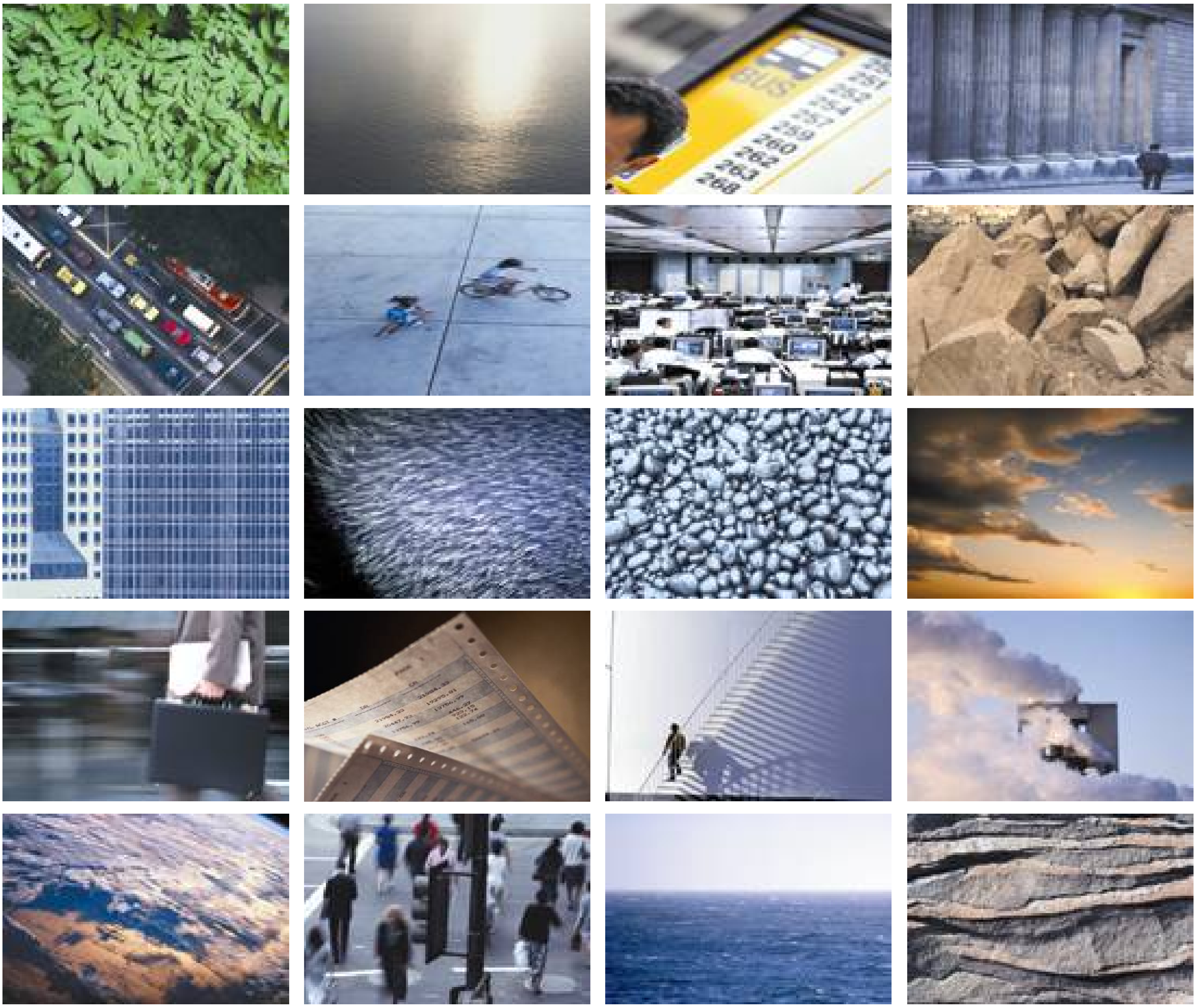

Eerste gefaseerd bodemsaneringsproject - Zevende tussentijds verslag bodemsaneringswerken

Periode juli 2015 - juli 2016

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

15 september 2017

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RAPPORT

Eerste gefaseerd bodemsaneringsproject - *Zevende tussentijds verslag bodemsaneringswerken*

Periode juli 2015 – juli 2016

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

Projectnr. : 0341743

Ref. : R01 0341743 TTV7 v2

Voor en namens ERM nv

Opgemaakt door: Ruben Maes
Tine Mandonx
Mattias Verbeeck
Nanda Hermes

Goedgekeurd door: Dirk Nuyens
Functie: Principal Partner
Handtekening:

Datum: 15 september 2017

INHOUDSTAFEL

DEEL 3 *BIJLAGEN*

Bijlage 1 OVAM conformverklaring eerste gefaseerd bodemsaneringsproject
Bijlage 2 Milieukundig dagboeken
Bijlage 3 Analysecertificaten
Bijlage 4 Overzicht veldmetingen en analyseresultaten
Bijlage 5 Statistische evaluatie analyseresultaten Blokkersdijk
Bijlage 6 Schematische weergave pompput en inspectiekamer

DEEL 4 **KAARTMATERIAAL**

Kaart 1 Situering onderzoekslocatie op topografische kaart
Kaart 2 Overzichtsplan onderzoekslocatie
Kaart 3 Detailplan ligging grondhopen
Kaart 4 Detailplannen monsternamelocaties
Kaart 5 Onttrekkingsfilters grondwateronttrekking

LIJST VAN AFKORTINGEN

BTEX	Benzeen, tolueen, ethylbenzeen en xylenen
ds	Droge stof
FC	Organofluoroverbindingen
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
WWTP	Bedrijfswaterzuiveringsinstallatie 3M (“waste water treatment plant”)

DEEL 1: ADMINISTRATIEVE GEGEVENS

Eerste gefaseerd bodemsaneringsproject -
Zevende tussentijds verslag bodemsaneringswerken
Periode juli 2015 - juli 2016

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

25 augustus 2017

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ADMINISTRATIEVE GEGEVENS

In Tabel 0.1 zijn de administratieve gegevens van het rapport samengevat. De administratieve gegevens van het perceel waarop de bodemsaneringswerken van toepassing zijn, zijn opgenomen in Tabel 0.2.

Tabel 0.1 Administratieve gegevens van het rapport

Titel:	Eerste gefaseerd bodemsaneringsproject - Zevende tussentijds verslag bodemsaneringswerken: Periode juli 2015 – juli 2016 3M Belgium bvba Canadastraat 11 te 2070 Zwijndrecht
Referentie EBSD:	R01 0341743 TTV7 v1.docx
Rapportdatum:	25 augustus 2017
Onderzoekslocatie:	3M Belgium bvba
- Straat + nummer of omschrijving:	Canadastraat 11
- Postcode:	2070
- Fusiegemeente:	Zwijndrecht
- Deelgemeente:	Zwijndrecht
Aanleiding:	☐ Overdracht grond ☐ Sluiting bedrijf ☐ Stopzetting activiteit ☐ Overdracht grond + sluiting bedrijf ☐ Periodieke verplichting ☒ Decretaal verplicht ☐ Decretaal vrijwillig ☐ Vrijwillig door derden ☐ Ambtshalve[1] ☐ Verspreiding ☐ Onteigening ☐ Onbekend
Naam opdrachtgever:	3M Belgium bvba
- Straat + nummer:	Hermeslaan 7
- Postcode:	1831
- Fusiegemeente:	Machelen
- Land:	België
- Telefoon:	-
- Fax:	-
- E-mail:	-

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[1] Ambtshalve: indien de bodemsaneringsdeskundige van de OVAM de opdracht heeft gekregen het beschrijvend bodemonderzoek op te stellen.

Titel:	Eerste gefaseerd bodemsaneringsproject - Zevende tussentijds verslag bodemsaneringswerken: Periode juli 2015 – juli 2016 3M Belgium bvba Canadastraat 11 te 2070 Zwijndrecht
Referentie EBSD:	R01 0341743 TTV7 v1.docx
Rapportdatum:	25 augustus 2017
Hoedanigheid:	☒ Eigenaar ☐ Gebruiker ☒ Exploitant ☐ Optredend in opdracht van de eigenaar/gebruiker/exploitant ☐ Andere: …………
Naam contactpersoon:	Charlotte Tack
- Telefoon:	[redacted]
- Fax:	[redacted]
- E-mail:	[redacted]
Naam contactpersoon ter plaatse:	Charlotte Tack
- Telefoon:	[redacted]
- Fax:	[redacted]
- E-mail:	[redacted]
Naam bodemsaneringsdeskundige:	Environmental Resources Management - ERM nv
Naam contactpersoon:	Nanda Hermes
- Telefoon:	[redacted]
- Fax:	-
- E-mail:	[redacted]
Dossiernummer OVAM:	732

Tabel 0.2 *Identificatie van de betrokken kadastrale percelen*

						Persoon (Eigenaar / gebruiker / exploitant)								
Gemeentenummer	Sectie	Perceelnummer	Adres	Gemeente	Oppervlakte perceel (m^2)	Periode Van	Periode Tot	Type (3)	Naam	Adres	Letter (4)	Huidig bestemmingstype (1)	Toekomstig bestemmingstype (1)	Grondwaterkwetsbaarheid (2)
11056	A	467E	Canadastraat 11	Zwijndrecht	321 598			Ex	3M Belgium bvba	Canadastraat 11 2070 Zwijndrecht	A	V	V	Ca1/v

Overige percelen: *zie conformverklaring BSP in Bijlage 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 strengste code.

(3) Bij het type voor de eigenaars en gebruikers geeft u aan of de betrokkene 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: RAPPORT

Eerste gefaseerd bodemsaneringsproject -
Zevende tussentijds verslag bodemsaneringswerken
Periode juli 2015 - juli 2016

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

25 augustus 2017

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EXECUTIVE SUMMARY

Environmental Resources Management – ERM nv (ERM) was contracted by 3M Belgium bvba (3M) to conduct the environmental supervision of the implementation of the phased remedial action plan (RAP), in accordance with the Flemish Soil Decree, at the 3M facility located in Zwijndrecht (Belgium).

Site location map showing the 3M site, the Blokkersdijk nature reserve and the Schelde River

Between 1994 and 2006, certified remedial experts Lisec nv and Arcadis Gedas nv conducted the Preliminary and Descriptive Soil Investigations at the 3M site. During these investigations, soil with elevated levels of fluorchemicals (FC's) and mercury, and groundwater impacted with FC's and aromatic compounds (mainly Xylenes), were identified. In 2008, the first phase of the remedial action plan (RAP) was submitted by Arcadis Gedas nv and in early 2009 the Flemish Soil Agency (OVAM) approved the RAP.

Currently, the first phase of the RAP is being implemented to remediate groundwater containing FC's in the production area (Building 16) and the waste water treatment plant (WWTP) area. In addition, the Blokkersdijk nature reserve and the second aquifer are being monitored regularly to assess the potential for migration of FC's from the 3M property to these areas. A second phase of the RAP will address the FC's present near the southern site boundary and in the "Palingbeek" (a ditch), but only after the civil works related to the Oosterweel connection (a road infrastructure project) have been finalized and the impact of the FC concentrations on the new ditch is known.

Progress on the remedial actions is reported on a yearly basis to OVAM. This interim report contains an evaluation and summary of the results of the remedial actions and monitoring undertaken between July 2015 and July 2016.

In the period between July 2015 and July 2016, the following remedial and monitoring actions were performed at the site:

1. Operation and Maintenance (O&M) of the installed pump and treat (P&T) system in the two source areas (Building 16 and WWTP). The source removal system was installed to reduce FC concentrations in groundwater and mitigate the FC migration in these two areas. Groundwater extracted from the wells in these two source areas is directed to the plant’s wastewater treatment system for removal of FCs. Samples of the groundwater discharged from the extraction wells are taken regularly to monitor the efficiency of the P&T system.

2. Water quality at the following locations is monitored periodically:

   - Groundwater and pumping wells in the FC source areas (Building 16 and WWTP);
   - Second aquifer wells (groundwater);
   - Groundwater wells along the southern site boundary;
   - Two wells (pH, conductivity and TPH) in the tank farm area (near Building 03);
   - Blokkersdijk wells and pond, including 3M pond (groundwater and surface water);
   - Surface water in the Paling and Tophat ditches; and
   - Stormwater collector pit and effluent WWTP.

3. Calculation of the PFOS mass flux to the Schelde River via the 3M sewer system and the Paling and Tophat ditches.

4. Evaluation of the analytical results of the water samples reported by 3M Environmental Laboratory versus the analytical results reported by SGS laboratory for the duplicate samples.

In addition, the following related activities have been conducted during this period:

5. Visual inspection of the soil pile impacted with FCs and Mercury at the request from OVAM.

6. Decommissioning of the Zwijndrecht Z-wells located south of the E34 motorway (near Zwijndrecht) after the final evaluation report was approved by OVAM.

7. Environmental follow up of the following environmental incident at the site: PTBA spill between Building 016 and 003 (August 2015).

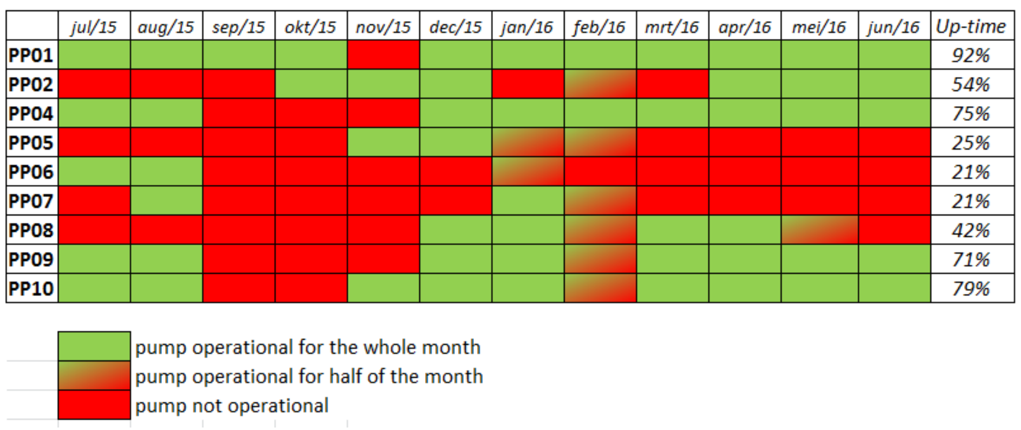
Follow-up of the P&T System

In 2010, the P&T system in the two source areas (Building 16 and WWTP area) was initiated with the startup of one extraction well in each area. Five additional extraction wells were installed in the source areas in late 2010 and started fully operating in August 2011. In early 2012, two additional extraction wells were installed in the WWTP area. These wells became fully operational in May 2012. At present, the system comprises nine extraction wells, three wells to remediate groundwater in the Building 16 source area and six wells in the WWTP source area.

ERM inspects the P&T system regularly to verify that it is operating correctly, to check on sand and iron build up in the extraction wells and to supervise the cleaning of the wells, if needed. In addition, water samples are taken from the wells (in 2013: bimonthly for the seven oldest wells and monthly for the two newest wells; as from 2014: quarterly for all nine wells) to track contaminant concentrations in these wells and groundwater levels are monitored to assess the impact of the P&T system on the natural groundwater levels and flow.

The ERM inspections show that the extraction system requires regular maintenance to remove sand and iron deposits accumulating in the wells. The average up-time of the remedial system was estimated to be 53% for this reporting period(2014-15: 75% and 2013-14: 71%), but the up-time varies from well to well: minimum 21% for well PP06 and PP07 and maximum 92% for wells PP01 as shown in the table below.

P&T system – Monthly summary of system up-time

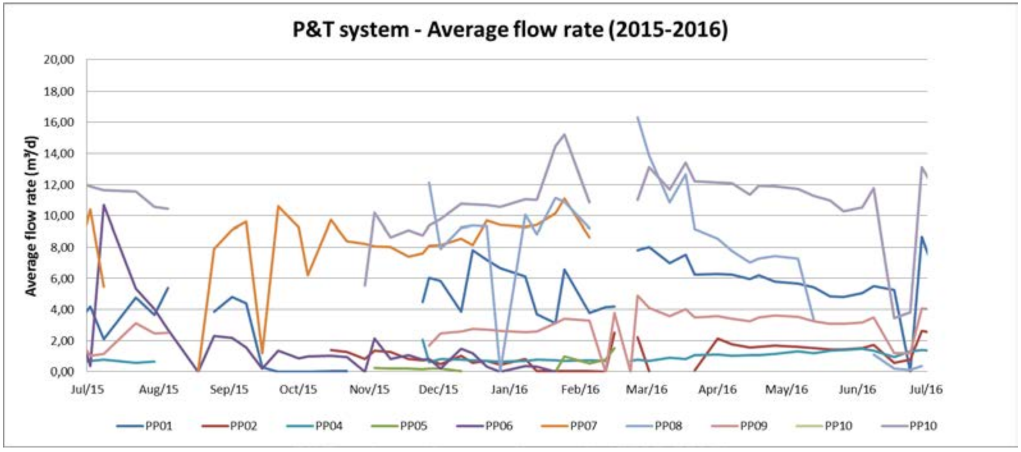


	jul/15	aug/15	sep/15	okt/15	nov/15	dec/15	jan/16	feb/16	mrt/16	apr/16	mei/16	jun/16	Up-time
PP01	🟩	🟩	🟩	🟩	🟥	🟩	🟩	🟩	🟩	🟩	🟩	🟩	*92%*
PP02	🟥	🟥	🟥	🟩	🟩	🟩	🟥	▨	🟥	🟩	🟩	🟩	*54%*
PP04	🟩	🟩	🟥	🟥	🟥	🟩	🟩	🟩	🟩	🟩	🟩	🟩	*75%*
PP05	🟥	🟥	🟥	🟥	🟩	🟩	▨	▨	🟥	🟥	🟥	🟥	*25%*
PP06	🟩	🟩	🟥	🟥	🟥	🟥	▨	🟥	🟥	🟥	🟥	🟥	*21%*
PP07	🟥	🟩	🟥	🟥	🟥	🟥	🟩	▨	🟥	🟥	🟥	🟥	*21%*
PP08	🟥	🟥	🟥	🟥	🟥	🟩	🟩	▨	🟩	🟩	▨	🟥	*42%*
PP09	🟩	🟩	🟥	🟥	🟥	🟩	🟩	▨	🟩	🟩	🟩	🟩	*71%*
PP10	🟩	🟩	🟥	🟥	🟩	🟩	🟩	▨	🟩	🟩	🟩	🟩	*79%*

🟩	pump operational for the whole month
▨	pump operational for half of the month
🟥	pump not operational

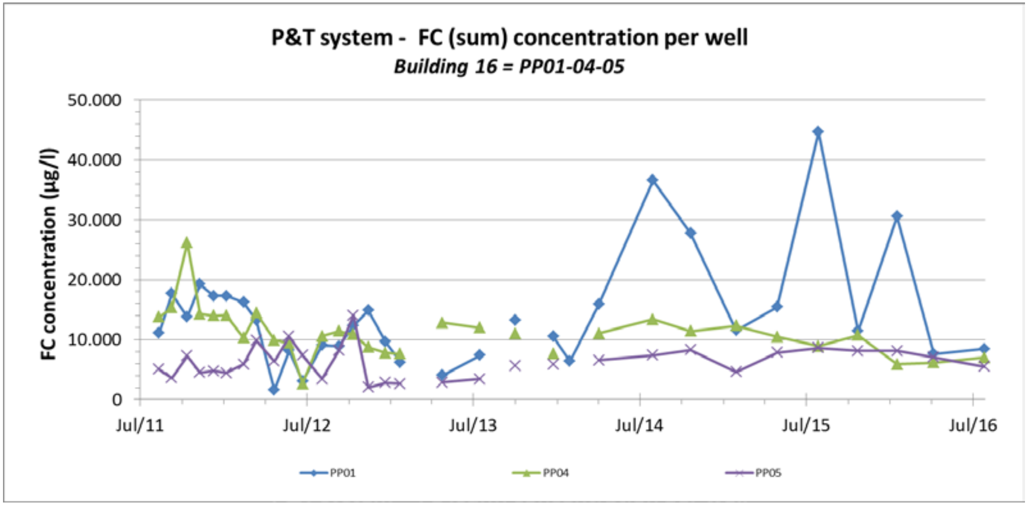
The average flow rate of the system (excluding well PP05 that operates intermittently) varies between minimum 0,03 m^3/day (well PP02) and maximum 16,3 m^3/day (well PP10) as shown in the following table.

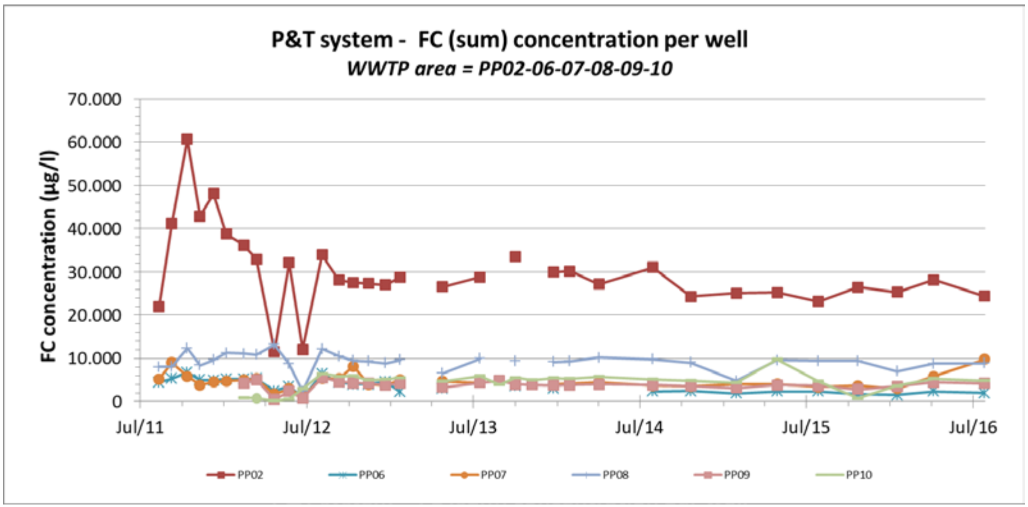
P&T system – Average flow P&T wells (July 2015 till July 2016)



The average groundwater FC concentration ranges between 1,87 mg/L (PP06) to 26,1 mg/L (PP02), depending on the extraction well, but the concentrations fluctuate significantly (see graph below) and further follow up is needed. The long-term trend in FC concentrations in most of the extraction wells is stable, although fluctuations can occur.

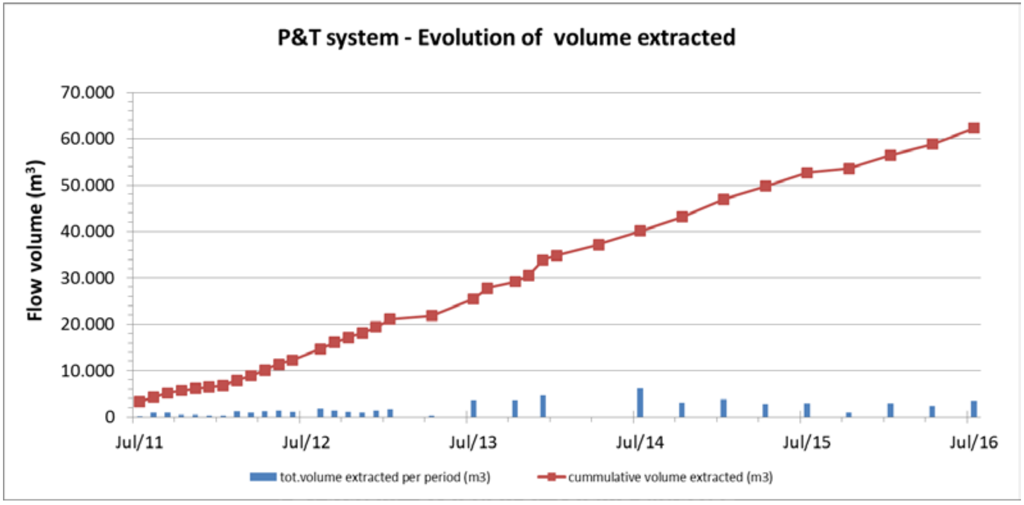
P&T system – Concentration Trends in Building 16 and WWTP area

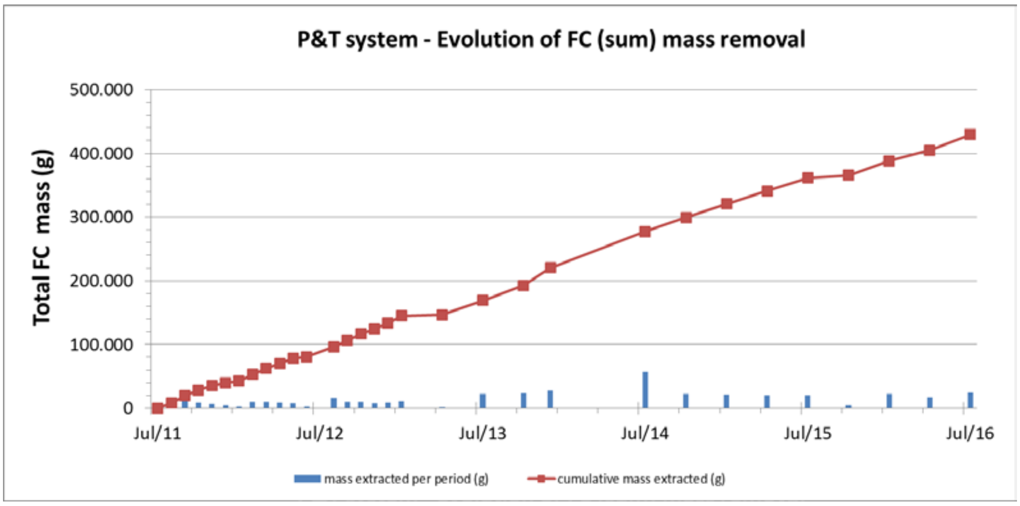




Between July 2011 and June 2016, approximately 62.316 m³ of contaminated groundwater was extracted and approximately 429,4 kg of FC's were removed, as shown in the graphs below.

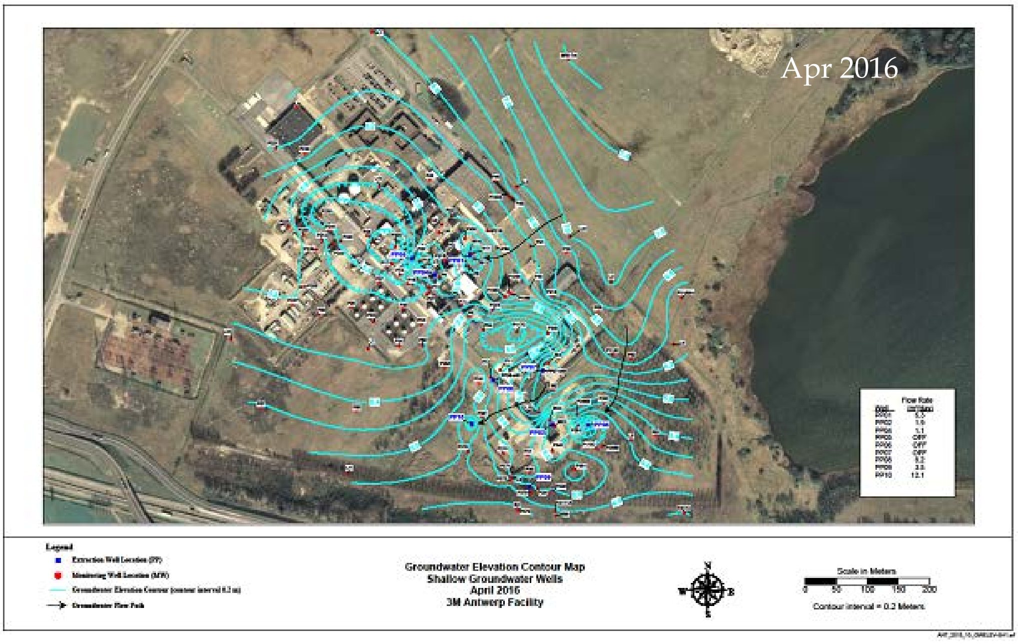
P&T system – Efficiency Trends





The system has a clear impact on the natural groundwater flow pattern of the first aquifer, as is shown in the following figure.

P&T system – Impact on groundwater flow pattern of the first aquifer



The stability of the buildings in the extraction areas was monitored on a six-monthly basis by an independent geotechnical expert to ensure no subsidence is occurring. The stability monitoring done in 2013 indicated that the settlement caused by the extraction system stayed within the predefined limits. Based on the recommendations of an independent geotechnical expert (Grontmij nv), a final measurement will be done when all pumping wells are operational to verify the findings of 2013 and to evaluate if the monitoring is necessary.

In accordance with the RAP specifications, ERM recommends that regular inspections of the system (at a minimum monthly frequency) and quarterly measurement of the contaminant concentrations in the extraction wells be continued. Groundwater levels will be measured quarterly to allow regular assessment of the impact of the P&T system on the natural groundwater flow pattern.

ERM further recommends evaluating the operation of the extraction system to assess the need for a potential extension of the remedial system to improve contaminant capture and up-time.

Monitoring Groundwater, Surface Water and Storm Water Quality

The remedial objectives of the monitoring program are to verify that the P&T system is mitigating the horizontal migration in the first aquifer in the areas of concern (Blokkersdijk nature reserve and the Paling ditch at the southern site boundary) as well as the vertical migration of contaminants from the first to the second aquifer. As part of this monitoring program, ground-, surface and storm water samples are taken and analyzed for FC's.

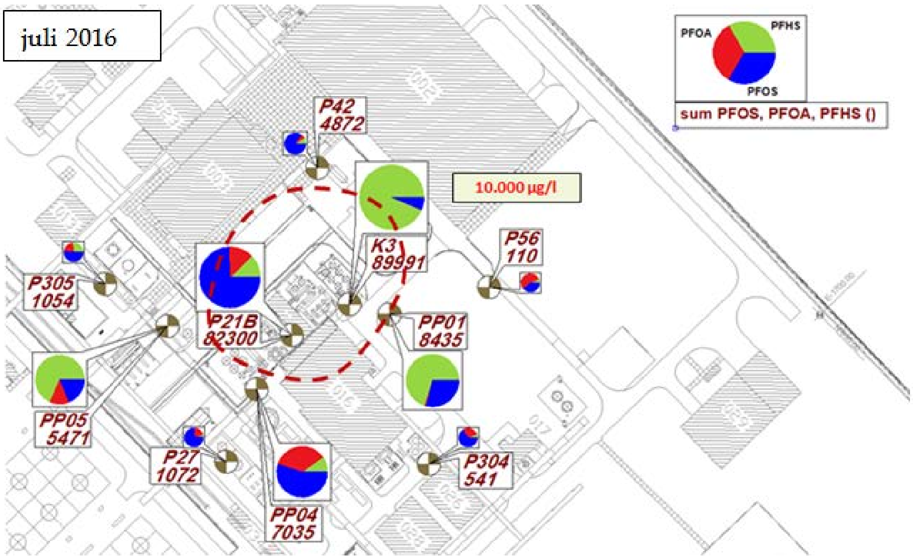
To evaluate the impact of the P&T system on groundwater quality of the first phreatic aquifer in the two **FC source areas** the following monitoring wells are sampled periodically:

- In the Building 16 area: two groundwater wells are sampled annually, four groundwater wells are sampled semi-annually and 1 well is sampled quarterly, and;
- In the WWTP area: Six groundwater wells are sampled annually, ten wells sampled semi-annually and one well is sampled quarterly.

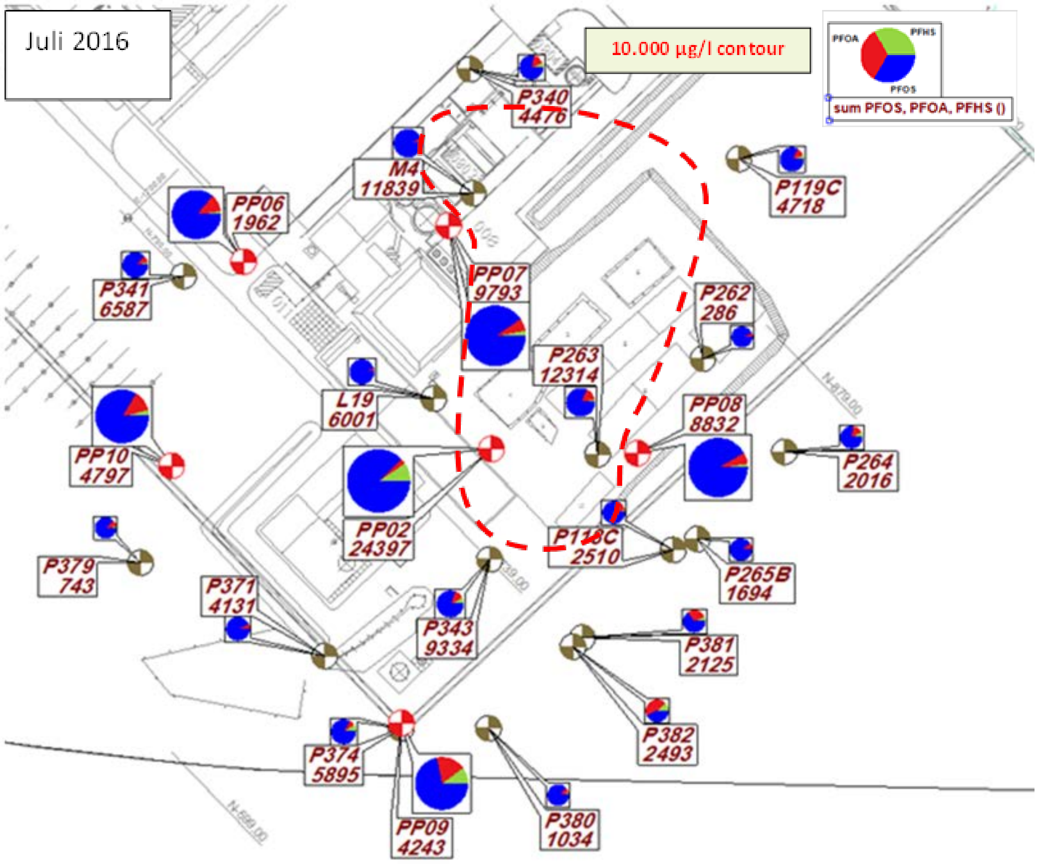
The results of the July 2016 monitoring are shown in the figures below with the sum FC-concentration contour (10.000 µg/L).

FC Source Areas - Results July 2016 Monitoring

Building 16 source area



WWTP source area



The general stable/decreasing trend in FC concentrations suggests that the shallow P&T system installed in the two source areas is reducing the concentrations of FC's in groundwater in the Building 16 and WWTP area.

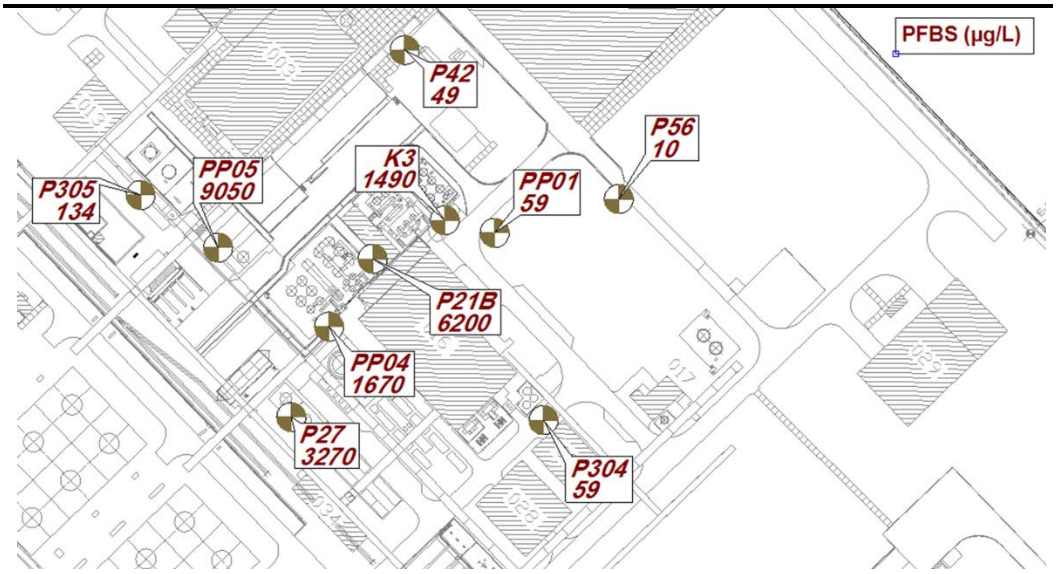
Based on a review of the historic monitoring results, groundwater concentrations in the Building 16 area are stable or decreasing, except for the FC concentrations in well P21B. The extent of the sum of FC concentrations contour appears to be equal to the contour based on the results of the previous monitoring round (2014-15).

In the WWTP area, groundwater FC concentrations in most of the wells are stable or decreasing, except for wells P119C and P263. The PFOS concentration in these wells has increased. The extent of the sum of FC concentrations contour has decreased compaired to the contour of the July 2015 results, but appears to be equal to the contours based on the October 2015 and January 2016 monitoring results.

The use of PFOS on-site was eliminated in 2002 and was replaced with PFBS; PFBS has a significantly lower toxicity than PFOS. Prior to the start of the production of PFBS, this compound was extensively screened by 3M for potential (eco)toxicological effects. PFBS replaced PFOS because it has a significantly shorter elimination half-life, is persistent but does not bio-accumulate, and has a high PNEC value (10.040 µg/L) compared to PFOS. Recent groundwater analytical data indicates the presenceof PFBS in the area of Building 16. The highest concentrations was measured in extraction well

PP05 in 2014. During the July 2016 monitoring event, additional wells were sampled and analyzed for PFBS to assess the extent of PFBS present in groundwater. The results of this additional monitoring are shown in the following figure. The highest groundwater concentrations are again measured near extraction well PP05, in monitoring well P21B. However, the concentrations in extraction well PP05 have continued to decrease since the measurement of 2014. Concentrations in well P21B are stable.

FC Source Area near Building 16 – PFBS Results July 2016 Monitoring



Building 16 source area

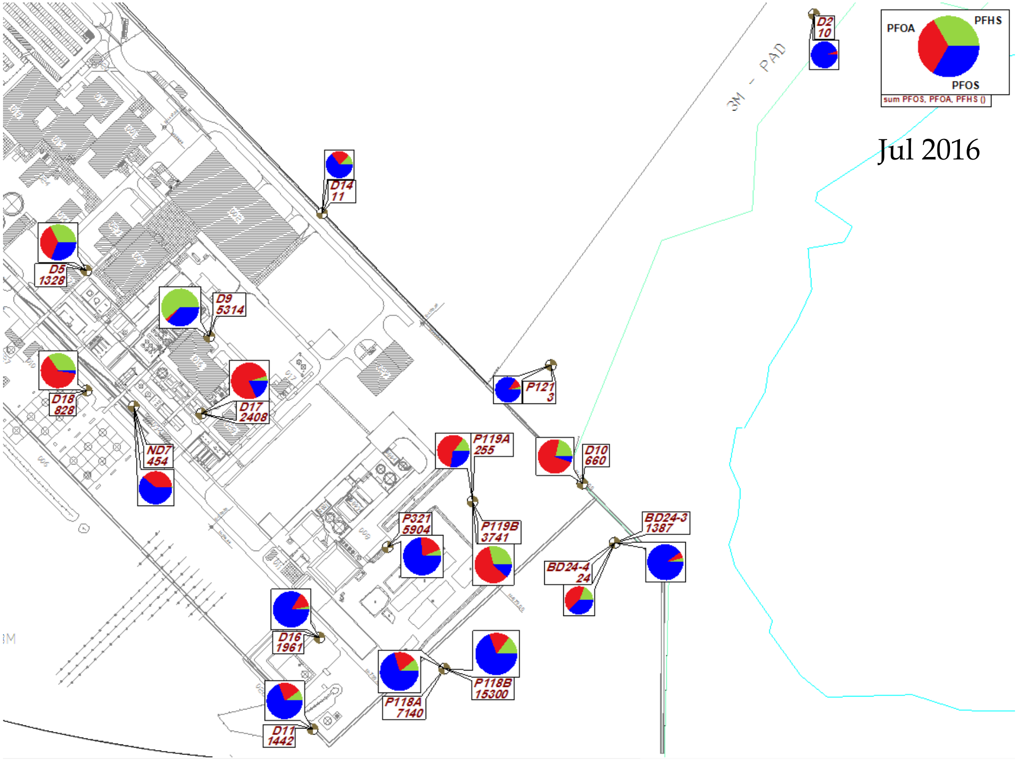
The cause of the PFBS detection and the observed concentration increase is, at present, unknown, but will continue to be investigated.

ERM recommends continuing the current groundwater monitoring program for wells completed in the shallow (1st) aquifer. In addition ERM recommends assessing the need for further optimization of the P&T system in the first aquifer as some wells show increasing concentration trends.

To monitor the groundwater quality of the **second aquifer**, six wells are sampled annually, three wells are sampled on a semi-annual basis and one well on a quarterly basis. Two wells are located in, or downgradient, of the Building 16 source area and eight wells in, or downgradient, of the WWTP. The monitoring results are used to evaluate the effectiveness of the shallow P&T system in mitigating the vertical migration of contaminants from the first aquifer to the second aquifer.

The results of the July 2016 monitoring are shown in the following figure. In order to better evaluate the evolution of PFC concentrations in the second aquifer, 8 additional monitoring wells located over the site, with a screen in the 2nd aquifer were monitored in July 2016.

Second Aquifer - Results July 2016 Monitoring



The evaluation of the monitoring results shows that the groundwater concentrations in the second aquifer fluctuate and are increasing in several wells in the WWTP area. The lower uptime of the groundwater extraction system may be a contributing factor. Efforts have been made to improve system uptime to enhance capture. Continued monitoring of these wells is also needed to assess the long-term trends and effectiveness. The optimization of the remedial system is expected to further decrease the potential for downward contaminant migration.

Seven of the eight additional wells that were sampled in July 2016, had higher concentrations than measured in the past. To assess the potential impact of lab changes and natural concentration fluctions, further monitoring is recommended to verify this observation.

The FC concentrations are below the five time limit value based on the concentrations measured during startup of the remediation system (mid 2011). At the northeast property boundary (downgradient from the source areas in the first aquifer) concentrations are below 10% of the solubility, meaning that remedial criteria specified in the RAP are being met.

Based on the results of the monitoring, ERM recommends that the impact of the P&T system on the FC concentrations in the second aquifer would be assessed through continued monitoring. In addition, ERM recommends:

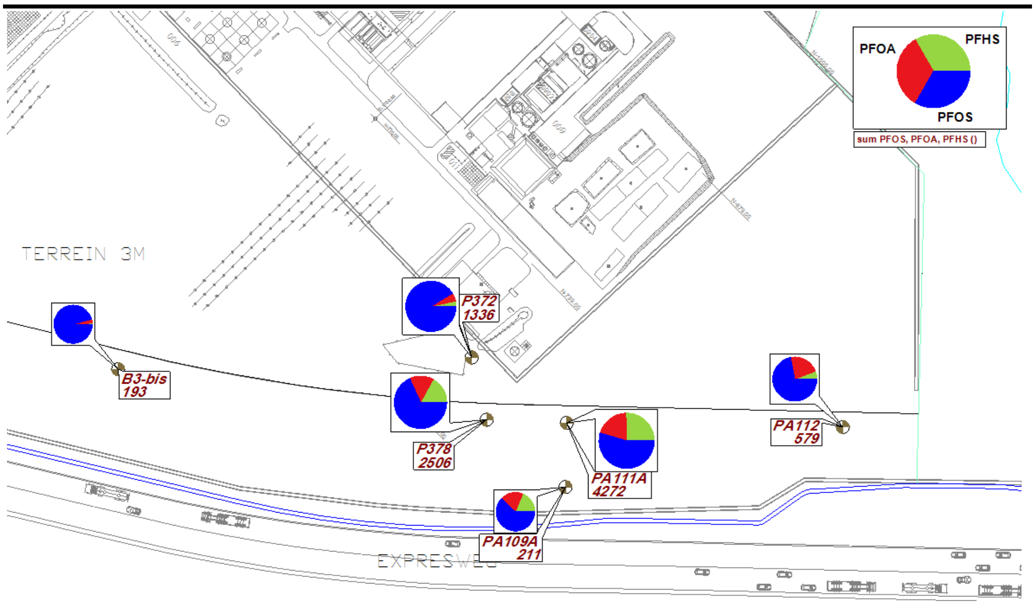
- To keep the current monitoring frequency for monitoring wells that were already in the 2016 monitoring program;

- Add seven of the wells, that were sampled for the first time in July 2016 (D5, ND7, D16, D17, D18, P119A & P119B), to the annual monitoring to verify the findings; and

- To optimize of the operation of P&T system in the first aquifer, as several wells show increasing concentration trends.

The groundwater quality along the **<u>southern site boundary</u>** is currently being monitored semi-annually in seven wells. The results of the July 2016 monitoring are shown in the following figure. Well B3 was replaced in October 2015 by a new well (B3-bis).

Southern Site Boundary - Results July 2016 Monitoring



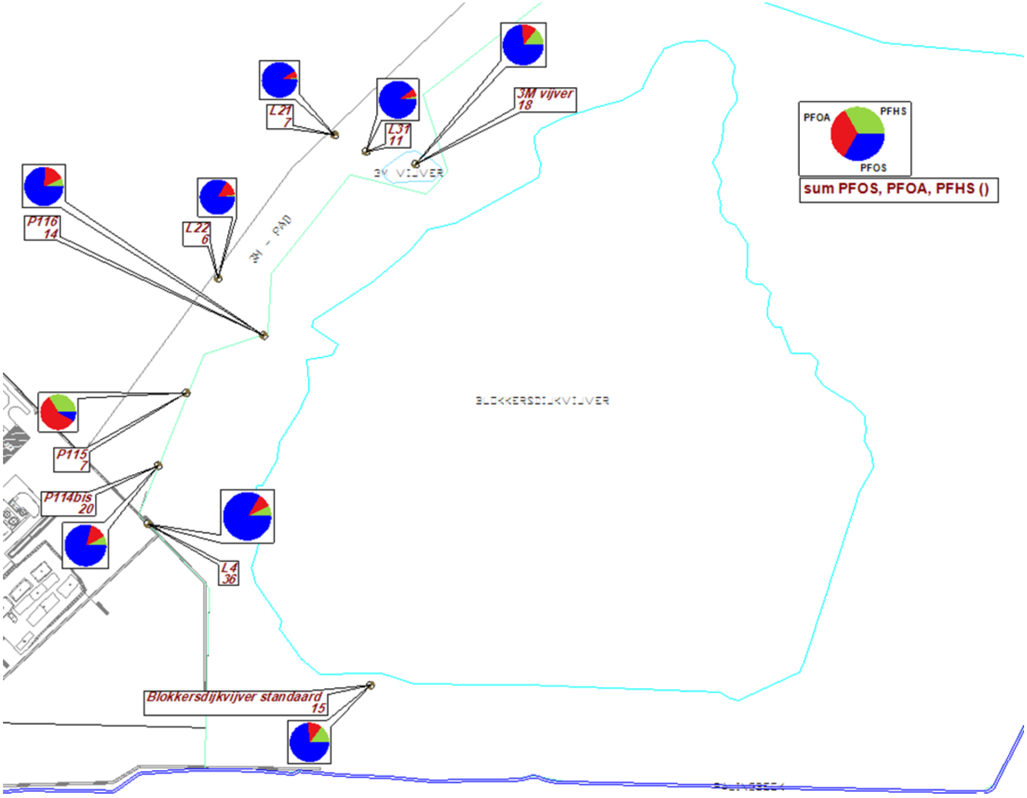
The evaluation of the monitoring results shows that, in general, the groundwater concentrations over time are decreasing or stable, but concentrations may fluctuate. The results suggest that the shallow P&T system installed at the WWTP source area is preventing contaminant migration to the southern site boundary. ERM recommends continuing the current groundwater monitoring program (semi-annual sampling).

The groundwater quality in the **<u>tank farm area</u>** (near Building 3) is currently monitored annually for <u>TPH</u>. The delineation investigation showed that pH, conductivity and TPH were higher in wells P18 and P28 compared to other wells in the tank farm area. The measured TPH concentration in well P28 has been significantly increased compared to previous measurements (12.000 µg/L).

The source of the TPH contamination is unknown but may be caused by interference from another impact; the elevated pH and conductivity are thought to be related to (minor) spills from the nearby KOH tank. Further historical and analytical investigation is ongoing. It is also recommended to continue the annual monitoring for TPH in wells P18 and P28.

Groundwater and surface water quality in, and close to, the **Blokkersdijk nature reserve** is monitored via eight wells and two ponds. Seven groundwater wells and two surface water locations are monitored quarterly to statistically evaluate the trend of FC concentrations. The results of the July 2016 monitoring are shown in the figure below.

Blokkersdijk Nature Reserve - Results July 2016 Monitoring



After the approval of the remedial action plan in 2009, periodic sampling has been conducted to collect water samples in the Blokkersdijk nature reserve pond, the 3M pond and selected wells adjacent to these two ponds. Analytical results from these water samples have been used for the PFOS trend analysis as described in the approved remedial action plan.

Since the start of the remedial monitoring, different laboratories have been used to analyze the collected water samples (due to quality issues) and concern has been raised that the use of multiple laboratories over the period of data collection could have an impact on the results of the PFOS trend analysis. An evaluation in 2014 showed that lab changes did confound the results of the PFOS trend analysis and the significant upward trends in three locations (L21, P114, and 3M vijver) appeared to be due to changes in analytical laboratories, but this needed to be confirmed. For this reason only data analyzed by 3M is utilized for future trend analysis.

When the PFOS concentration trends were evaluated for only the data analyzed by 3M Environmental Lab, a significant increasing trend is observed in well L21 and a significant decreasing trend in well L22. None of the other wells evaluated showed a statistically significant trend in PFOS. However, as the concentrations in L21 have been decreasing the last two sampling periods it is recommended to continue quarterly sampling in all the sample locations

in the Blokkersdijk area, to populate the data set.The graphical and statistical trend analysis should be continued.
There is no need for additional remedial actions in this area as there is no significant increasing trend observed in the Blokkersdijk pond.

However, a geohydrological investigation conducted in 2014, in the area of the 3M path, tentatively concluded that FC mass may leach from the old chemical sewer to the first phreatic aquifer and that the old sewer line could be a secondary contaminant source. The location of the sewer line, 3M path, nature reserve and 3M site is shown in the aerial picture underneath.

