemsaneringsdeskundige;
- de vervuilde ontgraven grond laten verwerken in een centrum voor grondreiniging;
- de bewijzen van afvoer van de verontreinigde grond en/of de verwerkingsattesten van de verontreinigde grond verwerven;
- controlestaalnames van bodem en grondwater (putbodem en -wanden) laten uitvoeren door een erkende bodemsaneringsdeskundige, conform de geldende richtlijnen van de OVAM;
- de saneringswerkzaamheden uitvoeren volgens de geldende codes van goede praktijk;
- het evaluatierapport, opgesteld door een bodemsaneringsdeskundige conform de betreffende richtlijn, aan de OVAM via het webloket bezorgen;
- de OVAM informeren binnen de termijn vermeld in dit besluit of tijdig in te lichten bij eventuele vertragingen;
- de bodemsaneringsdeskundige dient er op toe te zien dat de opgelegde (vergunningsplichtige) maatregelen overeenkomen met de effectief uit te voeren (vergunningsplichtige) maatregelen. Indien nodig vraagt hij een nieuw aangepast besluit op aan de bevoegde overheid.

Als die maatregelen handelingen, inrichtingen of activiteiten omvatten die meldings- of vergunningsplichtig zijn krachtens titel V van het decreet van 5 april 1995 houdende algemene bepalingen inzake milieubeleid of krachtens titel IV, hoofdstuk II, van de Vlaamse Codex Ruimtelijke Ordening, geldt de beslissing als meldingsakte of omgevingsvergunning.

Besluit:

Artikel 1. Het schadegeval dat zich heeft voorgedaan op 10 december 2018 op de grond, kadastraal bekend als:

Gemeente-nummer	Afdeling	Sectie	Grondnr.	Bisnr.	Exp.1	Exp.2
11056	ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	467		E	

en gelegen in de Canadalaan 11 te Zwijndrecht, wordt gekwalificeerd als een schadegeval dat valt onder het toepassingsgebied van de schadegevallenregeling, vermeld in artikel 74 tot en met 82 van het decreet van 27 oktober 2006 betreffende de bodemsanering en de bodembescherming.

Art. 2. De volgende maatregelen ter behandeling van de (mogelijke) bodemverontreiniging ingevolge het schadegeval op de grond, vermeld in artikel 1, moeten **vóór 17 juni 2019** worden uitgevoerd :

- de erkend bodemsaneringsdeskundige laten verifiëren of er een bodemverontreiniging ontstaan is die een verdere aanpak vereist conform het Bodemdecreet;
- afhankelijk van de bevindingen van de erkende bodemsaneringsdeskundige: overgaan tot de ontgraving van de verontreinigde grond, indien nodig een waterzuivering plaatsen, waarbij het afvalwater geloosd wordt op oppervlaktewater/bodem (herinfiltratie)/riolering en/of andere saneringsmaatregelen onder leiding van de erkende bodemsaneringsdeskundige;
- de vervuilde ontgraven grond laten verwerken in een centrum voor grondreiniging;
- de bewijzen van afvoer van de verontreinigde grond en/of de verwerkingsattesten van de verontreinigde grond verwerven;
- controlestalenames van bodem en grondwater (putbodem en -wanden) laten uitvoeren door een erkende bodemsaneringsdeskundige, conform de geldende richtlijnen van de OVAM;
- de saneringswerkzaamheden uitvoeren volgens de geldende codes van goede praktijk;
- het evaluatierapport, opgesteld door een bodemsaneringsdeskundige conform de betreffende richtlijn, aan de OVAM via het webloket bezorgen;
- de OVAM informeren binnen de termijn vermeld in dit besluit of tijdig in te lichten bij eventuele vertragingen;
- De bodemsaneringsdeskundige dient er op toe te zien dat de opgelegde (vergunningsplichtige) maatregelen overeenkomen met de effectief uit te voeren (vergunningsplichtige) maatregelen. Indien nodig vraagt hij een nieuw aangepast besluit op aan de bevoegde overheid.