Location 3M site, Blokkersdijk nature reserve, 3M path and Schelde River

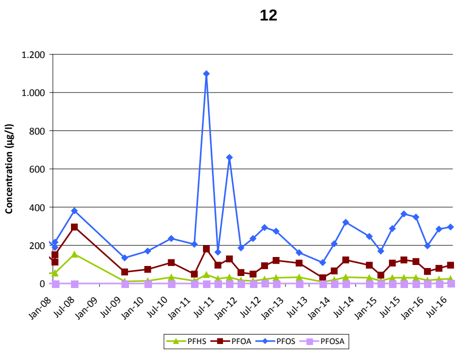
Further investigation (eg. mass calculations and groundwater modeling with contaminant migration) will be performed to assess the actual impact of the FC mass present in the sewer on the water quality of the first phreatic aquifer and nearby ponds.

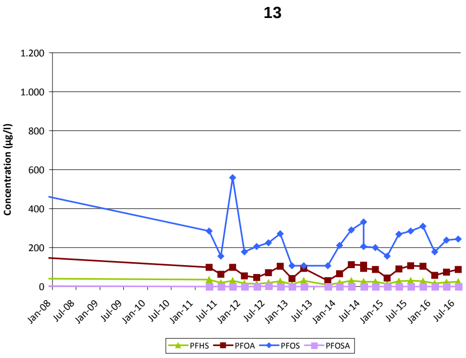
Based on the results of the monitoring, ERM recommends continuing the assessment of the FC concentrations in and close to the Blokkersdijk nature reserve through quarterly monitoring of the monitoring wells and the Blokkersdijk and 3M pond.

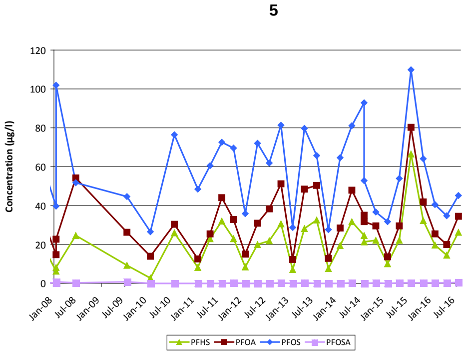
The quality of the surface water in the **Paling** and **Tophat ditches** is monitored quarterly for FC's at four different locations. The evaluation of the monitoring results shows that FC concentrations are stable at present, see graphs below, although concentrations can fluctuate. FC concentrations in the Tophat ditch are lower than those measured in the Paling ditch.

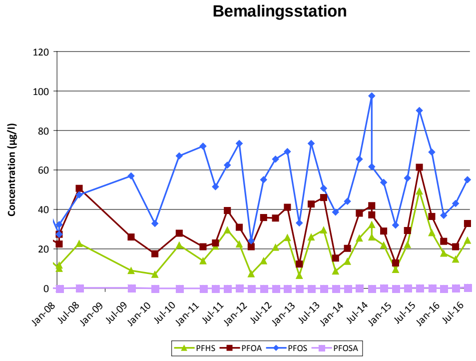
ERM recommends continuing the surface water monitoring at the four monitoring locations on a quarterly basis as the data is used to calculate the average PFOS mass flux to the Schelde River, and monitors the concentration trend in the Paling and Tophat ditch.

Paling and Tophat Ditch - Monitoring Results since 2008



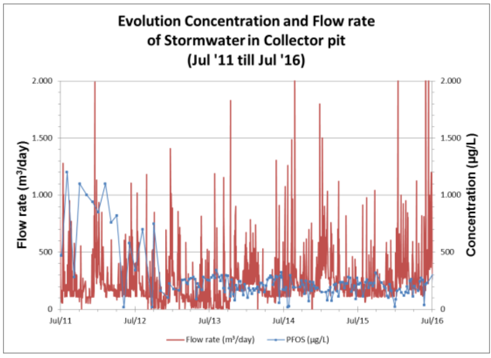


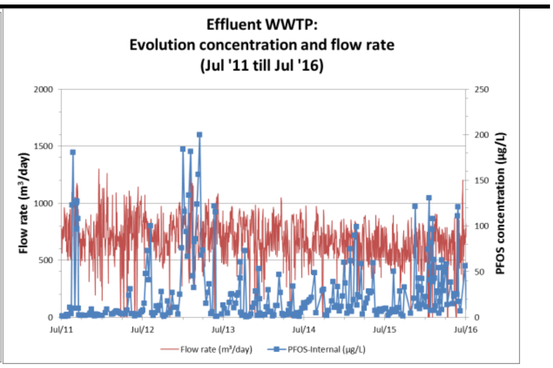




To monitor the PFOS mass flux leaving the 3M site due to discharge of the waste water treatment plant (WWTP) and infiltration of potentially contaminated groundwater into the storm sewer system, monthly samples are taken from the **WWTP** and **stormwater collector pit**. The monitoring results are also used to evaluate if the P&T system is effective in reducing the infiltration of contaminants into the stormwater sewer system. The collector pit results are less sensitive to fluctuations caused by rainwater and therefore more reliable.

Stormwater Collector Pit and WWTP - Monitoring Results





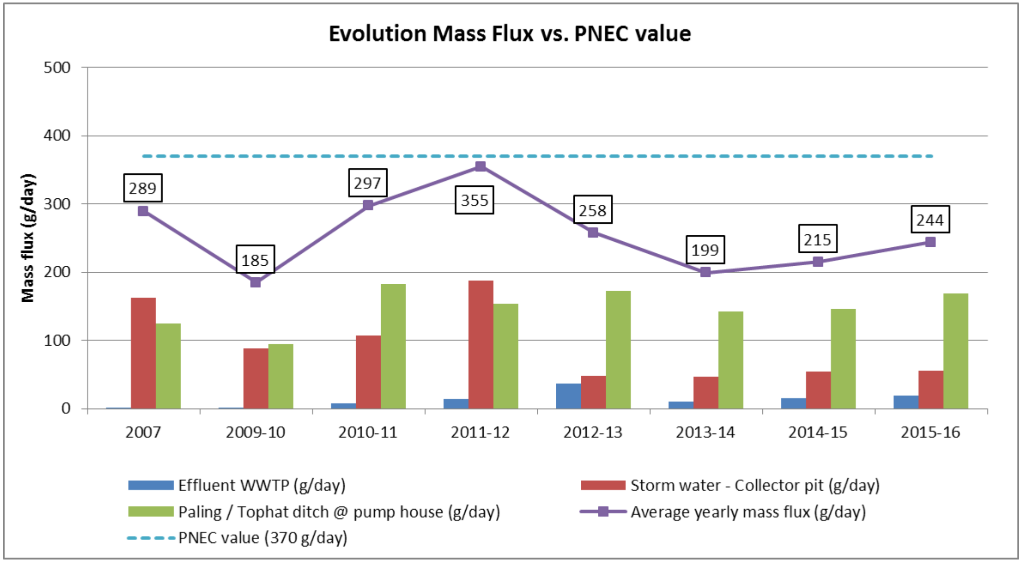
Since 2012-13, PFOS concentrations in the collector pit and waste water treatment plant have fluctuated, but show a stable trend (see figure above). The cleaning of the collector pit in 2012 has significantly decreased the mass flux, and therefore there is, at present, no need to install an additional treatment system (via GAC filters) on the effluent of the stormwater sewer.

The average PFOS concentration between July 2015 and July 2016 is 187 µg/L for the collector pit and 30,7 µg/L for the waste water treatment plant effluent discharged to the Schelde River. ERM recommends continuation of the weekly monitoring of the stormwater collector pit and waste water treatment plant by 3M, and quarterly monitoring by ERM.

PFOS Mass Flux to the Schelde River

The daily average mass flux of PFOS from the 3M site to the Schelde River was calculated and compared to the maximum allowable mass flux value based on the analytical results of samples collected between July 2015 and June 2016 from the effluent of the WWTP, the storm water collector pit and the Tophat ditch near the pump station. The calculated mass flux value (244 g/day) did not exceed the PNEC value (370 g/day). An overview of the calculated yearly mass flux of PFOS since 2007 is summarized in the following graph.

Average calculated Mass Flux of PFOS to the Schelde River



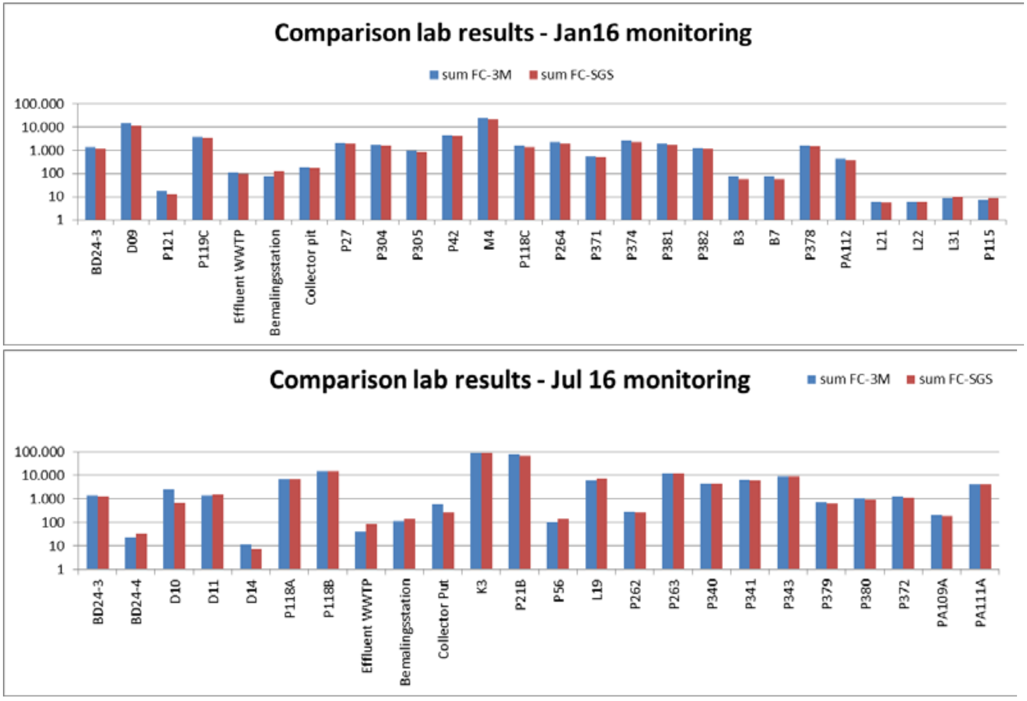
As discussed in previous reports, the higher mass flux calculated for the monitoring period 2011-2012 was likely caused by the accumulation of FC containing sludge at the bottom of the collector pit. This sludge was removed from the collector pit in September 2012. Since then, the FC concentrations in the collector pit have decreased, and also the average PFOS mass flux to the Schelde River decreased significantly. The monitoring of the yearly mass flux will be continued.

Evaluation of analytical results of water samples - SGS vs 3M Environmental Laboratory

Per the agreement reached with OVAM (meeting dd. 22 October 2013), all water samples taken in 2016 were analyzed by the 3M Environmental Laboratory in the USA. As verification, and to comply with local legislation, duplicate water samples were taken during the monitoring events and analyzed by SGS nv (an OVAM certified lab) as well as the 3M Environmental Laboratory. At least one duplicate sample is taken during one of the four monitoring rounds. For the sake of consistency in this report, only the 3M results are shown.

An evaluation of both data sets (see following graphs) shows that the results between both labs are comparable.

Lab results comparison (SGS vs 3M Environmental Laboratory)



For all monitoring events, average measured concentrations are higher for 3M lab compared to SGS:

- in October 2015 the 3M results were 34% higher;
- in January 2016 the 3M results were 15% higher ;
- in April 2016 the 3M results were 30% higher; and
- in July 2016 the 3M results were 22% higher.

The difference is likely due to different sample preparation techniques and analytical methods used by each laboratory (instrument parameters and calibration). The SGS result is in accordance with previous measured concentrations.

ERM recommends that in 2017 all water samples again be analyzed by the 3M Environmental Laboratory and the duplicate sampling to be spread out over the four monitoring events (similar to the monitoring in 2016). Duplicate water samples will be analyzed by the SGS laboratory during the monitoring period (January, April, July and October 2017).

Monitoring of the Soil Pile Impacted with FCs and Mercury

At OVAM's request, the visual inspection of the soil pile impacted with Fcs and mercury was done earlier than 2018. The inspection by ERM in 2016 showed that the condition of the liner was intact. The next visual inspection is scheduled for 2019.

Abandoning of the Zwijndrecht Z-wells

In 2015, OVAM approved the partial end evaluation investigation report for the area of the Zwijndrecht Z-wells. In 2016, the wells were abandoned in accordance with relevant regulations.

Environmental Follow-up of an Environmental Incident

On August 12, 2015, a minor leak of PTBA (perfluortributylamine) was noticed between Building 016 and Building 003.

The leak was closed and the asphalt was decontaminated with water. Part of the solution and the cleaning water leaked outside into the bare soil. The incident was reported to OVAM in accordance with local regulations.

The impacted area was immediately excavated in order to limit the impact of PTBA in soil. Two soil samples were taken to assess the impact of the leak: one sample of the excavated soil and one sample of at the bottom of the excavation pit. The analyses showed no exceedance of remedial target values.

Because it was a minor impact and the measured concentration of PTBA is below the relevant screening level, ERM concludes that no further actions are needed

The results and conclusions were reported to OVAM and approved by OVAM on April 11, 2016.

General Conclusions and Recommendations

The soil and groundwater contamination at the 3M site, as described in the approved first phase RAP, is being remediated in accordance with the RAP specifications. ERM recommends that the remedial actions and regular monitoring continue at the 3M site as summarized in the following table.

The P&T system, which has been operational since 2011, currently consists of nine extraction wells. Its main purpose is to reduce FC concentrations and mitigate horizontal and vertical contaminant migration. Between July 2011 and June 2016, approximately 62.316 m^3 of contaminated groundwater was extracted and approximately 429,4 kg of FC’s were removed.

In the periode from 1 July 2015 till 1 July 2016 the system had an average up-time of app. 53% (lower than previous periods), but the up-time varies from well to well. The long-term trend in FC concentrations in most of the extraction and monitoring wells is stable, although fluctuations can occur. Concentrations in some wells have increased and further follow up (monitoring) is needed.

The (potential) increasing trend in some of the Blokkersdijk monitoring wells and the 3M pond needs to be verified via further monitoring and statistical evaluation.

The yearly average mass flux of PFOS towards the Schelde River lies well below the relevant PNEC value and is comparable to previous results.

An incident occurred at the 3M site between July 2015 and July 2016. The sampling results identified no impact to soil and/or groundwater. Therefore, no further action is required. The impacted area will need to be assessed during the next periodic soil investigation.

ERM recommends continuing the remediation and groundwater monitoring program as summarized in the following table to assess the long-term FC concentration trends. In addition, ERM recommends evaluating the need for an extension of the remedial system to optimize the contaminant capture. During this evaluation, options to improve the operation of P&T system should be investigated. ERM also recommends continuing the assessment of the potential impact of the old chemical sewer.

The next interim report will be submitted to OVAM before the end of 2017, describing the remedial actions and monitoring results taken from July 2016 through July 2017.

■

Proposed Remedial Monitoring Program for 2017

Location	Number of Wells	Frequency	Well ID	Rationale	Remarks
P&T System	9	Quarterly	PP01, PP02,PP04, PP05, PP06, PP07, PP08, PP09, PP10	-	At minimum monthly inspections of the installation and extraction wells
Stability	-	Once (cancelation if confirmed by stability expert)	-		No unacceptable settlements and a technical review by independent stability indicate that there is no further need for monitoring. To be confirm by final inspection.
Soil Pile	-	Five-yearly	Visual inspection of soil pile	Inspection done in 2016: cover is in a good condition.	From 2013 onwards, one monitoring every five years since cover was replaced in 2012 and inspected in 2013; next inspection: 2018
FC Source Areas					
Building 16	2	Annual	K3, P56	-	PFBS will be added to the analyses to follow up the concentration trend
	4	Semi-annual	P27, P42, P304, P305		K1 is replaced by P27, as the concentrations in K1 have been low and stable since the start of the monitoring in 2008
	1	Quarterly	P21B		
WWTP	6	Annual	P262, P340, P341, P343, P379, P380	-	-
	10	Semi-annual	P118C, P264, P265C, P371, P374, P381, P382, L19, M4, P119C	-	P119C was moved from the Blokkersdijk monitoring to the monitoring of the WWTP source zone because of its location (downgradient from 3M path and upgradient from source zone)
	1	Quarterly	P263		
Second Aquifer	6	Annual	BD24-4, D10, D11, D14, P118A P118B	-	-
	3	Semi-annual	D9, BD24-3, P121	-	If increasing trend is not confirmed by two consecutive sampling rounds, decrease frequency back to annually
	7	Annual	D16, D17, D18, D5, ND7, P119A en P119B	Increased FC concentrations compared to 2006 monitoring data.	Additional monitoring to verify concentrations measured in July 2016.
	1	Quarterly	P321	-	-
Southern Site Boundary	7	Semi-annual	B3-bis, B7, PA111A, PA112, P378, PA109A, P372	-	Well B3 lost and replaced by well B3-bis in September 2015.

Location	**Number of Wells**	**Frequency**	**Well ID**	**Rationale**	**Remarks**
Tank Farm Area					
TPH	2	🟦 Annual	P18, P28	TPH monitoring at request of OVAM	Source investigation ongoing
Blokkersdijk Nature Reserve					
Wells	7	🟦 Quarterly	L21, L22, P115, L31, L4, P114bis, P116		P119C was moved from the Blokkersdijk monitoring to the monitoring of the WWTP source zone because of its location (downgradient from 3M path and upgradient from source zone)
Surface water	2	🟦 Quarterly	Blokkersdijk standard, 3M pond		
Surface water	1	🟧 Quarterly	Blokkersdijk east	Sampling location is again accessible	Two sampling locations are more representative.
Paling and Tophat Ditch	4	🟦 Quarterly	12, 13, 5, Pumping House	-	-
Storm Water	1	🟦 Quarterly	Collector pit	Monthly samples analyzed by 3M internal lab	Quarterly verification samples by OVAM certified laboratory (eg. SGS)
Effluent WWTP	1	🟦 Quarterly	Discharge line	Monthly samples analyzed by 3M internal lab	Quarterly verification samples by OVAM certified laboratory (eg. SGS)

Legend

🟧 Increased monitoring frequency
🟩 Decreased monitoring frequency
🟦 No change

1. *INLEIDING*

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 Zevende tussentijds verslag (TTV7) van de saneringswerken, zoals beschreven in het door de OVAM conform verklaarde bodemsaneringsproject (1e fase) (2) en de zes voorgaande tussentijdse verslagen (3), (4), (5), (6), (7) en (8). Het betreft de saneringswerken op het deel productiezone, voormalige slibbekkens/waterzuiveringsinstallatie, en het natuurgebied Blokkersdijk. De beschouwde saneringsperiode loopt van juli 2015 tot en met juli 2016.

De organofluorimpact ter hoogte van de zuidelijke perceelgrens en de Palingbeek wordt momenteel door middel van een grondwatermonitoring opgevolgd. Wanneer er meer duidelijkheid is over de impact van de werken in het kader van de Oosterweelverbinding op deze zone, zal – indien noodzakelijk – een saneringsaanpak voor de FC impact uitgewerkt worden in een tweede gefaseerd bodemsaneringsproject. De voorbereidende studiewerken voor het deelproject Linkeroever zijn opgestart en worden door 3M en ERM opgevolgd.

(2) "Eerste gefaseerd bodemsaneringsproject 3M Belgium NV, haven 1005 – Canadastraat 11, 2070 Zwijndrecht", opgesteld door Arcadis in oktober 2008

(3) "Eerste tussentijds verslag bodemsaneringswerken Eerste gefaseerd bodemsaneringsproject", opgesteld door ERM in september 2010

(4) "Tweede tussentijds verslag bodemsaneringswerken Eerste gefaseerd bodemsaneringsproject", opgesteld door ERM in oktober 2011

(5) "Derde tussentijds verslag bodemsaneringswerken Eerste gefaseerd bodemsaneringsproject", opgesteld door ERM in december 2012

(6) "Vierde tussentijds verslag bodemsaneringswerken Eerste gefaseerd bodemsaneringsproject", opgesteld door ERM in december 2013

(7) "Vijfde tussentijds verslag bodemsaneringswerken Eerste gefaseerd bodemsaneringsproject", opgesteld door ERM in februari 2015

(8) "Zesde tussentijds verslag bodemsaneringswerken Eerste gefaseerd bodemsaneringsproject", opgesteld door ERM in april 2016

2. *VOORSTUDIE*

2.1 *OMGEVINGSKENMERKEN*

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

Voor meer details in verband met de omgevingskenmerken wordt verwezen naar het eerste tussentijds verslag (3).

2.2 *BEKNOPTE HISTORIEK EN BESCHRIJVING VAN HET TERREIN*

Een overzicht van de onderzoekslocatie is weergegeven op Kaart 2.

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, niet-gefluoreerde verbindingen en 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 vergunningen en de historiek wordt verwezen naar het conform verklaarde bodemsaneringsproject (2).

2.3 *GEOLOGISCHE EN HYDROGEOLOGISCHE GEGEVENS*

Het geologisch kader is al uitvoerig beschreven in het eerste tussentijds verslag (3). In het tweede tussentijds verslag (4) zijn de dijken ter hoogte van de bluswatertestzone en de diepte van de Polderklei beter in kaart gebracht.

Op basis van de meest recente metingen van de grondwaterstand (april 2016) blijkt dat het grondwater in de eerste aquifer voornamelijk in zuidelijke richting stroomt, meer bepaald richting Palingbeek. In de tweede aquifer stroomt het grondwater voornamelijk in noordelijke richting (richting Schelde). Ter hoogte van de 3M site is in de tweede aquifer een meer noordoostelijke stromingsrichting waargenomen (richting Schelde en Blokkersdijk).

2.4 RESULTATEN VOORMALIGE BODEMONDERZOEKEN EN/OF SANERINGEN

2.4.1 *Analyseresultaten*

In het eerste tussentijds verslag zijn de verschillende verontreinigingen op de 3M site besproken op basis van de resultaten van de voorgaande bodemonderzoeken. Het gaat hierbij om de volgende verontreinigingen die relevant zijn tijdens het opvolgen van het saneringsproject:

- Grondhoop met verhoogde kwik- en FC concentraties;
- Grondwaterverontreiniging met vluchtige aromaten ter hoogte van ondergrondse tanks;
- Bronzone FC verbindingen in grondwater zone Gebouw 16;
- Bronzone FC verbindingen in grondwater zone Waterzuiveringsinstallatie (WWTP);
- FC verontreiniging in grondwater 2^{e} aquifer;
- FC verontreiniging in de regenwaterriolering door insijpelend verontreinigd grondwater;
- FC verontreiniging in grond- en oppervlaktewater in het Blokkersdijk natuurgebied door atmosferische depositie;
- FC verontreiniging in het grondwater aan de zuidelijke grens, Palingbeek en Tophatgracht; en
- FC verontreiniging in het grondwater ten zuiden van de Expresweg (Z-peilbuizen) door uitloging van atmosferische depositie.

2.4.2 *Vuilvrachtberekening*

Bij de evaluatie van de Best Beschikbare Techniek voor de waterzuivering, uitgevoerd door EPAS (referentie *P.R.04.042.06*), is een veilige PFOS vuilvracht voor de Schelde geraamd van 370 g/dag (= PNEC waarde). Hieruit blijkt dat de vuilvracht aan FC verbindingen welke vóór de sanering naar de Schelde stroomde, aanvaardbaar was.

Tijdens de sanering is ieder jaar de totale vuilvracht die dagelijks naar de Schelde stroomt, berekend op basis van de gegevens verzameld tijdens de monitoringscampagne die ieder jaar loopt van 1 juli tot 30 juni.

In de onderstaande tabel zijn de resultaten van de voorbije jaren samengevat.

Tabel 2.1 ***Gemiddelde PFOS vuilvracht richting de Schelde (2007 tem. 2015)***

Vuilvracht naar de Schelde	**Debiet (m³/dag)**	**PFOS concentratie (µg/L)**	**Vuilvracht (g/dag)**
September 2007 (BSP fase 1)			
Effluent van het bedrijfsafvalwater	700	2,3	1,6
Regenwaterriolering	325	500	163
Palingbeek/Tophatgracht***	2.865	43,7	125
Totale vuilvracht			**290**

Vuilvracht naar de Schelde	**Debiet (m³/dag)**	**PFOS concentratie (µg/L)**	**Vuilvracht (g/dag)**
juli 2009 – juni 2010 (TTV1)			
Effluent van het bedrijfsafvalwater	700	2,3	1,6
Effluent van de grondwaterextractie	11,5*	2,3**	0,03
Regenwaterriolering	212	417	88
Palingbeek/Tophatgracht***	2.865	33	95
Totale vuilvracht			**185**
juli 2010 – juni 2011 (TTV2)			
Effluent van het bedrijfsafvalwater (incl. opgepompt grondwater)	677	12	8
Regenwaterriolering	256	418	107
Palingbeek/Tophatgracht***	2.865	63,5	182
Totale vuilvracht			**297**
juli 2011 – juni 2012 (TTV3)			
Effluent van het bedrijfsafvalwater (incl. opgepompt grondwater)	724	19	14
Regenwaterriolering	246	760	187
Palingbeek/Tophatgracht***	2.865	53,7	154
Totale vuilvracht			**355**
juli 2012 – juni 2013 (TTV4)			
Effluent van het bedrijfsafvalwater (incl. opgepompt grondwater)	729	50,3	37
Regenwaterriolering	188	256	48
Palingbeek/Tophatgracht***	2.865	60	173
Totale vuilvracht			**258**
juli 2013 – juni 2014 (TTV5)			
Effluent van het bedrijfsafvalwater (incl. opgepompt grondwater)	692	14,0	10
Regenwaterriolering	216	217	47
Palingbeek/Tophatgracht***	2.865	49,75	143
Totale vuilvracht			**200**
juli 2014 – juni 2015 (TTV6)			
Effluent van het bedrijfsafvalwater (incl. opgepompt grondwater)	643	24	15,6
Regenwaterriolering	293	183	53,6
Palingbeek/Tophatgracht***	2.865	51	145,5
Totale vuilvracht			**215**

* Debiet in kader van pilootproef

** Algemene waarde voor effluent volgens 3M; lozingsnorm is 30 µg/L

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

De berekende vuilvrachten schommelen als gevolg van de schommelende concentraties in het oppervlaktewater, regenwaterriolering en/of bedrijfsafvalwater.

De verhoogde vuilvrachtwaarde berekend voor de periode juli 2011 – juni 2012 is zeer waarschijnlijk het gevolg van een incident in 2011 waarbij slib van de bedrijfswaterzuivering in de collectorput van de regenwaterriolering terecht is gekomen. Midden 2011 is dit slib geruimd en sindsdien zijn de PFOS concentraties in de regenwatercollectorput terug gedaald (zie paragraaf 4.10).

3. *ALGEMENE BESCHRIJVING SANERINGSWERKEN*

3.1 *ALGEMEEN*

In het eerste gefaseerd bodemsaneringsproject (2) zijn de selectie en uitwerking beschreven van de saneringsaanpak voor de verontreinigingen met organofluorverbindingen (FC's) en vluchtige aromaten (BTEX) op de site van 3M, waarvoor een saneringsnoodzaak bestaat. De saneringswerken uitgevoerd tussen juli 2009 en juni 2015 zijn beschreven in de zes voorgaande tussentijdse verslagen (3), (4), (5), (6), (7) en (8).

Het tweede gefaseerd bodemsaneringsproject zal de verontreiniging met FC verbindingen in het grondwater ter hoogte van het zuidelijk 3M terreingedeelte en de relatie met de Palingbeek behandelen. Daar deze werken een belangrijke impact kunnen hebben op de waterhuishouding op deze locatie, dient een saneringsaanpak afgestemd te worden op deze werken. De voorbereidende studiewerken voor het deelproject Linkeroever zijn opgestart en worden door 3M en ERM opgevolgd. Wanneer er meer duidelijkheid is over de impact van de werken in het kader van de Oosterweelverbinding op deze zone, kan – indien noodzakelijk - een saneringsaanpak voor de FC impact uitgewerkt worden in een tweede gefaseerd bodemsaneringsproject. In afwachting van een eventueel bodemsaneringsproject wordt de organofluorimpact ter hoogte van de zuidelijke perceelgrens en de Palingbeek momenteel opgevolgd door middel van een halfjaarlijkse grondwatermonitoring.

3.2 *SANERINGSWERKEN ZOALS VOORZIEN IN HET EERSTE GEFASEERD BODEMSANERINGSPROJECT*

3.2.1 *Algemene saneringsdoelstelling*

Als uitgangspunt en referentiekader is hieronder de saneringsaanpak zoals opgenomen in het eerste gefaseerd bodemsaneringsproject samengevat. Merk op dat als gevolg van de bekomen resultaten en wijzigende uitgangspunten een aantal zaken niet langer van toepassing zijn (zie ook paragraaf 3.3).

Algemeen gesteld dient het 3M terrein aanzien te worden als één milieutechnische eenheid waarbij de verontreiniging (zowel in grond als in grondwater) in de bronzones wordt beheerd. In totaliteit zal er bijgevolg naar gestreefd worden om de verontreiniging op de site te behouden en, in zoverre ook technisch/financieel mogelijk, te reduceren en hierbij de verspreiding van de verontreiniging buiten de terreingrenzen te verminderen.

Er is voorzien de verontreiniging op de site te beheren door volgende maatregelen:

- 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 vermindering van de FC verbindingen-vuilvracht naar de Schelde.

Daarnaast zal verontreiniging aanwezig buiten de terreingrenzen door middel van een grondwatermonitoring opgevolgd worden.

De invulling van deze doelstelling zoals opgenomen in het bodemsaneringsproject is hieronder per onderdeel weergegeven.

3.2.2 *Grondhoop verontreinigd met kwik en FC verbindingen*

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

Na de eerste vijf jaar wordt overgeschakeld naar een vijfjaarlijkse controle.

3.2.3 *Gestimuleerde biologische afbraak van de vluchtige aromatenverontreiniging ter hoogte van de ondergrondse tanken*

3.2.3.1 *Doelstelling*

Het verspreidingsrisico is niet langer aanwezig wanneer de concentraties lager zijn dan twee keer de bodemsaneringsnorm. Concreet betekent dit:

- Tolueen: 1.400 µg/L;
- Ethylbenzeen: 600 µg/L; en
- Xyleen: 1.000 µg/L.

3.2.3.2 *Injectie*

Er is voorzien om de aromatenverbindingen te saneren door het zuurstofgehalte te verhogen waardoor de aerobe biodegradatie gestimuleerd wordt. Er is een onderscheid gemaakt tussen de verontreiniging binnen en buiten de polyurethaan-inkuiping. Binnen de inkuiping zal het zuurstofgehalte verhoogd worden door injectie van lucht (biosparging), buiten de inkuiping zal een calciumperoxidesuspensie geïnjecteerd worden om het zuurstofgehalte te verhogen.

Volgende fases zijn bepaald:

- Fase1: Nulmonitoring en controle van het verspreidingspatroon;
- Fase 2: Installatie van injectieputten;
- Fase 3: Uitvoering injectie:
  - Pulserende injectie met lucht gedurende vijf jaar; en

- Vijf injectierondes over vijf jaar met calciumperoxide.

3.2.3.3 *Monitoring*

Een maand na de injectie zullen wekelijks het zuurstofgehalte, de pH, EC en temperatuur bepaald worden ter hoogte van een zestal peilbuizen. Om de twee weken zullen eveneens de concentraties aan BTEX bepaald worden. Na de eerste maand zullen de hoger vermelde veldparameters eenmaal per maand gemeten worden en de concentraties aan BTEX elke drie maanden.

3.2.4 *Grondwaterextractie zone Gebouw 16*

3.2.4.1 *Doelstelling*

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

3.2.4.2 *Gefaseerde opbouw grondwaterextractiesysteem*

Volgende fases zijn gedefinieerd in het bodemsaneringsproject:

- Fase 1: Plaatsing bijkomende monitoringspeilbuizen met een filter tot op de Polderklei;
- Fase 2: Opstart onttrekking op de horizontale drain in de productiezone. Het verwachtte onttrekkingdebiet is 2 m^3/dag. Het onttrokken grondwater zal over de grondwaterzuivering van 3M geleid worden;
- Fase 3: Installatie van drie verticale onttrekkingsputten in de productiezone. Het verwachtte onttrekkingdebiet is 4 m^3/dag. Het onttrokken grondwater zal over de grondwaterzuivering van 3M geleid worden;
- Fase 4: Evaluatie verspreidingspatroon en efficiëntie van het onttrekkingsysteem gedurende vijf jaar; en
- Fase 5: Eventuele uitbreiding van het onttrekkingsysteem in de productiezone.

De voorwaarden waarbij het extractiesysteem verder dient uitgebreid te worden zijn:

- De vuilvrachtverwijdering per tijdseenheid blijft behouden of neemt toe; en
- Het onttrekkingsdebiet bedraagt 30 L/u of meer.

3.2.4.3 *Monitoring*

De efficiëntie van de onttrekking zal worden nagegaan en opgevolgd middels een halfjaarlijkse bemonstering van een 13-tal peilbuizen gedurende twee jaar. Daarna wordt overgeschakeld op een jaarlijkse frequentie. De stalen worden geanalyseerd op FC verbindingen.

Het opgepompte grondwater zal de eerste twee jaar maandelijks bemonsterd en geanalyseerd worden. De stalen worden geanalyseerd op FC verbindingen. De bemonsteringsfrequentie alsook het analysepakket kan aangepast worden in functie van de bekomen resultaten.

Daarnaast zal in de eerste drie jaren maandelijks een staal genomen worden van het opgepompte grondwater uit de zone WWTP en de zone Gebouw 16, nadat deze stromen zijn samen gekomen en voordat ze over de grondwaterzuiveringsinstallatie worden geleid (zie ook paragraaf 3.2.5). De frequentie zal worden afgebouwd tot een tweemaandelijkse bemonstering na drie jaar en driemaandelijks na 6 jaar werking. Deze informatie dient meer inzicht te geven in de vuilvracht welke door de actief koolfilters dient verwijderd te worden. De stalen worden geanalyseerd op SOF (totaal oplosbaar FC), individuele FC verbindingen (PFOS, PFOA), anorganisch fluoride, BTEX, arseen, chroom, COD en N. De bemonsteringsfrequentie alsook het analysepakket kan aangepast worden in functie van de bekomen resultaten.

Het effluent van de zuiveringsinstallatie wordt door 3M op wekelijkse basis bemonsterd en geanalyseerd conform haar milieuvergunning.

3.2.5 *Grondwaterextractie zone WWTP*

3.2.5.1 *Doelstelling*

De sanering is erop gericht om de verspreiding van met FC verbindingen verontreinigd grondwater tegen te gaan vanuit deze zone in zowel horizontale als verticale richting (naar 2^e aquifer).

Meer bepaald voor wat betreft horizontale verspreiding houdt dit in dat een verspreiding van de concentraties hoger dan 10.000 µg/L voor de som van PFOS, PFOA en PFHS dient tegengegaan te worden.

Voor de tweede 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; en
- De concentraties aan 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 (zie ook paragraaf 3.2.6).

De oplosbaarheid voor de belangrijkste verbindingen is in onderstaande tabel weergegeven.

Tabel 3.1 ***Oplosbaarheid FC verbindingen***

Parameter	Oplosbaarheid (mg/L)	10% oplosbaarheid (µg/L)
PFOS	680	68.000
PFOA	9,5	950
PFHS	Niet bepaald	-
PFOSA	Niet bepaald	-

3.2.5.2 *Gefaseerde opbouw grondwaterextractiesysteem*

Volgende fases zijn gedefinieerd in het bodemsaneringsproject:

- Fase 1: Plaatsing bijkomende monitoringspeilbuizen voor afbakening van de 10.000 µg/L contour voor de som van PFOS, PFOA en PFHS;
- Fase 2: Installatie van 10 grondwateronttrekkingsputten ter hoogte van de zone WWTP. Het verwachtte onttrekkingsdebiet is 12 m^3/dag. Het onttrokken grondwater zal over de grondwaterzuivering van 3M geleid worden;
- Fase 3: Evaluatie van de efficiëntie van het onttrekkingssysteem gedurende vijf jaar; en
- Fase 4: eventuele uitbreiding van het onttrekkingssysteem.

De voorwaarden waarbij het extractiesysteem verder dient uitgebreid te worden zijn:

- Een uitbreiding van de 10.000 µg/L contourlijn voor de som van PFOA, PFHS en PFOS;
- Het uitblijven van een reductie van de massatransfer van FC verbindingen van de eerste naar de tweede aquifer (zie ook paragraaf 3.2.6).

3.2.5.3 *Monitoring*

De efficiëntie van de onttrekking zal worden nagegaan en opgevolgd middels een halfjaarlijkse bemonstering van een 11-tal peilbuizen gedurende twee jaar. Daarna wordt overgeschakeld op een jaarlijkse frequentie. De stalen worden geanalyseerd op FC verbindingen. De monitoring van de 2de aquifer is beschreven in paragraaf 3.2.6.

Het opgepompte grondwater zal de eerste twee jaar maandelijks bemonsterd en geanalyseerd worden. De stalen worden geanalyseerd op FC verbindingen. De bemonsteringsfrequentie alsook het analysepakket kan aangepast worden in functie van de bekomen resultaten.

Daarnaast zal in de eerste drie jaren maandelijks een staal genomen worden van het opgepompte grondwater uit de zone WWTP en de zone gebouw 016, nadat deze stromen zijn samengekomen en voordat ze over de grondwaterzuiveringsinstallatie worden geleid (zie ook paragraaf 3.2.4). De frequentie zal worden afgebouwd tot een tweemaandelijkse bemonstering na drie jaar en driemaandelijks na 6 jaar werking. Deze informatie dient meer inzicht te geven in de vuilvracht welke door de actief koolfilters dient verwijderd te worden. De stalen worden geanalyseerd op SOF, individuele FC verbindingen (PFOS, PFOA), anorganisch fluoride, BTEX, arseen, chroom, COD en N. De bemonsteringsfrequentie alsook het analysepakket kan aangepast worden in functie van de bekomen resultaten.

Het effluent van de zuiveringsinstallatie wordt door 3M op wekelijkse basis bemonsterd en geanalyseerd conform hun milieuvergunning.

3.2.6 *Monitoring 2^e^ aquifer*

De evolutie van de grondwaterconcentraties aan FC verbindingen in de 2^{e} aquifer zal middels het halfjaarlijks bemonsteren van een achttal peilbuizen opgevolgd worden. De stalen zullen geanalyseerd worden op FC verbindingen.

3.2.7 *Reductie van de FC impact in de regenwaterriolering*

3.2.7.1 *Doelstelling*

Vanuit risicogebaseerd oogpunt is het niet noodzakelijk 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 (zie paragraaf 2.4.2). De reductie van de vuilvracht in het regenwater kadert binnen de beheersing van de verontreiniging op het terrein van 3M.

3.2.7.2 *Zuiveren water uit regenwaterriolering*

Ter hoogte van het einde van de regenwaterriolering, nog vóór dit gemengd wordt met het effluent van het gezuiverde bedrijfsafvalwater, is in het bodemsaneringsproject voorzien om één of twee actief koolfilters te plaatsen. Het gemiddelde debiet is geraamd op 325 m^{3}/dag.

Bij de zuivering kan, omwille van kostenredenen, geen rekening gehouden worden met de grote debietsverschillen die kunnen optreden als gevolg van hevige buien en de hiermee veranderende samenstelling van de verhouding regenwater/grondwater. Het design is ingesteld op gemiddelde debieten met gemiddelde concentraties. In geval van een sterke stijging van debieten zal het water via een bypass ongezuiverd geloosd worden in de Schelde, wat niet leidt tot ecotoxicologische risico's voor de Schelde. Er is gesteld dat 90% van het totale volume dat op jaarbasis geloosd wordt via de regenwaterriolering zal behandeld worden.

Er zal worden gestreefd naar een effluentconcentratie van 30 µg/L voor PFOS.

3.2.7.3 *Monitoring*

Om de vuilvracht die het 3M terrein verlaat als gevolg van het infiltreren van potentieel verontreinigd grondwater in het regenwaterrioleringsstelsel, op te volgen, worden het debiet en de FC concentraties van het regenwater op regelmatige tijdstippen gemonitord. Hiervoor worden stalen genomen op vier verschillende locaties in de riolering en van de regenwatercollectorput net vóór het punt waar het regenwater wordt samengevoegd met het gezuiverde afvalwater dat in de Schelde wordt geloosd.

3.2.8 *Natuurgebied Blokkersdijk*

3.2.8.1 *Doelstelling*

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

3.2.8.2 *Monitoring*

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 zullen op regelmatige tijdstippen opgevolgd worden.
De monitoring van het grondwater dient te gebeuren door middel van het regelmatig bemonsteren van 6 peilbuizen ten westen van de vijvers. Deze peilbuizen zullen de eerste vijf jaar halfjaarlijks worden bemonsterd en vervolgens op jaarlijkse basis. De monitoring van de 3M vijver gebeurt op halfjaarlijkse basis. Het oppervlaktewater van de Blokkersdijkvijver zal op driemaandelijkse basis bemonsterd worden in de eerste drie jaar en daarna halfjaarlijks.

Door middel van een statistische analyse van de PFOS-concentraties in het water van de Blokkersdijkvijver dient aangetoond te worden dat de concentraties niet stijgen. Indien een 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 (zie verder).

Wanneer er voor het grondwater voldoende meetpunten in de tijd zijn verzameld, kan eveneens een trendanalyse worden uitgevoerd op de grondwaterconcentraties stroomopwaarts van Blokkersdijk. Indien een statistische stijging wordt waargenomen van de concentraties in het grondwater, zal de monitoringsfrequentie van de 3M vijver en de Blokkersdijkvijver opgedreven worden. Een statistisch significante stijging in het grondwater is niet noodzakelijk een aanleiding om tot een actieve saneringsvariant over te gaan. Hiervoor dient een statisch significante stijging van de PFOS concentratie aanwezig te zijn in de Blokkersdijkvijver.

3.2.8.3 *Actieve reservevariant*

Als back-up variant is een hydrogeologische barrière voorzien ter hoogte van het 3M pad (verbindingsweg tussen het 3M terrein en de Schelde). Voor meer details wordt verwezen naar het eerste gefaseerd bodemsaneringsproject (2).

Er dient opgemerkt te worden dat deze reservevariant enkel zinvol is indien de stijging van de concentraties aan PFOS in de Blokkersdijkvijver het gevolg zijn van de instroom van verontreinigd grondwater.

3.2.9 *Zuidelijke terreingrens, Palingbeek en Tophatgracht*

De aanpak van de verontreiniging aan de zuidelijke perceelgrens, inclusief de Palingbeek en de Tophatgracht zal in het kader van een tweede gefaseerd bodemsaneringsproject uitgewerkt worden. Momenteel zijn er nog teveel onduidelijkheden wat betreft timing en uitvoering van de werken in het kader van de Oosterweelverbinding en de impact hiervan op de verontreiniging en waterhuishouding in deze zone.

In afwachting van het tweede gefaseerd bodemsaneringsproject is voorzien om een 7-tal peilbuizen jaarlijks te bemonsteren. Daarnaast dient elke 6 maanden een oppervlaktewaterstaal genomen te worden ter hoogte van de Palingbeek, Tophatgracht en het pompstation ter hoogte van de Schelde. De stalen dienen te worden geanalyseerd op FC verbindingen.

3.2.10 *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, zullen 4 peilbuizen halfjaarlijks bemonsterd worden. De stalen zullen geanalyseerd worden op FC verbindingen.

3.2.11 *Vuilvracht richting de Schelde*

De geraamde vuilvracht van PFOS naar de Schelde indien de hoger vermelde saneringsaanpak wordt gevolgd, is samengevat in onderstaande tabel.

Tabel 3.2 ***Voorziene PFOS vuilvracht naar de Schelde zoals opgenomen in het eerste gefaseerd bodemsaneringsproject na sanering***

Vuilvracht naar de Schelde	**Debiet (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
Regenwaterriolering	325	30 (streefwaarde)	9,8
Palingbeek	2.865	43,7	125
Totale vuilvracht			**157**

Hierbij is uitgegaan van een PFOS-concentratie gelijk aan de lozingsnorm voor het effluent van de waterzuiveringsinstallatie (in werkelijkheid rond 2 µg/L) en een concentratie van 30 µg/L voor de regenwaterriolering. Deze laatste wordt beschouwd als een streefwaarde (zie ook paragraaf 3.2.7).

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 die dagelijks naar de Schelde stroomt, berekend op basis van de gegevens verzameld tijdens de monitoringscampagne die ieder jaar loopt van juli tot en met juni.

3.3 *WIJZIGINGEN IN HET BODEMSANERINGSPROJECT VOLGENS VOORGAANDE TUSSENTIJDSE VERSLAGEN*

Ten opzichte van de saneringsaanpak zoals voorzien in het eerste gefaseerd bodemsaneringsproject (2) zijn in de voorgaande tussentijdse verslagen (3), (4), (5), (6), (7) en (8) een aantal wijzigingen voorgesteld. Deze wijzigingen zijn toegelicht in onderstaande paragrafen. In paragraaf 3.3.11 wordt een overzicht gegeven van het monitoringsprogramma voor 2016.

3.3.1 *Grondhoop verontreinigd met kwik en FC verbindingen*

In 2012 is een nieuwe afdekking geplaatst waardoor het risico op uitloging door infiltrerend regenwater geminimaliseerd is. In het vierde tussentijds verslag is, na overleg met OVAM (dd. 22 oktober 2013) de controlefrequentie verlaagd naar vijfjaarlijks, met een eerstvolgende controle in 2018.

3.3.2 *Gestimuleerde biologische afbraak van de vluchtige aromatenverontreiniging ter hoogte van de ondergrondse tanken*

In het eerste tussentijds verslag (TTV1(3)) was al beslist dat een actieve saneringsaanpak voor de BTEX verontreiniging in het grondwater niet noodzakelijk was aangezien de concentraties in het grondwater sterk waren afgenomen en natuurlijke afbraak plaatsvindt. Bijgevolg werd een monitoring voorgesteld om de daling van de BTEX grondwaterconcentraties te bevestigen.

Uit de monitoring blijkt dat de concentraties blijven dalen, de concentraties liggen onder de relevante richtwaardes en dus is ook de saneringsdoelstelling behaald. Daarom is in het vierde tussentijds verslag voorgesteld om de monitoring van de BTEX concentraties in het grondwater stop te zetten (zie TTV4 (6)).

Tijdens het overleg met OVAM in oktober 2013 heeft OVAM de opmaak van een eindevaluatieonderzoek opgelegd voor de (voormalige) verontreiniging met BTEX in het grondwater. Het partieel eindevaluatieonderzoek met referentie R001-0243999-v2 is op 01/07/2015 door de OVAM conform verklaard (ref. BB-O-DS-20150289277).

Ter hoogte van twee peilbuizen (P18 en P28) zijn verhoogde waarden voor pH, geleidbaarheid en minerale olie in grondwater gemeten waarvoor momenteel geen verklaring gegeven kan worden. Interferentie door een andere verontreiniging kan niet worden uitgesloten. Verder historisch en analytisch brononderzoek ter hoogte van P18 en P28 is in uitvoering. De jaarlijkse monitoring van minerale olie in peilbuizen P18 en P28 blijft behouden. Het grondwater van beide peilbuizen wordt jaarlijks bemonsterd en geanalyseerd op minerale olie.

3.3.3 *Grondwaterextractie zone Gebouw 16 en zone WWTP*

De fasering en het design van de grondwateronttrekking zijn gewijzigd ten opzichte van wat oorspronkelijk was voorzien in het eerste gefaseerde bodemsaneringsproject.

3.3.3.1 *Fasering*

Voor beide bronzones verontreinigd met FC verbindingen is volgende gewijzigde fasering gehanteerd:

- Bijkomende hydrogeologische kartering in de brongebieden (zone Gebouw 16 en zone WWTP) door middel van ongeroerde staalname, korrelgrootteanalyses en waterpassing;
- Uitvoeren pilootproef (deel 1) gedurende twee dagen in elke kernzone middels een hiervoor voorziene grondwateronttrekkingsput (PP01 en PP02);
- Installatie bijkomende monitoringspeilbuizen ter verfijning van de contour van 10.000 µg/L voor de som van PFOS, PFOA en PFHS;
- Uitvoeren tweede deel pilootproef gedurende een drietal maanden, gebruik makend van onttrekkingsputten PP01 en PP02;
- Installatie en opstart van de eerste stap van de grondwateronttrekking;
- Evaluatie grondwateronttrekking na een aantal jaar onttrekken (nog uit te voeren); en
- Installatie en opstart van de tweede stap van de grondwateronttrekking indien nodig (nog uit te voeren).

3.3.3.2 *Design en opvolging grondwateronttrekkingssysteem*

Bij de uitwerking van het design van de grondwateronttrekkingsinstallatie diende rekening gehouden te worden met locatiespecifieke randvoorwaarden en beperkingen. Zo is geopteerd om voor een aantal grondwateronttrekkingsputten het effluent te leiden naar de dichtstbijzijnde afvalwaterriolering, eerder dan deze te koppelen op een collectorleiding per kernzone die op haar beurt naar de afvalwaterzuivering leidt. Dit is voornamelijk het geval op plaatsen waar, door aanwezigheid van gebouwen, (ondergrondse) leidingen, productieactiviteiten, enzovoort, het niet mogelijk is om de leidingen met elkaar te verbinden.

Als gevolg zijn er twee verschillende types van de grondwateronttrekkingsputten, meer bepaald deze waarbij een aansluiting is voorzien op een collectorleiding welke op haar beurt naar de grondwaterzuivering leidt en deze waarbij er enkel een leiding is tot aan de dichtstbijzijnde afvalwaterriolering.

Als gevolg van hoger vermelde opstelling, zal de opvolging van de onttrekkingsinstallatie (staalname, debiet, druk) enigszins verschillend zijn van wat is beschreven in het bodemsaneringsproject.

De geraamde onttrekkingsdebieten zoals berekend op basis van het grondwatermodel zijn in onderstaande tabel weergegeven. Hierbij is eveneens het debiet ingeschat in het kader van het eerste gefaseerd bodemsaneringsproject opgenomen.

Tabel 3.3 ***Overzicht geraamde onttrekkingsdebieten***

Zone	Debiet ingeschat in BSP		1e stap onttrekking		2e stap onttrekking	
	# filters	Debiet	# filters	Debiet	# filters	Debiet
Zone Gebouw 16						
Verticale filters	3	4 m³/dag	4	23 m³/dag	7	26 m³/dag
Drains	2	2 m³/dag	2	0,5 m³/dag	2	0,5 m³/dag
Zone WWTP	10	12 m³/dag	4	15 m³/dag	14	17 m³/dag

3.3.3.3 *Grondwatermonitoring*

De grondwatermonitoring ter hoogte van zone Gebouw 16 loopt intussen zes jaar, en in overeenstemming met het conform verklaarde bodemsaneringsproject, is in 2013 de monitoringsfrequentie verlaagd naar jaarlijks. Wel is toen aanbevolen om de oorzaak en stijging van de PFBS concentraties ter hoogte van de bronzone Gebouw 16 verder te onderzoeken en jaarlijks op te volgen. Eerder zijn al drie peilbuizen (P300, P5 en U11) geschrapt uit het monitoringsprogramma omdat de concentraties ter hoogte van deze peilbuizen stabiel zijn of een dalende trend vertonen. Peilbuis K4 was eveneens geschrapt uit het programma omdat deze beschadigd was en niet langer bruikbaar is.

Aangezien de grondwatermonitoring ter hoogte van zone WWTP intussen zes jaar loopt, is, in overeenstemming met het conform verklaarde bodemsaneringsproject, sinds 2013 de monitoringsfrequentie van de meeste peilbuizen verlaagd tot jaarlijks. Om een beeld te krijgen van de verontreinigingstoestand stroomopwaarts van de bronzone zijn twee nieuwe peilbuizen geplaatst (P381 en P382) en is peilbuis P264 toegevoegd aan het programma. De monitoringsfrequentie van peilbuizen P118C, P264, P265C, P381 en P382 is sinds 2013 halfjaarlijks. Sinds 2014 is de monitoringsfrequentie van peilbuizen P371 en P374 eveneens halfjaarlijks om een stijgende trend voor bepaalde FC's beter te kunnen opvolgen. Eerder zijn drie peilbuizen (P11, P265B en P361) geschrapt uit het monitoringsprogramma omdat de concentraties ter hoogte van deze peilbuizen een dalende trend vertonen en niet meer noodzakelijk zijn voor de monitoring van de WWTP bronzone.
In het vijfde tussentijds verslag (TTV5(7)) is aanbevolen om peilbuis P119C toe te voegen aan de monitoring van de WWTP bronzone in plaats van de monitoring van het Blokkersdijk natuurgebied, aangezien deze peilbuis stroomopwaarts ligt van de WWTP bronzone en stroomafwaarts van het natuurgebied.

In het zesde tussentijds verslag (TTV6(8)) is aanbevolen om de monitoringsfrequentie voor peilbuis P21B (zone Gebouw 16) en P263 (zone WWTP) te verhogen naar driemaandelijks om een stijgende trend voor bepaalde FC's beter te kunnen opvolgen.

3.3.4 *Monitoring 2e aquifer*

Aangezien voor de meeste peilbuizen in de bronzone in de tweede aquifer aan de saneringsdoelstelling is voldaan, is de monitoringsfrequentie in 2013 verlaagd naar jaarlijks. Voor peilbuis P321 blijft de verhoogde monitoringsfrequentie (driemaandelijks) voorlopig behouden om de afwijkende resultaten uit het verleden te verifiëren. De peilbuizen BD24-3, D10 en P118A worden sinds 2014 halfjaarlijks bemonsterd om de mogelijk stijgende trend te verifiëren.

3.3.5 *Reductie van de FC impact in de regenwaterriolering*

Omdat de concentraties gemeten in de regenwaterriolering zeer gevoelig zijn aan fluctuaties als gevolg van neerslag is, in overleg met OVAM, besloten om vanaf 2014 de driemaandelijkse monitoring van de regenwaterriolering (RW3, RW4, RW12 en RW13) te schrappen en te vervangen door de maandelijkse monitoring van de collectorput.

In 2012 is de collectorput gereinigd (het aanwezige slib is verwijderd) waarna de vuilvracht van de regenwaterriolering significant is gedaald en momenteel relatief stabiel is. Op basis van deze resultaten is besloten om momenteel nog geen bijkomende aktief koolfilters te plaatsen. Indien nodig zal deze beslissing geherevelueerd worden op basis van de metingen.

3.3.6 *Natuurgebied Blokkersdijk*

In het vierde tussentijds verslag (TTV4(6)) is aanbevolen om de FC concentraties in de peilbuizen L4, P114 en P116 (driemaandelijks), en P119C (halfjaarlijks) verder op te volgen. Bijkomend is voorgesteld om de monitoringsfrequentie van peilbuis L31 opnieuw naar halfjaarlijks te verlagen. In het vijfde tussentijds verslag (TTV5(7)) is aanbevolen om peilbuis P119C toe te voegen aan de monitoring van de WWTP bronzone, aangezien deze peilbuis stroomafwaarts ligt van het natuurgebied maar stroomopwaarts van de WWTP bronzone. De monitoringsfrequentie van de overige peilbuizen en van de Blokkersdijkvijver en de 3M blijft ongewijzigd (halfjaarlijks respectievelijk driemaandelijks). Omwille van de slechte toegankelijkheid van het staalnamepunt "Blokkersdijk oost" is in overleg met OVAM (dd. 22 oktober 2013) besloten om dit staalnamepunt uit de monitoring te schrappen.

Momenteel kan geen verklaring gegeven worden voor de vastgestelde concentratiestijging in enkele peilbuizen en de 3M vijver. Verder historisch en analytisch brononderzoek ter hoogte van het 3M pad is in uitvoering.

In het zesde tussentijds verslag (TTV6(8)) is aanbevolen om de monitoringsfrequentie voor peilbuizen L21, L22, P115 en L31 te verhogen naar driemaandelijks om de dataset voor de statistische evaluatie sneller aan te vullen.

In 2014 (zie TTV5 (7)) werd besloten om peilbuis P119C voortaan op te nemen in de evaluatie van de evolutie van FC concentraties ter hoogte van de WWTP zone bronzone, aangezien deze peilbuis stroomopwaarts ligt van de bronzone en stroomafwaarts van het natuurgebied Blokkersdijk.
Sinds april 2014 wordt het oppervlaktewater ter hoogte van de staalnamelocatie Blokkersdijk Oost niet meer bemonsterd omwille van de slechte toegankelijkheid.

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(8)) waarbij de resultaten van de statistische evaluatie van de volledige dataset zijn vergelijken met de resultaten van enkel de dataset van het 3M lab blijkt dat de wisseling van labo's invloed heeft gehad op de PFOS trend analyse. Daarom id voorgesteld om enkel nog de data uitgevoerd door het 3M Environmental laboratorium in de Verenigde Staten te gebruiken met controle analyses door SGS nv.

3.3.7 *Zuidelijke terreingrens*

De halfjaarlijkse monitoringsfrequentie voor de peilbuizen aan de zuidelijke terreingrens is ongewijzigd gebleven. Eerder werden wel de peilbuizen B2, P360A, P360B, P361, PA109B en PA111B uit het monitoringsprogramma geschrapt, omdat deze een stabiele of dalende trend vertoonden en werd peilbuis L6 vervangen door P372.

3.3.8 *Palingbeek en Tophatgracht*

De monitoringsfrequentie is in 2011 verhoogd naar driemaandelijks.

3.3.9 *Gebied ten zuiden van de Expresweg (Z-peilbuizen)*

In het eerste gefaseerd bodemsaneringsproject (2), dat in 2008 door Arcadis is opgesteld, is opgenomen om de grondwaterkwaliteit ten zuiden van de Expresweg verder op te volgen, ondanks het feit dat er in deze zone geen toename van verontreiniging verwacht wordt. Bijgevolg werd in het bodemsaneringsproject een monitoring voorgesteld om de evolutie van de FC grondwaterconcentraties op te volgen en om na te gaan of er een verdere toename van de organofluorconcentraties in het grondwater plaatsvindt in het gebied ten zuiden van de Expresweg.

Uit de monitoring blijkt dat de concentraties fluctueren maar globaal gezien stabiel blijven en vergelijkbaar zijn met de concentraties gemeten voor de start van de sanering in 2009. Er kan dan ook geconcludeerd worden dat de saneringsdoelstelling is behaald. Daarom is in het vierde tussentijds verslag voorgesteld om de monitoring van de organofluorconcentraties in het grondwater stop te zetten (zie TTV4 (6)).

Tijdens het overleg met OVAM in oktober 2013 heeft OVAM de opmaak van een gefaseerd eindevaluatieonderzoek opgelegd voor het gebied ten zuiden van de E34 Expressweg (Zwijndrecht Z-peilbuizen). Dit tweede gefaseerd eindevaluatieonderzoek met referentie R002-0243999-v2 is op 01/07/2015 door de OVAM conform verklaard (ref. BB-O-DS-20150289432).

3.3.10 *Vuilvracht richting de Schelde*

Geen wijzigingen.

3.3.11 *Monitoringsprogramma 2016*

Op basis van de resultaten uit de periode juli 2011 tot en met juli 2015 is het monitoringsprogramma zoals opgenomen in het conform verklaarde BSP fase 1 waar nodig of wenselijk aangepast. In de volgende tabel is een overzicht gegeven van het gewijzigde monitoringsprogramma voor de periode juli 2015 – juli 2016, met verantwoording. Voor meer details wordt verwezen naar het zesde TTV(8).

Tabel 3.4 *Monitoringsprogramma 2016*

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
P&T systeem	9	Driemaandelijks	PP01, PP02,PP04, PP05, PP06, PP07, PP08, PP09, PP10	-	Minstens maandelijkse inspectie van de installatie en de onttrekkingsputten
Stabiliteit	-	Eenmaal (stopzetting indien akkoord stabiliteitsdeskundige)	-	-	Geen onaanvaardbare zettingen en een technische evaluatie door onafhankelijke stabiliteitsdeskundige geeft aan dat er geen verder noodzaak is voor monitoring. Te bevestigen door eenmalige controle.
Grondhoop	-	Vijfjaarlijks	Visuele inspectie van grondhoop	Bovenafdek vervangen in 2012. Op vraag van OVAM is een tussentijdse controle uitgevoerd in 2016	Vanaf 2013 vijfjaarlijkse monitoring, aangezien bovenafdek in 2012 vervangen is; eerst volgende controle: 2018
FC Bronzones					
Gebouw 16	2	Jaarlijks	K3, P56	-	PFBS zal worden toegevoegd aan het analysepakket om de concentraties verder op te volgen
	4	Halfjaarlijks	P27, P42, P304, P305	-	P27 ter vervanging van K1 omdat de concentraties in peilbuis K1 sinds de start van de monitoring in 2008 stabiel en laag liggen
	1	Driemaandelijks	P21B		Stijgende concentratietrend ter hoogte van P21B waargenomen.
WWTP	6	Jaarlijks	P262, P340, P341, P343, P379, P380	-	-
	10	Halfjaarlijks	P118C, P264, P265C, P371, P374, P381, P382, L19, M4, P119C	-	P119C van Blokkersdijk monitoring naar bronzone monitoring gezien de locatie (stroomafwaarts van 3M pad, maar stroomopwaarts van bronzone)
	1	Driemaandelijks	P263		-
Tweede aquifer	6	Jaarlijks	BD24-4, D11, D14, P118B, D10, P118A	-	-
	7	Toegevoegd	D5, ND7, D16, D17, D18,	Bijkomend bemonstering in juli	

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
			P119A, P119B	2016 als bijkomende controle van de grondwaterkwaliteit van de tweede aquifer	
	3	Halfjaarlijks	D9, BD24-3, P121		Indien de stijgende trend niet wordt bevestigd bij twee opeenvolgende monitoringsrondes, zal de monitoringsfrequentie terug worden verlaagd naar jaarlijks
	1	Driemaandelijks	P321	-	-
Zuidelijke terreingrens	7	Halfjaarlijks	B3-bis, B7, P372, P378, PA109A, PA111A, PA112	-	Peilbuis B3 is beschadigd en vervangen door peilbuis B3-bis in september 2015
Ondergrondse tanken					
Minerale olie (MO)	2	Jaarlijks	P18, P28	Monitoring op vraag van OVAM	Brononderzoek lopende
Blokkersdijk Natuurgebied					
Peilbuizen	4	Driemaandelijks	L21, L22, P115, L31		-
	3	Driemaandelijks	L4, P114bis, P116	-	-
Oppervlaktewater	2	Driemaandelijks	Blokkersdijk standaard, 3M vijver	-	-
		Toegevoegd	Blokkersdijk oost	Locatie terug bereikbaar	
Palingbeek en Tophat gracht	4	Driemaandelijks	12, 13, 5, Bemalingsstation	-	-
Regenwater	1	Maandelijks	Collector put	Maandelijkse stalen geanalyseerd door 3M labo te Zwijndrecht	Driemaandelijkse verificatie van de stalen door SGS
Effluent WWTP	1	Driemaandelijks	Lozingspijp	Maandelijkse stalen geanalyseerd door 3M labo te Zwijndrecht	Driemaandelijkse verificatie van de stalen door SGS

Legende

Verhoogde monitoringsfrequentie

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen

🟩 Verlaagde monitoringsfrequentie

🟦 Geen wijzigingen

4. UITGEVOERDE MONITORING EN RESULTATEN

4.1 ALGEMEEN

De monsternamewerkzaamheden zijn door ERM en Servaco uitgevoerd conform de CMA-procedures (Compendium voor monsterneming en analyse, meest recente versie). Tijdens de grondwaterbemonstering zijn temperatuur, zuurtegraad (pH) en elektrische geleidbaarheid (conductiviteit) gemeten en zijn de grondwaterstanden in alle peilbuizen gepeild.

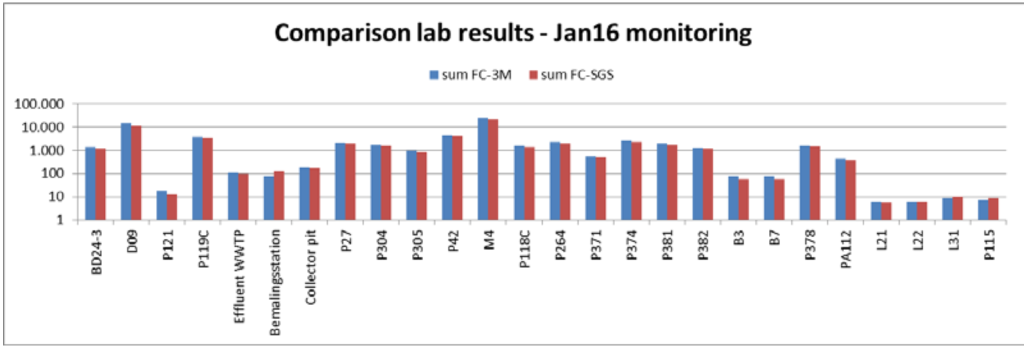
De wateranalyses van alle monitoringsrondes zijn opnieuw geanalyseerd door het 3M Environmental Laboratory en alle waterstalen zijn minstens éénmaal door het labo SGS nv geanalyseerd ter controle. De controle staalname en analyses zijn opnieuw gespreid over de vier monitoringsperiodes (in plaats van één controle tijdens de monitoring van juli).

In de figuren en grafieken opgenomen in dit verslag zijn, omwille van consistentie, enkel de analyseresultaten van het 3M Environmental Laboratory gebruikt.

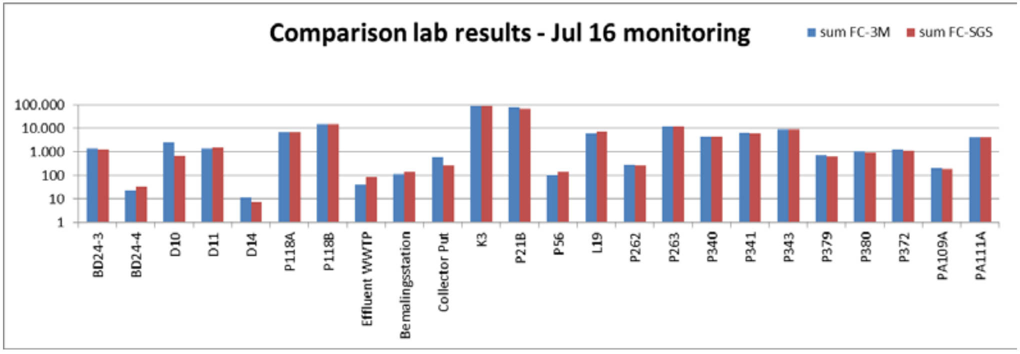
Uit een evaluatie van de datasets voor desbetreffende periode (SGS vs. 3M labo) volgt dat de resultaten van beide labo's in de meeste gevallen vergelijkbaar zijn (zie Grafiek 4.1). In oktober 2015 lagen de 3M resultaten 34% hoger, in januari 2016 15% hoger, in april 2016 30% hoger en in juli 2016 22% hoger.

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.

Grafiek 4.1 Vergelijking som FC resultaten SGS vs 3M Environmental Laboratory 2016



Vervolg Grafiek 4.1



ERM stelt voor dat in de eerstvolgende periode (juli 2016 – juli 2017) opnieuw alle waterstalen geanalyseerd worden door het 3M Environmental Laboratory, en dat de duplicaatstaalname en evaluatie verdeeld wordt over de vier monitoringsrondes net als in desbetreffende periode. Alle waterstalen zullen gedurende de monitoringsperiode van 1 jaar eenmaal door het SGS laboratorium geanalyseerd worden.

4.2 GRONDHOOP VERONTREINIGD MET KWIK EN FC VERBINDINGEN

4.2.1 *Achtergrond*

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 het plan op Kaart 3 is de locatie van deze hoop aangeduid.

Volgens de gegevens opgenomen in het beschrijvend bodemonderzoek opgesteld door de erkend bodemsaneringsdeskundige Arcadis Gedas (ref. 11/003094A, dd.30/06/2006) is de grondhoop voorzien van een boven- en een onderafdek en tonen de uitloogtesten aan dat “uitloging naar het grondwater zeer beperkt tot onbestaande” is.

De toestand van de grondhoop, en meer specifiek het bovenafdek, wordt sinds 2010 jaarlijks visueel door de erkend bodemsaneringsdeskundige ERM gecontroleerd.

Tijdens een inspectie in augustus 2011 bleek dat de afdekfolie over de hoop grond in slechte staat was en daarom is in maart 2012 door de saneringsaannemer DEC nv, onder supervisie van ERM, een nieuwe afdekking aangebracht op de grondhoop. De nieuwe afdekking minimaliseert het risico op uitloging door infiltrerend regenwater.

In juni 2013 is opnieuw een jaarlijkse controle van de afdekking uitgevoerd door ERM: De folie is in goede staat en vertoont geen tekortkomingen. Op basis van deze observatie, en het feit dat de folie van het bovenafdek in 2012 is vervangen, is in het vierde tussentijdsverslag (TTV4), na overleg met OVAM (dd. 22 oktober 2013), besloten de monitoringsfrequentie te verlagen van jaarlijks naar vijfjaarlijks.

4.2.2 *Uitgevoerde werken*

Op vraag van OVAM dd. 13 april 2016 werd het afdek op 26 april 2016 opnieuw controleerd door ERM. Tijdens het controlebezoek zijn visueel geen scheuren of schade aan de afdekking waargenomen. De bevindingen alsook een foto-rapportage zijn opgenomen in een afzonderlijk milieukundig dagboek dat is opgenomen in ***Bijlage 2***.

4.2.3 *Conclusies en aanbevelingen*

De afdekking bevindt zich nog in een goede staat. ERM stelt voor om de volgende controleronde uit te voeren in 2018.

4.3 *FC BRONZONES: GEBOUW 16 EN WWTP*

4.3.1 *Uitgevoerd veldwerk*

Om de FC verontreiniging in het grondwater te monitoren worden de peilbuizen in de bronzones (gebouw 016 en zone WWTP) jaarlijks bemonsterd door ERM. Om de potentiële verticale verspreiding van de verontreiniging buiten de dijk op te volgen, worden een aantal peilbuizen halfjaarlijks of driemaandelijks bemonsterd.

Een overzicht van de bemonsterde peilbuizen is weergegeven in onderstaande tabel. De locaties van deze peilbuizen zijn aangeduid op plan op Kaart 4A en 4B.

Tabel 4.1 ***Overzicht bemonsterde peilbuizen bronzones***

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum				Analyse
		okt 2015	jan 2016	apr 2016	juli 2016	
Zone Gebouw 16						
K3	3,6-5,6				X	FC inclusief PFBS
P21B	3,5-5,5		X	X	X	FC inclusief PFBS
P27	0,9-2,9		X		X	FC inclusief PFBS
P304	4,0-6,0	X	X		X	FC inclusief PFBS
P305	3,5-5,5		X		X	FC inclusief PFBS
P42	3,0-5,0		X		X	FC inclusief PFBS
P56	1,2-3,2				X	FC inclusief PFBS
Zone WWTP						
P118C	2,5-4,5		X		X	FC
P119C	2,5-4,5		X		X	FC
L19	2,8-4,8		X		X	FC
M4	1,0-3,0		X		X	FC
P262	6,2-7,2				X	FC
P263	7,4-8,4		X	X	X	FC
P264	5,0-5,5		X		X	FC
P265B	2,8-3,8				X	FC
P265C	4,5-5,5		Niet *		Niet *	FC

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum				Analyse
		okt 2015	jan 2016	apr 2016	juli 2016	
P340	4,0-6,0				X	FC
P341	3,5-5,5				X	FC
P343	4,8-6,8				X	FC
P371	1,5-3,5		X		X	FC
P374	5,0-6,0		X		X	FC
P379	3,5-5,5				X	FC
P380	3,5-5,5				X	FC
P381	5,0-6,0		X		X	FC
P382	2,5-3,5		X		X	FC

* Peilbuis P265C is beschadigd en niet langer bruikbaar. Tijdens de monitoringsronde juli 2016 is peilbuis P265B bemonsterd.

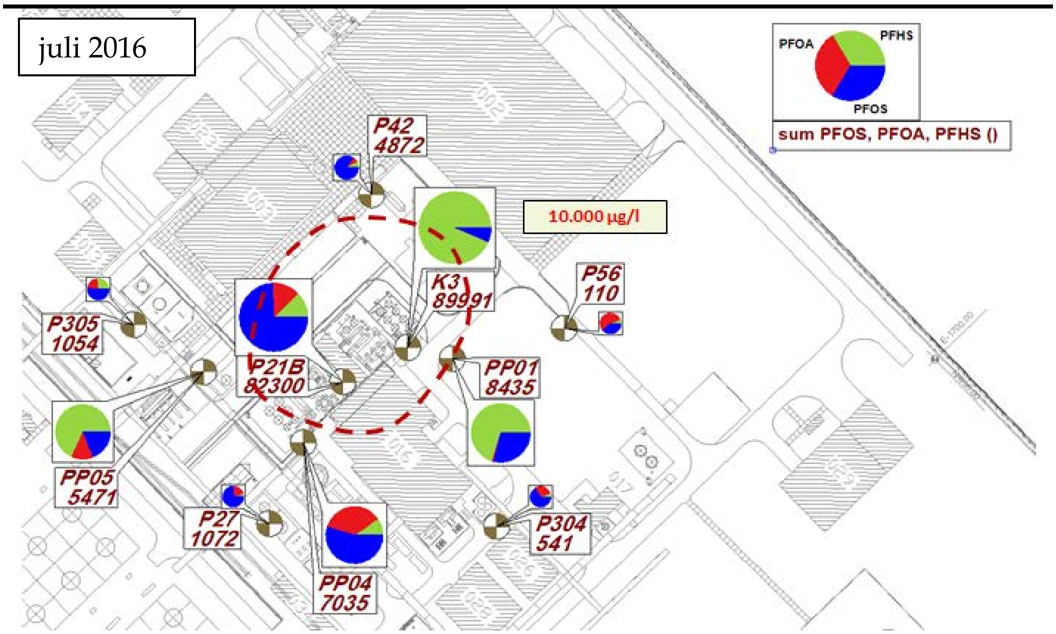
4.3.2 *Bespreking resultaten*

De analyseverslagen zijn toegevoegd in Bijlage 3. Een toetsingstabel met deze resultaten is opgenomen in Bijlage 4 samen met de resultaten van de veldmetingen.

4.3.2.1 *Gebouw 16*

De grondwateranalyseresultaten voor de som van PFOS, PFHS en PFOA van juli 2016 ter hoogte van gebouw 016 zijn op de volgende figuur voorgesteld. De rode lijn stelt de contour van 10.000 µg/L voor de som van PFOS, PFHS en PFOA voor.

Figuur 4.1 Schematische voorstelling FC-concentraties in grondwater thv. Gebouw 16



De evolutie van de concentraties aan FC's in het grondwater ter hoogte van gebouw 016 is voorgesteld in onderstaande grafieken.

Grafiek 4.2 Evolutie FC concentraties t.h.v. Gebouw 16

P304 (4.0 - 6.0 m bgl)

P305 (3.5 - 5.5 m bgl)

P21B (3.5 - 5.5 m bgl)

P42 (3.0 - 5.0 m bgl)

K3 (3.6 - 5.6 m bgl)

P56 (1.2 - 3.2 m bgl)

P27 (0.9 - 2.9 m bgl)

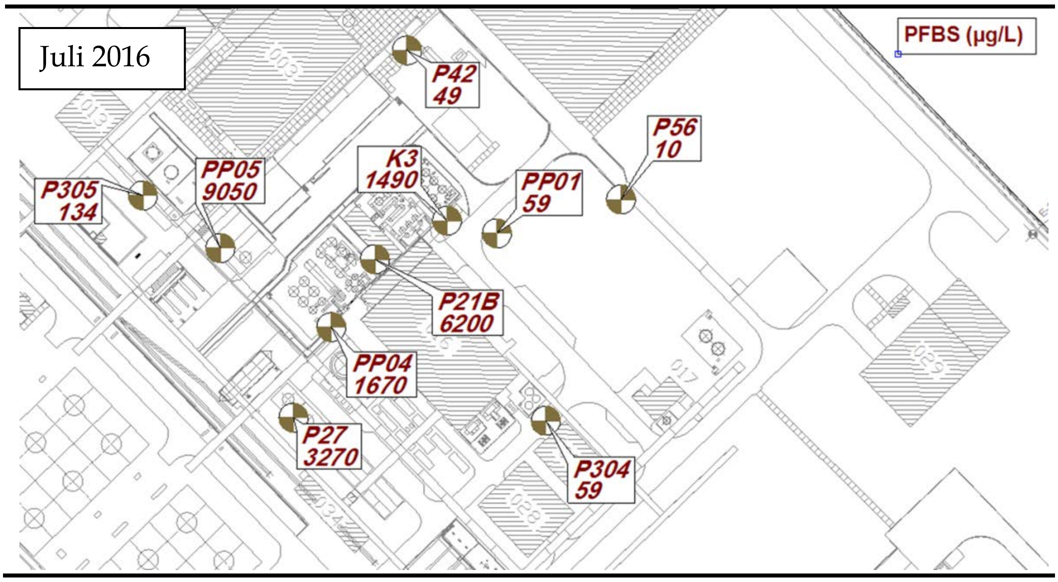
In het algemeen kan gesteld worden dat de FC concentraties in het grondwater ter hoogte van gebouw 016 dalen of stabiel zijn in de tijd, maar de concentraties fluctueren. De concentratie aan PFOS ter hoogte van peilbuis P21B lijkt over het algemeen een licht stijgende trend te vertonen. Deze peilbuis is centraal gelegen in de 10.000 µg/L contour ter hoogte van gebouw 16. Verdere monitoring is noodzakelijk om aan te tonen of deze trend significant is.

In vergelijking met de vorige monitoringsperiodes lijkt de omvang van de som FC concentratiecontour (10.000 µg/L) stabiel te zijn.
De algemene stabiele/dalende trend in FC-concentraties en som FC contour lijkt erop te duiden dat het onttrekkingssysteem de FC-concentraties in het grondwater in de bronzone van gebouw 016 effectief capteert.

De productie van PFOS is in 2002 stopgezet en vervangen door PFBS dat een significant lagere toxiciteit heeft dan PFOS. Alvorens de productie van PFBS te starten, is deze verbinding door 3M uitgebreid gescreend op mogelijke (eco)toxicologische effecten. PFBS vervangt PFOS omdat het significant sneller afbreekt, persistent is maar niet bioaccumuleert en een hoge PNEC-waarde (10.400 µg/L) heeft in vergelijking met PFOS.

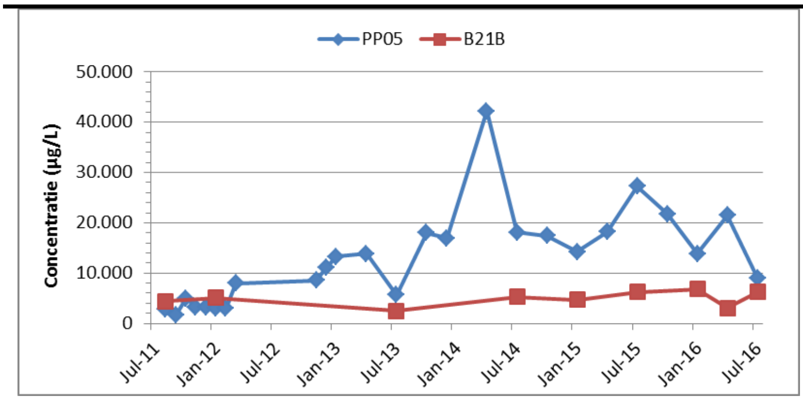
De resultaten voor PFBS van juli 2016 in het grondwater ter hoogte van gebouw 016 zijn op de volgende figuur voorgesteld. De hoogste concentraties worden gemeten ter hoogte van pompput PP05 en peilbuis P21B.

Figuur 4.2 Schematische voorstelling PFBS-concentraties in grondwater thv. gebouw 016



De evolutie van de PFBS concentratie in het grondwater ter hoogte van pompput PP05 en peilbuis P21B is voorgesteld in onderstaande grafiek.

Grafiek 4.3 Evolutie PFBS concentraties t.h.v. PP05 en P21B (Gebouw 16)

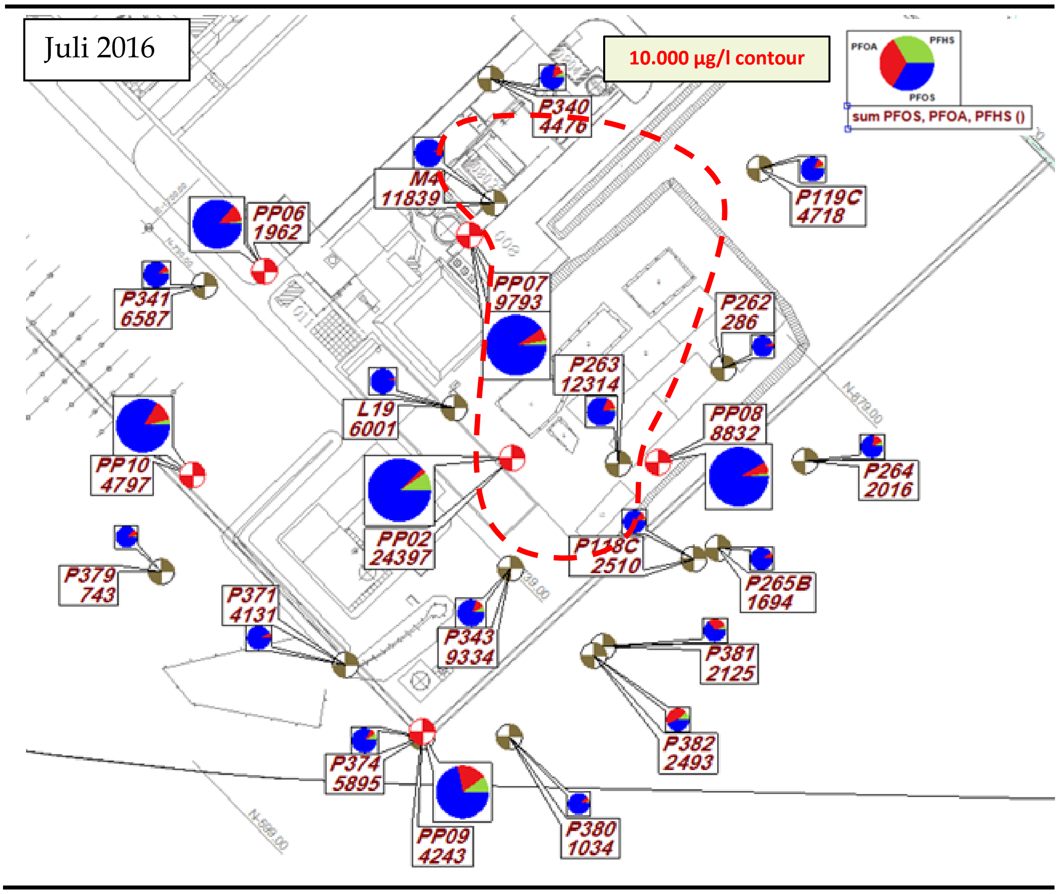


Uit de grafiek blijkt dat de concentratie in pompput PP05 opnieuw verder daalt. De oorzaak van de verhoogde PFBS detectie gemeten in 2014 is momenteel onvoldoende gekend, en wordt verder onderzocht.

4.3.2.2 *Zone WWTP*

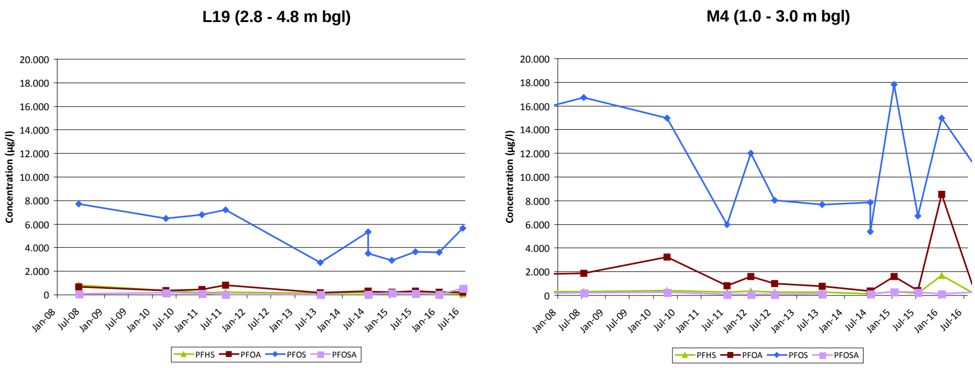
De grondwateranalyseresultaten voor de som van PFOS, PFHS en PFOA van juli 2016 ter hoogte van de WWTP zijn op de volgende figuur voorgesteld. De rode lijn stelt de concentratiecontour van 10.000 µg/L voor de som van PFOS, PFHS en PFOA voor.

Figuur 4.3 Schematische voorstelling FC-concentraties in grondwater thv. WWTP

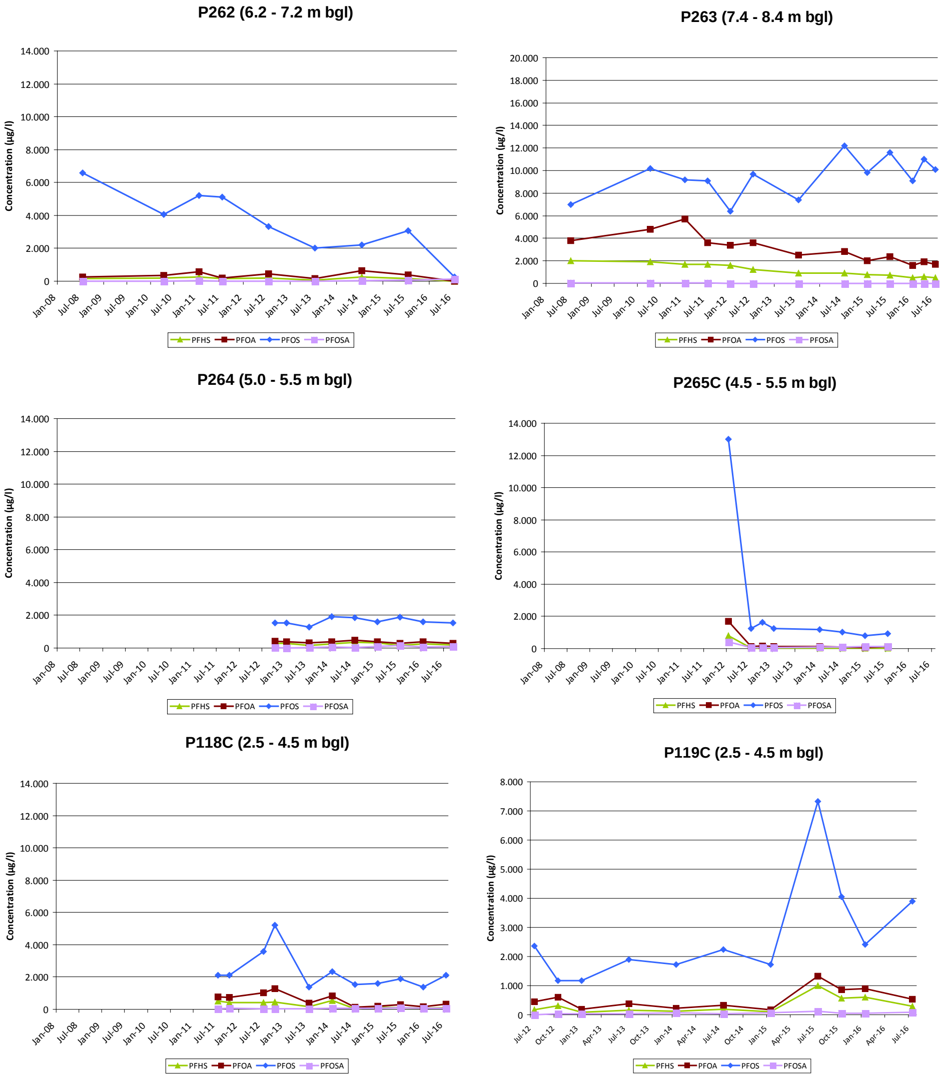


De evolutie van de concentraties aan PFOS, PFHS, PFOA en PFOSA in het grondwater ter hoogte van zone WWTP is voorgesteld in de volgende grafieken.

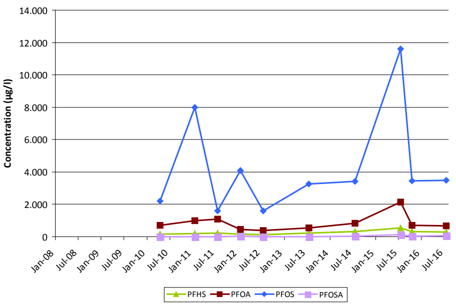
Grafiek 4.4 Evolutie FC concentraties t.h.v. zone WWTP



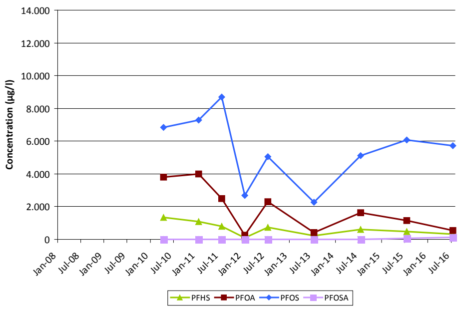
Vervolg Grafiek 4.4



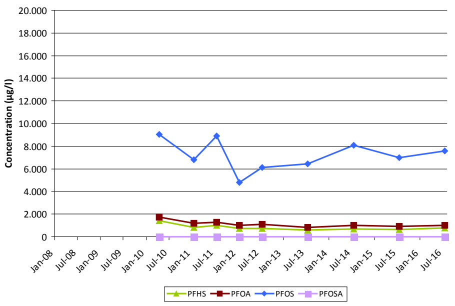
P340 (4.0 - 6.0 m bgl)



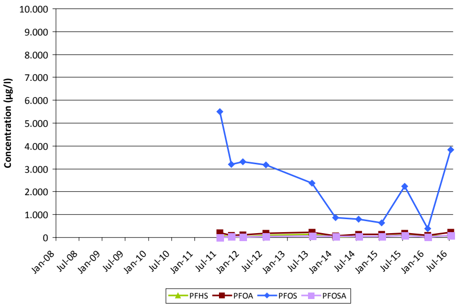
P341 (3.6 - 5.6 m bgl)



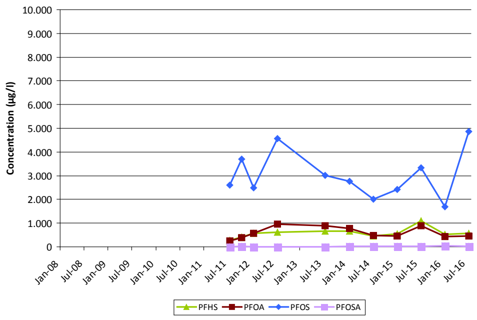
P343 (4.8 - 6.8 m bgl)



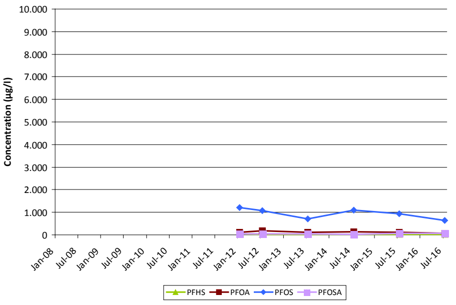
P371 (1.5 - 3.5 m bgl)



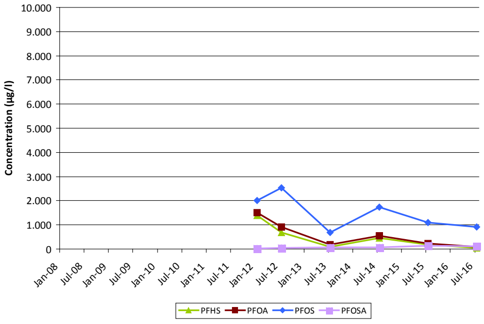
P374 (5.0 - 6.0 m bgl)

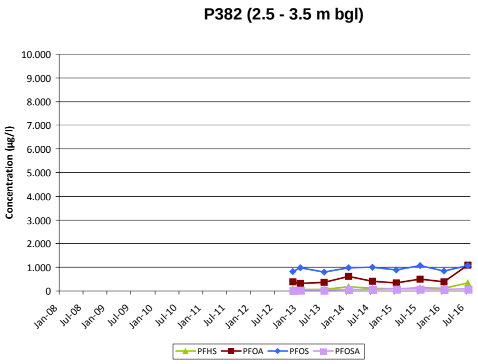


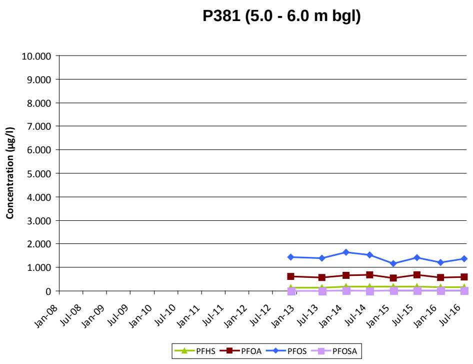
P379 (3.5 - 5.5 m bgl)



P380 (3.5 - 5.5 m bgl)







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. Enkel ter hoogte van peilbuizen P263 en P119C kan een stijgende PFOS concentratie worden waargenomen.

Tijdens de monitoring van januari 2015 en januari 2016 zijn er verhoogde concentratiesgemeten ter hoogte van peilbuis M4. Deze concentraties zijn van dezelfde grootte-orde als de gemeten concentraties in de periode voor de opstart van de P&T. Verdere monitoring is noodzakelijk om een eventuele stijgende trend te bevestigen.

De omvang van de som FC concentratiecontour (10.000 µg/L) is in omvang afgenomen in vergelijking met de contour van vorige monitoringsperiode (2014-15) en is vergelijkbaar met de contour van oktober 2015 en januari 2016.

4.3.3 Conclusies en aanbevelingen

De algemene stabiele/dalende trend in FC-concentraties lijkt erop te duiden dat het onttrekkingssysteem de FC-concentraties in het grondwater in de twee bronzones (Gebouw 016 en WWTP) effectief capteert. Echter de potentiële concentratiestijgingen in enkele peilbuizen kan er op duiden dat de captatie lokaal niet volledig is.

Om het effect van het onttrekkingssysteem op de FC concentraties in de twee bronzones te evalueren, wordt aanbevolen om de periodieke monitoring van peilbuizen te behouden en om een mogelijke optimalisatie van het saneringssysteem te bekijken.

De volgende volledige monitoring in de twee bronzones zal plaatsvinden **in juli 2017**.

4.4 TWEEDE AQUIFER

4.4.1 *Uitgevoerd veldwerk*

Een overzicht van de bemonsterde peilbuizen in de tweede aquifer is weergegeven in onderstaande tabel. De locaties van deze peilbuizen zijn aangeduid op Kaart 4C. De stalen zijn door 3M en SGS geanalyseerd op FC verbindingen.

Tabel 4.2 Overzicht bemonsterde peilbuizen tweede aquifer

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum				Analyse
		Okt. 2015	Jan. 2016	Apr. 2016	Jul. 2016	
Zone Gebouw 16						
D9	13,0-15,0	X	X		X	FC
D14	15,5-16,5				X	FC
D17 *	14,0-15,0				X	FC
D18 *	14,0-15,0				X	FC
D5 *	15,0-16,0				X	FC
ND7 *	15,0-16,0				X	FC
Zone WWTP						
D10	15,0-16,0				X	FC
D11	14,9-15,9				X	FC
BD24-3	17,0-18,0		X		X	FC
BD24-4	22,0-24,0				X	FC
P121	23,5-24,5	X	X		X	FC
P321	15,5-15,6	X	X	X	X	FC
D16 *	14,0-15,0				X	FC
D2 *	24,3-25,3				X	FC
P118A	22,5-23,5				X	FC
P118B	13,0-14,0				X	FC
P119A *	23,5-24,5				X	FC
P119B *	13,0-14,0				X	FC

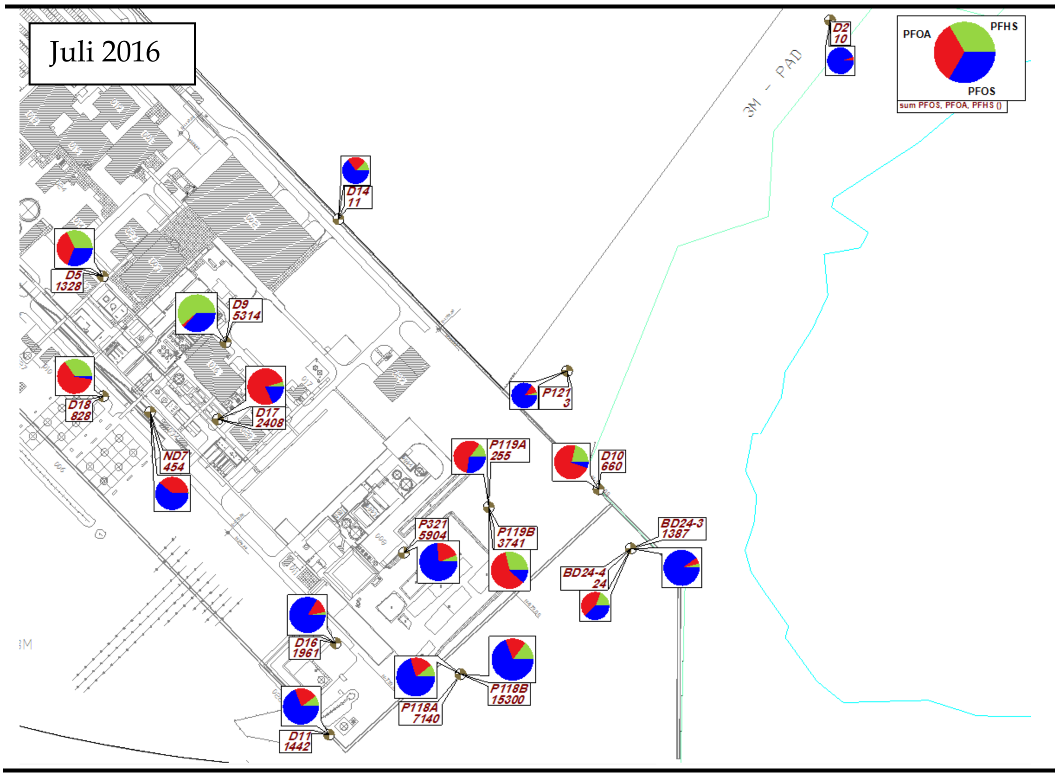
** Peilbuizen bijkomend bemonsterd in juli 2016 ter controle van de concentratie evolutie in de tweede aquifer.*

4.4.2 *Bespreking resultaten*

De analyseverslagen zijn toegevoegd in Bijlage 3. Een toetsingstabel met deze resultaten is opgenomen in Bijlage 4 samen met de resultaten van de veldmetingen.

De grondwateranalyseresultaten voor de som van PFOS, PFHS en PFOA van juli 2016 in de tweede aquifer zijn op de onderstaande figuur voorgesteld.

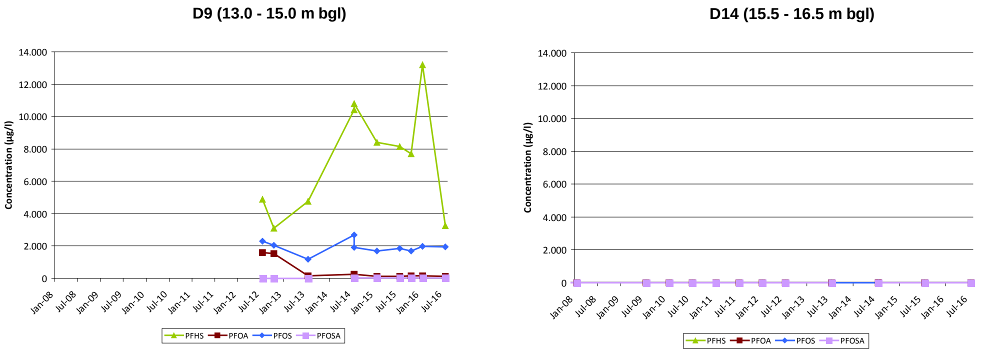
Figuur 4.4 *Schematische voorstelling FC-concentraties in grondwater tweede aquifer*



De evolutie van de concentraties aan PFOS, PFHS, PFOA en PFOSA in het grondwater van de tweede aquifer is voorgesteld in de volgende grafieken.