Deze maatregelen moeten worden uitgevoerd door 3M Belgium bvba.

Art. 3. De maatregelen, vermeld in artikel 2, kunnen ten allen tijde door een beslissing van de OVAM aangepast of opgeheven worden.

Hoogachtend,



20180725169

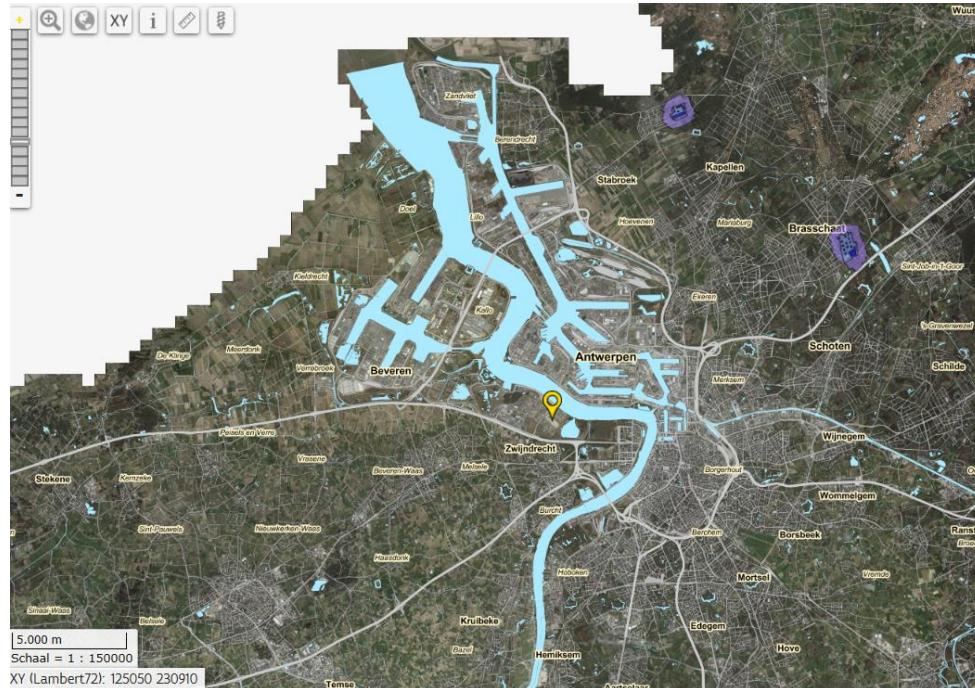
Ann Cuyckens
Afdelingshoofd

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OVERZIETH



DETAIL



Legende

- Winning
- Beschermingszone type I
- Beschermingszone type II
- Beschermingszone type III

: grondwatervergunningen (huidig)

Locatie: XY (Lambert72) = 147474 213864 / GPS (Lat/Long) = 51,2347 4,3326 / Z (DHM II) = 7,34 m TAW

Dichtstbijzijnde adres: Canadalaan 11, 2070 Zwijndrecht

Grondwatervergunningen (huidige):