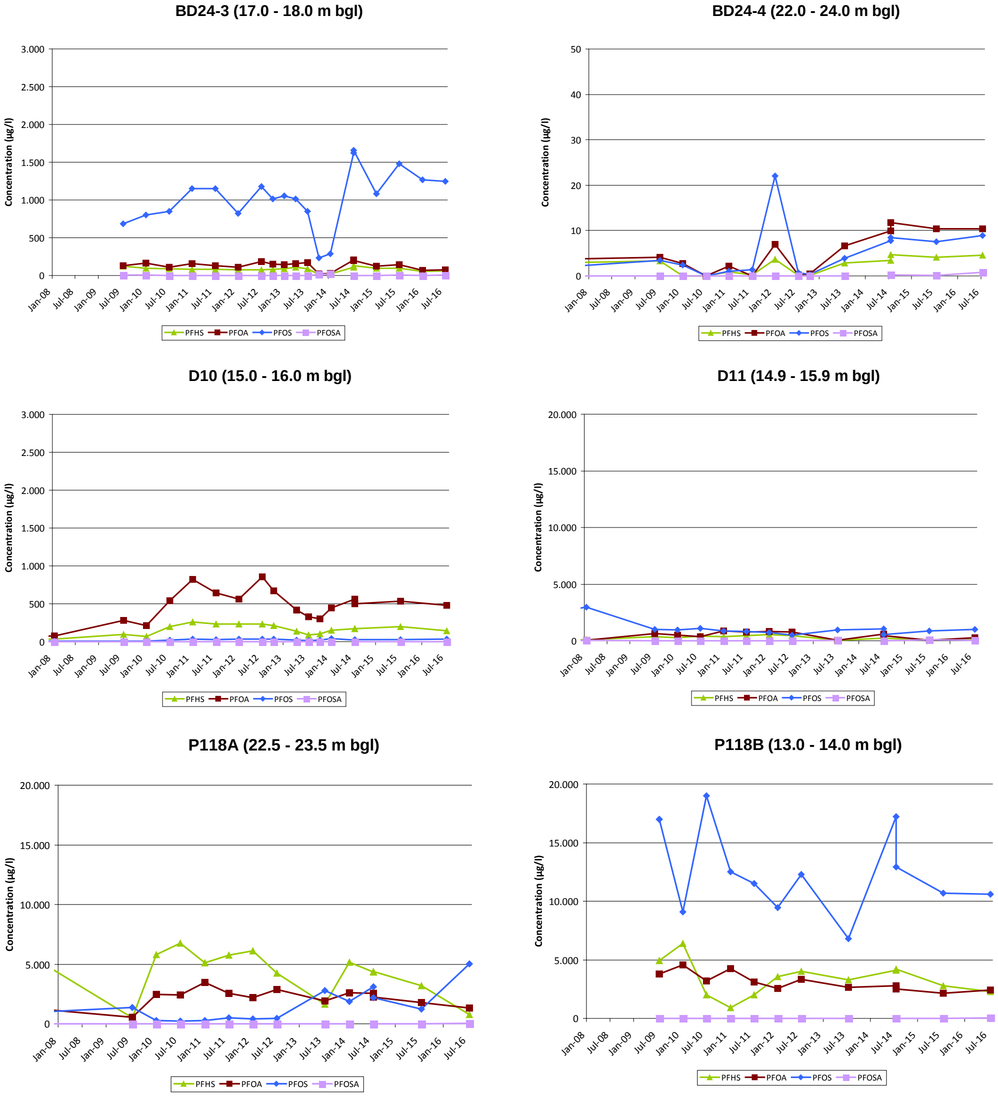
Grafiek 4.5 Evolutie FC concentraties in tweede aquifer t.h.v. Gebouw 16 en zone WWTP

Bronzone Gebouw 16


D9 (13.0 - 15.0 m bgl)

D14 (15.5 - 16.5 m bgl)

Bronzone WWTP


BD24-3 (17.0 - 18.0 m bgl)

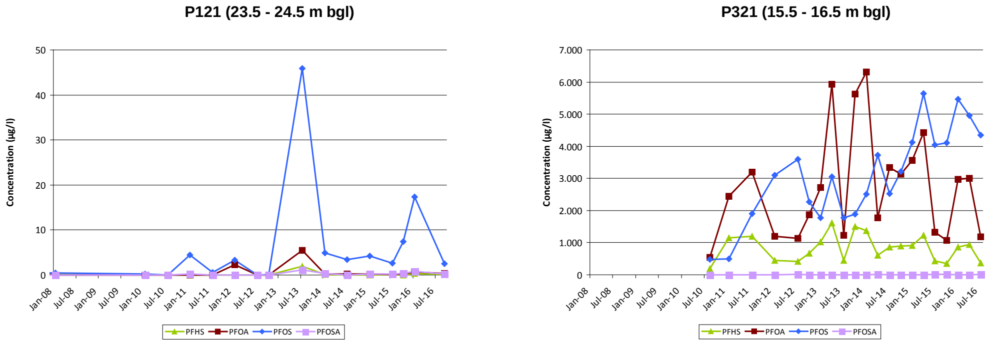
BD24-4 (22.0 - 24.0 m bgl)

D10 (15.0 - 16.0 m bgl)

D11 (14.9 - 15.9 m bgl)

P118A (22.5 - 23.5 m bgl)

P118B (13.0 - 14.0 m bgl)

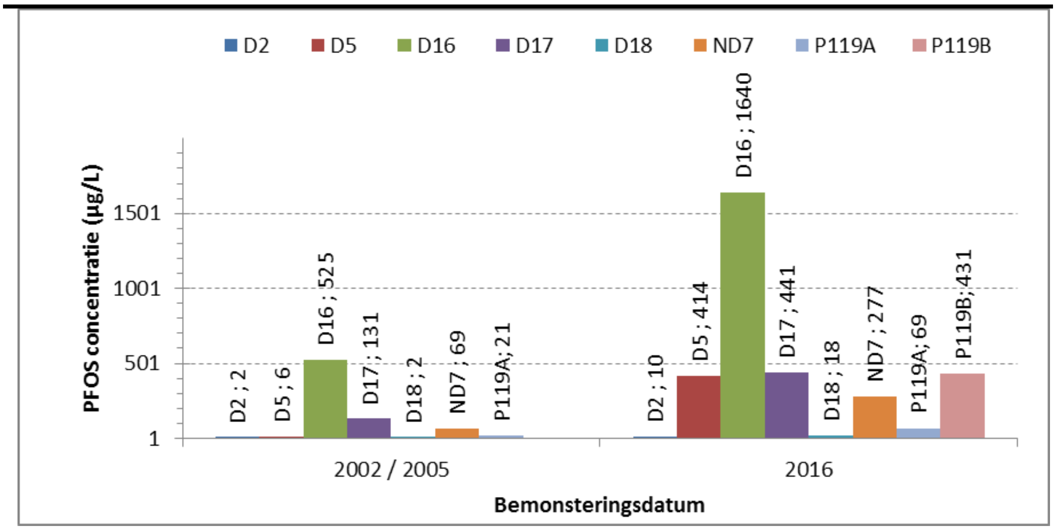


Ter hoogte van de WWTP tonen de FC-concentraties in aantal peilbuizen (P321, BD24-3 BD24-4, D10 en P118A) een stijgende trend, maar concentraties fluctueren significant. In de overige peilbuizen lijken de concentraties aan FC verbindingen in het grondwater stabiel te zijn in de tijd.

Ter hoogte van gebouw 016 (peilbuis D9) is in januari 2016 een verhoogde concentratie aan PFHS in grondwater gemeten. Deze verhoogde concentratie is niet bevestigd tijdens metingen in juli 2016. Verdere monitoring dient aan te wijzen of er eventueel een stijgende PFHS concentratietrend is.

In juli 2016 zijn een aantal peilbuizen met filterstelling in de 2de aquifer bemonsterd. Het betreft peilbuizen D2, D5, D16, D17, D18, ND7, P119A en P119B. De gemeten concentraties in juli 2016 zijn vergeleken met de concentraties gemeten in 2002 of 2005 (Peilbuis P119A). Met uitzondering van peilbuis P119B (hiervan werden geen oude analyseresultaten teruggevonden) zijn in het grondwater ter hoogte van deze peilbuizen (beperkt) verhoogde concentraties gemeten voor een of meerdere organofluorverbindingen (zie grafiek hieronder).

Grafiek 4.6 Evolutie FC concentraties in tweede aquifer t.h.v. Gebouw 16 en zone WWTP



4.4.3 *Conclusies en aanbevelingen*

De gemeten concentraties in de onderzochte peilbuizen in de tweede aquifer zijn lager dat het vijfvoud van de concentraties bij de opstart van de sanering.

Ter hoogte van de noordoostelijke perceelgrens (stroomafwaarts van de bronzones in de eerste aquifer) zijn de concentraties aan FC verbindingen lager dan 10% van de oplosbaarheid. Hieruit volgt dat wordt voldaan aan de saneringsdoelstellingen voor de tweede aquifer.

De noodzaak om deze saneringsdoelstellingen op te volgen (door monitoring) blijft behouden omdat in meerdere peilbuizen een potentiele concentratiestijging voor FC's wordt gemeten. Een mogelijke optimalisatie van het saneringssysteem moet worden bekeken om de potentiele migratie van verontreiniging van de eerste naar de tweede aquifer verder te minimaliseren.

Verder wordt aanbevolen om de periodieke monitoring van peilbuizen in de tweede aquifer te behouden om het effect van het onttrekkingssysteem op de FC concentraties in de tweede aquifer te kunnen blijven evalueren.

In juli 2016 zijn een achttal bijkomende peilbuizen bemonsterd en de concentraties vergeleken met eerder gemeten concentraties. In vrijwel alle peilbuizen lag de concentratie FC's hoger dan eerder gemeten, er wordt bijgevolg geadviseerd om deze peilbuizen in juli 2017 opnieuw te bemonsteren om deze observatie te verifieren, en om na te gaan wat de mogelijke impact is van de verschillende labo's die zijn gebruikt voor de analyse en de natuurlijke concentratie fluctuaties. In het volgende tussentijdse rapport zullen de resultaten voor deze peilbuizen verder worden besproken.

De eerst volgende volledige monitoring zal plaatsvinden **in juli 2017**.

4.5 ZUIDELIJKE TERREINGRENS

4.5.1 Uitgevoerd veldwerk

Een overzicht van de bemonsterde peilbuizen langs de zuidelijke 3M perceelgrens is weergegeven in onderstaande tabel. De locaties van deze peilbuizen zijn aangeduid op Kaart 4D. De stalen zijn door 3M en SGS geanalyseerd op FC verbindingen.

Voormalige peilbuis B3 is op 7 oktober 2015 door Servaco nv (onder begeleiding van ERM) herplaatst op een hoger gelegen deel iets ten noorden van de locatie van peilbuis B3. De nieuwe peilbuis is B3-bis genoemd.

Tabel 4.3 *Overzicht bemonsterde peilbuizen zuidelijke terreingrens*

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum		Analyse
		januari 2016	juli 2016	
B3-bis *	2,5-3,5 **	X	X	FC
B7	1,8-3,8	X	X	FC
P372	4,8-5,8	X	X	FC
P378	3,5-5,5	X	X	FC
PA109A	5,0-6,0	X	X	FC
PA111A	4,0-6,0	X	X	FC
PA112	3,5-5,5	X	X	FC

* Peilbuis B3 is vervangen door B3-bis in oktober 2015.

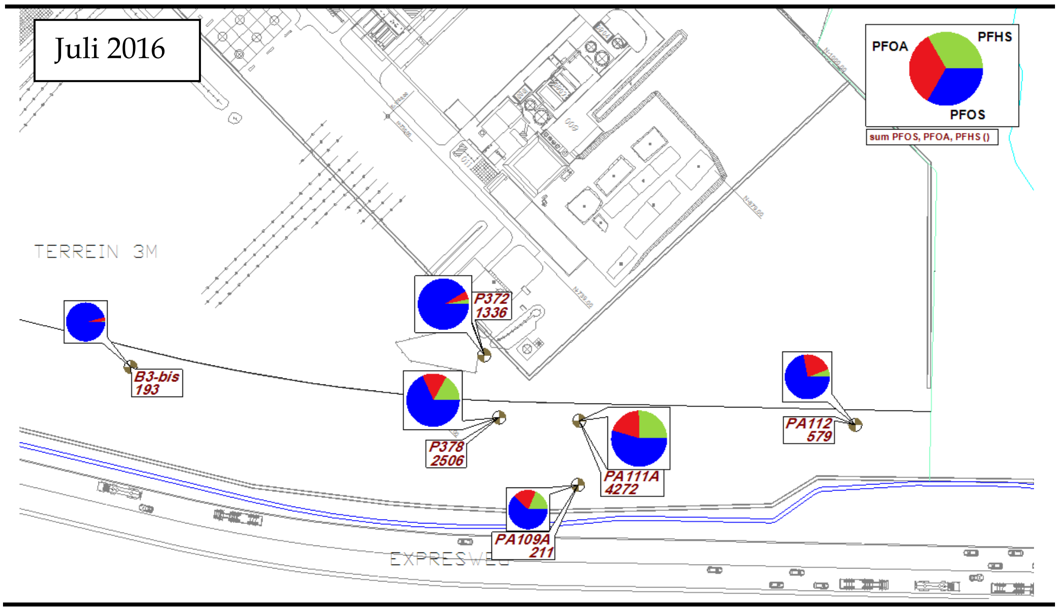
** Filterdiepte van B3-bis t.o.v. maaiveldniveau is aangepast in vergelijking met filterdiepte van voormalige peilbuis B3 (0,4-1,4 m-mv). Het maaiveldniveau ligt 2 meter hoger ter hoogte van de peilbuis P3-bis.

4.5.2 *Bespreking resultaten*

De analyseverslagen zijn toegevoegd in Bijlage 3. Een toetsingstabel met deze resultaten is opgenomen in Bijlage 4 samen met de resultaten van de veldmetingen.

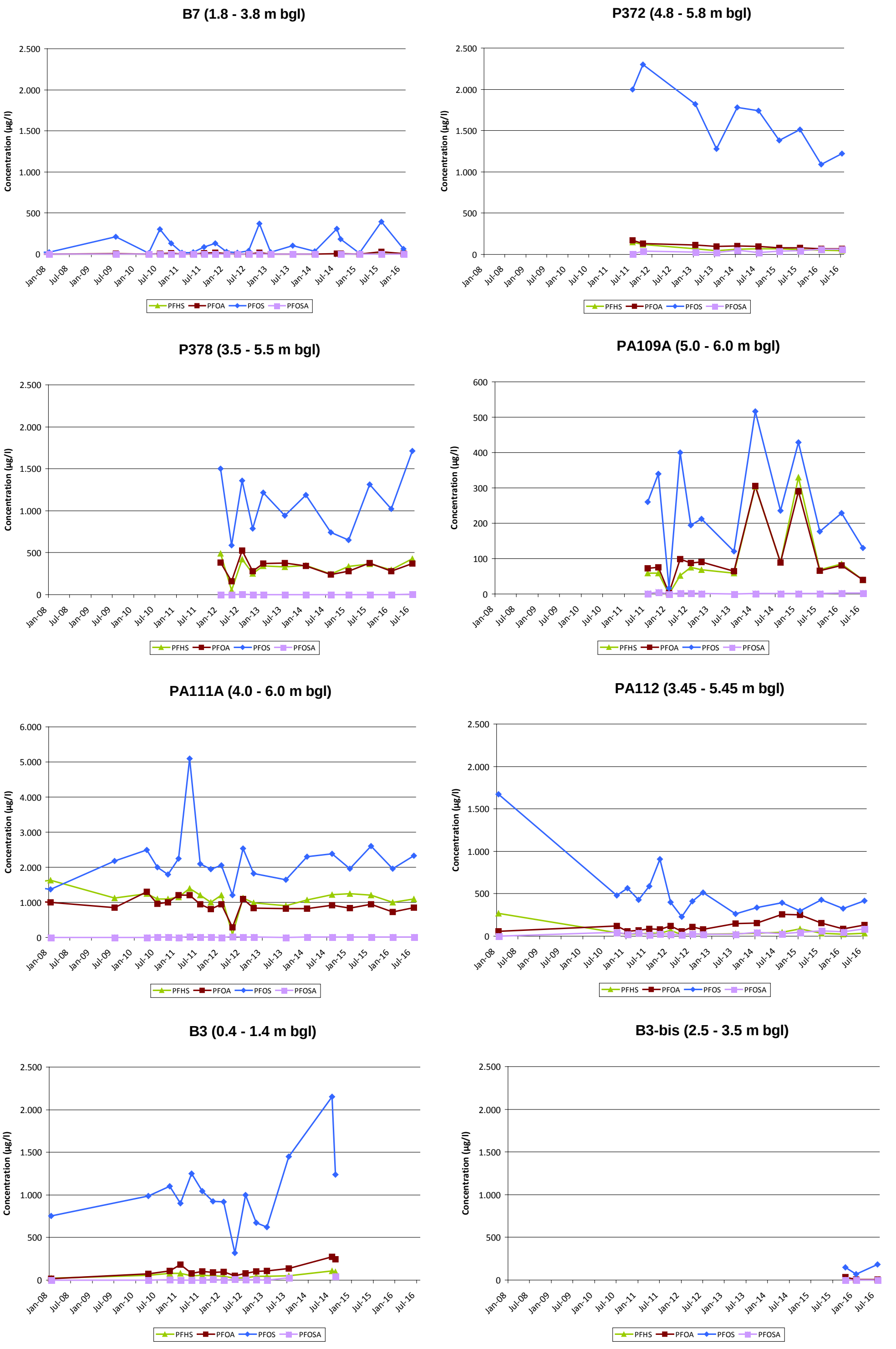
De grondwateranalyseresultaten voor de som van PFOS, PFHS en PFOA van juli 2016 ter hoogte van de zuidelijke perceelgrens zijn op de onderstaande figuur voorgesteld.

Figuur 4.5 *Schematische voorstelling FC-concentraties in grondwater t.h.v. zuidelijke perceelgrens*



De evolutie van de FC concentraties in het grondwater ter hoogte van de zuidelijke perceelgrens is voorgesteld in onderstaande grafieken.

Grafiek 4.7 Evolutie FC concentraties t.h.v. zuidelijke perceelgrens



Ter hoogte van peilbuizen B7, P372, PA109A, PA111A, PA112 en B3-bis vertonen de FC-concentraties in het grondwater ter hoogte van de zuidelijke perceelgrens een stabiele of dalende trend met fluctuaties in de tijd. Een uitzondering hierop is peilbuis P378, waar de fluctuerende concentraties een stijgende trend lijken te vertonen. Verdere metingen dienen deze mogelijk stijgende trend nog te bevestigen.

4.5.3 *Conclusies en aanbevelingen*

Algemeen kan gesteld worden dat de gemeten concentraties FC verbindingen in grondwater ter hoogte van de zuidelijke perceelgrens in dezelfde lijn liggen als de concentraties gemeten tijdens voorgaande bemonsteringen. De resultaten lijken erop te duiden dat het onttrekkingssysteem, geïnstalleerd ter hoogte van de WWTP bronzone, de migratie van de verontreiniging naar de zuidelijke perceelgrens beperkt.

De halfjaarlijkse monitoring blijft behouden en de volgende monitoring zal plaatsvinden **in januari 2017**.

4.6 GEBIED TEN ZUIDEN VAN DE EXPRESWEG (Z-PEILBUIZEN)

Tijdens het overleg met OVAM in oktober 2013 heeft OVAM de opmaak van een gefaseerd eindevaluatieonderzoek opgelegd voor de zone ten zuiden van de Expresweg (Z-peilbuizen). Dit tweede gefaseerd eindevaluatieonderzoek met referentie R002-0243999-v2.0 is op 01/07/2015 door de OVAM conform verklaard.

Alle peilbuizen in deze zone zijn definitief buitengebruik gesteld conform de wettelijke voorschriften door Servaco onder begeleiding van ERM op 8 december 2015 en 23 februari 2016. Verdere details betreffende het buiten gebruik stellen staan beschreven in een afzonderlijk milieukundig dagboek opgenomen in ***Bijlage 2***.

4.7 ZONE ONDERGRONDSE TANKEN

Het doel van de grondwatermonitoring was om na te gaan of er een verdere toename van de concentraties aan vluchtige aromaten in het grondwater plaatsvindt ter hoogte van de zone met ondergrondse tanks. Sinds de start van de sanering in juli 2009 zijn geen concentraties aan tolueen, ethylbenzeen en xylenen gemeten die de overeenkomstige terugsaneerwaarden overschrijden. De saneringsdoelstelling (zie paragraaf 3.2.1) voor vluchtige aromaten in grondwater is behaald.

Tijdens het overleg met OVAM in oktober 2013 heeft OVAM de opmaak van een eindevaluatieonderzoek opgelegd voor de (voormalige) verontreiniging met BTEX in het grondwater. Het rapport van het eindevaluatieonderzoek met referentie R001-0243999 is op 24/10/2014 aan OVAM overgemaakt en conform verklaard op 1 juli 2015.

Bij de periodieke grondwatermonitoring is echter gemeten dat in twee peilbuizen de concentratie minerale olie verhoogd is. Deze verhoogde concentraties worden momenteel gemonitord en de oorzaak wordt verder onderzocht.

4.7.1 *Uitgevoerd veldwerk*

In onderstaande tabel is een overzicht gegeven van de bemonsterde peilbuizen. De locaties van deze peilbuizen zijn aangeduid op Kaart 4A. De stalen zijn door SGS geanalyseerd op minerale olie.

Tabel 4.4 *Overzicht bemonsterde peilbuizen zone ondergrondse tanks*

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum Juli 2016	Analyses
P18	1,4-3,4	X	MO
P28	1,1-3,1	X	MO

4.7.2 *Bespreking resultaten*

De analyseverslagen zijn toegevoegd in Bijlage 3.

4.7.2.1 *Veldparameters*

De pH en geleidbaarheid van peilbuizen P18 en P28 zijn niet tijdens de bemonstering gemeten omdat er, omwille van sterk verhoogde waarden, een reële kans bestaat op beschadiging van de veldmeetapparatuur.

4.7.2.2 *Minerale olie*

De concentratie aan minerale olie in het grondwater ter hoogte van peilbuis P18 bedraagt 250 µg/L.

De concentratie aan minerale olie in het grondwater ter hoogte van peilbuis P28 bedraagt 12.000 µg/L. Deze concentratie overschrijdt ruim de bodemsaneringsnorm van 500 µg/L en de gemeten concentratie in 2016 is hoger dan de voorgaande gemeten concentraties aan minerale olie in grondwater ter hoogte van deze peilbuis (range 500 – 2.100 µg/L).

De dominante olie fracties zijn de C12-C20 en C20-C30 fracties. Verdere opvolging van de concentraties aan minerale olie is aangewezen.

4.7.3 *Conclusies en aanbevelingen*

De verontreiniging met minerale olie, pH en geleidbaarheid is afgeperkt en gelokaliseerd rond peilbuizen P18 en P28. De hoogst gemeten concentratie minerale olie in 2016 is significant gestegen ten opzichte van voorgaande metingen.

Momenteel kan geen verklaring voor de gemeten verhoogde waarden aan minerale olie in het grondwater worden gegeven. Interferentie door een andere verontreiniging kan niet worden uitgesloten. Verder historisch en analytisch brononderzoek ter hoogte van P18 en P28 is in uitvoering. De jaarlijkse monitoring van minerale olie in peilbuizen P18 en P28 blijft behouden en de volgende monitoring zal plaatsvinden **in juli 2017**.

4.8 NATUURGEBIED BLOKKERSDIJK

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

Al enige tijd wordt in enkele peilbuizen en de 3M-vijver een stijgende trend voor PFOS waargenomen. Echter, deze peilbuizen en de 3M vijver liggen stroomopwaarts van de meest nabij gelegen, gekende bronzone: de WWTP zone. Daarom is een geohydrologisch onderzoek uitgevoerd om na te gaan of in deze monitoringszone een potentiële FC- bron aanwezig is die de stijgende concentratietrends kan verklaren. Daarnaast is de methode van de statistische analyse van de PFOS-concentraties herbekeken om na te gaan of de initiële aannames waarop de analyse is gebaseerd nog geldig zijn (zie conform verklaarde bodemsaneringsproject (1e fase) (2).

De resultaten van de grond- en oppervlaktewatermonitoring, de herevaluatie van de statistische analyse van de PFOS-concentraties en het nader brononderzoek worden hierna beschreven.

4.8.1 *Monitoring grond- en oppervlaktewater*

4.8.1.1 *Uitgevoerd veldwerk*

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

In onderstaande tabel is een overzicht gegeven van de grondwater en oppervlaktewater monitoring ter hoogte van het 3M pad ten westen van het natuurgebied Blokkersdijk. De locaties van de peilbuizen en de monsternamepunten voor het oppervlaktewater zijn aangeduid op Kaart 4E. De grondwater- en oppervlaktewaterstalen zijn geanalyseerd op FC verbindingen door 3M en SGS.

Tabel 4.5 ***Overzicht staalnamelocaties natuurgebied Blokkersdijk***

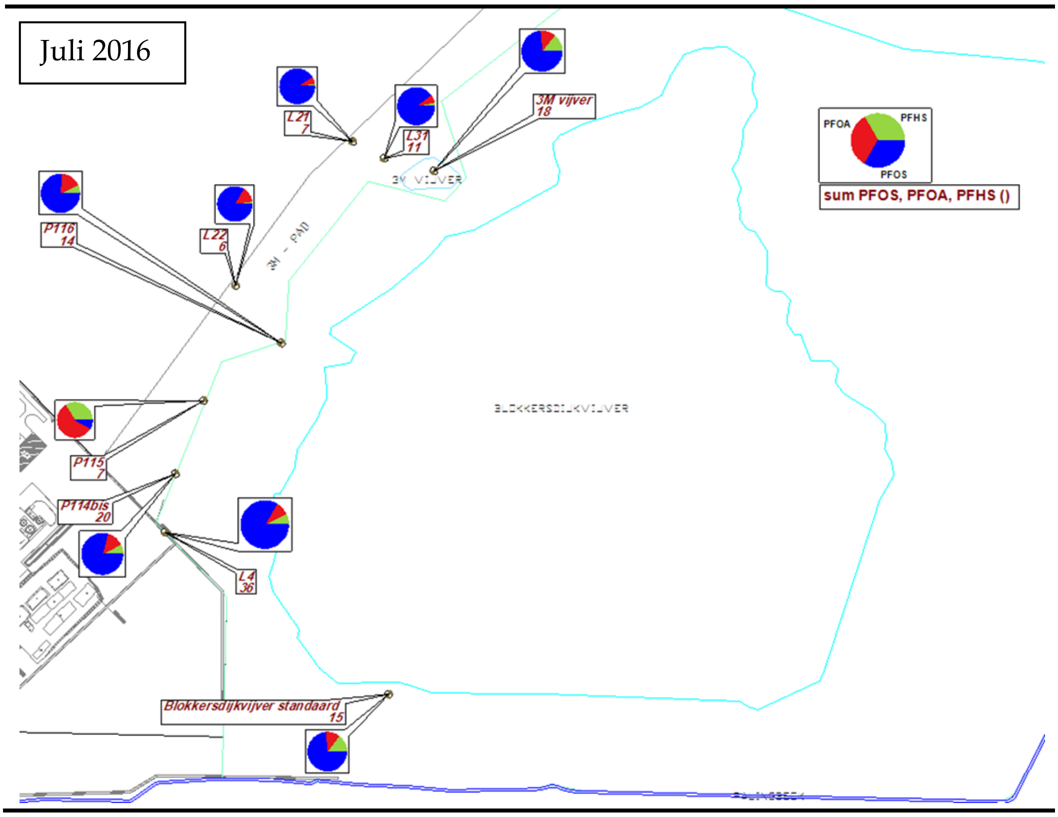
Locatie	Filterdiepte (m-mv)	Bemonsteringsdatum				Analyse
		Okt. 2015	Jan. 2016	Apr. 2016	Jul. 2016	
L21	3,0-5,0		X	X	X	FC
L22	2,0-4,0		X	X	X	FC
L31	1,2-3,2		X	X	X	FC
L4	2,0-4,0	X	X	X	X	FC
P114bis	3,6-4,6	X	X	X	X	FC
P115	3,3-4,3		X	X	X	FC
P116	2,5-3,5	X	X	X	X	FC
3M vijver	-	X	X	X	X	FC
Blokkersdijk standaard	-	X	X	X	X	FC

4.8.1.2 *Resultaten*

De analyseverslagen zijn toegevoegd in Bijlage 3. Een toetsingstabel met deze resultaten is opgenomen in Bijlage 4 samen met de resultaten van de veldmetingen.

Op onderstaande figuren zijn de meest recent (juli 2016) gemeten concentraties voor de som van PFOS, PFHS en PFOA in het grondwater langs de westkant van het natuurgebied Blokkersdijk en in het oppervlaktewater in de vijvers weergegeven.

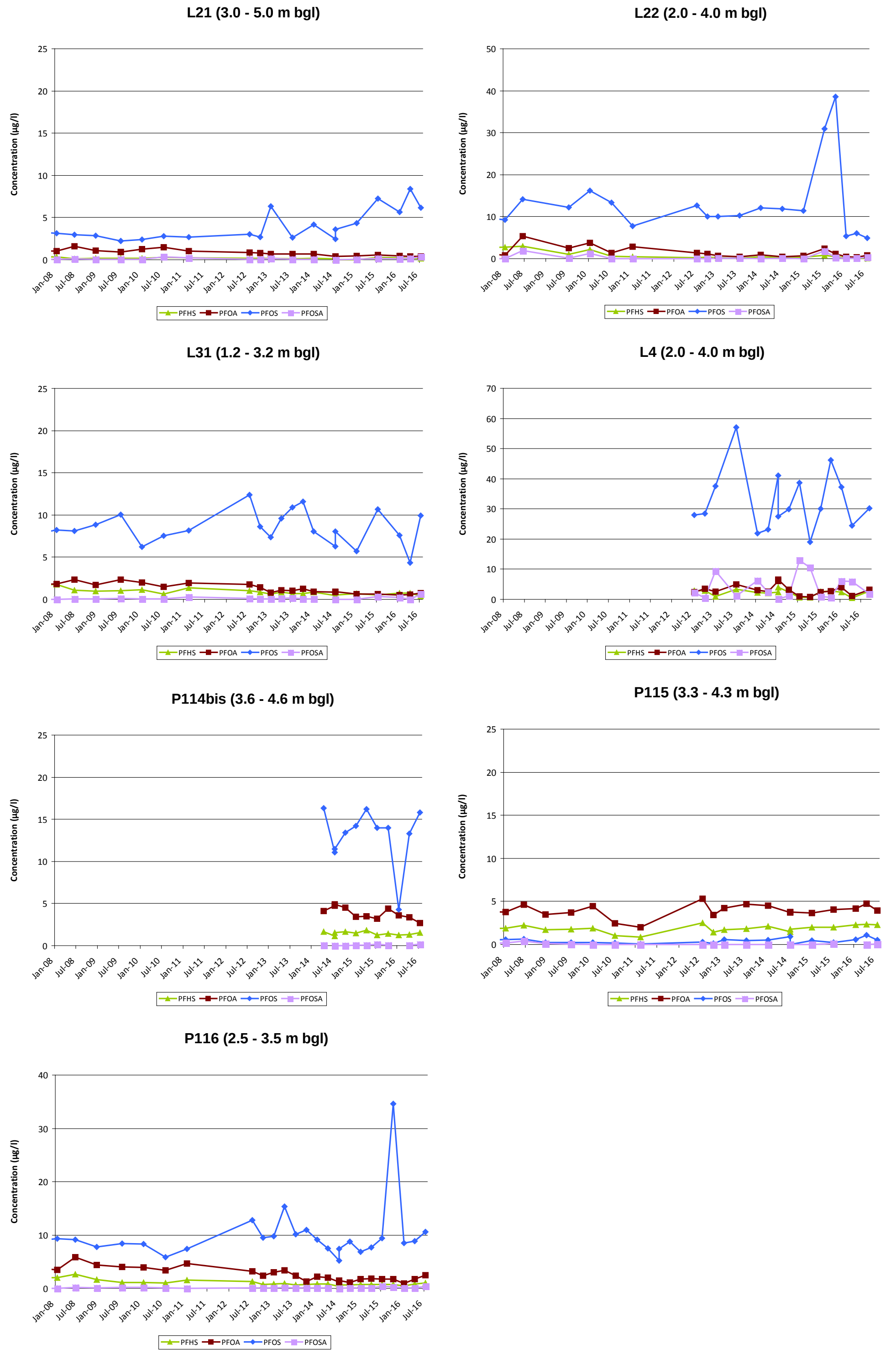
Figuur 4.6 Schematische voorstelling FC-concentraties in grondwater thv. natuurgebied Blokkersdijk



Algemeen kan worden gesteld dat de concentraties in grond- en oppervlaktewater relatief laag zijn.

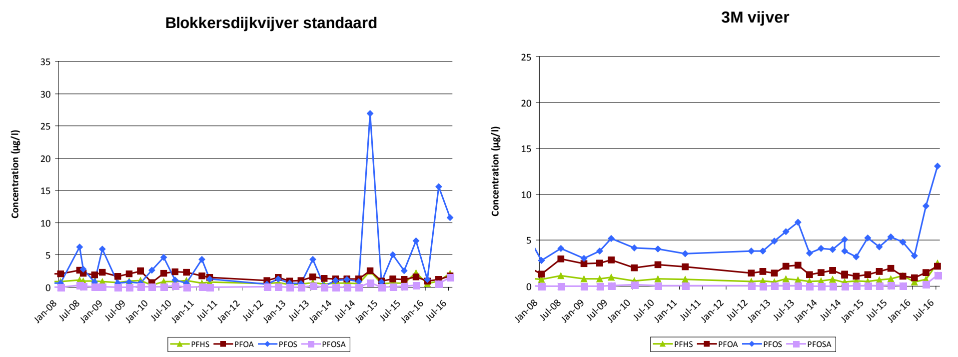
De evolutie van de FC concentraties in de peilbuizen in het 3M pad, dat langs de westkant van het natuurgebied loopt, is voorgesteld in onderstaande grafieken.

Grafiek 4.8 Evolutie FC concentraties in grondwater t.h.v. 3M pad (westelijk van Blokkersdijk natuurgebied)



De evolutie van de FC concentraties in het oppervlaktewater van de Blokkersdijkvijver en de 3M vijver is in de onderstaande grafieken weergegeven.

Grafiek 4.9 *Evolutie FC concentraties in oppervlaktewater Blokkersdijk natuurgebied*



4.8.2 *Statistische evaluatie PFOS-concentraties*

Na de goedkeuring van het bodemsaneringsproject (1e fase) (2) in 2009 zijn er periodiek waterstalen genomen van een aantal monitoringspeilbuizen en de twee vijvers in het natuurreservaat 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 worden 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 labo de meest representatieve is.
Uit de evaluatie (zie ***Bijlage 5***) blijkt dat in peilbuis L21 een significant stijgende trend wordt waargenomen en in peilbuis L22 een significant dalende trend.

4.8.3 *Conclusies en aanbevelingen*

De PFOS concentratie trend in de Blokkersdijk peilbuizen, de 3M vijver en de Blokkersdijk vijver worden verder opgevolgd via regelmatige monitoring en statistische evaluatie voort te zetten met enkel de data van het 3M Environmental lab, omdat de herevaluatie van de statistische analyses heeft aangetoond dat het gebruik van meerdere labo's de resultaten van de PFOS trend analyse beïnvloedt. Uit de evaluatie blijkt dat in peilbuis L21 een significant stijgende trend wordt waargenomen en in peilbuis L22 een significant dalende trend.
Momenteel is geen noodzaak voor bijkomende saneringsacties in deze zone aangezien er geen stijgende concentratietrend is waargenomen in de

Blokkersdijk vijver. Echter, uit de resultaten van het geohydrologisch onderzoek uitgevoerd in 2014 blijkt de oude chemische riolering die ter hoogte van het 3M pad loopt mogelijk een secundaire verontreinigingsbron is. Verder onderzoek (o.m. massa berekeningen en grondwatermodellering met stoftransport) zijn nodig om de werkelijke impact van de – in de oude riolering – aanwezige FC massa op de freatische aquifer en nabij gelegen vijvers na te gaan.

De volgende volledige monitoring zal plaats vinden **in januari 2017**.

4.9 PALINGBEEK EN TOPHAT GRACHT

4.9.1 Uitgevoerd veldwerk

Om de FC vuilvracht naar de Schelde op te volgen wordt het oppervlaktewater van de Palingbeek en de Tophatgracht en het oppervlaktewater ter hoogte van het bemalingsstation driemaandelijks bemonsterd. De staalnamelocaties zijn aangeduid op Kaart 4F. De stalen zijn door 3M en SGS geanalyseerd op FC verbindingen.

Tabel 4.6 Overzicht staalnamelocaties Palingbeek en Tophatgracht

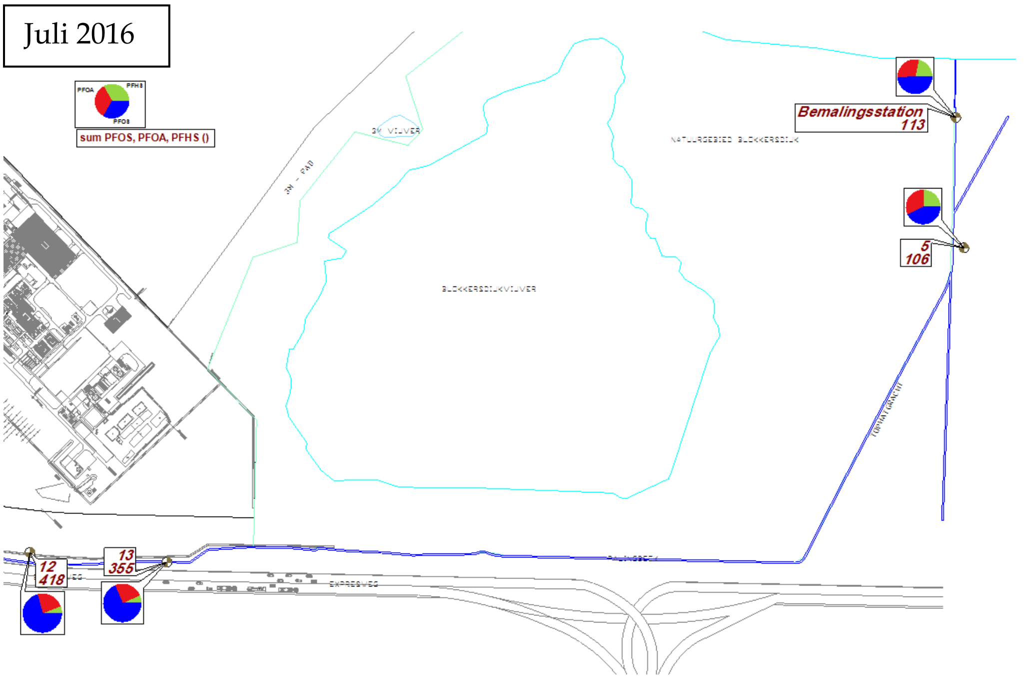
Locatie	Bemonsteringsdatum				Analyse
	Okt. 2015	Jan. 2016	Apr. 2016	Jul. 2016	
12	X	X	X	X	FC
13	X	X	X	X	FC
5	X	X	X	X	FC
Bemalingsstation	X	X	X	X	FC

4.9.2 Bespreking resultaten

De analyseverslagen zijn toegevoegd in Bijlage 3. In de toetsingstabellen in Bijlage 4 is een overzicht gegeven van de uitgevoerde chemische analyses voor oppervlaktewater en van de veldmetingen.

De analyseresultaten van het oppervlaktewater voor de som van PFOS, PFHS en PFOA van juli 2016 zijn op de volgende figuur voorgesteld.

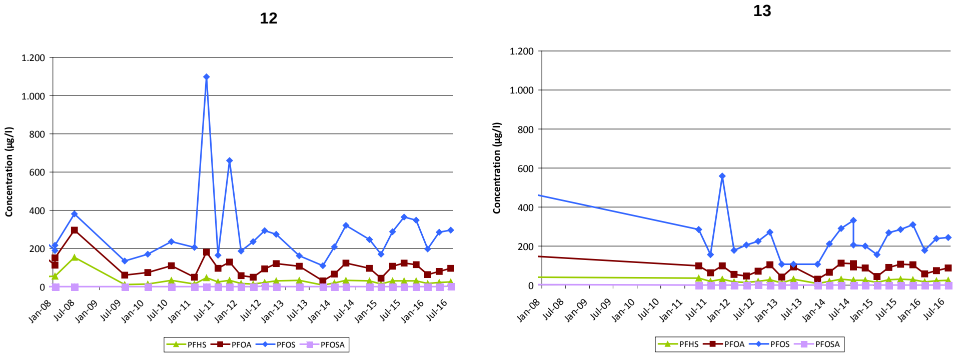
Figuur 4.7 *Schematische voorstelling FC-concentraties in grondwater t.h.v. Palingbeek en de Tophat gracht*



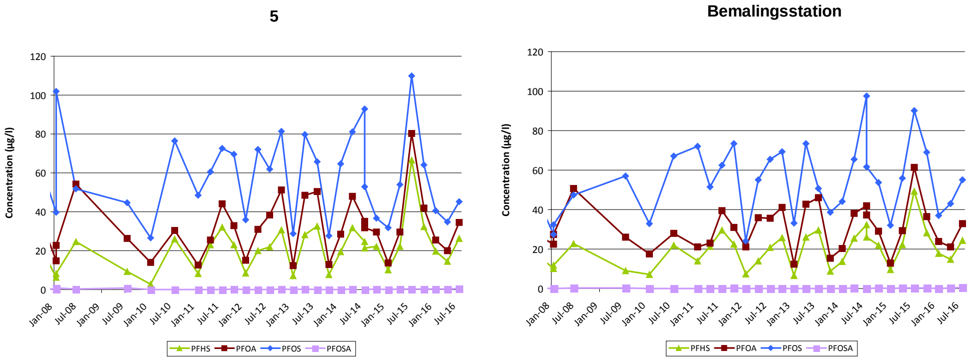
De evolutie van de FC concentraties in het oppervlaktewater van de Palingbeek en de Tophatgracht wordt weergegeven in onderstaande grafieken.

Grafiek 4.10 Evolutie FC concentraties in oppervlaktewater Palingbeek en de Tophat gracht

Staalnamepunten Palingbeek



Staalnamepunten Tophat gracht



De FC-concentraties ter hoogte van de Palingbeek vertonen globaal gezien een stabiel verloop sinds 2012, maar de concentraties kunnen fluctueren. De concentraties gemeten in de Tophat gracht zijn lager dan de concentraties in de Palingbeek.

4.9.3 *Vuilvrachtberekening*

De dagelijkse vuilvracht aan PFOS welke, berekend op basis van het jaarlijks gemiddelde van de analyseresultaten ter hoogte van het bemalingsstation, via de Palingbeek en Tophat gracht in de Schelde terecht komt, bedraagt 171 g voor de periode 1 juli 2015 tot en met 30 juni 2016. In onderstaande tabel is een overzicht gegeven van de berekende vuilvracht per jaar sinds de start van de monitoring in 2009.

Tabel 4.7 Gemiddelde PFOS vuilvrachtberekening in oppervlaktewater

Periode	Debiet (m^3/dag)	PFOS concentratie (µg/L)	Vuilvracht (g/dag)
juli 2009 tem. juni 2010	2 865	45,0	129
juli 2010 tem. juni 2011	2 865	63,5	182
juli 2011 tem. juni 2012	2 865	53,7	154
juli 2012 tem. juni 2013	2 865	60,3	173
juli 2013 tem. juni 2014	2 865	49,8	143
juli 2014 tem. juni 2015	2 865	50,8	146
juli 2015 tem. juni 2016	2 865	**59,8**	**171**

4.9.4 *Conclusies en aanbevelingen*

De FC concentraties in de grachten zijn het hoogst ter hoogte van het 3M terrein (Palingbeek, locaties 12 en 13) en nemen af naarmate de afstand tot het 3M terrein groter wordt (Tophat gracht). 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 Tophat gracht (locatie 5) en het bemalingsstation zijn vergelijkbaar en lijken licht te stijgen.

Aanbevolen wordt om de driemaandelijkse bemonsteringsfrequentie te behouden en de potentiële stijging in de Tophat gracht verder op te volgen.

Gezien de sterk beperkte bijdrage van de component PFOSA aan de somparameter FC, wordt voorgesteld om de analyse op PFOSA te reduceren tot 1 maal per jaar.

De volgende monitoring zal plaats vinden **in oktober 2016**.

4.10 *REGENWATERCOLLECTORPUT EN BEDRIJFSWATERZUIVERING*

Na mondeling akkoord van OVAM is sinds oktober 2013 de driemaandelijkse monitoring van de regenwaterriolering (RW3, RW4, RW12 en RW13) geschrapt en vervangen door de periodieke monitoring van de collectorput. De collectorput is de verzamelput waarin al het water van de regenwaterriolering wordt verzameld voor het, samen met het gezuiverde bedrijfsafvalwater, in de Schelde geloosd wordt. De resultaten van de collectorput zijn minder gevoelig voor fluctuaties veroorzaakt door neerslag en daardoor meer betrouwbaar.

De resultaten van de monitoring van de regenwatercollectorput en de bedrijfswaterzuivering worden hierna besproken.

4.10.1 *Uitgevoerd veldwerk*

Om de invloed van de lopende grondwatersanering op de concentraties in de regenwaterriolering na te gaan, worden door 3M wekelijks stalen genomen van de regenwatercollectorput en geanalyseerd op FC's. Ook van de bedrijfswaterzuivering worden wekelijks stalen genomen en geanalyseerd. Het debiet van de regenwatercollectorput en de bedrijfswaterzuivering wordt dagelijks door 3M opgevolgd.

Daarnaast worden elk kwartaal waterstalen genomen door ERM, die geanalyseerd worden op de FC verbindingen PFOS en PFOA door het laboratorium van SGS.

De locatie van de collectorput en de bedrijfswaterzuivering is aangeduid op Kaart 4G.

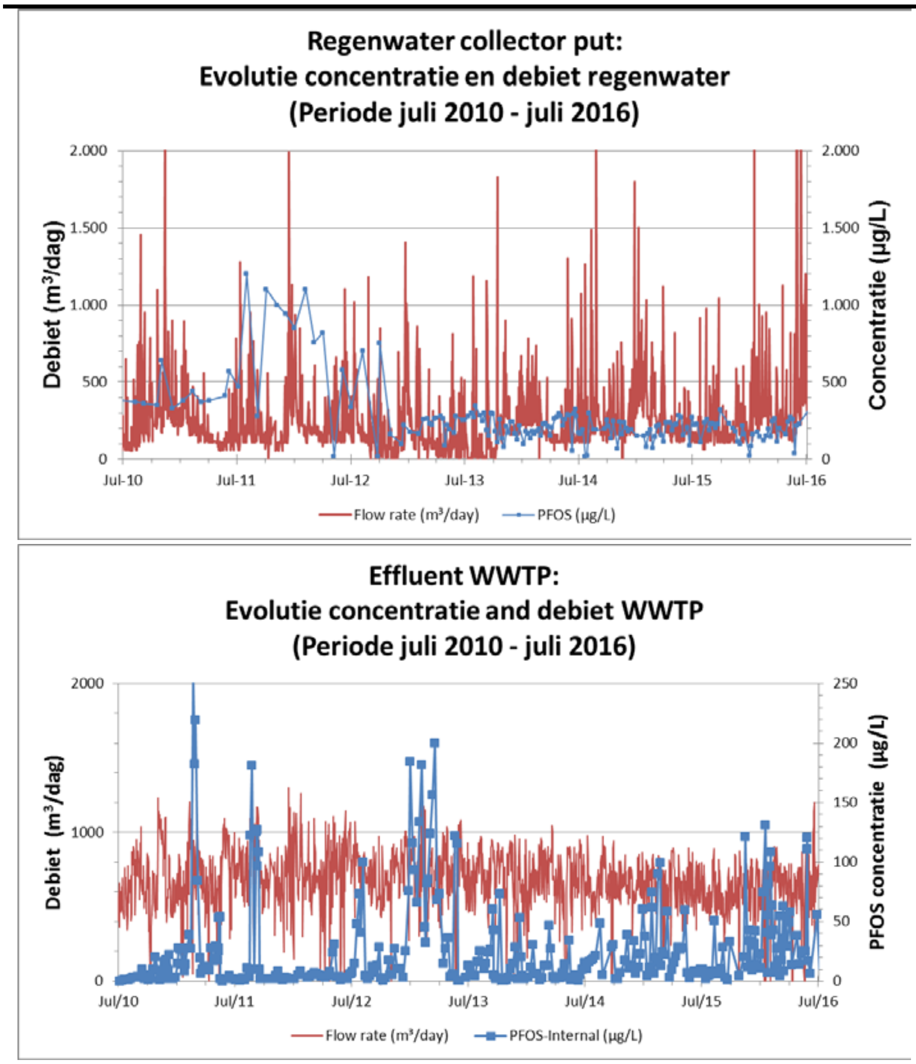
4.10.2 *Bespreking resultaten, conclusies en aanbevelingen*

De analyseverslagen zijn toegevoegd in Bijlage 3. Een toetsingstabel met deze resultaten is opgenomen in Bijlage 4.

4.10.2.1 *Debiet regenwatercollectorput versus PFOS concentratie*

In onderstaande grafiek is de evolutie van concentratie aan PFOS ter hoogte van de regenwatercollectorput weergegeven.

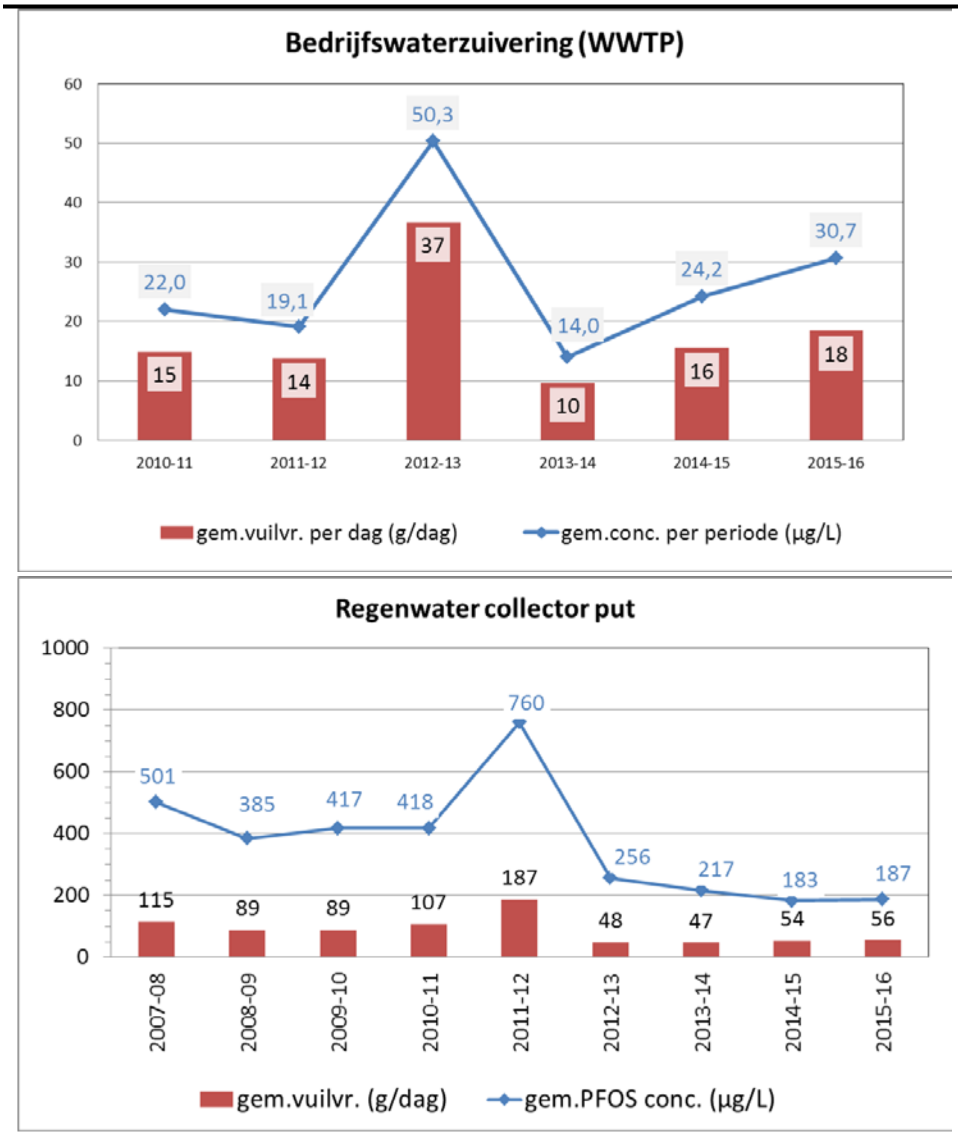
Grafiek 4.11 Evolutie PFOS concentratie in collector put en bedrijfswaterzuivering



Sinds november 2012 vertonen de PFOS concentraties in de collectorput een stabiel verloop. De concentraties kunnen echter fluctueren. De gemiddelde concentratie PFOS in de periode juli 2015 – juni 2016 bedraagt 187 µg/L. De gemiddelde PFOS concentratie in de bedrijfswaterzuivering effluent in dezelfde periode bedraagt 30,7 µg/L.

De jaarlijkse vuilvracht aan PFOS welke via de regenwatercollectorput en de bedrijfswaterzuivering in de Schelde terecht komt is weergegeven in onderstaande tabel. De gemiddelde dagelijkse vuilvracht is berekend uit de waarden bekomen per staalname interval.

Grafiek 4.12 PFOS Vuilvrachtberekening regenwatercollectorput en bedrijfswaterzuivering



Deze gegevens maken deel uit van de PFOS vuilvrachtberekening naar de Schelde (zie paragraaf 4.11). De hogere vuilvracht gemeten in de collectorput in 2011 en 2012 is waarschijnlijk te wijten aan het slib dat in de collectorput aanwezig was. Intussen is dit slib verwijderd, en is de vuilvracht terug gedaald en relatief stabiel.

Aanbevolen wordt om de dagelijkse opvolging van het debiet en de maandelijkse monitoring van de bedrijfswaterzuivering en het hemelwater in de collectorput, maandelijks door 3M en elk kwartaal door ERM/SGS, te behouden.

4.11 PFOS VUILVRACHT RICHTING SCHELDE

De vermoedelijke 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 vastgelegd op 370 g/dag en wordt

gebruikt als toetsingswaarde om de gemiddelde dagelijks PFOS vuilvracht te evalueren.
De gemiddelde dagelijkse PFOS vuilvracht wordt berekend aan de hand van de concentraties gemeten over een periode van één jaar in de regenwatercollectorput, het effluent van de bedrijfswaterzuivering en het pompstation in de Tophatgracht. Deze berekende vuilvrachtwaarde wordt vergeleken met de hierboven vermelde toetsingswaarde (PNEC: 370 g/dag).

Op basis van gegevens bekomen uit de monitoringscampagne van 1 juli 2015 tot en met 30 juni 2016 is de vuilvracht die dagelijks naar de Schelde stroomt berekend (zie
Tabel 4.8).

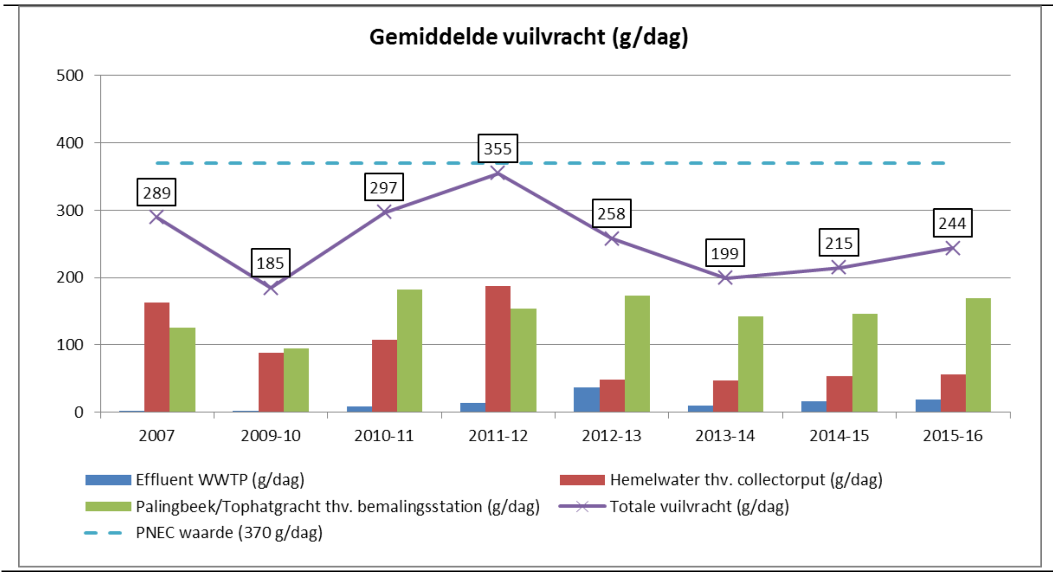
Tabel 4.8 *Gemiddelde PFOS vuilvracht richting de Schelde (juli 2015 tem. juni 2016)*

Influx naar de Schelde	**Gemiddeld debiet (m^3/dag)**	**Gemiddelde PFOS concentratie (µg/L)**	**Gemiddelde vuilvracht (g/dag)**
Effluent van het bedrijfsafvalwater (incl. opgepompt grondwater)	603	35	19
Regenwaterriolering	299	187	56
Palingbeek/Tophatgracht*	2.865	60	169
Totale vuilvracht			**244**

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

In de onderstaande grafiek zijn de gemiddelde berekende PFOS concentraties en vuilvrachten van de voorbije jaren en het huidige jaar samengevat.

Grafiek 4.13 *Evolutie gemiddelde PFOS concentratie en vuilvracht naar de Schelde*



Voor de periode juli 2015 tot en met juni 2016 bedraagt de berekende totale PFOS vuilvracht naar de Schelde 244 g/dag. Dit betekent dat de berekende vuilvracht de PNEC waarde (370 g/dag) niet overschrijdt.

4.12 *VOORSTEL MONITORINGSPROGRAMMA 2016-17*

In de volgende tabel is een overzicht gegeven van het voorgestelde monitoringsprogramma voor de periode juli 2016 – juli 2017.

Tabel 4.9 Monitoringsprogramma 2017

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
P&T systeem	9	Driemaandelijks	PP01, PP02,PP04, PP05, PP06, PP07, PP08, PP09, PP10	-	Minstens maandelijkse inspectie van de installatie en de onttrekkingsputten
Stabiliteit	-	Eenmaal (stopzetting indien akkoord stabiliteitsdeskundige)	-	-	Geen onaanvaardbare zettingen en een technische evaluatie door onafhankelijke stabiliteitsdeskundige geeft aan dat er geen verder noodzaak is voor monitoring. Te bevestigen door eenmalige controle.
Grondhoop	-	Volgende inspectie 2018	Visuele inspectie van grondhoop	Inspectie in 2016 uitgevoerd: bovenafdek is OK	Vanaf 2013 vijfjaarlijkse monitoring, aangezien bovenafdek in 2012 vervangen is; eerst volgende controle: 2018
FC Bronzones					
Gebouw 16	2	Jaarlijks	K3, P56	-	PFBS zal worden toegevoegd aan het analysepakket om de concentraties verder op te volgen
	4	Halfjaarlijks	P27, P42, P304, P305	-	P27 ter vervanging van K1 omdat de concentraties in peilbuis K1 sinds de start van de monitoring in 2008 stabiel en laag liggen
	1	Driemaandelijks	P21B		Stijgende concentratietrend ter hoogte van P21B waargenomen.
WWTP	6	Jaarlijks	P262, P340, P341, P343, P379, P380	-	-
	10	Halfjaarlijks	P118C, P264, P265C, P371, P374, P381, P382, L19, M4, P119C	-	P119C van Blokkersdijk monitoring naar bronzone monitoring gezien de locatie (stroomafwaarts van 3M pad, maar stroomopwaarts van bronzone)
	1	Driemaandelijks	P263		-
Tweede aquifer	6	Jaarlijks	BD24-4, D11, D14, P118B, D10, P118A	-	

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
	1	Jaarlijks	D2	Geen indicatie voor stijgende concentratietrend	D2 toont voorlopig geen stijgende concentratietrend FC's. Peilbuis enkel jaarlijks bemonsterd.
	3	Halfjaarlijks	D9, BD24-3, P121	-	Indien de stijgende trend niet wordt bevestigd bij twee opeenvolgende monitoringsrondes, zal de monitoringsfrequentie terug worden verlaagd naar jaarlijks
	7	Halfjaarlijks	D16, D17, D18, D5, ND7, P119A en P119B	Bijkomende staalnames om potentieel stijgende concentratietrend voor FC flux naar 2de aquifer beter te begrijpen.	Peilbuizen zijn in juli 2016 bemonsterd geweest. Bijkomende monitoring nodig om concentratietrend te begrijpen.
	1	Driemaandelijks	P321	-	-
Zuidelijke terreingrens	7	Halfjaarlijks	B3-bis, B7, P372, P378, PA109A, PA111A, PA112	-	Peilbuis B3 is beschadigd en vervangen door peilbuis B3-bis in september 2015.
Ondergrondse tanken					
Minerale olie (MO)	2	Jaarlijks	P18, P28	Monitoring op vraag van OVAM	Brononderzoek lopende
Blokkersdijk Natuurgebied					
Peilbuizen	7	Driemaandelijks	L21, L22, P115, L31, L4, P114bis, P116	-	-
Oppervlaktewater	2	Driemaandelijks	Blokkersdijk standaard, 3M vijver		
	1	Driemaandelijks	Blokkersdijk oost	Bemonsteringslocatie opnieuw bereikbaar	Voorkeur om Blokkersdijk op 2 locaties te bemonsteren langs 2 zijden. Gezien de omvang van de vijver is dit meer representatief.
Palingbeek en Tophat gracht	4	Driemaandelijks	12, 13, 5, Bemalingsstation	-	-
Regenwater	1	Maandelijks	Collector put	Maandelijkse stalen geanalyseerd door 3M labo te Zwijndrecht	Driemaandelijkse verificatie van de stalen door SGS.

Locatie	**Aantal peilbuizen**	**Frequentie**	**Peilbuis ID**	**Verantwoording**	**Opmerkingen**
Effluent WWTP	1	Driemaandelijks	Lozingspijp	Maandelijkse stalen geanalyseerd door 3M labo te Zwijndrecht	Driemaandelijkse verificatie van de stalen door SGS.

Legende

🟧 Verhoogde monitoringsfrequentie

🟩 Verlaagde monitoringsfrequentie

🟦 Geen wijzigingen

5. *GRONDWATERONTTREKKING BRONZONE*

5.1 *OPVOLGING ONTTREKKINGSSYSTEEM*

De hieronder besproken resultaten hebben betrekking op de periode van juli 2015 tot juli 2016.

5.1.1 *Beschrijving onttrekkingssysteem*

In 2010 is het P&T systeem in de bronzones opgestart met de inwerking stelling van één pompput in elke bronzone. Eind 2010 zijn in totaal vijf bijkomende pompputten geïnstalleerd. Het P&T systeem was volledig operationeel in augustus 2011. Begin 2012 zijn twee bijkomende pompputten geïnstalleerd in de WWTP bronzone. Deze pompputten zijn sinds mei 2012 operationeel.

In totaal zijn er negen pompputten in bedrijf (voor locaties zie Kaart 5). Er zijn drie pompputten in de "zone gebouw 016" en zes pompputten in de "zone WWTP". In de regel is het filterend gedeelte van de pompputten geplaatst tot net boven de polderklei, behalve bij pompputten PP09 en PP10, die in een zone staan waar geen polderklei is aangetroffen. De polderklei op de site bevindt zich op een variabele diepte, maar algemeen geldt: hoe dichter bij de Schelde, hoe dieper de polderklei ligt ten opzichte van het maaiveld. De pompputten hebben bijgevolg allemaal een verschillende diepte t.o.v. het maaiveld. In onderstaande tabel is een overzicht gegeven.

Tabel 5.1 Technisch overzicht pompputten

Pompput	Locatie	Diepte (m-mv)	Gemiddeld jaardebiet (l/min)
PP01	Gebouw 16	2,0-5,5	3,26
PP04	Gebouw 16	2,0-6,0	0,63
PP05	Gebouw 16	1,7-5,7	0,49
PP02	WWTP	3,2-6,2	0,76
PP06	WWTP	1,3-5,3	1,60
PP07	WWTP	2,0-6,0	5,63
PP08	WWTP	3,2-7,2	5,35
PP09	WWTP	3,0-6,0	1,94
PP10	WWTP	3,0-6,0	7,57

* *Geen constant debiet door softwareproblemen*

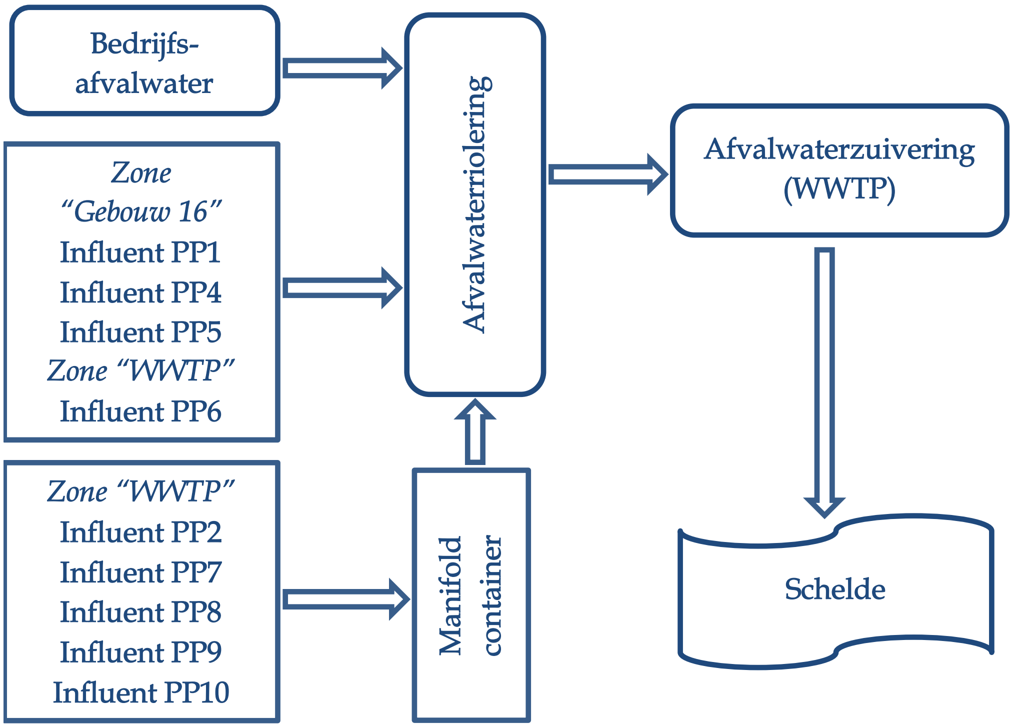
Elke pompput beschikt over een niveausensor (zie schematisch overzicht in Bijlage 6). De niveausensor is net boven de inlaat van de pomp gehangen. De inlaat van de pomp mag nooit droog komen, daarom wordt steeds een minimale waterkolom van 20 cm aangehouden. De onttrekking in de betreffende pompput wordt stilgelegd als het niveau in de pompput onder dit laagst toelaatbare niveau komt te liggen. Van zodra het water terug voldoende gestegen is, start de onttrekking automatisch terug op. In onderstaande tabel is een overzicht gegeven van de huidige ingestelde schakelniveaus. In de pompputten die veel water geven, wordt het niveau waarop de pompen terug aangeschakeld worden iets hoger ingesteld. In onderstaande tabel worden deze schakelniveaus weergegeven.

Tabel 5.2 *Schakelniveaus pompputten*

Naam pompput	Laagste Schakelniveau (m + onderkant filter)	Hoogste schakelniveau (m+ onderkant filter)
PP01	0,2	1,0
PP02	0,2	0,8
PP04	0,2	0,8
PP05	0,2	0,8
PP06	0,2	1,0
PP07	0,2	1,2
PP08	0,2	1,2
PP09	0,2	0,8
PP10	0,2	1,2

De pompputten geplaatst in de zone van gebouw 016 zijn rechtstreeks aangesloten op de chemische riolering van 3M. In de zone WWTP zijn de pompputten, met uitzondering van PP06, eerst aangesloten op een manifold container om daarna te lozen in de chemische riolering. De chemische riolering komt terecht in een bufferbekken, waarna het water behandeld wordt in de waterzuivering van 3M. In onderstaande schema is weergegeven hoe de betreffende putten aangesloten zijn op de chemische riolering van 3M.

Figuur 5.1 *Schematische weergave aansluiting van P&T putten op chemische riolering*



Naast de niveausensoren, beschikt elke pompput over een debietmeter en een drukmeter. Alle instrumentatie wordt ingemeten via het centrale systeem van

3M. Op termijn zullen de geregistreerde data automatisch verzonden worden naar de consultant.

5.1.2 *Opvolging sanering*

Er worden verschillende aspecten van het onttrekkingssysteem opgevolgd:

- De onttrokken debieten;
- De concentraties in het onttrokken grondwater; en
- De zettingsmetingen.

5.1.2.1 *Opvolging onttrokken debieten*

Op regelmatige tijdstippen gaat ERM ter plaatse om de werking van de installatie te controleren en om meterstanden te noteren. Tijdens deze bezoeken worden ook grondwaterstanden genoteerd in omliggende peilbuizen (zie paragraaf 5.2). In de periode van 1 juli 2015 tot 1 juli 2016 heeft ERM bezoeken uitgevoerd op de data vermeld in onderstaande tabel.
ERM komt ook steeds ter plaatse tijdens of kort na interventies door de aannemer, deze data zijn eveneens mee opgenomen in onderstaande tabel.

Tabel 5.3 Data controlebezoeken

Controlebezoeken ERM	Controlebezoeken ERM	Controlebezoeken ERM
02-07-15	18-11-15	18-03-16
08-07-15	24-11-15	22-03-16
22-07-15	27-11-15	01-04-16
30-07-15	11-12-15	07-04-16
05-08-15	16-12-15	15-04-16
18-08-15	22-12-15	19-04-16
25-08-15	28-12-15	26-04-16
02-09-15	08-01-16	06-05-16
08-09-15	13-01-16	13-05-16
15-09-15	21-01-16	20-05-16
22-09-15	25-01-16	26-05-16
01-10-15	05-02-16	03-06-16
05-10-15	12-02-16	08-06-16
15-10-15	16-02-16	17-06-16
22-10-15	23-02-16	24-06-16
30-10-15	26-02-16	29-06-16
03-11-15	02-03-16	
10-11-15	11-03-16	

5.1.2.2 *Opvolging concentraties in het onttrokken grondwater*

Het onttrokken grondwater in de pompputten wordt driemaandelijks bemonsterd. In de periode waarop dit tussentijds rapport betrekking heeft, zijn de pompputten bemonsterd op data vermeld in onderstaande tabel.

Tabel 5.4 Data bemonstering pompputten

Datum	Bemonsterde pompputten
22/10/2015	PP01, PP02, PP05, PP06, PP07, PP08, PP09 & PP10
16/01/2016	PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 & PP10
5/04/2016	PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 & PP10
26/07/2016	PP01, PP02, PP04, PP05, PP08, PP09 & PP10 (*)

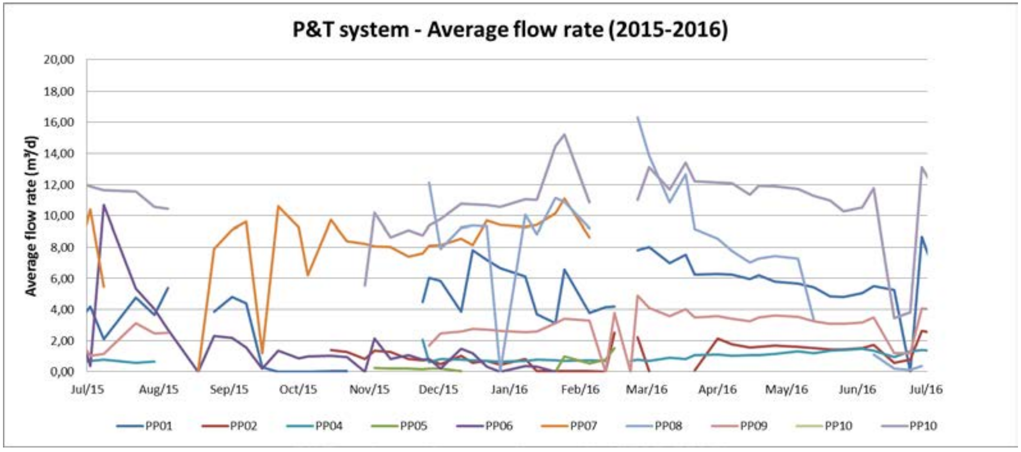
(*) de stalen van pompputten PP06 en PP07 zijn verloren gegaan en de pompputten zijn op 16/09/2016 herbemonsterd

5.1.3 Resultaten

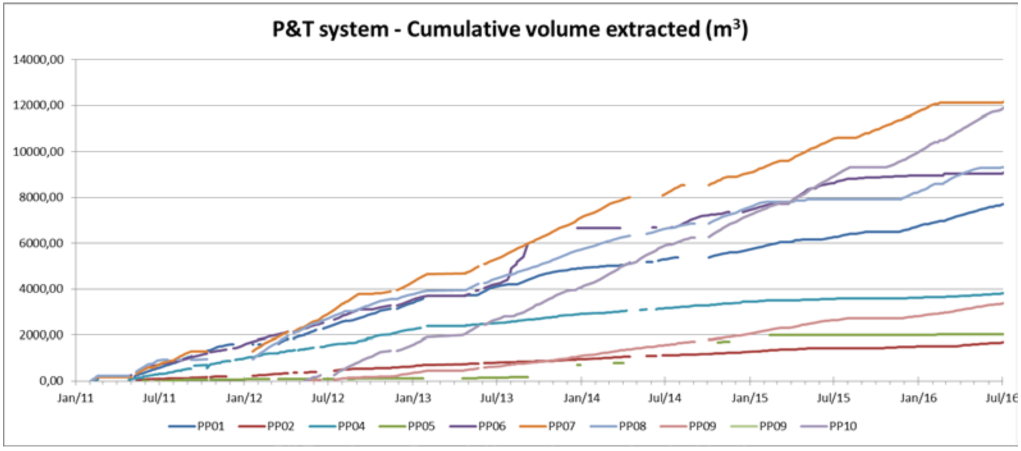
5.1.3.1 *De onttrokken debieten*

In onderstaande grafieken wordt getoond wat de gemiddelde en cumulatieve verpompte debieten zijn in de periode van 1 juli 2015 tot 1 juli 2016.

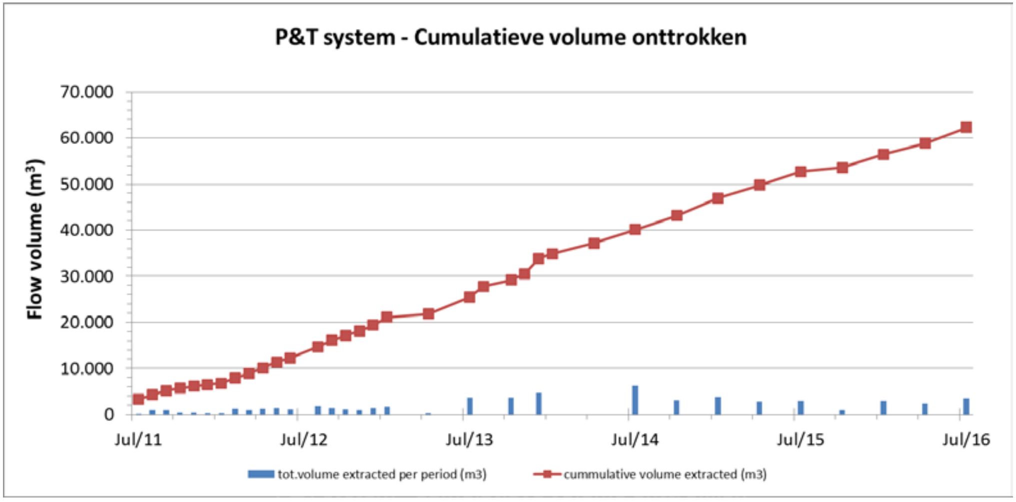
Grafiek 5.1 Gemiddeld debiet per onttrekkingsput (juli 2015 tot juli 2016)



Grafiek 5.2 Cumulatief debiet per onttrekkingsput



Grafiek 5.3 Cumulatief debiet van het totaal saneringssysteem



Uit hoger getoonde grafieken kunnen volgende zaken worden afgeleid:

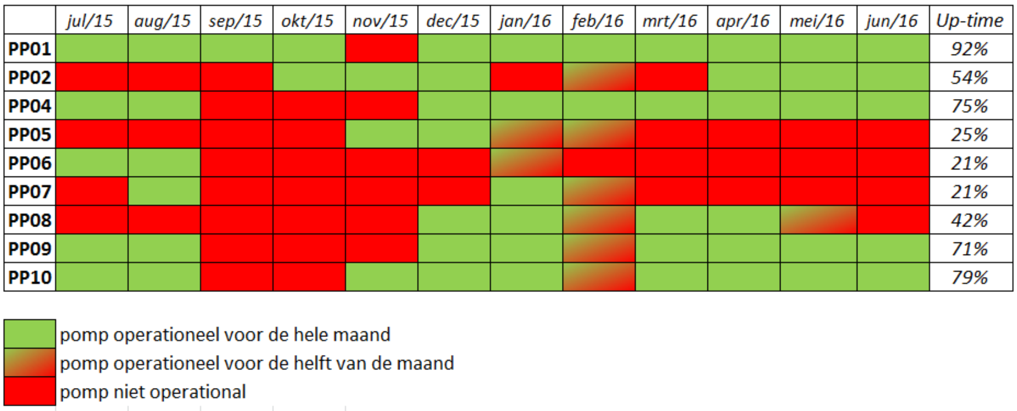
- Het onttrokken debiet per put (uitgezonderd PP05 die intermitterend werkt) verschilt aanzienlijk en varieert tussen minimaal 0,03 m³/dag (PP02) en maximaal 16,3 m³/dag (PP10);

- De onttrekking in PP05 (debiet < 1 m³/dag) werkt niet goed om verschillende redenen:
  - De toestroming in de pompput is zeer gering; en
  - De sturing op de pomp moet geregeld gereset worden (softwareprobleem).

- Verder zijn nog een aantal kleinere onderbrekingen geweest van de installatie door technische storingen. De volgende storingen komen frequent voor:
  - Pompwaaiers en debietmeters verstopt of afgeschuurd, hetzij door fijn zand, hetzij door ijzerslib; en
  - Afgaan van de thermische beveiliging van de pomp.

  Op basis van de frequente inspecties is geraamd dat de installatie een up-time heeft van gemiddeld circa 53% (14-15: 73%; 13-14: 71%). Echter, zoals uit onderstaande Tabel 5.5 blijkt is er een groot verschil tussen de werking van de verschillende pompen. Pompput 6 en 7 hebben slechts een up-time van 21%, terwijl pompput PP01 een up-time heeft van 92%.

Tabel 5.5 Werking pompputten



	jul/15	aug/15	sep/15	okt/15	nov/15	dec/15	jan/16	feb/16	mrt/16	apr/16	mei/16	jun/16	Up-time
PP01	🟩	🟩	🟩	🟩	🟥	🟩	🟩	🟩	🟩	🟩	🟩	🟩	*92%*
PP02	🟥	🟥	🟥	🟩	🟩	🟩	🟥	▨	🟥	🟩	🟩	🟩	*54%*
PP04	🟩	🟩	🟥	🟥	🟥	🟩	🟩	🟩	🟩	🟩	🟩	🟩	*75%*
PP05	🟥	🟥	🟥	🟥	🟩	🟩	▨	▨	🟥	🟥	🟥	🟥	*25%*
PP06	🟩	🟩	🟥	🟥	🟥	🟥	▨	🟥	🟥	🟥	🟥	🟥	*21%*
PP07	🟥	🟩	🟥	🟥	🟥	🟥	🟩	▨	🟥	🟥	🟥	🟥	*21%*
PP08	🟥	🟥	🟥	🟥	🟥	🟩	🟩	▨	🟩	🟩	▨	🟥	*42%*
PP09	🟩	🟩	🟥	🟥	🟥	🟩	🟩	▨	🟩	🟩	🟩	🟩	*71%*
PP10	🟩	🟩	🟥	🟥	🟩	🟩	🟩	▨	🟩	🟩	🟩	🟩	*79%*

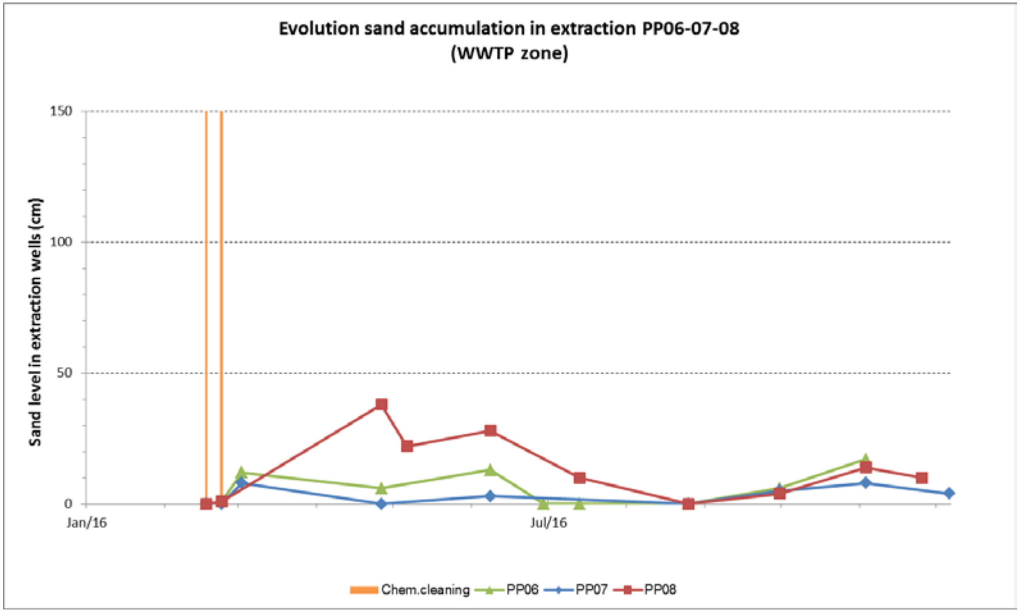
🟩	pomp operationeel voor de hele maand
▨	pomp operationeel voor de helft van de maand
🟥	pomp niet operational

- Tussen juli 2015 en juli 2016 is ongeveer 9.639 m³ verontreinigd grondwater onttrokken (tegen circa 12.535 m3 (2014-15) en 14.625 m³ (2013-14)). In totaal is sinds juli 2011 circa 62.316 m³ grondwater onttrokken.

Om de saneringsinstallatie maximaal draaiende te houden, zijn volgende acties genomen:

- Het niveau van fijn zand in de onttrekkingsfilters wordt regelmatig opgevolgd en indien nodig wordt het fijn zand verwijderd met behulp van een slangenpomp of ingebouwde boosterpompjes. Pompputten PP06, PP07 en PP08 blijken het meest gevoelig te zijn aan zandophoping. In volgende grafiek is de evolutie van de zandniveaus in de hoger vermelde putten weergegeven.

Grafiek 5.4 Evolutie zandaccumulatie in onttrekkingsputten PP06-07-08



- Bij technische defecten aan de pomp wordt de aannemer gecontacteerd om de pomp te reviseren.

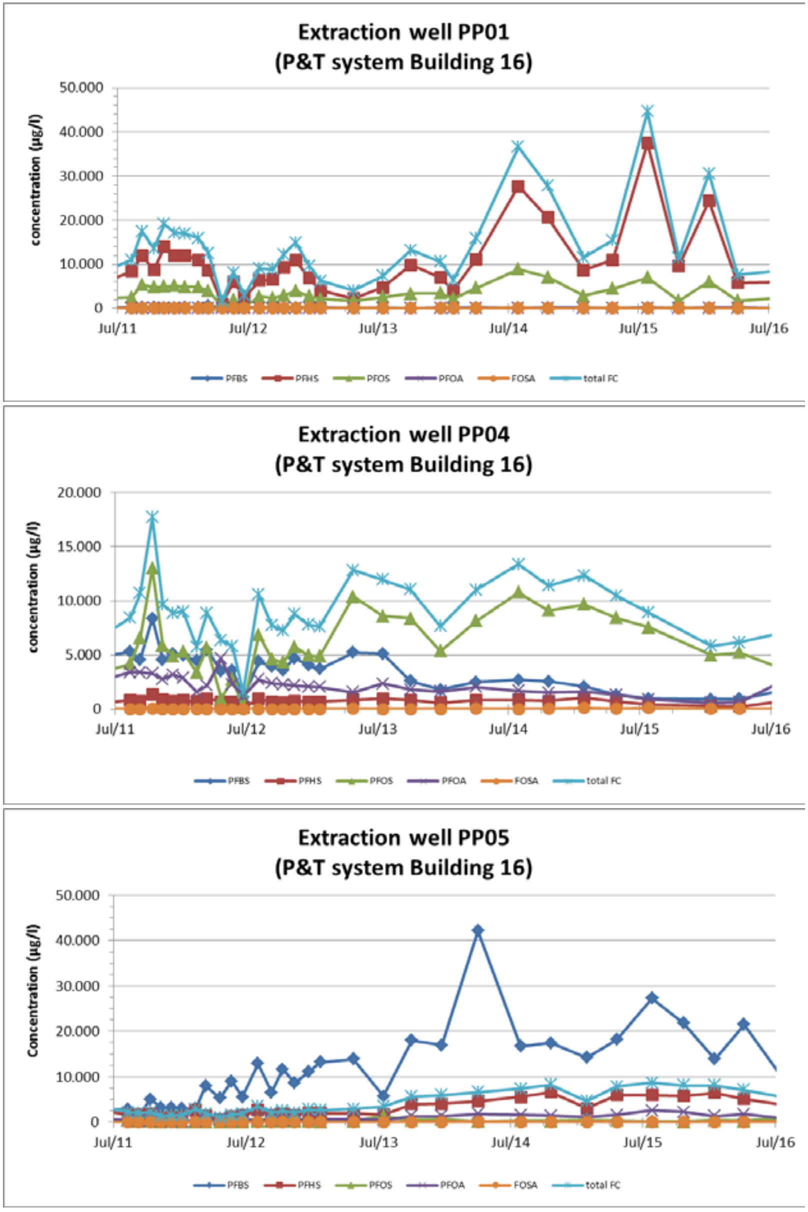
- Bij elektrische problemen of software problemen wordt de technische dienst van 3M gecontacteerd en gevraagd om de betreffende problemen op te lossen. Bij 3M is een vaste contactpersoon/technieker aangesteld om eventuele problemen op te lossen.

- Indien blijkt uit de data-opvolging dat het rendement van een bepaalde onttrekkingsput daalt zonder een aantoonbare technische reden, wordt er van uit gegaan dat het filterpakket verstopt begint te raken met ijzerslib. Er wordt dan over gegaan tot een chemische reiniging van de onttrekkingsput. 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. In augustus 2015 en februari 2016 werd een chemische reiniging uitgevoerd.

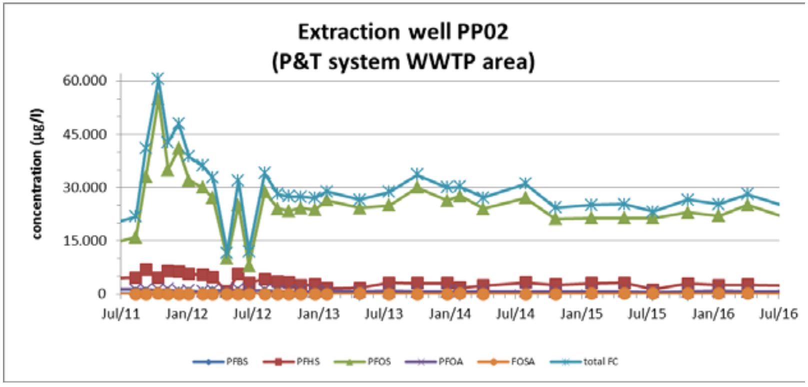
5.1.3.2 De concentraties in het onttrokken grondwater

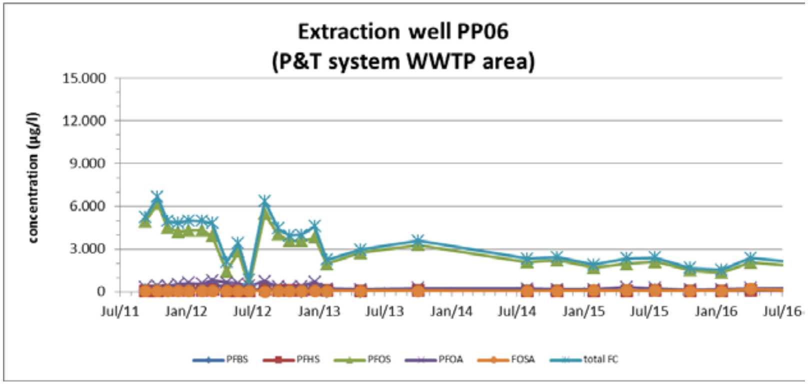
In onderstaande grafieken is per pompput weergegeven wat de verpompte concentraties zijn. In de laatste grafiek is de som van FC’s (PFOS, PFOA en PFHS) weergegeven per pompput.

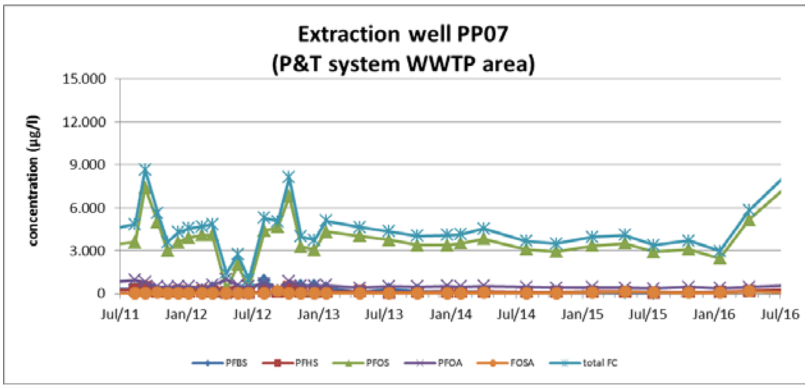
Grafiek 5.5 Evolutie FC concentraties t.h.v. gebouw 016 (pompput PP01, PP04 en PP05)

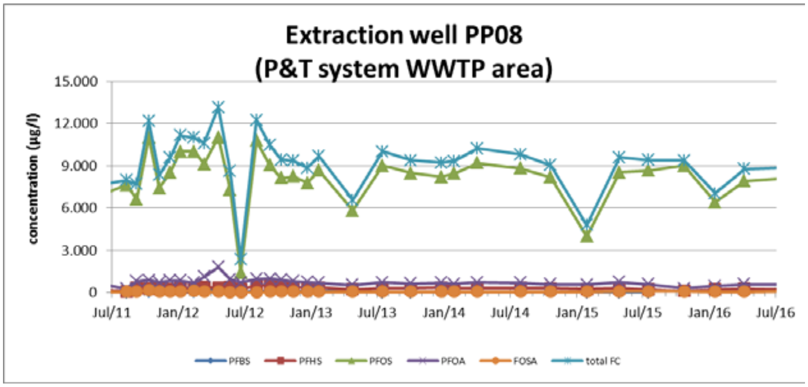


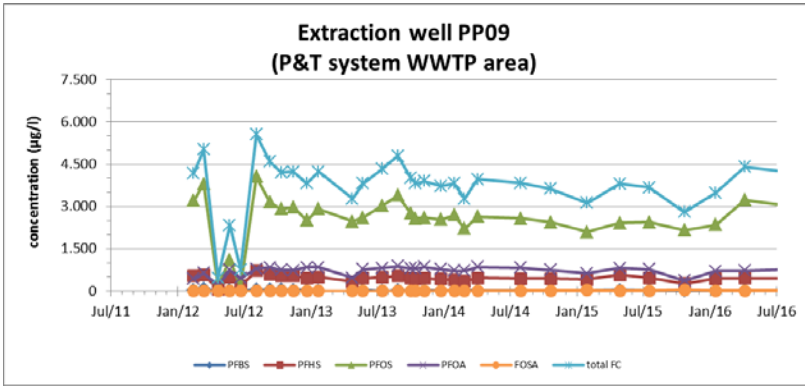
Grafiek 5.6 Evolutie FC concentraties t.h.v. WWTP zone (pompput PP02, PP06, PP07, PP08, PP09 en PP10)

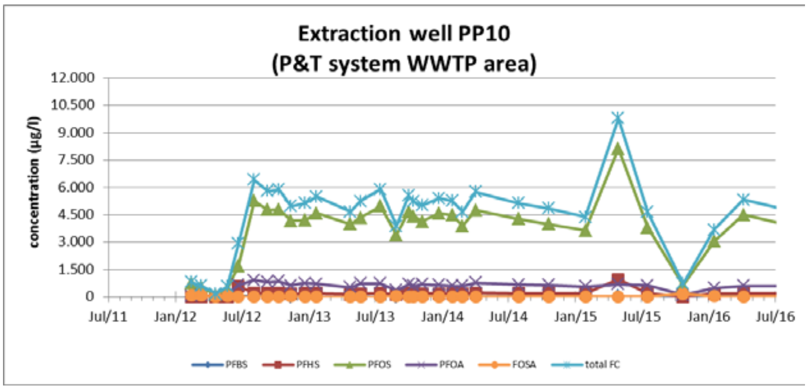



Extraction well PP06
(P&T system WWTP area)
concentration (µg/l)
15.000
12.000
9.000
6.000
3.000
0
Jul/11
Jan/12
Jul/12
Jan/13
Jul/13
Jan/14
Jul/14
Jan/15
Jul/15
Jan/16
Jul/16
PFBS
PFHS
PFOS
PFOA
FOSA
total FC

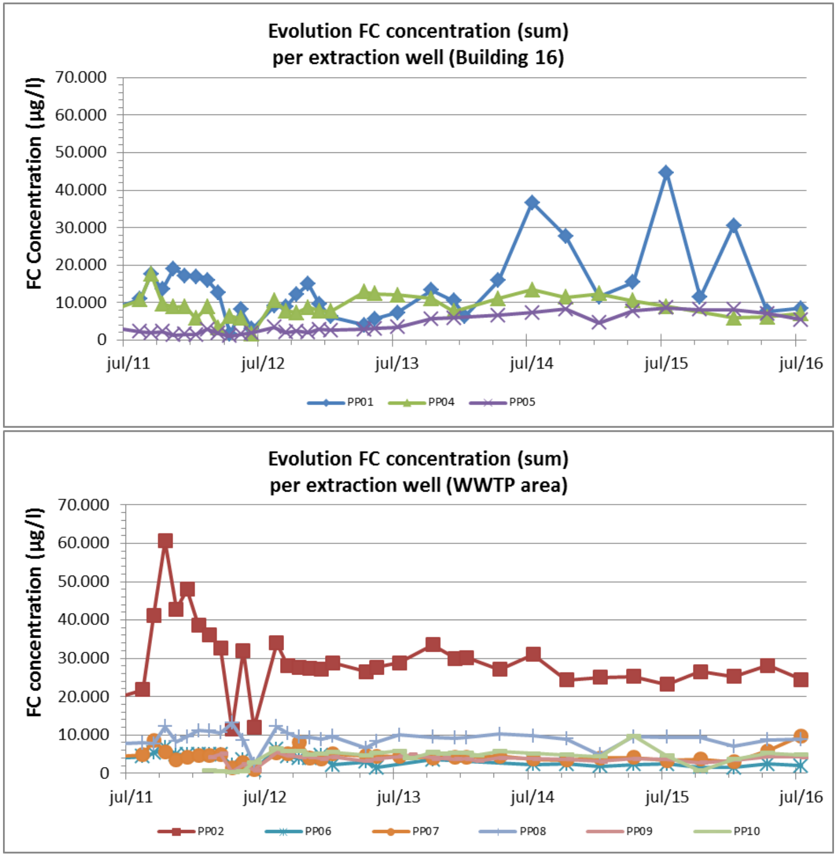

Extraction well PP07
(P&T system WWTP area)
concentration (µg/l)
15.000
12.000
9.000
6.000
3.000
0
Jul/11
Jan/12
Jul/12
Jan/13
Jul/13
Jan/14
Jul/14
Jan/15
Jul/15
Jan/16
Jul/16
PFBS
PFHS
PFOS
PFOA
FOSA
total FC


Extraction well PP08
(P&T system WWTP area)
concentration (µg/l)
15.000
12.000
9.000
6.000
3.000
0
Jul/11
Jan/12
Jul/12
Jan/13
Jul/13
Jan/14
Jul/14
Jan/15
Jul/15
Jan/16
Jul/16
PFBS
PFHS
PFOS
PFOA
FOSA
total FC

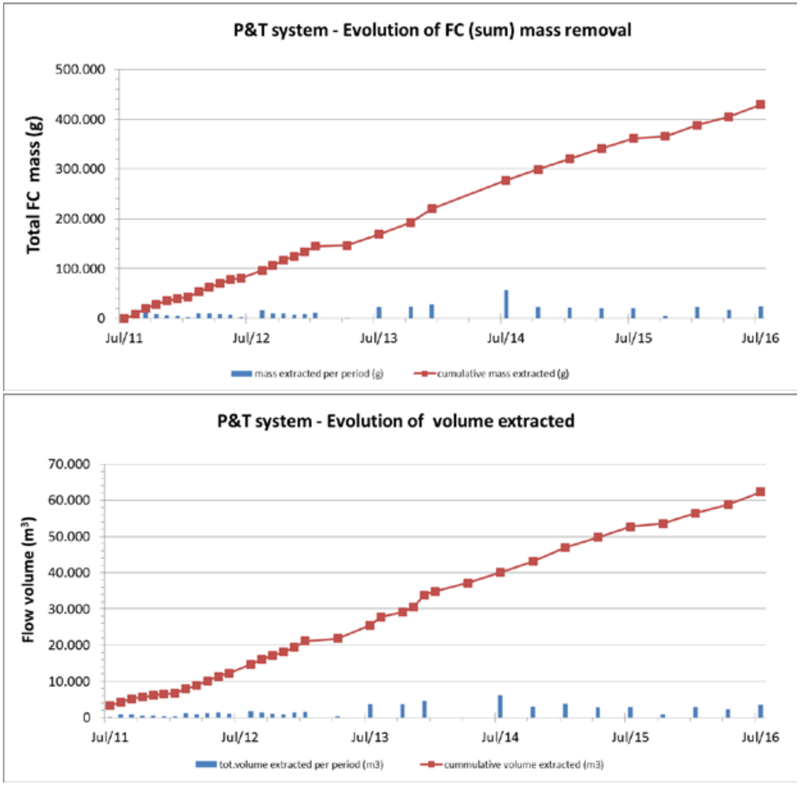

Extraction well PP09
(P&T system WWTP area)
concentration (µg/l)
7.500
6.000
4.500
3.000
1.500
0
Jul/11
Jan/12
Jul/12
Jan/13
Jul/13
Jan/14
Jul/14
Jan/15
Jul/15
Jan/16
Jul/16
PFBS
PFHS
PFOS
PFOA
FOSA
total FC


Extraction well PP10
(P&T system WWTP area)
concentration (µg/l)
12.000
10.500
9.000
7.500
6.000
4.500
3.000
1.500
0
Jul/11
Jan/12
Jul/12
Jan/13
Jul/13
Jan/14
Jul/14
Jan/15
Jul/15
Jan/16
Jul/16
PFBS
PFHS
PFOS
PFOA
FOSA
total FC

Grafiek 5.7 *Overzicht evolutie som FC concentraties (PFOS, PFOA en PFHS) t.h.v. gebouw 016 en WWTP zone*



Grafiek 5.8 *Cumulatieve vrachtverwijdering per onttrekkingsput en van het totaal saneringssysteem*



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 016 (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 PP01 en PP02. Ter hoogte van PP01 fluctueert de totale FC concentratie. De oorzaak hiervan is tot op heden onbekend. De totale FC concentratie is stabiel ter hoogte van PP02.

- De opgepompte concentraties aan som FC's (PFOS, PFOA en PFHS) fluctueren, maar in de meeste pompputten zijn de concentratietrends min of meer stabiel:
  - De gemeten concentraties aan som FC's (PFOS, PFOA en PFHS) in PP06, PP07, PP09 en PP10 liggen doorgaans rond 5.000 µg/L.
  - Voor PP04 en PP08 ligt de gemeten concentratie aan som FC's rond 10.000 µg/L.
  - In pompput PP02 ligt de som FC concentratie rond de 30.000 µg/L.
  - Enkel in PP05 en PP07 is een stijging van de concentratie gemeten. In beide pompputten bedraagt de concentratie in juli 2016 circa 10.000 µg/L.

- Tussen juli 2015 en juni 2016 is ongeveer 67,9 kg FC's verwijderd. In totaal is sinds juli 2011 circa 429,5 kg FC verwijderd.

5.1.3.3 *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 (8)). Belangrijke randvoorwaarde voor stopzetting is dat de opgepompte debieten in de toekomst, nooit de tot op heden opgepompte debieten mogen overschrijden.

5.1.4 Besluit

In totaal is in de periode juli 2015 tot juli 2016 ongeveer 9.639 m^3 verontreinigd grondwater onttrokken en circa 67,9 kg FC verwijderd. Uit de analyses blijkt dat in de meeste pompputten PFOS de belangrijkste component is. Uitzonderingen hierop zijn pompput PP01, waar PFHS de hoogste gemeten concentratie is en PP05 waar PFBS de belangrijkste FC parameter is. Over het algemeen zijn de concentraties in de pompputten stabiel wat erop duidt dat de verontreiniging globaal gezien gecapteerd wordt door het P&T systeem. Echter, in enkele pompputten worden stijgende concentraties gemeten wat erop kan duiden dat de captatie lokaal niet volledig is. Een mogelijke uitbreiding van het saneringssysteem om de captatie te optimaliseren moet worden geëvalueerd.