Watnr	Exploitant	Adres expl	Postcode exploitant	Gemeente exploitant	Nacebelsector	Klasse	Vlarem rubriek	Vergunningverlenende overheid	X inst	Y inst	Plaatsomschrijving	Diepte	Vergund aantal putten	Aquifercode	Aquiferbeschrijving	Begindatum vergunning	Einddatum vergunning	Vergund dagdebiet	Vergund jaardebiet
ANT-ZI004266	[REDACTED]	2017	ZWIJNDRECHT	Vervaardiging van diverse chemische producten	2	53.8.2<17	Gemeentebestuur ZWIJNDRECHT	147475,00	213863,00	1-A3-456c t.e.m h,k t.e.m. n.p.r,t,x,v,467c	4,50	1	0160	Pleistocene afzettingen	25/01/2001	25/01/2021		8760,00	
Watnr	Exploitant	Adres expl	Postcode exploitant	Gemeente exploitant	Nacebelsector	Klasse	Vlarem rubriek	Vergunningverlenende overheid	X inst	Y inst	Plaatsomschrijving	Diepte	Vergund aantal putten	Aquifercode	Aquiferbeschrijving	Begindatum vergunning	Einddatum vergunning	Vergund dagdebiet	Vergund jaardebiet
ANT-gw2/2441	[REDACTED]	2070	ZWIJNDRECHT	Fruitteelt	A	99	Gemeentebestuur ZWIJNDRECHT	147190,00	213055,00	1-A-524l	9,00	1	0160	Pleistocene afzettingen	05/05/1998	05/05/2018	12,00	168,00	
Watnr	Exploitant	Adres expl	Postcode exploitant	Gemeente exploitant	Nacebelsector	Klasse	Vlarem rubriek	Vergunningverlenende overheid	X inst	Y inst	Plaatsomschrijving	Diepte	Vergund aantal putten	Aquifercode	Aquiferbeschrijving	Begindatum vergunning	Einddatum vergunning	Vergund dagdebiet	Vergund jaardebiet
ANT-gw2/4759	[REDACTED]	2070	ZWIJNDRECHT	Rundveehouderij	2	53.8.2<17	Gemeentebestuur ZWIJNDRECHT	147081,00	212997,00	A-532g	5,00	1	0160	Pleistocene afzettingen	20/06/2006	20/06/2026		847,00	
Watnr	Exploitant	Adres expl	Postcode exploitant	Gemeente exploitant	Nacebelsector	Klasse	Vlarem rubriek	Vergunningverlenende overheid	X inst	Y inst	Plaatsomschrijving	Diepte	Vergund aantal putten	Aquifercode	Aquiferbeschrijving	Begindatum vergunning	Einddatum vergunning	Vergund dagdebiet	Vergund jaardebiet
ANT-gw2/2442	[REDACTED]	9120	BEVEREN	Fruitteelt	A	99	Gemeentebestuur ZWIJNDRECHT	147035,00	213005,00	1-A-540a	9,00	1	0160	Pleistocene afzettingen	05/05/1998	05/05/2018	10,00	1000,00	

INDAVER

3M Belgium bvba
Mevr. Ingrid Wouters
Canadastraat 11 Haven 1005
B-2070 ZWIJNDRECHT

Certificaat

Bijlage bij factuurnr.	Certificaatnr.	Pag.
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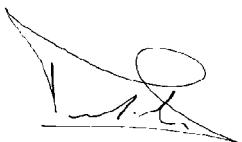
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Indaver Logistics nv
Boomsesteenweg 199
B-2830 WILLEBROEK

Delnr / Matnr / Omschrijving	EURAL code	Aanleverings Datum	R/D	Gewicht
87763171 531654 ABF10121 VERONTR.GROND	17 05 03*	2019.01.03	D10	5.220 KG

Indaver n.v., Ketenislaan 1, Burcht Singelberg, Blok D, Haven 1548, BE- 9130 Kallo, verklaart hierbij de hogervermelde afvalstoffen conform de haar opgelegde vergunningsvoorraarden afgevoerd te hebben naar de hierboven vermelde inrichting voor verdere recyclage of verwerking.



Paul De Bruycker
CEO



Michel Decorte
CFO

aktiviteitennr. Indaver:

- ophaler: 833 E30 - vervoerder: 833 E30

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Maatschappelijke zetel: Burcht Singelberg, Blok D, (Lady Hedwige Tower) - Haven 1548, Ketenislaan 1 - 9130 Kallo
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KBC 409-0507001-26; IBAN: BE59 4090 5070 0126; BIC: KREDBEBB