Tijdens de meetcampagne in juli 2015 en januari 2016 is ter hoogte van pompput PP01 een sterk verhoogde concentratie aan PFHS gemeten. Deze verhoogde concentratie dient verder te worden opgevolgd maar wordt niet bevestigd tijdens de meest recente meetcampagnes in april en juli 2016.

Het saneringssysteem is storingsgevoelig wat resulteert in een daling van de 'up-time'. Om de onderbrekingen tot een minimum te beperken is er een regelmatige opvolging nodig van:

- Het fijn zand dat zich opstapelt op de bodem van de onttrekkingsfilters;
- De ijzeraanslag in de pomp en het leidingwerk; en
- De goede werking van het leidingwerk, met bijzondere aandacht voor de terugslagkleppen en debietsmeters.

Op basis van de vaststellingen tijdens de controlebezoeken, dient regelmatig een campagne georganiseerd te worden om:

- Het fijn zand uit de onttrekkingsfilters te verwijderen; en/of
- Een (chemische) regeneratie van de pompputten te realiseren om het ijzerslib (en biologisch slib) te verwijderen; en
- De pompen en leidingwerk te onderhouden.

Uit een kwantitatieve evaluatie van de velddata blijkt dat de up-time van het saneringssysteem gemiddeld 53% bedraagt in de periode juli 2015-juni 2016, maar dat er een groot verschil is tussen de putten onderling: min. 21% in PP06 en PP07 en max. 92% in PP01. De up-time ligt behoorlijk lager dan in de voorbije jaren (14-15: 73%; 13-14: 71%). Bij de evaluatie van de potentiële optimalisatie van het systeem moeten worden nagegaan hoe de werking van het systeem verbeterd kan worden.

In overeenstemming met de bepalingen in het BSP beveelt ERM aan dat de regelmatige inspecties van het onttrekkingssysteem en staalname van de pompputten verdergezet worden. De inspecties dienen minimum op maandelijkse basis te worden uitgevoerd. De staalname frequentie van de pompputten (met analyse van FC-concentraties) dient behouden te blijven op driemaandelijks. Verder wordt aanbevolen te evalueren of de captatie en de werking van het saneringssysteem kunnen worden verbeterd door een systeemoptimalisatie.

5.2 *MONITORING GRONDWATERSTANDEN*

Om de seizoenale fluctuaties van het grondwaterpeil en de invloed van de pompputten beter te kunnen opvolgen, is opgenomen in TTV5 (8) dat vanaf 2015 elk kwartaal een dipronde zal worden uitgevoerd samen met de bemonsteringscampagnes voorzien in januari, april, juli en oktober.

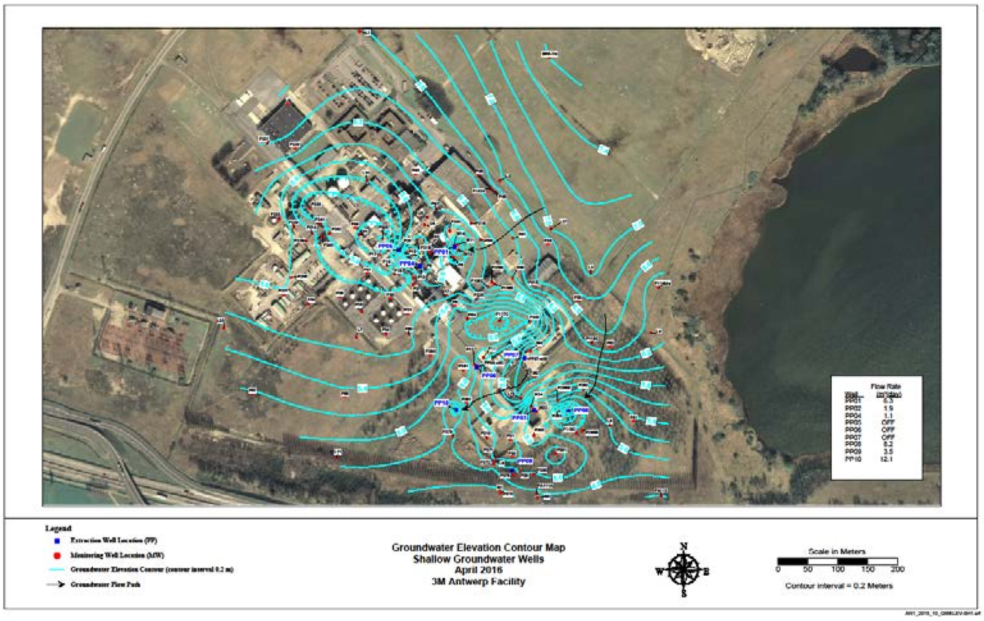
In de periode besproken in voorliggend rapport zijn dan ook de volgende diprondes uitgevoerd op:

- 17 juli 2015;
- 19 oktober 2015;
- 11 januari 2016; en
- 4 april 2016.

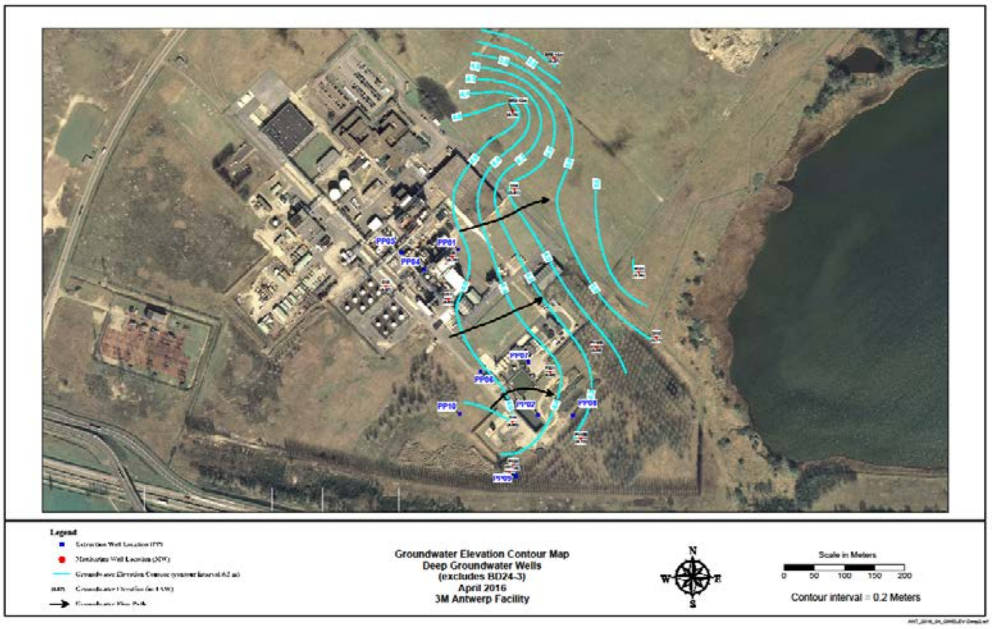
Op 4 april 2016 is een algemene dipronde uitgevoerd waarbij de grondwaterstand in een groot gedeelte van de peilbuizen op en in de onmiddellijke nabijheid van de site is opgemeten. De peilbuizen hebben een filter in de eerste freatische aquifer of de tweede aquifer.
In juli 2015, oktober 2015 en januari 2016 is een beperkte ronde uitgevoerd waarbij enkel peilbuizen in de eerste freatische aquifer zijn opgemeten.

De grondwatercontouren die opgemaakt werden op basis van de meetresultaten van de meest recente algemene diprondes (april 2016) zijn weergegeven in Figuur 5.2.

Figuur 5.2 Grondwatercontouren april 2016



Grondwatercontour eerste freatische aquifer april 2016



Grondwatercontour tweede aquifer april 2016

In de eerste aquifer is een duidelijke invloedszone van de pompputten op te merken, deze komt min of meer overeen met de voorspelde toestand van het voor de sanering opgestelde grondwatermodel (zie TTV4 (6)).

De grondwaterstanden zullen volgende periode verder worden opgevolgd met een ongewijzigde frequentie van vier maal per jaar.

5.3 *VOORZIENE WERKEN VOOR VOLGEND JAAR*

5.3.1 *Grondwateronttrekking*

De grondwateronttrekking zal in het volgende jaar verder opgevolgd worden. Volgende zaken zullen hierbij uitgevoerd worden:

- Regelmatige controles en opvolging die de goede werking van de installatie garanderen;

- Optimaliseren van de digitale data-transfer tussen de 3M systemen en ERM:
  - Instantaan debiet;
  - Cumulatief debiet;
  - Waterkolom boven de pomp;
  - Druk op de afvoerleiding; en
  - Up-time saneringssysteem.

- Regeneratie van de onttrekkingsfilters volgens een optimale frequentie:
  - Chemische regeneratie; en
  - Verwijderen van fijn inert materiaal.

- Opvolgen van de opgepompte concentraties in het grondwater, zodat een vuilvracht bepaald kan worden. Gezien de sterk beperkte bijdrage van de component PFOSA aan de somparameter FC, wordt voorgesteld om de analyse op PFOSA te reduceren tot 1 maal per jaar.

- Opvolgen van de grondwaterstanden om de invloed van het onttrekkingssysteem op het natuurlijke grondwaterniveau en de grondwaterstroming te bepalen.

- Evaluatie van de werking van het huidige P&T systeem om de captatie van de verontreiniging te optimaliseren. Bij deze evaluatie moet ook worden nagegaan hoe de werking van het systeem verbeterd kan worden (minder storingen en hogere up-time).

6. *VOORZIENE WERKEN IN VOLGENDE JAAR VAN DE SANERING*

6.1 *GRONDWATERSANERING*

Het P&T systeem zal minimaal maandelijks worden geïnspecteerd (geen wijziging).

Er wordt voorgesteld om de driemaandelijkse monitoring van het opgepompte water te behouden voor alle pompputten.

Voor een verdere gedetailleerde beschrijving wordt verwezen naar paragraaf 5.3.1.

6.2 *MONITORING GRONDWATER EN OPPERVLAKTEWATER*

In Tabel 4.9 is per zone 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 en -contouren te bepalen en de mogelijke evolutie ervan op te volgen.

6.3 *PLANNING*

De planning van de werken voor het volgende jaar is hieronder samengevat weergegeven.

- Afronden van de evaluatie over de noodzaak van een optimalisatie van het saneringssysteem;
- Voortzetten van een grondwateronttrekking in de twee bronzones;
- Monitoring grondwaterconcentraties conform frequentie vermeld in Tabel 4.9;
- Driemaandelijkse opvolging van de grondwaterpeilen; en,
- September - November 2017: Opstellen en indienen 8^e^ tussentijds verslag.

Op basis van de voortgang van het dossier van de Oosterweelverbinding zullen mogelijks bijkomende werkzaamheden dienen te worden uitgevoerd in het kader van het eerste en tweede gefaseerde bodemsaneringsproject.

7. EOHWIJZIGINGEN TEN OPZICHTE VAN BODEMSANERINGSPROJECT

Voor de saneringsaanpak zoals voorzien in het eerste gefaseerd bodemsaneringsproject, opgesteld door Arcadis op 29 oktober 2008 (referentie *11/003460*), wordt verwezen naar paragraaf 3.2.

Voor de wijzigingen in de saneringsaanpak zoals beschreven in de voorgaande tussentijdse saneringsverslagen (periode van juli 2009 tot juni 2014) wordt verwezen naar paragraaf 3.3.

De wijzigingen in de saneringsaanpak van juli 2015 tot en met juni 2016 zijn in detail toegelicht in hoofdstuk 4 en 5. Algemeen kan gesteld worden dat het aantal monitoringspeilbuizen en de keuze ervan voor verschillende zones in beperkte mate is gewijzigd. Dit was meestal een gevolg van het aanpassen van de monitoringsfrequentie in functie van de concentratietrends. Een overzicht van het nieuw voorgestelde monitoringsprogramma is gegeven in Tabel 4.9.

Gezien de sterk beperkte bijdrage van de component PFOSA aan de somparameter FC, wordt voorgesteld om de analyse op PFOSA stop te zetten.

Het betreft steeds een **kleine wijziging** van het eerste gefaseerd bodemsaneringsproject.

8. CALAMITEITEN

8.1 PTBA INCIDENT TER HOOGTE VAN GEBOUW 017

Op 12 augustus 2015 is een lek waargenomen in een bovengrondse leiding, gelegen in een leidingsbrug tussen gebouw 016 en gebouw 003. Het vrijgekomen product betreft een gefluorineerd mengsel, met als hoofdcomponent PTBA (perfluortributylamine).

Door 3M is geraamd dat ongeveer een veertigtal liter van het product PTBA is vrijgekomen onder de vorm van een nevel, die deels is neergedaald op een met kiezel verharde ondergrond, een geasfalteerde weg en een open betonnen rioleringssleuf (van de chemische riolering). Geschat wordt door 3M dat ongeveer 25 kg product op de met kiezel verharde bodem is terecht gekomen (de rest is verneveld of in de chemische riolering terecht gekomen).

Na vaststelling is het lek onmiddellijk gedicht. Het vrijgekomen product op het asfalt is verwijderd met water. Het spoelwater en het product dat terecht kwam in de riolering is via de betonnen rioleringssleuf (chemische riolering) afgevoerd naar de bedrijfswaterzuivering.

Om de impact naar de bodem van de eerder beperkte lekkage van PTBA te onderzoeken is op 13 augustus 2015 in de namiddag gestart met het selectief ontgraven van de zone waar het product is terechtgekomen. Het betreft een zone van 1m bij 0,5 m en een totale diepte van 0,2 m. Vervolgens is door ERM een mengstaal van de grond genomen van de ontgraven grond en een monster van de bodem van de ontgravingsput. De stalen zijn geanalyseerd door het erkende laboratorium Servaco.

Voor de bepaling van de saneringsdoelstelling is door ERM een toetsingswaarde voor een standaard bodem en industrieel bodemgebruik bepaald. De concentratie PTBA gemeten in het staal genomen van de bodem van de ontgravingsput en het mengstaal van de ontgraven en tijdelijk gestockeerde grond (B) overschrijden de berekende saneringsnorm en daarvan afgeleide richtwaarde niet. Bijgevolg is geoordeeld dat er geen verdere maatregelen of een beschrijvend bodemonderzoek nodig zijn.

De afgegraven steenslag en grond zijn, in overleg met en na goedkeuring van OVAM, terug in het ontgravingsvak aangebracht.

Het incident is op 19 augustus 2015 (binnen de 14 dagen na vaststelling) via het standaardformulier "Melding schadegeval bij klasse I-inrichtingen" gemeld aan OVAM en de milieu-inspectie.

De resultaten en besluiten zijn in een 'Evaluatierapport schadegeval' aan OVAM gerapporteerd (referentie R03-0287933-v1.0). Dit onderzoek is op 11 april 2016 door OVAM conform verklaard (referentieDS-LB-KB-20160209703).

In de conformverklaring is opgenomen dat "*voor dit schadegeval met een gefluorineerd mengsel, met als hoofdcomponent PTBA (perfluortributylamine), ontstaan op 12 augustus 2015 afdoende maatregelen werden genomen en geen beschrijvend bodemonderzoek dient uitgevoerd te worden volgens het Bodemdecreet*".

9. TWEEDE FASE BODEMSANERINGSPROJECT

Het tweede gefaseerd bodemsaneringsproject zal de verontreiniging met FC verbindingen in het grondwater ter hoogte van het zuidelijk 3M terreingedeelte en de relatie met de Palingbeek behandelen. Aangezien de werken in het kader van de Oosterweelverbinding een belangrijke impact kunnen hebben op de waterhuishouding op deze locatie, dient een saneringsaanpak afgestemd te worden op deze werken. De voorbereidende studiewerken voor het deelproject Linkeroever zijn opgestart en worden door 3M en ERM opgevolgd. Wanneer er meer duidelijkheid is over de impact van de werken in het kader van de Oosterweelverbinding op deze zone, kan – indien noodzakelijk - een saneringsaanpak voor de FC impact uitgewerkt worden in een tweede gefaseerd bodemsaneringsproject.
In afwachting van een eventueel bodemsaneringsproject wordt de organofluorimpact ter hoogte van de zuidelijke perceelgrens en de Palingbeek momenteel opgevolgd door middel van een halfjaarlijkse grondwatermonitoring.

In afwachting van het tweede gefaseerd bodemsaneringsproject worden de FC-concentraties ter hoogte van de zuidelijke perceelgrens opgevolgd door middel van regelmatige grondwater- en oppervlaktewaterstaalnames.

10. BESLUIT

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 Zevende tussentijds verslag (TTV7) van de saneringswerken, zoals beschreven in het door de OVAM conform verklaarde bodemsaneringsproject (1^{e} fase) en de zes voorgaande tussentijdse verslagen. Het betreft de saneringswerken op het deel productiezone, voormalige slibbekkens/waterzuiveringsinstallatie, natuurgebied Blokkersdijk en de 2de aquifer. De beschouwde saneringsperiode loopt van juli 2015 tot en met juli 2016.

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.

In het kader van het conformverklaarde BSP (fase 1) zijn in de periode van juli 2015 tot en met juni 2016 de volgende zaken uitgevoerd:

- Opvolging en onderhoud van het grondwatersaneringssysteem in twee bronzones (Gebouw 016 en WWTP zone). Het systeem omvat negen onttrekkingsputten (PP01-02-04-05-06-07-08-09-10). Het voornaamste doel van de sanering is om de horizontale en verticale verspreiding van de verontreiniging ter hoogte van de bronzones van Gebouw 016 en de WWTP tegen te gaan. Er worden elk kwartaal stalen genomen van de pompputten om de efficiëntie van het saneringssysteem op te volgen;

- Periodieke grondwatermonitoring ter hoogte van de twee kernzones (Gebouw 016 en WWTP), de 2de aquifer, de zuidelijke perceelgrens, de zone met de ondergrondse tanks en het natuurgebied Blokkersdijk. En de periodieke monitoring van de kwaliteit van het oppervlaktewater van de Palingbeek, Tophatgracht, 3M vijver en Blokkersdijkvijver;

- Het berekenen van gemiddelde PFOS vuilvracht die in de Schelde geloosd wordt via de regenwater- en bedrijfsriolering, en de grachten (Palingbeek en Tophat gracht);

- Het vergelijken van de analyseresultaten van de standaard waterstalen geanalyseerd door het 3M Environmental Laboratory en de duplicaat stalen geanalyseerd door SGS;

- Buiten gebruik stellen van de Z-peilbuizen gelegen aan de overkant van de E34 snelweg (Expresweg) en de voormalige BTEX verontreiniging in de zone van de ondergrondse tanken. Deze twee partiële eindevaluatieonderzoeken zijn op 1 juli 2015 door OVAM conform verklaard;

- Opvolging van de vooruitgang van het Oosterweelverbindingsproject aangezien deze werken in belangrijke mate de timing en uitwerking van het 2de gefaseerd bodemsaneringsproject bepalen;

- Hervaluatie van de initiele aannames waarop de PFOS trend analyse is gebaseerd, rekening houdend met de mogelijke impact van het gebruik van verschillende labo's tijdens de monitoringsperiode; en

- Begeleiden van de mitigerende maatregelen genomen naar aanleiding van het incident waarbij op 12 augustus 2015 onder de pijpenbrug tussen gebouw 016 en gebouw 003 een beperkte hoeveelheid PTBA (perfluortributylamine)is vrijgekomen, en het opstellen van een rapport verkennend onderzoek voor OVAM na afloop van de werken.

Uit de onderzoeksverrichtingen en saneringswerken uitgevoerd in de voorbije periode (juli 2015-juli 2016) kunnen volgende conclusies getrokken worden:

- De grond- en grondwaterverontreiniging bij 3M, zoals beschreven in het eerste bodemsaneringsproject, wordt gesaneerd in overeenstemming met de bepalingen in het bodemsaneringsproject.

- Het P&T system, dat operationeel is sinds 2010, bestaat momenteel uit negen pompputten. In totaal is in de periode juli 2015 tot juli 2016 ongeveer 9.639 m^3 verontreinigd grondwater onttrokken en circa 67,9 kg FC verwijderd. In totaal is sinds juli 2011 circa 429 kg FC 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. Echter, in enkele pompputten en peilbuizen worden stijgende concentraties gemeten, wat erop kan duiden dat de captatie lokaal niet volledig is. Uit veldobservaties blijkt verder dat het saneringssysteem storingsgevoelig is en in de periode juli 2015-juli 2016 een up-time heeft van gemiddeld 53% wat lager is dan voorgaande jaren. Er is wel een groot verschil tussen de putten onderling: min. 21% in PP06 en PP07 en max. 92% in PP01.
  ERM raadt aan om de saneringsacties en regelmatige monitoring voort te zetten zoals samengevat in Tabel 4.9 (p.54). De potentiële stijgende concentraties moeten verder geverifieerd worden door monitoring. Verder wordt aanbevolen om te evalueren of een uitbreiding van het saneringssysteem nodig is om de captatie van de verontreiniging te optimaliseren. Bij deze evaluatie moet ook worden nagegaan hoe de werking van het systeem verbeterd kan worden.

- De PFOS concentratie trend in de Blokkersdijk peilbuizen, de 3M vijver en de Blokkersdijk vijver worden verder opgevolgd via regelmatige monitoring en statistische evaluatie voort te zetten met enkel de data van het 3M Environmental lab, omdat de herevaluatie van de statistische analyses heeft aangetoond dat het gebruik van meerdere labo's de resultaten van de PFOS trend analyse beïnvloedt. Uit de evaluatie blijkt dat in peilbuis L21 een significant stijgende trend wordt waargenomen en in peilbuis L22 een significant dalende trend.
  Momenteel is geen noodzaak voor bijkomende saneringsacties in deze zone aangezien er geen stijgende concentratietrend is waargenomen in de Blokkersdijk vijver.

- De gemiddelde berekende vuilvracht van PFOS naar de Schelde voor de periode 2015 – 2016 ligt in lijn met eerder berekende vuilvrachten. De hogere vuilvracht voor de periode 2011 – 2012 was waarschijnlijk het gevolg van het slib dat onderin de collectorput van de regenwaterriolering aanwezig was. Intussen is dit slib geruimd en zijn de PFOS concentraties in de regenwaterriolering en de berekende gemiddelde vuilvracht opnieuw gedaald.
  De berekende gemiddelde vuilvrachten zijn steeds onder de maximaal toegelaten vuilvracht van 370 g/dag gebleven.

- In de periode juli 2015 - juli 2016 heeft zich één milieu-incident voorgedaan op de site van 3M. De analyseresultaten toonden geen impact in deze zone. Bijgevolg zijn er geen verdere acties vereist.

- In afwachting van het tweede gefaseerd bodemsaneringsproject worden de FC-concentraties ter hoogte van de zuidelijke perceelgrens opgevolgd door middel van regelmatige grondwater- en oppervlaktewater-staalnames. Wanneer er meer duidelijkheid is over de impact van de werken in het kader van de Oosterweelverbinding op deze zone, zal – indien noodzakelijk - een saneringsaanpak voor de FC impact uitgewerkt worden in een tweede gefaseerd bodemsaneringsproject. De voorbereidende studiewerken voor het deelproject Linkeroever zijn opgestart en worden door 3M en ERM opgevolgd.

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

Het volgende tussentijds rapport met de bespreking van de saneringswerken en monitoringsresultaten van de periode juli 2016 tot en met juli 2017 zal aan OVAM overgemaakt worden tegen eind 2017.

■

DEEL 3: BIJLAGEN

Eerste gefaseerd bodemsaneringsproject -
Zevende tussentijds verslag bodemsaneringswerken
Periode juli 2015 - juli 2016

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

25 augustus 2017

www.erm.com

Bijlage 1

OVAM conformverklaring eerste gefaseerd bodemsaneringsproject

Bijlage 2

Milieukundig dagboeken

- inspectie afdek
- buitengebruikstellen Z-peilbuizen

Bijlage 3

Analysecertificaten

Bijlage 4

Overzicht veldmetingen en analyseresultaten

Bijlage 5

Statistische evaluatie analyseresultaten Blokkersdijk

Bijlage 6

Schematische weergave pompput en inspectiekamer

DEEL 4: KAARTMATERIAAL

Eerste gefaseerd bodemsaneringsproject -
Zevende tussentijds verslag bodemsaneringswerken
Periode juli 2015 - juli 2016

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

25 augustus 2017

www.erm.com

Kaart 1

Situering onderzoekslocatie op topografische kaart

Kaart 2

Overzichtsplan onderzoekslocatie

Kaart 3

Detailplan ligging grondhopen

Kaart 4

Detailplannen monsternamelocaties

A. Monsternamelocaties Gebouw 16
B. Monsternamelocaties Gebouw WWTP
C. Monsternamelocaties 2de aquifer
D. Monsternamelocaties Zuidelijke grens
E. Monsternamelocaties Blokkersdijk
F. Monsternamelocaties Paling- en Tophatbeek
G. Monsternamelocaties Collector put en lozingspunt WWTP

Kaart 5

Onttrekkingsfilters grondwateronttrekking

DEEL 3: BIJLAGEN

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Canadastraat 11 te 2070 Zwijndrecht

25 augustus 2017

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Bijlage 1

OVAM conformverklaring eerste gefaseerd bodemsaneringsproject

OPENBARE VLAAMSE AFVALSTOFFENMAATSCHAPPIJ

STATIONSSTRAAT 110
2800 MECHELEN

TEL. 015 284 284
FAX 015 203 275

WWW.OVAM.BE

Aangetekend met ontvangstbewijs 2009500832

3M Belgium NV

Haven 1005, Canadastraat 11
2070 ZWIJNDRECHT

Uw Bericht Van ·
Uw Kenmerk ·
Bijlagen · 1 bundel
Mechelen · 09 FEB. 2009

Afdeling · Bodembeheer
Dienst · Bodemonderzoek en -sanering Oost
Contactpersoon · Dominique Suys [redacted]
Ons Kenmerk · BB-O-DS/2009500832 (Dossiernummer: 732)

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, Canadastraat 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 milieuheffingen vastgesteld op 0 EUR per ton. Dit tarief van milieuheffing is geldig voor afvalstoffen afkomstig van bodemsaneringsoperaties, voor het storten op een daartoe 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 meebrengen of onmogelijk zijn.

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

OVAM
de heer Filip De Naeyer
Diensthoofd
Stationsstraat 110
2800 MECHELEN

Het bedrag aan milieuheffing 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

OPENBARE VLAAMSE AFVALSTOFFENMAATSCHAPPIJ

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 CANADASTRAAT 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
11813 ANTWERPEN 13 AFD	N	0477 A
11813 ANTWERPEN 13 AFD	N	0478 A
11813 ANTWERPEN 13 AFD	N	0479 A
11813 ANTWERPEN 13 AFD	N	0480 A
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
11813 ANTWERPEN 13 AFD	N	0485 A
11813 ANTWERPEN 13 AFD	N	0486 A
11813 ANTWERPEN 13 AFD	N	0487
11813 ANTWERPEN 13 AFD	N	0488 A
11813 ANTWERPEN 13 AFD	N	0490 A
11813 ANTWERPEN 13 AFD	N	0491
11813 ANTWERPEN 13 AFD	N	0492
11813 ANTWERPEN 13 AFD	N	0493
11813 ANTWERPEN 13 AFD	N	0494
11813 ANTWERPEN 13 AFD	N	0495
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
11813 ANTWERPEN 13 AFD	N	0509
11813 ANTWERPEN 13 AFD	N	0510
11813 ANTWERPEN 13 AFD	N	0511
11813 ANTWERPEN 13 AFD	N	0512
11813 ANTWERPEN 13 AFD	N	0513
11813 ANTWERPEN 13 AFD	N	0514
11813 ANTWERPEN 13 AFD	N	0515
11813 ANTWERPEN 13 AFD	N	0516
11813 ANTWERPEN 13 AFD	N	0517
11813 ANTWERPEN 13 AFD	N	0518
11813 ANTWERPEN 13 AFD	N	0519
11813 ANTWERPEN 13 AFD	N	0520
11813 ANTWERPEN 13 AFD	N	0521
11813 ANTWERPEN 13 AFD	N	0522
11813 ANTWERPEN 13 AFD	N	0523
11813 ANTWERPEN 13 AFD	N	0524
11813 ANTWERPEN 13 AFD	N	0525
11813 ANTWERPEN 13 AFD	N	0526
11813 ANTWERPEN 13 AFD	N	0527
11813 ANTWERPEN 13 AFD	N	0528
11813 ANTWERPEN 13 AFD	N	0529
11813 ANTWERPEN 13 AFD	N	0530
11813 ANTWERPEN 13 AFD	N	0531
11813 ANTWERPEN 13 AFD	N	0532
11813 ANTWERPEN 13 AFD	N	0533 B
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
11813 ANTWERPEN 13 AFD	N	0534/02 A
11813 ANTWERPEN 13 AFD	N	0543 A
11813 ANTWERPEN 13 AFD	N	0568
11813 ANTWERPEN 13 AFD	N	0569
11813 ANTWERPEN 13 AFD	N	0570
11813 ANTWERPEN 13 AFD	N	0571
11813 ANTWERPEN 13 AFD	N	0572
11813 ANTWERPEN 13 AFD	N	0573
11813 ANTWERPEN 13 AFD	N	0574
11813 ANTWERPEN 13 AFD	N	0575
11813 ANTWERPEN 13 AFD	N	0576
11813 ANTWERPEN 13 AFD	N	0577
11813 ANTWERPEN 13 AFD	N	0578
11813 ANTWERPEN 13 AFD	N	0579
11813 ANTWERPEN 13 AFD	N	0590 A
11813 ANTWERPEN 13 AFD	N	0610
11813 ANTWERPEN 13 AFD	N	0611 A

11813 ANTWERPEN 13 AFD	N	0613 A
11813 ANTWERPEN 13 AFD	N	0614 A
11813 ANTWERPEN 13 AFD	N	0615
11813 ANTWERPEN 13 AFD	N	0616
11813 ANTWERPEN 13 AFD	N	0617 A
11813 ANTWERPEN 13 AFD	N	0619 A
11813 ANTWERPEN 13 AFD	N	0620 A
11813 ANTWERPEN 13 AFD	N	0621 A
11813 ANTWERPEN 13 AFD	N	0622
11813 ANTWERPEN 13 AFD	N	0623 A
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 vluchtige 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 organoflourconcentraties. 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 gefaseerdbodemsaneringsproject 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 volledig gefaseerd 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 gevolgd.
- Tijdens de sanering moeten regelmatig analyses worden uitgevoerd en ter beschikking gehouden van de controlerende 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 natuurreservaat Blokkersdijk betreft recent onderzoek heeft uitgewezen (Universiteit Antwerpen) dat PFOS (perfluoroctaan 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 Vlarem 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 natuurreservaat 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 1ste 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 (2de 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 een zelfde 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 natuurreservaat (Vogelrichtlijngebied) liggen eenduidig meer naar het oosten. Er valt geen eigendom van 3M binnen het natuurreservaat.
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 dewelke 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 kon 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 trent 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 oppervlatewaterkwaliteit 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 herinfiltratienormen op voorwaarde dat het om een gesloten systeem gaat waarop de nodige controle wordt uitgevoerd.

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

(1) Opmerking: de herinfiltratienorm is de drinkwaternorm. Indien evenwel de terugsaneerwaarde strenger is, is dit de herinfiltratienorm. De herinfiltratiewaarde 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 onttrekking 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 geherinfiltreerd, Gelet op de strenge herinfiltratienormen en het feit dat het onttrokken grondwater opnieuw wordt geherinfiltreerd, 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 verwezelijking 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 meegedeeld.

- 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, Eindevaluatieonderoek en Nazorg'.
- De grondwateronttrekking is gekoppeld aan de strikte naleving van de eigen milieuvergunningsvoorwaarden 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 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 trent 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 oppervlatewaterkwaliteit 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.
-
- herinfiltratienormen (op voorwaarde dat het om een gesloten systeem gaat waarop de nodige controle wordt uitgevoerd):

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

(1) Opmerking: de herinfiltratienorm is de drinkwaternorm. Indien evenwel de terugsaneerwaarde strenger is, is dit de herinfiltratienorm. De herinfiltratiewaarde 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.

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, 0 9 FEB. 2009

Eddy Van Dyck
Afdelingshoofd

Bijlage 2

Milieukundig dagboeken

- inspectie afdek
- buitengebruikstellen Z-peilbuizen

DAILY BOOK ENVIRONMENTAL SUPERVISION
DAGBOEK MILIEUKUNDIG TOEZICHT*

Date: 26/04/2016

Project: 0341743 – 3M Belgium

OVAM dossier: 732

Site Location	Weather
Canadastraat 11 2070 Zwijndrecht	Cloudy hail and rain +/- 5°C

Distribution list	
3M	**Charlotte Tack ([redacted])**
Weston Solutions	**Dave Cairns ([redacted])**
ERM	**Mattias Verbeeck [redacted]), Erik Boeckx [redacted]), Nanda Hermes ([redacted]), Ruben Maes ([redacted]), Adriaan de Bourgraaf ([redacted])**

Present

Name	*Function*	*Firm*	*Hour in*	*Hour out*
Adriaan de Bourgraaf	Project consultant	ERM	7:20	08:50

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. The next check was scheduled for July 2018.
At the request from OVAM (dd. April 13th, 2016), an additional check was done this year.

Site visit – Status cover

During the visit on April 26th 2016, 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

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* conform OVAM document "Bodemsaneringswerken en nazorg, standaardprocedure, september 2015"

Environmental Observation

Field observations

Not applicable

Sampling and Analyse

Not applicable

Photolog

Map with photo locations is added as Annex 1.

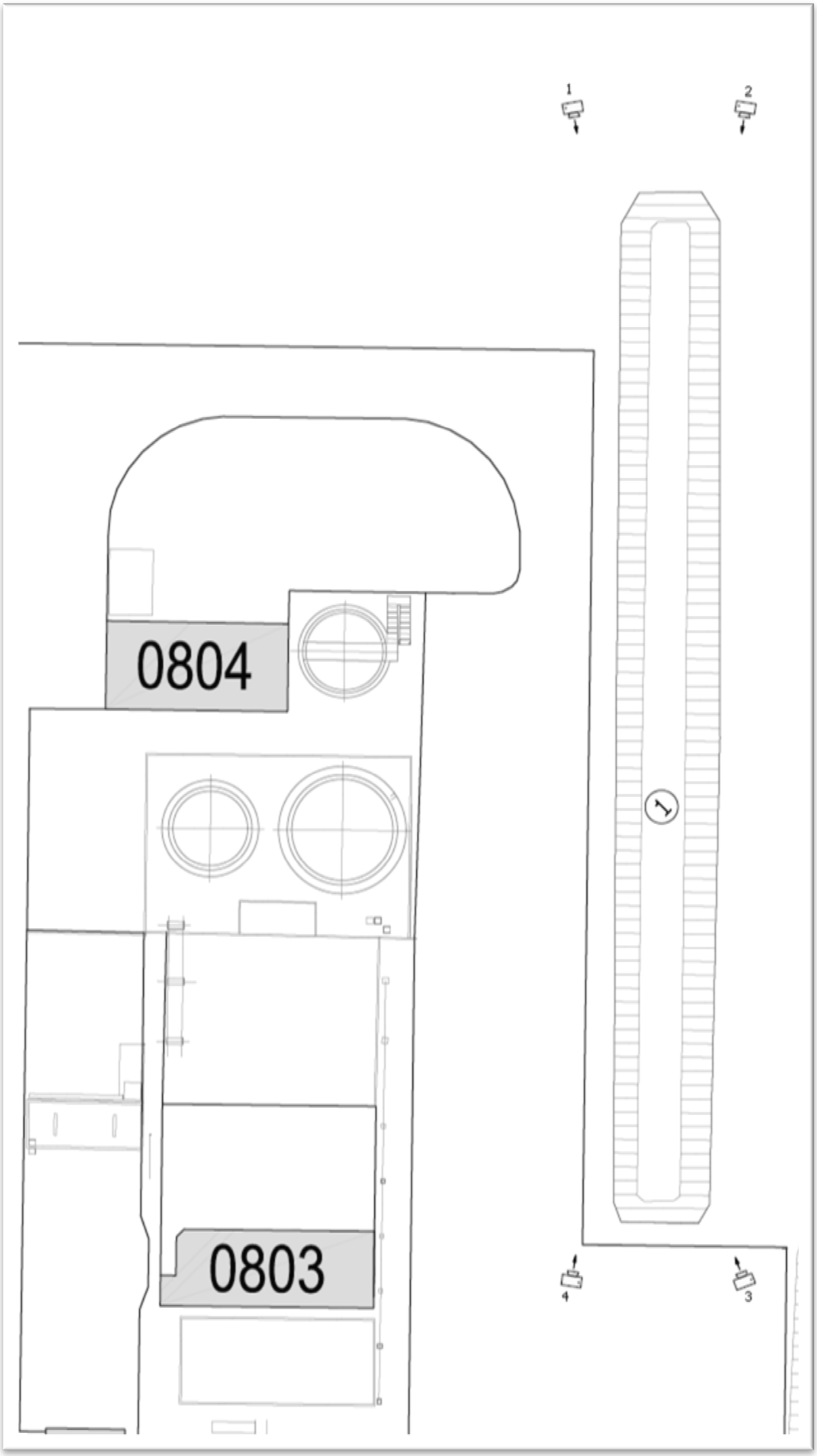
1. 	2. 
3. 	4. 

Additional comments

Not applicable

For the Environmental Consultant *(bodemsaneringsdeskundige)* Name and Signature Adriaan de Bourgraaf	**For the Client** Name and Signature

Annex 1: Map with photo locations



ERM	**DAILY BOOK ENVIRONMENTAL SUPERVISION** **DAGBOEK MILIEUKUNDIG TOEZICHT*** *Date: 08/12/2015, 08/01/2016, 23/02/2016* *Project*: 0287933 – 3M Belgium *OVAM dossier:* 732

Site Location	**Weather**
2070 Zwijndrecht	08/12/2015 : Sun, +/- 12°C *08/01/2016:* Cloudy, +/- 8°C *23/02/2016:* Sunny, +/- 5°C

Distribution list	
3M	**Charlotte Tack** [redacted]
Weston Solutions	**Dave Cairns (**[redacted]
ERM	**Mattias Verbeeck** [redacted]**), Erik Boeckx (**[redacted]**), Nanda Hermes (**[redacted]**), and Adriaan de Bourgraaf (**[redacted]**)**

Present				
Name	***Function***	***Firm***	***Start time***	***End time***
08/12/2015				
Adriaan de Bourgraaf	Project Engineer	ERM	08:00	12:00
Frederic Carton	Contractor	Servaco	08:00	12:00
08/01/2016				
Adriaan de Bourgraaf	Project Engineer	ERM	08:00	08:30
Bram	Land-surveyor	BuroEyckmans	08:00	08:30
23/02/2016				
Adriaan de Bourgraaf	Project Engineer	ERM	12:00	13:00
Servaco_contractor	Contractor	Servaco	12:00	13:00

Decommissioning of z-wells

3M asked ERM to decommission nine monitoring wells (Zwijndrecht Z-wells), located south of the 3M site and the E34 highway in Zwijndrecht.

The Z-wells are no longer included in the remedial monitoring program and could pose a risk by acting as preferential flow path for substances polluting the groundwater. To remove this risk it was decided to remove the wells.

Environmental supervision

Servaco (subcontractor) performed the decommissioning of all Z-wells. The works were supervised by ERM. Two wells could initially not be located and a landsurveyor (BuroEyckmans bvba) was contracted to locate the two missing wells. This was done on the 8th of January under supervision of ERM. Both wells were found:

- Z3-3m : located, damaged and jammed by metal cover; and
- Z3-10m: located, damaged and jammed by metal cover.

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* conform OVAM document "Bodemsaneringswerken en nazorg, standaardprocedure, september 2015"

All of the nine wells were decommissioned according to the Flemish environmental prescribed procedures (CMA/1/A.2.):

- Z3-3m (decommissioned on 23-Jan-16)
- Z3-10m (decommissioned on 23-Jan-16)
- Z6-3m (decommissioned on 8-Dec-15)
- Z1-3m (decommissioned on 8-Dec-15)
- Z1-10m (decommissioned on 8-Dec-15)
- Z4-8m (decommissioned on 8-Dec-15)
- Z2-3m (decommissioned on 8-Dec-15)
- Z2-10m (decommissioned on 8-Dec-15)
- Z5-8m (decommissioned on 8-Dec-15)

Health & Safety
Special care needs to be taken when lifting heavy loads for the decommission of the wells. This is indicated in the H&S Plan prepared by ERM for decommissioning of the Z-wells.

Environmental Observation

Field observations

Not applicable

Sampling and Analyse

Not applicable

Photo log

Decommissioning of Z3-10m & Z3-3m.

Decommissioning of Z3-10m & Z3-3m.

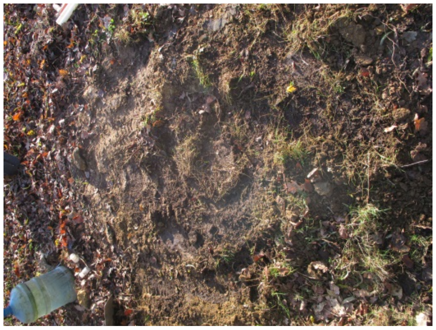
Decommissioning of Z6-8m.

Decommissioning of Z6-8m

Decommissioning of Z4-8m

Decommissioning of Z1-3m & Z1-10m

Result decommissioning Z1-3m &Z1-10m



Decommissioning Z2-3m & Z2-10m

Decommissioning Z2-3m & Z2-10m

Decommissioning Z5-8m

Result decommissioning Z5-8m

Localisation of Z3-3m & Z3-10m

Damaged and jammed well cover Z3

Location well next to screwdriver

Additional comments

No additional comments

For the Environmental Consultant *(bodemsaneringsdeskundige)* Name and Signature Adriaan de Bourgraaf	**For the Client** Name and Signature

Bijlage 3

Analysecertificaten

ADDENDUM to analytical report IAC15-05677

Beste Sandra,

Midden oktober staat een nieuwe bemonsteringscampagne ingepland bij 3M.
Zouden jullie tegen eind volgende week recipiënten kunnen aanleveren in ons kantoor te Antwerpen-Berchem?

ERM NV t.a.v. Ruben Maes/Nanda Hermes
Posthoflei 5 bus 6
2600 Antwerpen-Berchem

In bijlage van deze e-mail kunt u een lijst terugvinden van de recipiënten die we nodig hebben.
Er staat ook telkens bij vermeld welke parameters dienen te worden geanalyseerd.

Dank alvast bij voorbaat,

Ruben Maes
Consultant

Environmental Resources Management - ERM nv
Posthoflei 5 bus 6, 2600 Antwerpen-Berchem, Belgium

Cantersteen 47, 1000 Brussel, Belgium
Burg. De Raadtsingel 55, 3311JG Dordrecht, The Netherlands

☎ | [redacted]
📱 | [redacted]

✉ | [redacted]

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Zone	ID	Nature of monitoring	Well screen (mbgl)	Parameters to be analysed
Blokkersdijk nature reserve	3M vijver	surfacewater	n.a.	FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Blokkersdijk nature reserve	L4	groundwater	2,0-4,0	FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Blokkersdijk nature reserve	P116	groundwater	2,5-3,5	FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Blokkersdijk nature reserve	P119C	groundwater	2,5-4,5	FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Effluent WWTP	Effluent WWTP	waste water	n.a.	FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Extration wells P&T	PP01	remediation	2,6-5,6	FC-parameters (PFOS-PFOA-PFHS-PFOSA-PFBS)
Extration wells P&T	PP04	remediation	2,0-6,0	FC-parameters (PFOS-PFOA-PFHS-PFOSA-PFBS)
Extration wells P&T	PP06	remediation	1,3-5,3	FC-parameters (PFOS-PFOA-PFHS-PFOSA-PFBS)
Extration wells P&T	PP08	remediation	3,2-7,2	FC-parameters (PFOS-PFOA-PFHS-PFOSA-PFBS)
Extration wells P&T	PP10	remediation	3,0-6,0	FC-parameters (PFOS-PFOA-PFHS-PFOSA-PFBS)
Palingbeek & Tophatgracht	13	surfacewater	n.a.	FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Palingbeek & Tophatgracht	Bemalingsstation	surfacewater	n.a.	FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Sewer	Collector put	stormwater	n.a.	FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Southern site boundary	B3-bis	groundwater	0,4-1,4	FC-parameters (PFOS-PFOA-PFHS-PFOSA)

3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC15-05677-002

3M Lab Request Number: E15-0965

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: December 17th, 2015

Requester

Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000

1. Introduction - Summary.

At the request (Lab Request Number: E15-0965) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, one water sample was collected on October 20th, 2015 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 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 ± 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	0.881	2.03	9.42	0.107 (a)

(a): The recovery of FOSA for 3M Vijver is ±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 ($^{13}C_2$-PFHS, $^{13}C_4$-PFOA, $^{13}C_4$-PFOS, $^{13}C_7$-PFUdA) and internal standard solution ($^{13}C_3$-PFHS, $^{13}C_8$-PFOA, $^{13}C_8$-PFOS, $^{13}C_8$-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}C-PFC-SS	^{13}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}C-PFC Surrogate Recovery Standards
Solution B = 1500 ng/mL (nominal) ^{13}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 determination of suspended solids.

2.4. Analysis.

All solutions and extracts were analyzed for the PFCs (PFHS, PFOA, PFOS, FOSA) and the surrogate recovery standards ($^{13}C_2$-PFHS, $^{13}C_4$-PFOA, $^{13}C_4$-PFOS, $^{13}C_7$-PFUdA) and internal standards ($^{13}C_3$-PFHS, $^{13}C_8$-PFOA, $^{13}C_8$-PFOS, $^{13}C_8$-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
$^{13}C_7$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 ± 25% (± 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 ± 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 carryover.

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 ± 25% for accuracy.
All LCSs produced recoveries within the method acceptance criteria of ± 15% RPD for precision.
Table 7 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
10 November 2015	LCSL01	0.125	96.3	113	107	115	101	120	120	112
	LCSL02	0.125	93.3	120	112	115	104	118	117	122
	Average		**94.8**	**116**	**109**	**115**	**103**	**119**	**119**	**117**
	%RPD		**3.2**	**5.8**	**4.5**	**0.3**	**3.4**	**1.1**	**2.4**	**8.5**
	LCSH 01	2.5	111	100	114	121	86.4	114	106	98.9
	LCSH 02	2.5	104	99.4	112	119	91.3	113	111	105
	Average		**107**	**99.9**	**113**	**120**	**88.9**	**113**	**108**	**102**
	%RPD		**6.7**	**1.0**	**1.5**	**1.3**	**5.5**	**0.4**	**5.0**	**6.4**

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	$^{13}C_2$-PFHS	PFOA	$^{13}C_4$-PFOA
	ng/ml	%Rec	ng/ml	%Rec
Field Trip Blank	<0.025	95.1	<0.025	88.8
3M vijver	0.881	88.7	2.03	93.2

Table 9. Sample Results PFOS and FOSA.

	PFOS	$^{13}C_4$-PFOS	FOSA	$^{13}C_7$-PFuDA
	ng/ml	%Rec	ng/ml	%Rec
Field Trip Blank	<0.025	119	<0.025	79.2
3M vijver	9.42	111	0.107	32.1(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 ± 30% except for FOSA for the following sample locations:

- The accuracy for FOSA for 3M vijver is ± 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 Ruben Maes (ERM Belgium).

8. Signatures.

Geert De Smet,
Lab Operations Manager Date December 17th, 2015

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories Date December 17th, 2015

I.A.C.
A Division of SGS Belgium NV

3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC15-05677-001

3M Lab Request Number: E15-0965

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: December 17th, 2015

Requester

Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000

1. Introduction - Summary.

At the request (Lab Request Number: E15-0965) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected between October 20th and 22nd, 2015 from 12 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 ($C_7F_{15}{}^{13}CF_2SO3^-$)
- 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 ± 30%.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
L4		1.72	1.47	29.2	<0.598
P116		0.439	1.29	14.8	<0.201
P119C		450	736	2790	14.9
B3-bis		4.46	30.8	99.1	<1.09
Effluent WWTP		8.73	17.0	1.69	<0.201
Bemalingsstation		20.8	30.4	48.7	<0.598
Collector Put		15.9	49.0	116	3.22
PP01	23.8	7100	39.7	1184	34.7
PP06	15.8	19.2	102	1035	9.19
PP08	<11.0	88.6	279	6385	19.8
PP10	<11.0	<8.91	76.6	414	61.3
13		20.4	89.3	216	<1.09

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
P119C	100
B3-bis	100
Effluent WWTP	100
Bemalingsstation	100
Collector Put	100
PP01	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
L4	1.0	0.100	Solution A	5.0	-
P116	1.0	0.100	Solution A	1.0	
P119C	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
B3-bis	1.0	0.100	Solution A	10.0	-
Effluent WWTP	1.0	0.100	Solution A	1.0	-
Bemalingsstation	1.0	0.100	Solution A	5.0	-
Collector Put	1.0	0.100	Solution A	5.0	-
PP01	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP06	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
PP08	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP10	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
13	1.0	0.100	Solution A	10.0	-
Field Trip Blank	1.0	0.100	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	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, ^{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 ± 25% (± 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 0.105 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.150 to 12.8 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 ± 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 ± 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
10 Novembre 2015	LCS1	80	85.2	91.4	94.9	89.5	92.0	124	83.2

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 ± 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
2 July 2015	QC Lab Low inj1	10	107	97.2	124	114	108	91.5	98.4
	QC Lab High inj1	100	75.4	75.0	100	75.4	93.9	75.0	75.2
	QC Lab Low inj2	10	103	93.4	124	112	108	92.2	95.3
	QC Lab High inj2	100	75.4	75.6	109	79.6	94.9	76.1	75.7

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
L4		1.72	1.47	29.2	<0.598
P116		0.439	1.29	14.8	<0.201
P119C		450	736	2790	14.9
B3-bis		4.46	30.8	99.1	<1.09
Effluent WWTP		8.73	17.0	1.69	<0.201
Bemalingsstation		20.8	30.4	48.7	<0.598
Collector Put		15.9	49.0	116	3.22
PP01	23.8	7100	39.7	1184	34.7
PP06	15.8	19.2	102	1035	9.19
PP08	<11.0	88.6	279	6385	19.8
PP10	<11.0	<8.91	76.6	414	61.3
13		20.4	89.3	216	<1.09
Field Trip Blank	<0.999	<0.810	<0.844	<1.17	<1.09

Table 10. Sample Results ^{13}C-PFOA and ^{13}C-PFOS

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
L4	120	100	107	87.2
P116	56.1	93.8	44.3	72.0
P119C	122	102	102	82.9
B3-bis	122	102	103	84.0
Effluent WWTP	56.9	95.0	43.3	70.4
Bemalingsstation	117	98.2	106	86.1
Collector Put	117	97.5	104	84.3
PP01	115	96.1	111	90.5
PP06	108	90.3	112	90.9
PP08	107	89.7	100	81.4
PP10	112	93.4	103	83.7
13	116	96.9	103	83.5
Field Trip Blank	118	98.8	108	87.4

5. Conclusion.

- The recovery of ^{13}C-PFOA and ^{13}C-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 Ruben Maes (ERM Belgium).

8. Signatures.

Geert De Smet,
Lab Operations Manager
Date December 17th, 2015

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories
Date December 17th, 2015

I.A.C.
A Division of SGS Belgium NV

ADDENDUM to analytical report IAC15-07460

Beste Sandra,

In de tweede week van januari staat een nieuwe bemonsteringscampagne ingepland bij 3M. Zouden jullie voor donderdag 7 januari 2016 recipiënten (zie bijlage) kunnen aanleveren in ons kantoor te Antwerpen-Berchem?

ERM NV t.a.v. Ruben Maes/Nanda Hermes
Posthoflei 5 bus 6
2600 Antwerpen-Berchem

In bijlage van deze e-mail kunt u een lijst terugvinden van de recipiënten die we nodig hebben.
Er staat ook telkens bij vermeld welke parameters dienen te worden geanalyseerd.

Alvast bedankt,

Met vriendelijk groet,

Adriaan de Bourgraaf
Consultant

ERM Belgium
Posthoflei 5 bus 6
2600 Berchem (Antwerp)
Belgium

Tel: [redacted]
Mobile [redacted]

www.erm.com
[redacted]

Zone ID Nature of
monitoring
Analyse
Jan

2nd Aquifer BD24-3 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
2nd Aquifer D09 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
2nd Aquifer P121 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Blokkersdijk nature reserve L21 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Blokkersdijk nature reserve L22 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Blokkersdijk nature reserve L31 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Blokkersdijk nature reserve P115 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Source area - WWTP P119C groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Effluent WWTP Effluent WWTP waste water x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Palingbeek & Tophatgracht Bemalingsstation surfacewater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Sewer Collector put stormwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Source area - Building 16 P27 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA), PFBS
Source area - Building 16 P304 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA), PFBS
Source area - Building 16 P305 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA), PFBS
Source area - Building 16 P42 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA), PFBS
Source area - WWTP M4 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Source area - WWTP P118C groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Source area - WWTP P264 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Source area - WWTP P265C groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Source area - WWTP P371 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Source area - WWTP P374 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Source area - WWTP P381 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Source area - WWTP P382 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Southern site boundary B3 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Southern site boundary B7 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Southern site boundary P378 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Southern site boundary PA112 groundwater x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
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3M Corporate Environmental Programs.
Jim Kotsmith
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ANALYTICAL REPORT

IAC15-07460-002

3M Lab Request Number: E16-0190

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: March 7th, 2016

Requester

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1. Introduction - Summary.

At the request (Lab Request Number: E15-0190) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, one water sample was collected on January 14th, 2016 from 4 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 ± 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
L21	0.165	0.411	5.25	0.129
L22	0.193	0.432	5.70	0.113
L31	0.697	0.598	8.58	0.222
P115	2.83	5.35	0.757	0.025

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 ($^{13}C_2$-PFHS, $^{13}C_4$-PFOA, $^{13}C_4$-PFOS, $^{13}C_7$-PFUdA) and internal standard solution ($^{13}C_3$-PFHS, $^{13}C_8$-PFOA, $^{13}C_8$-PFOS, $^{13}C_8$-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}C-PFC-SS	^{13}C-PFC-IS
L21	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
L22	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
L31	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
P115	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0

Solution A = 1000 ng/mL (nominal) ^{13}C-PFC Surrogate Recovery Standards
Solution B = 1500 ng/mL (nominal) ^{13}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
L21	40	8
L22	40	8
L31	40	8
P115	40	8
Field Trip Blank	40	8

2.3. Determination of suspended solids in water.

No determination of suspended solids.

2.4. Analysis.

All solutions and extracts were analyzed for the PFCs (PFHS, PFOA, PFOS, FOSA) and the surrogate recovery standards ($^{13}C_2$-PFHS, $^{13}C_4$-PFOA, $^{13}C_4$-PFOS, $^{13}C_7$-PFUdA) and internal standards ($^{13}C_3$-PFHS, $^{13}C_8$-PFOA, $^{13}C_8$-PFOS, $^{13}C_8$-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
$^{13}C_7$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 ± 25% (± 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 ± 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 ± 25% for accuracy.
All LCSs produced recoveries within the method acceptance criteria of ± 15% RPD for precision.
Table 7 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
3 February 2016	LCSL01	0.125	93.1	113	102	125	93.1	120	115	106
	LCSL02	0.125	97.2	118	116	117	106	119	118	118
	Average		**95.2**	**115**	**109**	**121**	**100**	**120**	**116**	**112**
	%RPD		**4.3**	**4.6**	**13**	**6.2**	**13**	**1.4**	**2.3**	**11**
	LCSH 01	2.5	113	112	102	120	85.6	109	112	93.0
	LCSH 02	2.5	114	114	98.5	117	86.3	115	118	103
	Average		**114**	**113**	**100**	**118**	**86.0**	**112**	**115**	**98.2**
	%RPD		**0.3**	**2.4**	**3.9**	**2.5**	**0.8**	**5.0**	**4.9**	**11**

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	$^{13}C_2$-PFHS	PFOA	$^{13}C_4$-PFOA
	ng/ml	%Rec	ng/ml	%Rec
Field Trip Blank	<0.025	95.5	<0.025	99.1
L21	0.165	84.9	0.411	89.9
L22	0.193	95.9	0.432	106
L31	0.697	87.9	0.598	94.3
P115	2.83	89.8	5.35	98.0

Table 9. Sample Results PFOS and FOSA.

	PFOS	$^{13}C_4$-PFOS	FOSA	$^{13}C_7$-PFuDA
	ng/ml	%Rec	ng/ml	%Rec
Field Trip Blank	<0.025	89.8	<0.025	116
L21	5.25	90.1	0.129	80.6
L22	5.70	94.2	0.113	81.4
L31	8.58	98.8	0.222	73.3
P115	0.757	91.9	0.025	109

5. Conclusion.

- The recovery of the surrogate recovery standard fell within the method acceptance criteria of 70-130%.
- 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 Adriaan de Adriaan de Bourgraaf (ERM Belgium).

8. Signatures.

Geert De Smet,
Lab Operations Manager Date March 7th, 2016

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories Date March 7th, 2016

I.A.C.
A Division of SGS Belgium NV

3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
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USA

ANALYTICAL REPORT

IAC15-07460-001

3M Lab Request Number: E16-0190

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: March 7th, 2016

Requester

Jim Kotsmith
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1. Introduction - Summary.

At the request (Lab Request Number: E15-0190) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected between January 12th and 19th, 2016 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 ($C_7F_{15}{}^{13}CF_2SO3^-$)
- 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 ± 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
BD24-3		46.7	63.8	1132	<4.33
D09		9514	95.8	1734	<23.8
P121		0.939	<0.322	11.4	<0.400
P119C		567	748	2169	29.2
Effluent WWTP		32.7	21.4	39.7	<0.410
Bemalingsstation		30.9	37.1	62.6	<2.16
Collector Put		15.4	37.1	115	3.56
P27	2922	20.3	360	1622	14.8
P304	195	122	507	965	6.30
P305	83.6	201	163	491	<4.33
P42	42.9	446	283	3239	<13.0
M4		1517	8098 (a)	12493	56.2
P118C		61.7	129	1231	25.5
P264		240	318	1436	38.7
P371		54.1	82.2	376	9.37
P374		476	385	1517	8.10
P381		156	511	1050	<4.33
P382		103	329	785	37.1
B3		<0.833	3.31	56.1	<2.16
B7		3.69	6.64	46.7	<0.400
P378		265	253	963	<4.33
PA112		13.9	65.3	312	25.5

(a) The accuracy for PFOA for M4 is ±80%.

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
D09	100
P121	100
P119C	100
Effluent WWTP	100
Bemalingsstation	100
Collector Put	100
P27	100
P304	100
P305	100
P42	100
M4	100
P118C	100
P264	100
P265C	100
P371	100
P374	100
P381	100
P382	100
B3	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
BD24-3	1.0	0.100	Solution A	5.0	1.0mL extract + 1.0mL MeOH (*)
D09	1.0	0.100	Solution A	1.0	1.0mL extract + 10.0mL MeOH (*)
P121	1.0	0.05	Solution A	10.0	-
P119C	1.0	0.100	Solution A	5.0	1.0mL extract + 5.0mL MeOH (*)
Effluent WWTP	1.0	0.100	Solution A	1.0	-
Bemalingsstation	1.0	0.100	Solution A	5.0	-
Collector Put	1.0	0.100	Solution A	5.0	-
P27	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P304	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
P305	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
P42	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
M4	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P118C	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
P264	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
P371	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
P374	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
P381	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
P382	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
B3	1.0	0.100	Solution A	10.0	-
B7	1.0	0.05	Solution A	10.0	-
P378	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
PA112	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
Field Trip Blank	1.0	0.100	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	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, ^{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 ± 25% (± 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 0.0750 to 0.195 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.154 to 23.8 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 ± 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 ± 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
02 Febr 2016	LCS1	80	84.7	102	88.5	84.1	107	123	84.2

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 ± 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
2 July 2015	QC Lab Low inj1	10	119	118	120	124	101	88.2	112
	QC Lab High inj1	100	80.4	83.2	102	80.4	104	81.4	79.1
	QC Lab Low inj2	10	115	113	122	124	101	83.5	111
	QC Lab High inj2	100	75.3	78.2	96.9	75.6	98.7	75.1	75.4

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		46.7	63.8	1132	<4.33
D09		9514	95.8	1734	<23.8
P121		0.939	<0.322	11.4	<0.400
P119C		567	748	2169	29.2
Effluent WWTP		32.7	21.4	39.7	<0.410
Bemalingsstation		30.9	37.1	62.6	<2.16
Collector Put		15.4	37.1	115	3.56
P27	2922	20.3	360	1622	14.8
P304	195	122	507	965	6.30
P305	83.6	201	163	491	<4.33
P42	42.9	446	283	3239	<13.0
M4		1517	8098	12493	56.2
P118C		61.7	129	1231	25.5
P264		240	318	1436	38.7
P371		54.1	82.2	376	9.37
P374		476	385	1517	8.10
P381		156	511	1050	<4.33
P382		103	329	785	37.1
B3		<0.833	3.31	56.1	<2.16
B7		3.69	6.64	46.7	<0.400
P378		265	253	963	<4.33
PA112		13.9	65.3	312	25.5
Field Trip Blank	<2.11	<0.833	<1.74	<0.910	<2.16

Table 10. Sample Results ^{13}C-PFOA and ^{13}C-PFOS

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
BD24-3	135	113	105	85.2
D09	86.1	72.0	88.4	71.8
P121	62.2	104	47.1	76.4
P119C	101	84.4	118	95.7
Effluent WWTP	124	104	107	87.1
Bemalingsstation	123	103	112	90.9
Collector Put	136	113	121	98.2
P27	127	106	117	95.1
P304	119	99.8	113	92.0
P305	124	103	115	93.6
P42	118	98.9	118	95.7
M4	28.8	24.1(*)	86.3	70.1
P118C	136	113	107	86.7
P264	120	101	105	85.5
P371	117	98.0	110	89.0
P374	122	102	100	81.1
P381	118	98.9	115	93.5
P382	119	99.1	112	91.3
B3	135	113	117	95.2
B7	65.1	109	47.2	76.7
P378	118	99.0	104	84.6
PA112	131	110	114	93.0
Field Trip Blank	141	118	118	96.1

(*) recovery ^{13}C-PFOA < 70%

5. Conclusion.

- The recovery of ^{13}C-PFOA and ^{13}C-PFOS fell within the method acceptance criterion of 70-130%, except for one sample (recovery ^{13}C-PFOA for M4 is 24.1%)
- 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 Adriaan de Bourgraaf (ERM Belgium).

8. Signatures.

Geert De Smet,
Lab Operations Manager

Date March 7th, 2016

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date March 7th, 2016

I.A.C.
A Division of SGS Belgium NV

ADDENDUM to analytical report IAC15-07460

Beste Sandra,

In de week van maandag 4 april 2016 staat een nieuwe bemonsteringscampagne ingepland bij 3M.
Zou u voor vrijdag 25 maart de flesje die nodig zijn kunnen aanleveren in ons kantoor te Antwerpen-Berchem?

Ons adres is:
ERM NV t.a.v. Adriaan de Bourgraaf/Nanda Hermes
Posthoflei 5 bus 6
2600 Antwerpen-Berchem

In bijlage van deze e-mail kunt u een lijst terugvinden van de recipiënten die nodig zijn.
Er staat ook telkens bij vermeld welke parameters dienen te worden geanalyseerd.

Alvast bedankt.

Met vriendelijk groet,

Adriaan de Bourgraaf
Consultant

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Kind regards,

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2nd Aquifer P321 groundwater 15,5-16,5 16,7 quarterly x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Blokkersdijk nature reserve Blokkersdijkvijver –
standaard
surfacewater n.a. n.a. quarterly x FC-parameters (PFOS-PFOA-PFHS-PFOSA) sampling only on Thursday!
Blokkersdijk nature reserve P114bis groundwater 3,6-4,6 quarterly x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Effluent WWTP Effluent WWTP waste water n.a. quarterly x FC-parameters (PFOS-PFOA-PFHS-PFOSA) All results used for mass flux calculations -> SGS
Data aangevuld met maandelijkse staalname en analyse door
3M zwijndrecht; resultaten periodiek opvragen
Extration wells P&T PP02 remediation 3,2-6,2 quarterly x FC-parameters (PFOS-PFOA-PFHS-PFOSA-PFBS)
Extration wells P&T PP05 remediation 1,6-5,6 quarterly x FC-parameters (PFOS-PFOA-PFHS-PFOSA-PFBS)
Extreem hoge pH; Caution when sampling
Extration wells P&T PP07 remediation 2,0-6,0 quarterly x FC-parameters (PFOS-PFOA-PFHS-PFOSA-PFBS)
Extration wells P&T PP09 remediation 3,0-6,0 quarterly x FC-parameters (PFOS-PFOA-PFHS-PFOSA-PFBS)
Palingbeek & Tophatgracht 12 surfacewater n.a. quarterly x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Palingbeek & Tophatgracht 5 surfacewater n.a. quarterly x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Palingbeek & Tophatgracht Bemalingsstation surfacewater n.a. quarterly x FC-parameters (PFOS-PFOA-PFHS-PFOSA) All results used for mass flux calculations -> SGS
Sewer Collector put stormwater n.a. quarterly x FC-parameters (PFOS-PFOA-PFHS-PFOSA) All results used for mass flux calculations -> SGS
Data aangevuld met maandelijkse staalname en analyse door
3M zwijndrecht; resultaten periodiek opvragen

3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC16-01242-002

3M Lab Request Number: E16-0229

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: April 21st, 2016

Requester

Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000

1. Introduction - Summary.

At the request (Lab Request Number: E16-0229) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, one water sample was collected between April 5th and 7th, 2016 from 2 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 ± 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
Blokkersdijkvijver std (filter)	0.782	1.16	13.4	0.437 (a)
Blokkersdijkvijver std (niet filter)	0.476	0.963	1.69	0.032 (b)
P114bis	3.02	9.70	38.1	0.229 (c)

(a): The recovery of FOSA for BDV std (filter) is ±75%.
(b): The recovery of FOSA for BDV std (niet filter) is ±68%
(c): The recovery of FOSA for P114bis is ±36%

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 ($^{13}C_2$-PFHS, $^{13}C_4$-PFOA, $^{13}C_4$-PFOS, $^{13}C_7$-PFUdA) and internal standard solution ($^{13}C_3$-PFHS, $^{13}C_8$-PFOA, $^{13}C_8$-PFOS, $^{13}C_8$-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}C-PFC-SS	^{13}C-PFC-IS
Blokkersdijkvijver std (filter)	400	0.1	Solution A	0.25	-
		0.25	Solution B	-	1.0
Blokkersdijkvijver std (niet filter)	400	0.1	Solution A	0.25	-
		0.25	Solution B	-	1.0
P114bis	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0

Solution A = 1000 ng/mL (nominal) ^{13}C-PFC Surrogate Recovery Standards
Solution B = 1500 ng/mL (nominal) ^{13}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
Blokkersdijkvijver std (filter)	40	8
Blokkersdijkvijver std (niet filter)	40	8
P114bis	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)	0.029

2.4. Analysis.

All solutions and extracts were analyzed for the PFCs (PFHS, PFOA, PFOS, FOSA) and the surrogate recovery standards ($^{13}C_2$-PFHS, $^{13}C_4$-PFOA, $^{13}C_4$-PFOS, $^{13}C_7$-PFUdA) and internal standards ($^{13}C_3$-PFHS, $^{13}C_8$-PFOA, $^{13}C_8$-PFOS, $^{13}C_8$-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 5. 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 6. 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 7. 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 ± 25% (± 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 ± 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 ± 25% for accuracy.
All LCSs produced recoveries within the method acceptance criteria of ± 15% RPD for precision.
Table 8 summarize the LCS recovery results.

Table 8. 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
14 April 2016	LCSL01	0.125	115	89.0	104	124	81.2	121	95.4	106
	LCSL02	0.125	98.1	94.4	96.5	88.1	78.9	119	93.0	99.3
	Average		**106**	**91.7**	**100**	**106**	**80.1**	**120**	**94.2**	**103**
	%RPD		**16 (a)**	**5.9**	**7.1**	**34 (a)**	**2.9**	**1.8**	**2.5**	**6.2**
	LCSH 01	2.5	106	105	115	114	77.5	103	87.4	85.3
	LCSH 02	2.5	111	112	112	119	82.9	121	106	103
	Average		**108**	**109**	**113**	**116**	**80.2**	**112**	**96.6**	**94.4**
	%RPD		**4.1**	**6.8**	**2.7**	**4.3**	**6.7**	**16 (a)**	**19 (a)**	**19 (a)**

(a) The recovery of the RPD fell outside the method acceptance criterion of ±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 9 and 10 summarizes the results for the sample locations.

Table 9. Sample Results PFHS and PFOA.

	PFHS	$^{13}C_2$-PFHS	PFOA	$^{13}C_4$-PFOA
	ng/ml	%Rec	ng/ml	%Rec
Field Trip Blank	<0.025	85.4	<0.025	98.2
Blokkersdijkvijver std (filter)	0.782	84.9	1.16	98.0
Blokkersdijkvijver std (niet filter)	0.476	105	0.963	115
P114bis	3.02	114	9.70	114

Table 10. Sample Results PFOS and FOSA.

	PFOS	$^{13}C_4$-PFOS	FOSA	$^{13}C_7$-PFuDA
	ng/ml	%Rec	ng/ml	%Rec
Field Trip Blank	<0.025	93.8	<0.025	111
Blokkersdijkvijver std (filter)	13.4	77.6	0.437	25.6 (a)
Blokkersdijkvijver std (niet filter)	1.69	94.4	0.032	32.5 (a)
P114bis	38.1	116	0.229	64.8 (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 ± 30%, except for FOSA for the following sample locations:

- The accuracy for FOSA for BDV std (filter) is ± 75%.
- The accuracy for FOSA for BDV std (niet filter) is ± 68%.
- The accuracy for FOSA for P114bis is ± 36%.

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 Adriaan de Bourgraaf (ERM Belgium).

8. Signatures.

Geert De Smet,
Lab Operations Manager Date April 21st, 2016

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories Date April 21st, 2016

I.A.C.
A Division of SGS Belgium NV

SGS

3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC16-01242-001

3M Lab Request Number: E16-0229

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: April 21st, 2016

Requester

Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000

1. Introduction - Summary.

At the request (Lab Request Number: E16-0229) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected between April 5th and 7th, 2016 from 10 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 ($C_7F_{15}{}^{13}CF_2SO3^-$)
- 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 ± 30%.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
P321		603	2133	3310	<31.0
Effluent WWTP		18.5	17.4	53.3	0.554
Bemalingsstation		11.5	15.4	32.6	<1.55
Collector Put		16.3	47.8	186	8.53
PP02	28.5	1868	460	21118	<59.7
PP05	15960	3219	1090	184	<31.0
PP07	65.4	142	336	3979	79.2
PP09	20.8	312	486	2331	<31.0
12		12.9	50.1	206	<2.82
5		9.81	12.0	23.7	<1.55

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
Effluent WWTP	100
Bemalingsstation	100
Collector Put	100
PP02	100
PP05	100
PP07	100
PP09	100
12	100
5	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
P321	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
Effluent WWTP	1.0	0.100	Solution A	1.0	-
Bemalingsstation	1.0	0.100	Solution A	5.0	-
Collector Put	1.0	0.100	Solution A	5.0	-
PP02	1.0	0.100	Solution A	10.0	0.5mL extract + 10.0mL MeOH (*)
PP05	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP07	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP09	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
12	1.0	0.100	Solution A	10.0	-
5	1.0	0.100	Solution A	10.0	-
Field Trip Blank	1.0	0.100	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	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, ^{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 ± 25% (± 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 0.254 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.158 to 59.7 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 ± 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 ± 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
14 April 2016	LCS1	80	86.9	86.8	75.0	86.9	94.1	95.0	80.4

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 ± 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
2 July 2015	QC Lab Low inj1	10	124	99.2	120	124	120	87.7	102
	QC Lab High inj1	100	92.7	82.0	101	80.3	110	79.4	78.5
	QC Lab Low inj2	10	115	95.4	108	114	124	85.9	94.2
	QC Lab High inj2	100	86.2	75.0	96	76.9	108	78.7	75.2

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		603	2133	3310	<31.0
Effluent WWTP		18.5	17.4	53.3	0.554
Bemalingsstation		11.5	15.4	32.6	<1.55
Collector Put		16.3	47.8	186	8.53
PP02	28.5	1868	460	21118	<59.7
PP05	15960	3219	1090	184	<31.0
PP07	65.4	142	336	3979	79.2
PP09	20.8	312	486	2331	<31.0
12		12.9	50.1	206	<2.82
5		9.81	12.0	23.7	<1.55
Field Trip Blank	<0.855	<0.888	<1.12	<0.899	<2.82

Table 10. Sample Results ^{13}C-PFOA and ^{13}C-PFOS

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
P321	94.0	78.6	89.9	73.0
Effluent WWTP	64.8	108	44.1	71.6
Bemalingsstation	138	115	107	87.2
Collector Put	123	103	101	81.7
PP02	266	111	199	80.7
PP05	115	96.2	119	96.9
PP07	106	88.9	91.8	74.6
PP09	108	90.5	87.8	71.4
12	117	98.1	99.5	80.8
5	122	102	98.4	79.9
Field Trip Blank	132	110	108	87.6

5. Conclusion.

- The recovery of ^{13}C-PFOA and ^{13}C-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 Adriaan de Bourgraaf (ERM Belgium).

8. Signatures.

Geert De Smet,
Lab Operations Manager — Date April 21st, 2016

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories — Date April 21st, 2016

I.A.C.
A Division of SGS Belgium NV

ADDENDUM to analytical report IAC16-03846

Beste Sandra,

In de week van maandag 18 juli 2016 staat een nieuwe bemonsteringscampagne ingepland bij 3M.
Zou u voor vrijdag 15 juli de flesje die nodig zijn kunnen aanleveren in ons kantoor te Antwerpen-Berchem?

Ons adres is:
ERM NV t.a.v. Adriaan de Bourgraaf/Nanda Hermes
Posthoflei 5 bus 6
2600 Antwerpen-Berchem

In bijlage van deze e-mail kunt u een lijst terugvinden van de recipiënten die nodig zijn.
Er staat ook telkens bij vermeld welke parameters dienen te worden geanalyseerd.

Alvast bedankt.

Met vriendelijk groet,

Adriaan de Bourgraaf
Consultant

ERM Belgium
Posthoflei 5 bus 6
2600 Berchem (Antwerp)
Belgium

Tel: [redacted]
Mobile: [redacted]

www.erm.com
[redacted]

Zone ID Nature of
monitoring
Well screen
(mbgl)
Analyse Remarks

2nd Aquifer BD24-3 groundwater 17,0-18,0 x FC-parameters (PFOS-PFOA-PFHS-PFOSA) changed to quarterly from may 2016 onwards
2nd Aquifer BD24-4 groundwater 22,0-24,0 x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
2nd Aquifer D10 groundwater 15,0-16,0 x FC-parameters (PFOS-PFOA-PFHS-PFOSA) changed to semi-annually from May 2016 onwards, Close
follow-up on PFOA trend (+ PFOS)
2nd Aquifer D11 groundwater 14,9-15,9 x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
2nd Aquifer D14 groundwater 15,5-16,5 x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
2nd Aquifer P118A groundwater 22,5-23,5 x FC-parameters (PFOS-PFOA-PFHS-PFOSA) changed to annually from 2015 onwards
2nd Aquifer P118B groundwater 13,0-14,0 x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Effluent WWTP Effluent WWTP waste water n.a. x FC-parameters (PFOS-PFOA-PFHS-PFOSA) All results used for mass flux calculations -> SGS
Data aangevuld met maandelijkse staalname en analyse door
3M zwijndrecht; resultaten periodiek opvragen
Palingbeek & Tophatgracht Bemalingsstation surfacewater n.a. x FC-parameters (PFOS-PFOA-PFHS-PFOSA) All results used for mass flux calculations -> SGS
Sewer Collector put stormwater n.a. x FC-parameters (PFOS-PFOA-PFHS-PFOSA) All results used for mass flux calculations -> SGS
Data aangevuld met maandelijkse staalname en analyse door
3M zwijndrecht; resultaten periodiek opvragen
Source area - Building 16 K3 groundwater 3,6-5,6 x FC-parameters (PFOS-PFOA-PFHS-PFOSA), PFBS
Source area - Building 16 P21B groundwater 3,5-5,5 x FC-parameters (PFOS-PFOA-PFHS-PFOSA), PFBS Increased frequency to allow quarterly evaluation of the FCcontour
Source area - Building 16 P56 groundwater 1,2-3,2 x FC-parameters (PFOS-PFOA-PFHS-PFOSA), PFBS
Source area - WWTP L19 groundwater 2,8-4,8 x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Source area - WWTP P262 groundwater 6,2-7,2 x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Source area - WWTP P263 groundwater 7,4-8,4 x FC-parameters (PFOS-PFOA-PFHS-PFOSA) Increased frequency to allow quarterly evaluation of the FCcontour
Source area - WWTP P340 groundwater 4,0-6,0 x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Source area - WWTP P341 groundwater 3,6-5,6 x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Source area - WWTP P343 groundwater 4,8-6,8 x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Source area - WWTP P379 groundwater 3,5-5,5 x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Source area - WWTP P380 groundwater 3,5-5,5 x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Southern site boundary P372 groundwater 4,8-5,8 x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Southern site boundary PA109A groundwater 5,0-6,0 x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
Southern site boundary PA111A groundwater 4,0-6,0 x FC-parameters (PFOS-PFOA-PFHS-PFOSA)
UST tank farm P18 groundwater 1,4-3,4 x TPH Extreem hoge pH; Caution when sampling
Baseline samples (duplicates to 3M lab)
UST tank farm P28 groundwater 1,1-3,1 x TPH Extreem hoge pH; Caution when sampling
Baseline samples (duplicates to 3M lab)

Duplicate
sample for
ERM

3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC16-03846

3M Lab Request Number: E16-0554

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 17st, 2016

Requester

Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000

1. Introduction - Summary.

At the request (Lab Request Number: E16-0554) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected between August 20th and 29th, 2016 from 24 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 ($C_7F_{15}{}^{13}CF_2SO3^-$)
- 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 ± 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
BD24-3		54.3	112	1137	<2.80
BD24-4		4.36	12.5	17.8	<0.256
D10		142	551	22.1	<1.86
D11		160	400	969	28.04
D14		0.939	2.92	3.49	<0.256
P118A		755	1697	4467	<10.3
P118B		2710	2810	9944	<19.6
Effluent WWTP		3.28	1.70	80.3	0.604
Bemalingsstation		31.3	59.1	53.2	<0.512
Collector put		22.5	68.7	174	8.058
K3	1612	84316	<15.1	5615	<97.6
P21B	5909	8675	12826 (a)	48548	<97.6
P56	10.7	6.47	90.6	41.8	3.17
L19		68.9	380.0	7049	173
P262		<8.42	<1.59	266	39.2
P263		543	2734	9269	<19.8
P340		244	754	3375	32.0
P341		313	672	5129	27.0
P343		833	1428	6862	<10.3
P379		6.26	61.1	591	30.1
P380		34.2	98.6	763	52.5
P372		17.8	42.0	1126	23.8
PA109A		40.7	50.8	102	<0932
PA111A		1086	1154	1925	<10.3

(a): The recovery of PFOA for P21B is ± 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)
BD24-3	100
BD24-4	100
D10	100
D11	100
D14	100
P118A	100
P118B	100
Effluent WWTP	100
Bemalingsstation	100
Collector put	100
K3	100
P21B	100
P56	100
L19	100
P262	100
P263	100
P340	100
P341	100
P343	100
P379	100
P380	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
BD24-3	1.0	0.100	Solution A	10.0	1.0mL extract + 2.0mL MeOH (*)
BD24-4	1.0	0.05	Solution A	2.0	-
D10	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
D11	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
D14	1.0	0.05	Solution A	2.0	-
P118A	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P118B	1.0	0.100	Solution A	10.0	0.5mL extract + 10.0mL MeOH (*)
Effluent WWTP	1.0	0.100	Solution A	5.0	-
Bemalingsstation	1.0	0.100	Solution A	5.0	-
Collector put	1.0	0.100	Solution A	5.0	-
K3	1.0	0.500	Solution A	10.0	0.1mL extract + 10.0mL MeOH (*)
P21B	1.0	0.500	Solution A	10.0	0.1mL extract + 10.0mL MeOH (*)
P56	1.0	0.100	Solution A	10.0	-
L19	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P262	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P263	1.0	0.200	Solution A	10.0	0.5mL extract + 10.0mL MeOH (*)
P340	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
P341	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P343	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P379	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P380	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P372	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
PA109A	1.0	0.100	Solution A	10.0	-
PA111A	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
Field Trip Blank	1.0	0.100	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	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, ^{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 ± 25% (± 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 0.0130 to 0.0860 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.0397 to 99.9 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 ± 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 ± 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
10 August 2016	LCS1	80	121	106	121	91.4	107	107	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 ± 25% for accuracy, except for 13C-PFOA QC lab High inj1.
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 August 2016	QC Lab Low inj1	10	91.5	103	123	124	98.9	90.0	106
	QC Lab High inj1	100	81.5	75.2	104	60.7(a)	77.3	84.0	79.5
	QC Lab Low inj2	10	106	100	124	99.2	104	102	112
	QC Lab High inj2	100	121	92.1	124	75.0	97.2	90.6	76.7

(a): The recovery of ^{13}C-PFOA fell outside the method acceptance criterion of 75-125%.

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		54.3	112	1137	<2.80
BD24-4		4.36	12.5	17.8	<0.256
D10		142	551	22.1	<1.86
D11		160	400	969	28.04
D14		0.939	2.92	3.49	<0.256
P118A		755	1697	4467	<10.3
P118B		2710	2810	9944	<19.6
Effluent WWTP		3.28	1.70	80.3	0.604
Bemalingsstation		31.3	59.1	53.2	<0.512
Collector put		22.5	68.7	174	8.058
K3	1612	84316	<15.1	5615	<97.6
P21B	5909	8675	12826	48548	<97.6
P56	10.7	6.47	90.6	41.8	3.17
L19		68.9	380.0	7049	173
P262		<8.42	<1.59	266	39.2
P263		543	2734	9269	<19.8
P340		244	754	3375	32.0
P341		313	672	5129	27.0
P343		833	1428	6862	<10.3
P379		6.26	61.1	591	30.1
P380		34.2	98.6	763	52.5
P372		17.8	42.0	1126	23.8
PA109A		40.7	50.8	102	<0932
PA111A		1086	1154	1925	<10.3
Field Trip Blank	<0.488	<0.766	<0.144	<0.955	<0.932

Table 10. Sample Results ^{13}C-PFOA and ^{13}C-PFOS

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
BD24-3	144	126	139	113
BD24-4	72.9	127	70.3	114
D10	132	115	149	121
D11	148	129	151	122
D14	72.3	126	70.3	114
P118A	128	112	116	94.4
P118B	105	91.6	118	95.8
Effluent WWTP	149	130	154	125
Bemalingsstation	142	124	160	130
Collector put	144	126	140	114
K3	534	93.1	661	107
P21B	295	51.3 (a)	462	75.0
P56	142	123	158	128
L19	133	116	124	101
P262	128	111	129	105
P263	270	118	226	92.0
P340	138	120	104	84.8
P341	117	102	108	87.7
P343	113	98.5	125	102
P379	107	92.8	126	103
P380	117	102	135	109
P372	121	105	144	117
PA109A	138	120	143	116
PA111A	96.6	84.2	128	104
Field Trip Blank	140	122	155	126

(a): The recovery of ^{13}C-PFOA fell outside the method acceptance criterion of 70-130%.

All PFBS, PFHS, PFOA, PFOS and FOSA results have an accuracy of ± 30%, except for PFOA for the following sample location:

- The accuracy for PFOA for P21B is ± 50%.

5. Conclusion.

- The recovery of ^{13}C-PFOA and ^{13}C-PFOS fell within the method acceptance criterion of 70-130%, except for one sample (^{13}C-PFOA for sample 21B < 70%).
- 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 QC lab high inj 1 (^{13}C-PFOA is < 75%).

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 Adriaan de Bourgraaf (ERM Belgium).

8. Signatures.

Geert De Smet,
Lab Operations Manager

Date August 17th, 2016

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date August 17th, 2016

I.A.C.
A Division of SGS Belgium NV

3M Corporate Environmental Programs.
Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC16-03847

3M Lab Request Number: E16-0554

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 12th, 2016

Requester

Jim Kotsmith
3M Center, Bldg 224-5w-17
St. Paul, MN 55144-1000

1. Introduction.

At the request (Lab Request Number: E16-0554) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected on July 18th 2016 from 2 locations and analyzed for:

- Total Petroleum Hydrocarbons (TPH)

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

Samples were collected in a 1L glass container containing HCl as a preservative. All bottles were sent by SGS Belgium NV, Division IAC Laboratory.

2.2. Analysis.

2.2.1. Mineral oil.

Samples were analyzed according to CMA/3/R.1 and WAC/IV/B/025, the official Dutch method.

2.2.2. Volatile compounds.

Samples were analyzed according to CMA/3/E and WAC/IV/A/016, the official Dutch method.

3. Results.

Table 1-2 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	< 25	140	79	< 25	250
P28	1100	8000	2000	510	12000

*: no result; sample could not be analyzed due to severe matrixinterferences

Table 2. Volatile compounds.

Sample identification	**Concentration (in µg/L)**					
	Benzene	**Ethylbenzene**	**m- + p-xylene**	**o-xylene**	**Sum xylene**	**Toluene**
P18	0.58	< 0.50	< 0.30	< 0.15	< 0.45	< 0.50
P28	1.9	0.56	0.91	1.0	1.9	21

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.

Geert De Smet,
Lab Operations Manager

Date August 12th, 2016

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date August 12th, 2016

I.A.C.
A Division of SGS Belgium NV

Final Report

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

October 2015 Sampling

Laboratory Request Number: ISO15-0017

Report Date – Date of Last Signature

Testing Laboratory

3M Environment, Health, Safety, and Sustainability
3M Environmental 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 Environmental Laboratory

3M Environmental Laboratory Technical Director: William K. Reagen, Ph.D.
3M Principal Analytical Investigator: Susan Wolf
Report Author: Chelsie Grochow

Analytical Report ISO15-0017

Water Sample Analysis at 3M Antwerp, Belgium
October 2015 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M Environmental Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. Samples were collected between October 19, 2015 and October 22, 2015. Samples were returned to the 3M Environmental Laboratory on November 6, 2015 at ambient temperature for the analysis of Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO15-0017.

The 3M Environmental Laboratory prepared sample containers for twenty-six sampling locations. Each sample set consisted of a field sample and field sample duplicate. Twenty-one 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 prior to being sent to the field for sample collection. Sample location PP04 was not collected.

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.

ilac-MRA

ACCREDITED
Testing Cert. # 2052.01

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)**
ISO15-0017-001	D09; Sample	167	7720	1660	18.5
ISO15-0017-002	D09; Sample Dup	168	7670	1700	19.5
	Average	**168**	**7700**	**1680**	**19.0**
	%RPD Sample/Sample Dup	**0.60**	**0.65**	**2.4**	**5.3**
ISO15-0017-004	P121; Sample	0.301	<0.189	7.64	0.351
ISO15-0017-005	P121; Sample Dup	0.265	<0.189	7.28	0.269
	Average	**0.283**	**<0.189**	**7.46**	**0.310**
	%RPD Sample/Sample Dup	**13**	**NA**	**4.8**	**26 (3)**
ISO15-0017-007	3M vijver; Sample	1.07	1.19	4.95	0.0583
ISO15-0017-008	3M vijver; Sample Dup	1.05	1.12	4.60	0.0492
	Average	**1.06**	**1.16**	**4.78**	**0.0538**
	%RPD Sample/Sample Dup	**1.9**	**6.1**	**7.3**	**17**
ISO15-0017-010	Blokkersdijkvijver std; Sample	1.59	2.36	7.65	0.356
ISO15-0017-011	Blokkersdijkvijver std; Sample Dup	1.52	2.13	6.71	0.310
	Average	**1.56**	**2.25**	**7.18**	**0.333**
	%RPD Sample/Sample Dup	**4.5**	**10**	**13**	**14**
ISO15-0017-013	L22; Sample	1.09	0.505	38.5	0.214
ISO15-0017-014	L22; Sample Dup	1.09	0.453	38.6	0.218
	Average	**1.09**	**0.479**	**38.6**	**0.216**
	%RPD Sample/Sample Dup	**0.0**	**11**	**0.26**	**1.9**
ISO15-0017-016	L4; Sample	2.75	2.75	46.3	0.657
ISO15-0017-017	L4; Sample Dup	2.78	2.79	46.1	0.578
	Average	**2.77**	**2.77**	**46.2**	**0.618**
	%RPD Sample/Sample Dup	**1.1**	**1.4**	**0.43**	**13**
ISO15-0017-019	P114bis; Sample	4.39	1.44	13.9	0.0758
ISO15-0017-020	P114bis; Sample Dup	4.37	1.46	14.1	0.0596
	Average	**4.38**	**1.45**	**14.0**	**0.0677**
	%RPD Sample/Sample Dup	**0.46**	**1.4**	**1.4**	**24 (3)**
ISO15-0017-022	P116; Sample	1.59	0.688	29.1	0.219
ISO15-0017-023	P116; Sample Dup	1.84	0.772	40.0	0.285
	Average	**1.72**	**0.730**	**34.6**	**0.252**
	%RPD Sample/Sample Dup	**15**	**12**	**32 (3)**	**26 (3)**
ISO15-0017-025	P119C; Sample	900	596	4190	55.0
ISO15-0017-026	P119C; Sample Dup	809	543	3900	55.7
	Average	**855**	**570**	**4050**	**55.4**
	%RPD Sample/Sample Dup	**11**	**9.3**	**7.2**	**1.3**

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 ± 16%, PFBS ± 13%, PFHS ± 17%, and PFOS ± 16%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOSA ± 21%.

(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(4) The data uncertainty has been expanded for location PP02 for PFOA due to field matrix spike recovery. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA (2)**
ISO15-0017-027	Effluent WWTP; Sample	22.5	11.5	4.04	0.148
ISO15-0017-028	Effluent WWTP; Sample Dup	22.8	11.6	4.43	0.148
	Average	**22.7**	**11.6**	**4.24**	**0.148**
	%RPD Sample/Sample Dup	**1.3**	**0.87**	**9.2**	**0.0**
ISO15-0017-053	12; Sample	115	28.9	352	0.601
ISO15-0017-054	12; Sample Dup	115	28.7	342	0.475
	Average	**115**	**28.8**	**347**	**0.538**
	%RPD Sample/Sample Dup	**0.0**	**0.69**	**2.9**	**23 (3)**
ISO15-0017-056	13; Sample	103	27.3	302	0.733
ISO15-0017-057	13; Sample Dup	105	29.0	315	0.638
	Average	**104**	**28.2**	**309**	**0.686**
	%RPD Sample/Sample Dup	**1.9**	**6.0**	**4.2**	**14**
ISO15-0017-059	5; Sample	41.2	32.2	64.5	0.319
ISO15-0017-060	5; Sample Dup	42.7	32.4	63.9	0.272
	Average	**42.0**	**32.3**	**64.2**	**0.296**
	%RPD Sample/Sample Dup	**3.6**	**0.62**	**0.93**	**16**
ISO15-0017-062	Bemalingsstation; Sample	36.7	27.6	66.8	0.268
ISO15-0017-063	Bemalingsstation; Sample Dup	36.0	28.8	71.4	0.245
	Average	**36.4**	**28.2**	**69.1**	**0.257**
	%RPD Sample/Sample Dup	**1.9**	**4.3**	**6.7**	**9.0**
ISO15-0017-065	Collector put; Sample	59.8	19.4	163	9.99
ISO15-0017-066	Collector put; Sample Dup	59.1	19.2	156	10.2
	Average	**59.5**	**19.3**	**160**	**10.1**
	%RPD Sample/Sample Dup	**1.2**	**1.0**	**4.4**	**2.1**
ISO15-0017-068	P340; Sample	718	305	3770	33.1
ISO15-0017-069	P340; Sample Dup	678	303	3120	30.6
	Average	**698**	**304**	**3450**	**31.9**
	%RPD Sample/Sample Dup	**5.7**	**0.66**	**19**	**7.8**
ISO15-0017-070	B3-bis; Sample	36.4	7.76	149	0.653
ISO15-0017-071	B3-bis; Sample Dup	36.3	7.37	142	0.457
	Average	**36.4**	**7.57**	**146**	**0.555**
	%RPD Sample/Sample Dup	**0.28**	**5.2**	**4.8**	**35 (3)**
ISO15-0017-076	P321, Sample	1040	355	3980	17.4
ISO15-0017-077	P321, Sample Dup	1100	361	4230	12.6
	Average	**1070**	**358**	**4110**	**15.0**
	%RPD Sample/Sample Dup	**5.6**	**1.7**	**6.1**	**32 (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 ± 16%, PFBS ± 13%, PFHS ± 17%, and PFOS ± 16%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOSA ± 21%.

(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(4) The data uncertainty has been expanded location PP02 for PFOA due to field matrix spike recovery. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)				
3M LIMS ID	**Sample Description**	**PFOA**	**PFBS**	**PFHS**	**PFOS**	**PFOSA (2)**
ISO15-0017-030	PP01; Sample	40.6	35.9	9590	1720	86.8
ISO15-0017-031	PP01; Sample Dup	39.6	35.0	9710	1730	87.6
	Average	**40.1**	**35.5**	**9650**	**1730**	**87.2**
	%RPD Sample/Sample Dup	**2.5**	**2.5**	**1.2**	**0.58**	**0.92**
ISO15-0017-032	PP02; Sample	651	26.5	2860	22500	35.3
ISO15-0017-033	PP02; Sample Dup	660	27.1	3020	23200	31.0
	Average	**656 (4)**	**26.8**	**2940**	**22900**	**33.2**
	%RPD Sample/Sample Dup	**1.4**	**2.2**	**5.4**	**3.1**	**13**
ISO15-0017-037	PP05; Sample	2270	22500	5920	92.8	7.35
ISO15-0017-038	PP05; Sample Dup	2160	21100	5700	67.2	7.20
	Average	**2220**	**21800**	**5810**	**80.0**	**7.28**
	%RPD Sample/Sample Dup	**5.0**	**6.4**	**3.8**	**32 (3)**	**2.1**
ISO15-0017-040	PP06; Sample	130	26.0	37.5	1540	41.4
ISO15-0017-041	PP06; Sample Dup	120	23.4	34.3	1420	38.1
	Average	**125**	**24.7**	**35.9**	**1480**	**39.8**
	%RPD Sample/Sample Dup	**8.0**	**11**	**8.9**	**8.1**	**8.3**
ISO15-0017-043	PP07; Sample	462	113	136	3100	68.6
ISO15-0017-044	PP07; Sample Dup	460	116	136	3080	71.4
	Average	**461**	**115**	**136**	**3090**	**70.0**
	%RPD Sample/Sample Dup	**0.43**	**2.6**	**0.0**	**0.65**	**4.0**
ISO15-0017-045	PP08; Sample	270	12.8	93.7	9200	67.2
ISO15-0017-046	PP08; Sample Dup	282	14.8	100	8790	75.2
	Average	**276**	**13.8**	**96.9**	**9000**	**71.2**
	%RPD Sample/Sample Dup	**4.3**	**14**	**6.5**	**4.6**	**11**
ISO15-0017-048	PP09; Sample	363	16.0	265	2170	39.6
ISO15-0017-049	PP09; Sample Dup	378	15.9	270	2150	40.1
	Average	**371**	**16.0**	**268**	**2160**	**39.9**
	%RPD Sample/Sample Dup	**4.0**	**0.63**	**1.9**	**0.93**	**1.3**
ISO15-0017-050	PP10; Sample	88.3	3.69	17.2	625	171
ISO15-0017-051	PP10; Sample Dup	94.0	11.4	24.8	621	178
	Average	**91.2**	**7.5**	**21.0**	**623**	**175**
	%RPD Sample/Sample Dup	**6.3**	**102 (3)**	**36 (3)**	**0.64**	**4.0**

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 ± 16%, PFBS ± 13%, PFHS ± 17%, and PFOS ± 16%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOSA ± 21%.

(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(4) The data uncertainty has been expanded location PP02 for PFOA due to field matrix spike recovery. See section 4 for details.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M Environmental 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 between October 19, 2015 and October 22, 2015 in Nalgene^{TM} (high-density polyethylene) bottles prepared at the 3M Environmental 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. Collected sample bottles were returned to the laboratory at ambient temperature on November 6, 2015.

2.3 Sample Preparation

All samples were prepared for PFOA, PFBS, PFHS, and PFOS 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 9.9 mL of methanol. Diluted samples and LCSs were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

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 1.0 ng/mL). The laboratory control samples were then diluted with methanol in the same manner. The calibration standards and method blanks were also 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).

2.4 Analysis

All samples and quality control samples were analyzed for five target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). 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. 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 Kirk**	**ETS Buster**
Liquid Chromatograph	Agilent 1260	Agilent 1100
Analysis Method	ETS-8-044.3	ETS-8-044.3
Analysis Date	11/11/15	11/18/15, 11/20/15
Guard column	Betasil C18 (4.6 mm X 100 mm), 5 μ	Betasil C18 (4.6 mm X 100 mm), 5 μ
Analytical column	Betasil C18 (4.6 mm X 100 mm), 5 μ	Betasil C18 (4.6 mm X 100 mm), 5 μ
Injection Volume	5 μL	5 μL or 10 μL
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex API 4000
Ion Source	Turbo Spray	Turbo Spray
Polarity	Negative	Negative
Software	Analyst 1.6.2	Analyst 1.6.2

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis (ETS Kirk)				
Step Number	**Total Time (min)**	**Flow Rate (μL/min)**	**Percent A (2 mM ammonium acetate)**	**Percent B (Methanol)**
0	0.00	750	90.0	10.0
1	0.50	750	90.0	10.0
2	4.00	750	70.0	30.0
3	6.00	750	70.0	30.0
4	11.0	750	20.0	80.0
5	13.0	750	20.0	80.0
6	13.5	750	10.0	90.0
7	16.0	750	10.0	90.0
8	16.5	750	90.0	10.0
9	19.0	750	90.0	10.0
ETS-8-044.3 Analysis (ETS Buster)				
Step Number	**Total Time (min)**	**Flow Rate (μL/min)**	**Percent A (2 mM ammonium acetate)**	**Percent B (Methanol)**
0	0.00	750	97.0	3.0
1	0.50	750	97.0	3.0
2	4.00	750	70.0	30.0
3	6.00	750	70.0	30.0
4	11.0	750	20.0	80.0
5	13.0	750	20.0	80.0
6	13.5	750	10.0	90.0
7	16.0	750	10.0	90.0
8	16.5	750	97.0	3.0
9	19.0	750	97.0	3.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard (1)	Mass Transition Q1/Q3
PFOA	413/369	NA	NA
	413/219		
	413/169		
PFBS	299/99	NA	NA
	299/80		
PFHS	399/99	NA	NA
	399/80		
PFOS	499/99	NA	NA
	499/80		
	499/130		
PFOSA	498/78	[$^{13}C_8$]-PFOSA	506/78
[$^{13}C_4$]-PFOA	417/372	NA	NA
[$^{13}C_4$]-PFOS	503/80	NA	NA

NA = Not Applicable

Dwell time was 20 or 100 msec for each transition. The individual transitions were summed to produce a “total ion chromatogram” (TIC), which was used for quantitation.

(1) Internal standards were only used for the analysis of PFOSA.

3 Data Analysis

3.1 Calibration

11/11/15 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 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.

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 100 mL of 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 were analyzed on 11/18/15 and from 0.025 ng/mL to 1.0 ng/mL on 11/20/15. Prior to analysis, the calibration standards were diluted 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±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) 11/11/15 Analysis	LOQ, ng/mL (1) 11/18/15 Analysis	LOQ, ng/mL (1) 11/20/15 Analysis
PFOA	0.0192	NA	NA
PFBS	0.0200	NA	NA
PFHS	0.0189	NA	NA
PFOS	0.0464	NA	NA
PFOSA	NA	0.100	0.0250

NA = Not Applicable
(1) 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/solvent 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 [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS were also 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:

- The high level LCSs were prepared above the resulting ULOQ (50 ng/mL) for PFHS.
- One low level LCS was calculated as an outlier for PFHS. The outlier is not included in the RPD or average LCS calculations nor was it used to calculate analytical method uncertainty.
- The low level LCSs had an average recovery of 138% for PFOSA. The low LCSs were re-injected due to suspected carry-over, which resulted in a similar recovery. Therefore, the low LCSs were re-diluted on 11/20/15, which resulted in an average recovery of 121%. The re-diluted low LCSs were used to assess method uncertainty.