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Myanmar	Vietnam	

uw bericht van		afdeling	Bodembeheer
uw kenmerk		dienst/team	Team Lokale en bovenlokale besturen
bijlagen		contactpersoon	Klantenbeheer (██████████)
Mechelen	23.07.2019	ons kenmerk	BB-LB2-KB-20190429400 (Dossiernummer: 732)

Evaluatierapport na schadegeval: Zwijndrecht – Canadastraat 11

Geachte mevrouw,

De OVAM heeft het onderzoeksverslag van 12 juni 2019 met als titel 'Evaluatie rapport schadegeval, Incident Hydraulische olie - December 2018, Canadastraat 11 te 2070 Zwijndrecht', opgesteld door ERM NV, goed ontvangen. Het onderzoeksverslag werd uitgevoerd op de grond ter hoogte van de Canadastraat 11 in Zwijndrecht en is kadastral gekend als:

Gemeentenr.	Afdeling	Sectie	Grondnr.	Bisnr.	Exp.1	Exp.2
11056	ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	467		E	

Op 10 december 2018 is er ter hoogte van het terrein gelegen aan de Canadastraat 11 in Zwijndrecht een schadegeval ontstaan door een breuk van een hydraulische leiding van een boorinstallatie ter hoogte van een onverhard deel van het terrein. Hierbij kwam ongeveer 10-tal liter hydraulische olie vrij met mogelijke bodemverontreiniging tot gevolg.

Volgende maatregelen werden uitgevoerd:

- er werden absorberende korrels gestrooid
- de vrijgekomen grond (+/- 1,5 m³) is tijdelijk op een folie geplaatst.
- er werden staalnames uitgevoerd van de bodem

Uit de analyseresultaten van de controlestalen na de uitgevoerde saneringswerken blijkt dat er geen restverontreiniging meer aanwezig is, die een verdere aanpak vereist volgens het Bodemdecreet.

De OVAM is op basis van de resultaten van het onderzoeksverslag van 12 juni 2019 van oordeel dat er voor dit schadegeval met hydraulische olie , ontstaan op 10 december 2018 afdoende maatregelen werden genomen en geen beschrijvend bodemonderzoek dient uitgevoerd te worden volgens het Bodemdecreet. Het dossier met betrekking tot dit schadegeval wordt hierbij dan ook afgesloten.

Beroep

Binnen zestig dagen na ontvangst ervan kan u tegen dit besluit een verzoekschrift tot schorsing of nietigverklaring indienen bij de Raad van State, Wetenschapsstraat 33 in 1040 Brussel. Dat verzoekschrift moet aangetekend worden verzonden, moet gedagtekend en ondertekend worden

door de partij of door een advocaat en moet enkele verplichte vermeldingen bevatten:

- het opschrift “verzoekschrift tot nietigverklaring” als het niet eveneens een vordering tot schorsing bevat;
- de naam, hoedanigheid, woonplaats of zetel van de verzoekende partij en de gekozen woonplaats;
- het voorwerp van het beroep en een uiteenzetting van de feiten en de middelen;
- de naam en het adres van de verwerende partij.

Meer weten?

Voor bijkomende vragen kan u terecht:

- Bij de bodemsaneringsdeskundige die u heeft bijgestaan bij de uitvoering van de werken.
- Op www.ovam.be/schadegevallen.
- Bij het team klantenbeheer van de OVAM op [REDACTED] Wij beantwoorden uw vragen elke werkdag van 8.30 u tot 12.30 u en van 13.30 u tot 16.30 u. Op maandag zijn we bereikbaar vanaf 10 u, op vrijdag tot 16 u
- Of mail naar bodem@ovam.be.

Wij vragen u het dossiernummer **732** te vermelden als u contact opneemt met de OVAM.

Hoogachtend,



20190429400

Ann Cuyckens
Afdelingshoofd

Kopie:

3M Belgium bvba Canadastraat 11 2070 Zwijndrecht
Gemeente Zwijndrecht Binnenplein 1 2070 ZWIJNDRECHT

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Ierland	Thailand
Italië	VAE
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