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 11/11/15	**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-151110-1	100	104	104	100	101	101
LCS-151110-2	100	98.1	98.1	100	99.2	99.2
LCS-151110-3	100	96.7	96.7	100	95.3	95.3
Average ± %RSD	**99.6% ± 3.9%**			**98.5% ± 3.0%**		
LCS-151110-4	5000	5370	107	5000	5340	107
LCS-151110-5	5000	5120	102	5000	4940	98.8
LCS-151110-6	5000	5400	108	5000	5490	110
Average ± %RSD	**106% ± 3.0%**			**105% ± 5.5%**		
LCS-151110-7	35000	32500	92.9	35000	36000	103
LCS-151110-8	35000	32400	92.6	35000	35200	100
LCS-151110-9	35000	31800	90.9	35000	34500	98.6
Average ± %RSD	**92.1% ± 1.2%**			**101% ± 2.2%**		

ETS-8-044.3 External Calibration Analyzed 11/11/15	**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-151110-1	100	NA (1)	NA (1)	100	101	101
LCS-151110-2	100	101	101	100	97.0	97.0
LCS-151110-3	100	100	100	100	95.4	95.4
Average ± %RSD/RPD	**101% ± 1.0%**			**97.8% ± 2.9%**		
LCS-151110-4	5000	5280	106	5000	5580	112
LCS-151110-5	5000	4950	98.9	5000	4900	98.0
LCS-151110-6	5000	5300	106	5000	5490	110
Average ± %RSD	**104% ± 4.0%**			**107% ± 7.1%**		
LCS-151110-7	35000	>ULOQ	NA	35000	40200	115
LCS-151110-8	35000	>ULOQ	NA	35000	39100	112
LCS-151110-9	35000	>ULOQ	NA	35000	38200	109
Average ± %RSD	**NA (2)**			**112% ± 2.7%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCS is an outlier and was not included in the accuracy or precision calculations.
(2) LCSs prepared above the resulting ULOQ.
(3) LCSs do not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 11/11/15	**[$^{13}C_4$]-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-151110-1	99.8	108	108	95.6	104	109
LCS-151110-2	99.8	111	112	95.6	101	106
LCS-151110-3	99.8	106	106	95.6	102	107
Average ± %RSD	**109% ± 2.8%**			**107% ± 1.4%**		
LCS-151110-4	998	978	98.0	956	926	96.9
LCS-151110-5	998	983	98.5	956	918	96.1
LCS-151110-6	998	988	99.0	956	918	96.1
Average ± %RSD	**98.5% ± 0.51%**			**96.4% ± 0.48%**		

ETS-8-044.3 Internal Calibration Analyzed 11/18/15	**PFOSA**		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-151118-1	0.198	0.236	119
LCS-151118-2	0.198	0.255	129
LCS-151118-3	0.198	0.227	115
Average ± %RSD	**121% ± 6.0% (3)**		
LCS-151118-4	19.8	22.0	111
LCS-151118-5	19.8	21.7	110
LCS-151118-6	19.8	21.7	109
Average ± %RSD	**110% ± 0.91%**		
LCS-151118-7	139	149	107
LCS-151118-8	139	145	104
LCS-151118-9	139	148	106
Average ± %RSD	**106% ± 1.4%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCS is an outlier and was not included in the accuracy or precision calculations.
(2) LCSs prepared above the resulting ULOQ.
(3) LCSs do not meet acceptance criteria of 100 ± 20%.

3.7 Analytical Method 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-12-012.3. 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%.

The method uncertainty for PFOSA using internal calibration was calculated at ±11% following ETS-12-012.3; however, the method uncertainty was expanded to ±21% based on the average recovery of the low level LCSs.

Table 8. Analytical Method Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	7.76	±16%
PFBS	External	6.46	±13%
PFHS	External	8.54	±17%
PFOS	External	8.11	±16%
PFOSA	Internal	NA	±21%

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 contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. The stock solutions of PFOS and PFOA used to prepare the field matrix spikes were prepared from a different reference source than the one used to prepare the calibration standards. 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 $^{13}C_4$-PFOA and $^{13}C_4$-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles prior to sample collection. The $^{13}C_4$- PFOA was selected to represent the C8 perfluorocarboxylic acid. The $^{13}C_4$-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids and PFOSA. 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.

$$\boxed{\text{FMS Recovery} = \frac{(\text{Sample Concentration of FMS} - \text{Average Concentration : Field Sample \& Field Sample Dup.})}{\text{Spike Concentration}} * 100\%}$$

Table 9. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
D09, PP05	FMS	19.2	20.0	20.0	18.5	20.0
P121, Blokkersdijkvijver – standard, L4, P114bis, P116	FMS	1.92	2.00	2.00	1.85	2.00
Effluent WWTP	FMS	11.9	12.0	12.0	11.9	2.00
PP10	FMS	519	520	520	519	20.0
PP06, B3-bis	FMS	198	200	200	196	50.0
12, 13, 5, Bemalingsstation	FMS	202	202	202	202	2.00
Collector put	FMS	207	208	208	206	20.0
P321	FMS	2020	2020	2020	2020	20.0
3M vijver, L22 (1)	FMS	1.72	1.79	1.79	1.66	1.79
PP02 (1)	FMS	449	450	450	449	17.3
PP08 (1)	FMS	173	175	175	171	43.7
Trip Blank	Low	11.9	12.0	12.0	11.9	2.00
	High	198	200	200	196	50.0

(1) FMS concentration was adjusted due to sample bottle being overfilled by greater than 10%.

4 Data Summary and Discussion

Tables 10-31 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. The field matrix spike concentrations were selected based on previous results for these sampling locations. The spike level, at times, 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. Surrogate recovery standards are summarized in Table 32. All surrogate recovery standards met method acceptance criteria.

Sample location PP02: Field matrix spike recovery was 134% for PFOA. Since the %bias of the FMS sample (±34%) is greater than the overall method uncertainty (±16%), the data uncertainty has been expanded to ±34% for PFOA.

Sample location PP10: PFBS had a sample/sample duplicate RPD of 102%. Since the RPD was greater than 50%, the samples were re-analyzed which confirmed the high RPD values. Results from the initial analysis are reported.

Table 10. Location ID: D09

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-001	D09; Sample	167	NA	7720	NA
ISO15-0017-002	D09; Sample Dup	168	NA	7670	NA
ISO15-0017-003	D09; FMS	189	NC	7840	NC
Average Concentration (ng/mL) ± %RPD		**168 ng/mL ± 0.60%**		**7700 ng/mL ± 0.65%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-001	D09; Sample	1660	NA	18.5	NA
ISO15-0017-002	D09; Sample Dup	1700	NA	19.5	NA
ISO15-0017-003	D09; FMS	1760	NC	42.2	116
Average Concentration (ng/mL) ± %RPD		**1680 ng/mL ± 2.4%**		**19.0 ng/mL ± 5.3%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 11. Location ID: P121

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-004	P121; Sample	0.301	NA	<0.189	NA
ISO15-0017-005	P121; Sample Dup	0.265	NA	<0.189	NA
ISO15-0017-006	P121; FMS	2.14	96.9	2.18	109
Average Concentration (ng/mL) ± %RPD		**0.283 ng/mL ± 13%**		**<0.189 ng/mL**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-004	P121; Sample	7.64	NA	0.351	NA
ISO15-0017-005	P121; Sample Dup	7.28	NA	0.269	NA
ISO15-0017-006	P121; FMS	8.85	NC	2.32	101
Average Concentration (ng/mL) ± %RPD		**7.46 ng/mL ± 4.8%**		**0.310 ng/mL ± 26% (1)**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Sample/sample duplicate RPD does not meet acceptance criteria of ≤20%.

Table 12. Location ID: 3M vijver

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO15-0017-007	3M vijver; Sample	1.07	NA	1.19	NA
ISO15-0017-008	3M vijver; Sample Dup	1.05	NA	1.12	NA
ISO15-0017-009	3M vijver; FMS	2.92	108	3.17	112
Average Concentration (ng/mL) ± %RPD		**1.06 ng/mL ± 1.9%**		**1.16 ng/mL ± 6.1%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO15-0017-007	3M vijver; Sample	4.95	NA	0.0583	NA
ISO15-0017-008	3M vijver; Sample Dup	4.60	NA	0.0492	NA
ISO15-0017-009	3M vijver; FMS	6.52	NC	1.83	99.0 (1)
Average Concentration (ng/mL) ± %RPD		**4.78 ng/mL ± 7.3%**		**0.0538 ng/mL ± 17%**	

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 13. Location ID: Blokkersdijkvijver standard

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO15-0017-010	Blokkersdijkvijver std; Sample	1.59	NA	2.36	NA
ISO15-0017-011	Blokkersdijkvijver std; Sample Dup	1.52	NA	2.13	NA
ISO15-0017-012	Blokkersdijkvijver std; FMS	3.45	98.9	4.15	95.3
Average Concentration (ng/mL) ± %RPD		**1.56 ng/mL ± 4.5%**		**2.25 ng/mL ± 10%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO15-0017-010	Blokkersdijkvijver std; Sample	7.65	NA	0.356	NA
ISO15-0017-011	Blokkersdijkvijver std; Sample Dup	6.71	NA	0.310	NA
ISO15-0017-012	Blokkersdijkvijver std; FMS	9.31	NC	2.29	97.9
Average Concentration (ng/mL) ± %RPD		**7.18 ng/mL ± 13%**		**0.333 ng/mL ± 14%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 14. Location ID: L22

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-013	L22; Sample	1.09	NA	0.505	NA
ISO15-0017-014	L22; Sample Dup	1.09	NA	0.453	NA
ISO15-0017-015	L22; FMS	3.05	114	2.43	109
Average Concentration (ng/mL) ± %RPD		**1.09 ng/mL ± 0.0%**		**0.479 ng/mL ± 11%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-013	L22; Sample	38.5	NA	0.214	NA
ISO15-0017-014	L22; Sample Dup	38.6	NA	0.218	NA
ISO15-0017-015	L22; FMS	38.8	NC	1.98	98.3
Average Concentration (ng/mL) ± %RPD		**38.6 ng/mL ± 0.26%**		**0.216 ng/mL ± 1.9%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 15. Location ID: L4

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-016	L4; Sample	2.75	NA	2.75	NA
ISO15-0017-017	L4; Sample Dup	2.78	NA	2.79	NA
ISO15-0017-018	L4; FMS	4.84	108	4.94	109
Average Concentration (ng/mL) ± %RPD		**2.77 ng/mL ± 1.1%**		**2.77 ng/mL ± 1.4%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-016	L4; Sample	46.3	NA	0.657	NA
ISO15-0017-017	L4; Sample Dup	46.1	NA	0.578	NA
ISO15-0017-018	L4; FMS	50.1	NC	2.61	99.6
Average Concentration (ng/mL) ± %RPD		**46.2 ng/mL ± 0.43%**		**0.618 ng/mL ± 13%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 16. Location ID: P114bis

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-019	P114bis; Sample	4.39	NA	1.44	NA
ISO15-0017-020	P114bis; Sample Dup	4.37	NA	1.46	NA
ISO15-0017-021	P114bis; FMS	6.08	NC	3.59	107
Average Concentration (ng/mL) ± %RPD		**4.38 ng/mL ± 0.46%**		**1.45 ng/mL ± 1.4%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-019	P114bis; Sample	13.9	NA	0.0758	NA
ISO15-0017-020	P114bis; Sample Dup	14.1	NA	0.0596	NA
ISO15-0017-021	P114bis; FMS	17.8	NC	2.06	99.6 (1)
Average Concentration (ng/mL) ± %RPD		**14.0 ng/mL ± 1.4%**		**0.0677 ng/mL ± 24% (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 does not meet acceptance criteria of ≤20%.

Table 17. Location ID: P116

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-022	P116; Sample	1.59	NA	0.688	NA
ISO15-0017-023	P116; Sample Dup	1.84	NA	0.772	NA
ISO15-0017-024	P116; FMS	3.58	97.3	2.76	102
Average Concentration (ng/mL) ± %RPD		**1.72 ng/mL ± 15%**		**0.730 ng/mL ± 12%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-022	P116; Sample	29.1	NA	0.219	NA
ISO15-0017-023	P116; Sample Dup	40.0	NA	0.285	NA
ISO15-0017-024	P116; FMS	33.0	NC	2.29	102
Average Concentration (ng/mL) ± %RPD		**34.6 ng/mL ± 32% (1)**		**0.252 ng/mL ± 26% (1)**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Sample/sample duplicate RPD does not meet acceptance criteria of ≤20%.

Table 18. Location ID: Effluent WWTP

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-027	Effluent WWTP; Sample	22.5	NA	11.5	NA
ISO15-0017-028	Effluent WWTP; Sample Dup	22.8	NA	11.6	NA
ISO15-0017-029	Effluent WWTP; FMS	33.5	91.1	22.7	92.9
Average Concentration (ng/mL) ± %RPD		**22.7 ng/mL ± 1.3%**		**11.6 ng/mL ± 0.87%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-027	Effluent WWTP; Sample	4.04	NA	0.148	NA
ISO15-0017-028	Effluent WWTP; Sample Dup	4.43	NA	0.148	NA
ISO15-0017-029	Effluent WWTP; FMS	13.8	80.7	2.23	104 (1)
Average Concentration (ng/mL) ± %RPD		**4.24 ng/mL ± 9.2%**		**0.148 ng/mL ± 0.0%**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.

Table 19. Location ID: PP02

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-032	PP02; Sample	651	NA	26.5	NA	2860	NA
ISO15-0017-033	PP02; Sample Dup	660	NA	27.1	NA	3020	NA
ISO15-0017-034	PP02; FMS	1260	134 (1)	574	122 (3)	3770	NC
Average Concentration (ng/mL) ± %RPD		**656 ng/mL ± 1.4% (2)**		**26.8 ng/mL ± 2.2%**		**2940 ng/mL ± 5.4%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-032	PP02; Sample	22500	NA	35.3	NA
ISO15-0017-033	PP02; Sample Dup	23200	NA	31.0	NA
ISO15-0017-034	PP02; FMS	28600	NC	52.2	110
Average Concentration (ng/mL) ± %RPD		**22900 ng/mL ± 3.1%**		**33.2 ng/mL ± 13%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%.
(2) Data uncertainty has been expanded to ±34%.
(3) FMS concentration greater than 10 times the sample concentration.

Table 20. Location ID: PP05

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-037	PP05; Sample	2270	NA	22500	NA	5920	NA
ISO15-0017-038	PP05; Sample Dup	2160	NA	21100	NA	5700	NA
ISO15-0017-039	PP05; FMS	2120	NC	20400	NC	5520	NC
Average Concentration (ng/mL) ± %RPD		**2220 ng/mL ± 5.0%**		**21800 ng/mL ± 6.4%**		**5810 ng/mL ± 3.8%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-037	PP05; Sample	92.8	NA	7.35	NA
ISO15-0017-038	PP05; Sample Dup	67.2	NA	7.20	NA
ISO15-0017-039	PP05; FMS	80.8	NC	26.6	96.6
Average Concentration (ng/mL) ± %RPD		**80.0 ng/mL ± 32% (1)**		**7.28 ng/mL ± 2.1%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Sample/sample duplicate RPD does not meet acceptance criteria of ≤20%.

Table 21. Location ID: PP06

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-040	PP06; Sample	130	NA	26.0	NA	37.5	NA
ISO15-0017-041	PP06; Sample Dup	120	NA	23.4	NA	34.3	NA
ISO15-0017-042	PP06; FMS	327	102	230	103	230	97.1
Average Concentration (ng/mL) ± %RPD		**125 ng/mL ± 8.0%**		**24.7 ng/mL ± 11%**		**35.9 ng/mL ± 8.9%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-040	PP06; Sample	1540	NA	41.4	NA
ISO15-0017-041	PP06; Sample Dup	1420	NA	38.1	NA
ISO15-0017-042	PP06; FMS	1750	NC	94.0	109
Average Concentration (ng/mL) ± %RPD		**1480 ng/mL ± 8.1%**		**39.8 ng/mL ± 8.3%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 22. Location ID: PP08

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-045	PP08; Sample	270	NA	12.8	NA	93.7	NA
ISO15-0017-046	PP08; Sample Dup	282	NA	14.8	NA	100	NA
ISO15-0017-047	PP08; FMS	489	123	199	106 (1)	291	111
Average Concentration (ng/mL) ± %RPD		**276 ng/mL ± 4.3%**		**13.8 ng/mL ± 14%**		**96.9 ng/mL ± 6.5%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-045	PP08; Sample	9200	NA	67.2	NA
ISO15-0017-046	PP08; Sample Dup	8790	NA	75.2	NA
ISO15-0017-047	PP08; FMS	9060	NC	114	98.0
Average Concentration (ng/mL) ± %RPD		**9000 ng/mL ± 4.6%**		**71.2 ng/mL ± 11%**	

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 23. Location ID: PP10

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-050	PP10; Sample	88.3	NA	3.69	NA	17.2	NA
ISO15-0017-051	PP10; Sample Dup	94.0	NA	11.4	NA	24.8	NA
ISO15-0017-052	PP10; FMS	597	97.4	524	99.3 (1)	519	95.8 (1)
Average Concentration (ng/mL) ± %RPD		**91.2 ng/mL ± 6.3%**		**7.55 ng/mL ± 102% (2)**		**21.0 ng/mL ± 36% (2)**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-050	PP10; Sample	625	NA	171	NA
ISO15-0017-051	PP10; Sample Dup	621	NA	178	NA
ISO15-0017-052	PP10; FMS	1120	95.8	197	NC
Average Concentration (ng/mL) ± %RPD		**623 ng/mL ± 0.64%**		**175 ng/mL ± 4.0%**	

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.
(2) Sample/sample duplicate RPD does not meet acceptance criteria of ≤20%.

Table 24. Location ID: 12

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-053	12; Sample	115	NA	28.9	NA
ISO15-0017-054	12; Sample Dup	115	NA	28.7	NA
ISO15-0017-055	12; FMS	314	98.6	240	105
Average Concentration (ng/mL) ± %RPD		**115 ng/mL ± 0.0%**		**28.8 ng/mL ± 0.69%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-053	12; Sample	352	NA	0.601	NA
ISO15-0017-054	12; Sample Dup	342	NA	0.475	NA
ISO15-0017-055	12; FMS	560	106	2.41	93.6
Average Concentration (ng/mL) ± %RPD		**347 ng/mL ± 2.9%**		**0.538 ng/mL ± 23% (1)**	

NA = Not Applicable
(1) Sample/sample duplicate RPD does not meet acceptance criteria of ≤20%.

Table 25. Location ID: 13

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-056	13; Sample	103	NA	27.3	NA
ISO15-0017-057	13; Sample Dup	105	NA	29.0	NA
ISO15-0017-058	13; FMS	286	90.1	219	94.5
Average Concentration (ng/mL) ± %RPD		**104 ng/mL ± 1.9%**		**28.2 ng/mL ± 6.0%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-056	13; Sample	302	NA	0.733	NA
ISO15-0017-057	13; Sample Dup	315	NA	0.638	NA
ISO15-0017-058	13; FMS	502	95.9	2.60	95.7
Average Concentration (ng/mL) ± %RPD		**309 ng/mL ± 4.2%**		**0.686 ng/mL ± 14%**	

NA = Not Applicable

Table 26. Location ID: 5

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-059	5; Sample	41.2	NA	32.2	NA
ISO15-0017-060	5; Sample Dup	42.7	NA	32.4	NA
ISO15-0017-061	5; FMS	223	89.7	225	95.4
Average Concentration (ng/mL) ± %RPD		**42.0 ng/mL ± 3.6%**		**32.3 ng/mL ± 0.62%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-059	5; Sample	64.5	NA	0.319	NA
ISO15-0017-060	5; Sample Dup	63.9	NA	0.272	NA
ISO15-0017-061	5; FMS	225	79.7	2.31	101
Average Concentration (ng/mL) ± %RPD		**64.2 ng/mL ± 0.93%**		**0.296 ng/mL ± 16%**	

NA = Not Applicable

Table 27. Location ID: Bemalingsstation

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-062	Bemalingsstation; Sample	36.7	NA	27.6	NA
ISO15-0017-063	Bemalingsstation; Sample Dup	36.0	NA	28.8	NA
ISO15-0017-064	Bemalingsstation; FMS	212	87.0	212	91.0
Average Concentration (ng/mL) ± %RPD		**36.4 ng/mL ± 1.9%**		**28.2 ng/mL ± 4.3%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-062	Bemalingsstation; Sample	66.8	NA	0.268	NA
ISO15-0017-063	Bemalingsstation; Sample Dup	71.4	NA	0.245	NA
ISO15-0017-064	Bemalingsstation; FMS	244	86.6	2.22	98.2
Average Concentration (ng/mL) ± %RPD		**69.1 ng/mL ± 6.7%**		**0.257 ng/mL ± 9.0%**	

NA = Not Applicable

Table 28. Location ID: Collector Put

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-065	Collector put; Sample	59.8	NA	19.4	NA
ISO15-0017-066	Collector put; Sample Dup	59.1	NA	19.2	NA
ISO15-0017-067	Collector put; FMS	257	95.6	229	101 (1)
Average Concentration (ng/mL) ± %RPD		**59.5 ng/mL ± 1.2%**		**19.3 ng/mL ± 1.0%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-065	Collector put; Sample	163	NA	9.99	NA
ISO15-0017-066	Collector put; Sample Dup	156	NA	10.2	NA
ISO15-0017-067	Collector put; FMS	364	99.3	30.3	101
Average Concentration (ng/mL) ± %RPD		**160 ng/mL ± 4.4%**		**10.1 ng/mL ± 2.1%**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.

Table 29. Location ID: B3-bis

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-070	B3-bis; Sample	36.4	NA	7.76	NA
ISO15-0017-071	B3-bis; Sample Dup	36.3	NA	7.37	NA
ISO15-0017-072	B3-bis; FMS	228	96.8	203	97.7 (1)
Average Concentration (ng/mL) ± %RPD		**36.4 ng/mL ± 0.28%**		**7.57 ng/mL ± 5.2%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-070	B3-bis; Sample	149	NA	0.653	NA
ISO15-0017-071	B3-bis; Sample Dup	142	NA	0.457	NA
ISO15-0017-072	B3-bis; FMS	310	83.8	51.2	101 (1)
Average Concentration (ng/mL) ± %RPD		**146 ng/mL ± 4.8%**		**0.555 ng/mL ± 35% (2)**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.
(2) Sample/sample duplicate RPD does not meet acceptance criteria of ≤20%.

Table 30. Location ID: P321

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-076	P321, Sample	1040	NA	355	NA
ISO15-0017-077	P321, Sample Dup	1100	NA	361	NA
ISO15-0017-078	P321, FMS	3000	95.5	2510	107
Average Concentration (ng/mL) ± %RPD		**1070 ng/mL ± 5.6%**		**358 ng/mL ± 1.7%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-076	P321, Sample	3980	NA	17.4	NA
ISO15-0017-077	P321, Sample Dup	4230	NA	12.6	NA
ISO15-0017-078	P321, FMS	6100	NC	29.1	70.5
Average Concentration (ng/mL) ± %RPD		**4110 ng/mL ± 6.1%**		**15.0 ng/mL ± 32% (1)**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Sample/sample duplicate RPD does not meet acceptance criteria of ≤20%.

Table 31. Trip Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-073	Trip Blank; Sample	<0.192	NA	<0.200	NA	<0.189	NA
ISO15-0017-074	Trip Blank; FMS Low	12.2	102	12.2	102	11.9	99.2
ISO15-0017-075	Trip Blank; FMS High	210	106	212	106	208	104

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0017-073	Trip Blank; Sample	<0.464	NA	<0.100	NA
ISO15-0017-074	Trip Blank; FMS Low	11.9	100	2.11	106
ISO15-0017-075	Trip Blank; FMS High	205	104	50.6	101

NA = Not Applicable

Table 32. Surrogate Recovery Standard Results (1)

		Percent Recovery (%)	
3M LIMS ID	**Sample Description**	**[$^{13}C_4$]-PFOA**	**[$^{13}C_4$]-PFOS**
ISO15-0017-001	D09; Sample	103	93.0
ISO15-0017-002	D09; Sample Dup	102	97.8
ISO15-0017-003	D09; FMS	102	98.5
ISO15-0017-004	P121; Sample	107	108
ISO15-0017-005	P121; Sample Dup	103	99.7
ISO15-0017-006	P121; FMS	103	104
ISO15-0017-007	3M vijver; Sample	113	111
ISO15-0017-008	3M vijver; Sample Dup	113	112
ISO15-0017-009	3M vijver; FMS	116	111
ISO15-0017-010	Blokkersdijkvijver std; Sample	115	113
ISO15-0017-011	Blokkersdijkvijver std; Sample Dup	125	120
ISO15-0017-012	Blokkersdijkvijver std; FMS	106	107
ISO15-0017-013	L22; Sample	115	114
ISO15-0017-014	L22; Sample Dup	104	99.2
ISO15-0017-015	L22; FMS	94.2	89.5
ISO15-0017-016	L4; Sample	114	111
ISO15-0017-017	L4; Sample Dup	105	107
ISO15-0017-018	L4; FMS	110	108
ISO15-0017-019	P114bis; Sample	106	102
ISO15-0017-020	P114bis; Sample Dup	99.5	95.9
ISO15-0017-021	P114bis; FMS	106	106
ISO15-0017-022	P116; Sample	115	113
ISO15-0017-023	P116; Sample Dup	98.7	98.7
ISO15-0017-024	P116; FMS	105	104
ISO15-0017-025	P119C; Sample	113	103
ISO15-0017-026	P119C; Sample Dup	103	94.7
ISO15-0017-027	Effluent WWTP; Sample	94.8	95.3
ISO15-0017-028	Effluent WWTP; Sample Dup	106	103
ISO15-0017-029	Effluent WWTP; FMS	96.4	97.0
ISO15-0017-030	PP01; Sample	99.9	102
ISO15-0017-031	PP01; Sample Dup	101	98.7
ISO15-0017-032	PP02; Sample	101	91.6
ISO15-0017-033	PP02; Sample Dup	105	97.2
ISO15-0017-034	PP02; FMS	103	93.4
ISO15-0017-037	PP05; Sample	104	103
ISO15-0017-038	PP05; Sample Dup	105	98.7
ISO15-0017-039	PP05; FMS	101	99.7
ISO15-0017-040	PP06; Sample	103	103
ISO15-0017-041	PP06; Sample Dup	102	99.6
ISO15-0017-042	PP06; FMS	109	108

(1) The surrogate recovery standards $^{13}C_4$-PFOA and $^{13}C_4$-PFOS were added to the samples during sample preparation.

Table 32 continued. Surrogate Recovery Standard Results (1)

		Percent Recovery (%)	
3M LIMS ID	**Sample Description**	**[$^{13}C_4$]-PFOA**	**[$^{13}C_4$]-PFOS**
ISO15-0017-043	PP07; Sample	100	95.9
ISO15-0017-044	PP07; Sample Dup	106	102
ISO15-0017-045	PP08; Sample	98.9	88.2
ISO15-0017-046	PP08; Sample Dup	103	89.5
ISO15-0017-047	PP08; FMS	99.1	89.6
ISO15-0017-048	PP09; Sample	102	96.2
ISO15-0017-049	PP09; Sample Dup	114	112
ISO15-0017-050	PP10; Sample	98.7	97.5
ISO15-0017-051	PP10; Sample Dup	100	99.4
ISO15-0017-052	PP10; FMS	100	101
ISO15-0017-053	12; Sample	102	99.3
ISO15-0017-054	12; Sample Dup	93.3	92.1
ISO15-0017-055	12; FMS	95.5	89.7
ISO15-0017-056	13; Sample	100	93.7
ISO15-0017-057	13; Sample Dup	103	97.2
ISO15-0017-058	13; FMS	98.2	96.0
ISO15-0017-059	5; Sample	97.0	95.0
ISO15-0017-060	5; Sample Dup	98.2	97.0
ISO15-0017-061	5; FMS	94.1	93.0
ISO15-0017-062	Bemalingsstation; Sample	92.2	89.8
ISO15-0017-063	Bemalingsstation; Sample Dup	100	101
ISO15-0017-064	Bemalingsstation; FMS	91.3	91.7
ISO15-0017-065	Collector put; Sample	92.5	91.3
ISO15-0017-066	Collector put; Sample Dup	96.4	95.2
ISO15-0017-067	Collector put; FMS	95.6	93.3
ISO15-0017-068	P340; Sample	102	99.5
ISO15-0017-069	P340; Sample Dup	95.7	95.9
ISO15-0017-070	B3-bis; Sample	99.1	99.7
ISO15-0017-071	B3-bis; Sample Dup	99.5	98.2
ISO15-0017-072	B3-bis; FMS	97.3	96.5
ISO15-0017-076	P321, Sample	96.4	96.0
ISO15-0017-077	P321, Sample Dup	95.5	91.0
ISO15-0017-078	P321, FMS	95.1	92.9
ISO15-0017-073	Trip Blank; Sample	101	98.2
ISO15-0017-074	Trip Blank; FMS Low	106	103
ISO15-0017-075	Trip Blank; FMS High	103	100

(1) The surrogate recovery standards $^{13}C_4$-PFOA and $^{13}C_4$-PFOS 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 Environmental 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 10-32 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M Environmental Laboratory standard operating procedures.

7 Signatures

Chelsie Grochow, 3M Report Author

Susan T. Wolf, 3M Principal Analytical Investigator

William K. Reagen, Ph.D., 3M Environmental Laboratory Technical Director

The 3M Environmental 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 Environmental Laboratory.

Final Report

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

January 2016 Sampling

Laboratory Request Number: ISO16-0001

Report Date – Date of Last Signature

Testing Laboratory

3M Environment, Health, Safety, and Sustainability
3M Environmental Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Nicole Cauberghe
3M Belgium EHS Operations
3M Belgium; ZW018/0/33
Phone: [redacted]

ACCREDITED
Testing Cert. # 2052.01

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 Environmental Laboratory

3M Environmental Laboratory Technical Director: William K. Reagen, Ph.D.
3M Principal Analytical Investigator: Susan Wolf
Report Author: Chelsie Grochow

Analytical Report ISO16-0001

Water Sample Analysis at 3M Antwerp, Belgium
January 2016 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M Environmental Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. Samples were collected between January 11, 2016 and January 19, 2016. Samples were returned to the 3M Environmental Laboratory on February 1, 2016 at ambient temperature for the analysis of Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO16-0001.

The 3M Environmental Laboratory prepared sample containers for fifty-one sampling locations. Each sample set consisted of a field sample and field sample duplicate. Thirteen 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 prior to being sent to the field for sample collection. Sample location P265C was dry and therefore not collected.

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.

ilac-MRA
ACCREDITED
Testing Cert. # 2052.01

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)**
ISO16-0001-001	BD24-3; Sample	69.2	54.8	1270	2.79
ISO16-0001-002	BD24-3; Sample Dup	70.6	55.6	1270	2.85
	Average	**69.9**	**55.2**	**1270**	**2.82**
	%RPD Sample/Sample Dup	**2.0**	**1.4**	**0.0**	**2.1**
ISO16-0001-004	D09; Sample	162	13300	1970	21.5
ISO16-0001-005	D09; Sample Dup	160	13000	1980	21.1
	Average	**161**	**13200**	**1980**	**21.3**
	%RPD Sample/Sample Dup	**1.2**	**2.3**	**0.51**	**1.9**
ISO16-0001-006	P121; Sample	0.631	0.364	17.5	0.747
ISO16-0001-007	P121; Sample Dup	0.648	0.390	17.2	0.778
	Average	**0.640**	**0.377**	**17.4**	**0.763**
	%RPD Sample/Sample Dup	**2.7**	**6.9**	**1.7**	**4.1**
ISO16-0001-008	P321; Sample	2970	870	5460	0.497
ISO16-0001-009	P321; Sample Dup	2960	855	5480	0.421
	Average	**2970**	**863**	**5470**	**0.459**
	%RPD Sample/Sample Dup	**0.34**	**1.7**	**0.37**	**17**
ISO16-0001-010	3M vijver; Sample	0.959	0.434	3.28	<0.0250
ISO16-0001-011	3M vijver; Sample Dup	0.915	0.456	3.30	<0.0250
	Average	**0.937**	**0.445**	**3.29**	**<0.0250**
	%RPD Sample/Sample Dup	**4.7**	**4.9**	**0.61**	**NA**
ISO16-0001-012	Blokkersdijkvijver std; Sample	0.946	0.578	1.27	<0.0250
ISO16-0001-013	Blokkersdijkvijver std; Sample Dup	0.896	0.531	1.18	<0.0250
	Average	**0.921 (2)**	**0.555 (2)**	**1.23 (2)**	**<0.0250**
	%RPD Sample/Sample Dup	**5.4**	**8.5**	**7.3**	**NA**
ISO16-0001-014	L21; Sample	0.446	0.206	5.56	0.133
ISO16-0001-015	L21; Sample Dup	0.462	0.195	5.73	0.128
	Average	**0.454 (2)**	**0.201 (2)**	**5.65 (2)**	**0.131**
	%RPD Sample/Sample Dup	**3.5**	**5.5**	**3.0**	**3.8**
ISO16-0001-017	L22; Sample	0.456	0.218	5.56	0.113
ISO16-0001-018	L22; Sample Dup	0.485	0.207	5.21	0.118
	Average	**0.471 (2)**	**0.213 (2)**	**5.39 (2)**	**0.116**
	%RPD Sample/Sample Dup	**6.2**	**5.2**	**6.5**	**4.3**
ISO16-0001-019	L31; Sample	0.552	0.762	7.90	0.230
ISO16-0001-020	L31; Sample Dup	0.510	0.761	7.31	0.221
	Average	**0.531 (2)**	**0.762 (2)**	**7.61 (2)**	**0.226**
	%RPD Sample/Sample Dup	**7.9**	**0.13**	**7.8**	**4.0**

NA = Not Applicable
NR = Not Reportable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 19%, PFBS ± 15%, PFHS ± 17%, and PFOS ± 17%.

(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 ± 16%, and PFOSA ± 17%.

(3) Sample not reportable for PFOSA due to analyte area counts not meeting method blank criteria. See section 4 of the report for additional information.

(4) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(5) The data uncertainty has been expanded to ±37% for PFOA, ±38% for PFBS, and ±40% for PFHS due to field matrix spike recovery.

Table 1 continued. Sample Results Summary [1]

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA [2]**
ISO16-0001-021	L4; Sample	3.93	2.40	38.2	5.96
ISO16-0001-022	L4; Sample Dup	4.03	2.40	36.3	6.29
	Average	**3.98**	**2.40**	**37.3**	**6.13**
	%RPD Sample/Sample Dup	**2.5**	**0.0**	**5.1**	**5.4**
ISO16-0001-023	P114bis; Sample	3.67	1.29	4.35	NR [3]
ISO16-0001-024	P114bis; Sample Dup	3.57	1.27	4.22	<0.0250
	Average	**3.62**	**1.28**	**4.29**	**<0.0250**
	%RPD Sample/Sample Dup	**2.8**	**1.6**	**3.0**	**NA**
ISO16-0001-025	P115; Sample	4.10	2.35 [2]	0.566	<0.0250
ISO16-0001-026	P115; Sample Dup	4.25	2.25 [2]	0.622	<0.0250
	Average	**4.18 [2]**	**2.30 [2]**	**0.594 [2]**	**<0.0250**
	%RPD Sample/Sample Dup	**3.6**	**4.3**	**9.4**	**NA**
ISO16-0001-028	P116; Sample	0.921	0.507	8.60	0.0889
ISO16-0001-029	P116; Sample Dup	0.879	0.489	8.42	0.0907
	Average	**0.900**	**0.498**	**8.51**	**0.0898**
	%RPD Sample/Sample Dup	**4.7**	**3.6**	**2.1**	**2.0**
ISO16-0001-030	P119C; Sample	884	600	2410	54.1
ISO16-0001-031	P119C; Sample Dup	891	599	2400	52.3
	Average	**888**	**600**	**2410**	**53.2**
	%RPD Sample/Sample Dup	**0.79**	**0.17**	**0.42**	**3.4**
ISO16-0001-032	Effluent WWTP; Sample	26.6	32.7	48.9	1.43
ISO16-0001-033	Effluent WWTP; Sample Dup	25.7	32.4	48.6	1.39
	Average	**26.2**	**32.6**	**48.8**	**1.41**
	%RPD Sample/Sample Dup	**3.4**	**0.92**	**0.62**	**2.8**
ISO16-0001-055	12; Sample	62.9	17.1	196	0.195
ISO16-0001-056	12; Sample Dup	62.0	17.2	196	0.212
	Average	**62.5**	**17.2**	**196**	**0.204**
	%RPD Sample/Sample Dup	**1.4**	**0.58**	**0.0**	**8.4**
ISO16-0001-057	13; Sample	58.7	16.7	178	0.200
ISO16-0001-058	13; Sample Dup	58.2	16.6	175	0.180
	Average	**58.5**	**16.7**	**177**	**0.190**
	%RPD Sample/Sample Dup	**0.86**	**0.60**	**1.7**	**11**
ISO16-0001-059	5; Sample	25.3	19.4	38.4	0.151
ISO16-0001-060	5; Sample Dup	25.9	19.7	42.5	0.142
	Average	**25.6**	**19.6**	**40.5**	**0.147**
	%RPD Sample/Sample Dup	**2.3**	**1.5**	**10**	**6.1**

NA = Not Applicable
NR = Not Reportable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 19%, PFBS ± 15%, PFHS ± 17%, and PFOS ± 17%.

(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 ± 16%, and PFOSA ± 17%.

(3) Sample not reportable for PFOSA due to analyte area counts not meeting method blank criteria. See section 4 of the report for additional information.

(4) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(5) The data uncertainty has been expanded to ±37% for PFOA, ±38% for PFBS, and ±40% for PFHS due to field matrix spike recovery.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA (2)**
ISO16-0001-061	Bemalingsstation; Sample	23.7	17.5	39.7	0.137
ISO16-0001-062	Bemalingsstation; Sample Dup	23.7	18.1	34.5	0.132
	Average	**23.7**	**17.8**	**37.1**	**0.135**
	%RPD Sample/Sample Dup	**0.0**	**3.4**	**14**	**3.7**
ISO16-0001-064	Collector pit; Sample	42.9	14.7	115	8.29
ISO16-0001-065	Collector pit; Sample Dup	44.6	15.3	127	8.24
	Average	**43.8**	**15.0**	**121**	**8.27**
	%RPD Sample/Sample Dup	**3.9**	**4.0**	**9.9**	**0.60**
ISO16-0001-077	L19; Sample	207	203	3660	45.2
ISO16-0001-078	L19; Sample Dup	209	202	3590	39.9
	Average	**208**	**203**	**3630**	**42.6**
	%RPD Sample/Sample Dup	**0.96**	**0.49**	**1.9**	**12**
ISO16-0001-080	M4; Sample	8570	1670	14500	139
ISO16-0001-081	M4; Sample Dup	8490	1690	15400	145
	Average	**8530**	**1680**	**15000**	**142**
	%RPD Sample/Sample Dup	**0.94**	**1.2**	**6.0**	**4.2**
ISO16-0001-082	P118C; Sample	154	69.2	1370	62.4
ISO16-0001-083	P118C; Sample Dup	152	69.1	1360	62.2
	Average	**153**	**69.2**	**1370**	**62.3**
	%RPD Sample/Sample Dup	**1.3**	**0.14**	**0.73**	**0.32**
ISO16-0001-084	P263; Sample	1550	518	8590	10.9
ISO16-0001-085	P263; Sample Dup	1650	529	9550	10.9
	Average	**1600**	**524**	**9070**	**10.9**
	%RPD Sample/Sample Dup	**6.3**	**2.1**	**11**	**0.0**
ISO16-0001-086	P264; Sample	372	262	1630	71.1
ISO16-0001-087	P264; Sample Dup	366	262	1590	76.4
	Average	**369**	**262**	**1610**	**73.8**
	%RPD Sample/Sample Dup	**1.6**	**0.0**	**2.5**	**7.2**
ISO16-0001-091	P371; Sample	90.9	58.5	393	27.1
ISO16-0001-092	P371; Sample Dup	90.4	56.8	399	27.5
	Average	**90.7**	**57.7**	**396**	**27.3**
	%RPD Sample/Sample Dup	**0.55**	**2.9**	**1.5**	**1.5**
ISO16-0001-093	P374; Sample	416	508	1670	35.2
ISO16-0001-094	P374; Sample Dup	432	519	1720	35.8
	Average	**424**	**514**	**1700**	**35.5**
	%RPD Sample/Sample Dup	**3.8**	**2.1**	**2.9**	**1.7**

NA = Not Applicable
NR = Not Reportable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 19%, PFBS ± 15%, PFHS ± 17%, and PFOS ± 17%.

(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 ± 16%, and PFOSA ± 17%.

(3) Sample not reportable for PFOSA due to analyte area counts not meeting method blank criteria. See section 4 of the report for additional information.

(4) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(5) The data uncertainty has been expanded to ±37% for PFOA, ±38% for PFBS, and ±40% for PFHS due to field matrix spike recovery.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA (2)**
ISO16-0001-095	P381; Sample	577	167	1200	15.6
ISO16-0001-096	P381; Sample Dup	569	167	1190	15.6
	Average	**573**	**167**	**1200**	**15.6**
	%RPD Sample/Sample Dup	**1.4**	**0.0**	**0.84**	**0.0**
ISO16-0001-098	P382; Sample	381	117	851	73.5
ISO16-0001-099	P382; Sample Dup	374	111	831	76.1
	Average	**378**	**114**	**841**	**74.8**
	%RPD Sample/Sample Dup	**1.9**	**5.3**	**2.4**	**3.5**
ISO16-0001-100	B3; Sample	7.02	1.59	69.5	0.131
ISO16-0001-101	B3; Sample Dup	7.11	1.67	66.1	0.119
	Average	**7.07 (2)**	**1.63 (2)**	**67.8**	**0.125**
	%RPD Sample/Sample Dup	**1.3**	**4.9**	**5.0**	**9.6**
ISO16-0001-102	B7; Sample	7.74	4.23	65.0	0.328
ISO16-0001-103	B7; Sample Dup	7.41	4.16	65.0	0.331
	Average	**7.58**	**4.20**	**65.0**	**0.330**
	%RPD Sample/Sample Dup	**4.4**	**1.7**	**0.0**	**0.91**
ISO16-0001-104	P372; Sample	69.7	49.9	1100	64.3
ISO16-0001-105	P372; Sample Dup	68.5	49.2	1080	63.9
	Average	**69.1**	**49.6**	**1090**	**64.1**
	%RPD Sample/Sample Dup	**1.7**	**1.4**	**1.8**	**0.62**
ISO16-0001-107	P378; Sample	276	294	1000	1.61
ISO16-0001-108	P378; Sample Dup	284	295	1040	1.58
	Average	**280**	**295**	**1020**	**1.60**
	%RPD Sample/Sample Dup	**2.9**	**0.34**	**3.9**	**1.9**
ISO16-0001-109	PA109A; Sample	81.7	84.9	232	2.18
ISO16-0001-110	PA109A; Sample Dup	80.6	83.8	226	1.97
	Average	**81.2**	**84.4**	**229**	**2.08**
	%RPD Sample/Sample Dup	**1.4**	**1.3**	**2.6**	**10**
ISO16-0001-111	PA111A; Sample	735	1010	1970	9.83
ISO16-0001-112	PA111A; Sample Dup	724	983	1940	9.88
	Average	**730**	**997**	**1960**	**9.86**
	%RPD Sample/Sample Dup	**1.5**	**2.7**	**1.5**	**0.51**
ISO16-0001-113	PA112; Sample	84.4	20.9	326	50.1
ISO16-0001-114	PA112; Sample Dup	84.4	21.2	330	49.5
	Average	**84.4**	**21.1**	**328**	**49.8**
	%RPD Sample/Sample Dup	**0.0**	**1.4**	**1.2**	**1.2**

NA = Not Applicable
NR = Not Reportable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 19%, PFBS ± 15%, PFHS ± 17%, and PFOS ± 17%.

(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 ± 16%, and PFOSA ± 17%.

(3) Sample not reportable for PFOSA due to analyte area counts not meeting method blank criteria. See section 4 of the report for additional information.

(4) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(5) The data uncertainty has been expanded to ±37% for PFOA, ±38% for PFBS, and ±40% for PFHS due to 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)**
ISO16-0001-035	PP01; Sample	80.2	134	24300	6150	40.4
ISO16-0001-036	PP01; Sample Dup	85.6	132	24400	5960	41.7
	Average	**82.9**	**133**	**24400**	**6060**	**41.1**
	%RPD Sample/Sample Dup	**6.5**	**1.5**	**0.41**	**3.1**	**3.2**
ISO16-0001-038	PP02; Sample	806	27.5	2450	21600	30.9
ISO16-0001-039	PP02; Sample Dup	829	28.7	2470	22400	31.3
	Average	**818**	**28.1**	**2460**	**22000**	**31.1**
	%RPD Sample/Sample Dup	**2.8**	**4.3**	**0.81**	**3.6**	**1.3**
ISO16-0001-040	PP04; Sample	585	936	271	4740	62.8
ISO16-0001-041	PP04; Sample Dup	625	859	278	5240	61.7
	Average	**605**	**898**	**275**	**4990**	**62.3**
	%RPD Sample/Sample Dup	**6.6**	**8.6**	**2.6**	**10**	**1.8**
ISO16-0001-042	PP05; Sample	1200	13200	6010	339	65.0
ISO16-0001-043	PP05; Sample Dup	1360	14500	6850	480	61.9
	Average	**1280**	**13850**	**6430**	**410**	**63.5**
	%RPD Sample/Sample Dup	**13**	**9.4**	**13**	**34 (4)**	**4.9**
ISO16-0001-044	PP06; Sample	139	13.9	44.9	1330	47.8
ISO16-0001-045	PP06; Sample Dup	137	13.2	45.0	1320	48.2
	Average	**138**	**13.6**	**45.0**	**1330**	**48.0**
	%RPD Sample/Sample Dup	**1.4**	**5.2**	**0.22**	**0.75**	**0.83**
ISO16-0001-046	PP07; Sample	403	111	116	2440	64.0
ISO16-0001-047	PP07; Sample Dup	388	116	112	2500	61.8
	Average	**396 (5)**	**114 (5)**	**114 (5)**	**2470**	**62.9**
	%RPD Sample/Sample Dup	**3.8**	**4.4**	**3.5**	**2.4**	**3.5**
ISO16-0001-049	PP08; Sample	439	21.7	196	6380	48.1
ISO16-0001-050	PP08; Sample Dup	426	20.9	196	6410	49.3
	Average	**433**	**21.3**	**196**	**6395**	**48.7**
	%RPD Sample/Sample Dup	**3.0**	**3.8**	**0.0**	**0.47**	**2.5**
ISO16-0001-051	PP09; Sample	708	29.4	435	2340	12.9
ISO16-0001-052	PP09; Sample Dup	693	28.5	431	2360	14.2
	Average	**701**	**29.0**	**433**	**2350**	**13.6**
	%RPD Sample/Sample Dup	**2.1**	**3.1**	**0.92**	**0.85**	**9.6**
ISO16-0001-053	PP10; Sample	474	72.1	167	3020	42.6
ISO16-0001-054	PP10; Sample Dup	483	72.6	169	3050	44.1
	Average	**479**	**72.4**	**168**	**3040**	**43.4**
	%RPD Sample/Sample Dup	**1.9**	**0.69**	**1.2**	**0.99**	**3.5**

NA = Not Applicable
NR = Not Reportable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 19%, PFBS ± 15%, PFHS ± 17%, and PFOS ± 17%.

(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 ± 16%, and PFOSA ± 17%.

(3) Sample not reportable for PFOSA due to analyte area counts not meeting method blank criteria. See section 4 of the report for additional information.

(4) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(5) The data uncertainty has been expanded to ±37% for PFOA, ±38% for PFBS, and ±40% for PFHS due to 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)**
ISO16-0001-066	P27; Sample	386	3690	31.5	1800	54.5
ISO16-0001-067	P27; Sample Dup	383	3700	31.7	1770	53.2
	Average	**385**	**3700**	**31.6**	**1790**	**53.9**
	%RPD Sample/Sample Dup	**0.78**	**0.27**	**0.63**	**1.7**	**2.4**
ISO16-0001-068	P21B; Sample	11800	6940	11200	54700	1.99
ISO16-0001-069	P21B; Sample Dup	11500	6760	10700	56000	2.05
	Average	**11700**	**6850**	**11000**	**55400**	**2.02**
	%RPD Sample/Sample Dup	**2.6**	**2.6**	**4.6**	**2.3**	**3.0**
ISO16-0001-070	P304; Sample	556	218	134	1040	19.1
ISO16-0001-071	P304; Sample Dup	569	224	138	1050	19.0
	Average	**563**	**221**	**136**	**1050**	**19.1**
	%RPD Sample/Sample Dup	**2.3**	**2.7**	**2.9**	**0.96**	**0.52**
ISO16-0001-072	P305; Sample	183	91.4	228	537	14.4
ISO16-0001-073	P305; Sample Dup	183	95.4	228	537	14.8
	Average	**183**	**93.4**	**228**	**537**	**14.6**
	%RPD Sample/Sample Dup	**0.0**	**4.3**	**0.0**	**0.0**	**2.7**
ISO16-0001-074	P42; Sample	340	50.0	479	3650	14.7
ISO16-0001-075	P42; Sample Dup	346	47.9	468	3700	15.0
	Average	**343**	**49.0**	**474**	**3680**	**14.9**
	%RPD Sample/Sample Dup	**1.7**	**4.3**	**2.3**	**1.4**	**2.0**

NA = Not Applicable
NR = Not Reportable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 19%, PFBS ± 15%, PFHS ± 17%, and PFOS ± 17%.
(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 ± 16%, and PFOSA ± 17%.
(3) Sample not reportable for PFOSA due to analyte area counts not meeting method blank criteria. See section 4 of the report for additional information.
(4) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
(5) The data uncertainty has been expanded to ±37% for PFOA, ±38% for PFBS, and ±40% for PFHS due to field matrix spike recovery.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M Environmental 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 between January 11, 2016 and January 19, 2016 in Nalgene^{TM} (high-density polyethylene) bottles prepared at the 3M Environmental 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. In addition, all sample bottles were pre-spiked with a mass-labeled internal standard mix at a nominal concentration of 1 ng/mL. Collected sample bottles were returned to the laboratory at ambient temperature on February 1, 2016.

2.3 Sample Preparation

All samples were prepared for PFOA, PFBS, PFHS, and PFOS 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 9.9 mL of methanol. Samples that required a 1:1000 dilution were prepared using a serial dilution by first diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol (1:100 dilution), then diluting 1 mL of the 1:100 dilution with 9.0 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 Blokkersdijkvijver std, L21, L22, L31, P115, and B3 were re-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). The surrogate recovery standards were added to the sample bottle following sample receipt at a nominal concentration of 0.1 ng/mL. 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 1.0 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.

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 1.0 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. The calibration standards and method blanks were also 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).

2.4 Analysis

All samples and quality control samples were analyzed for five target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). 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-12-010.1. 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 Kirk**	**ETS DaVinci**
Liquid Chromatograph	Agilent 1260	Agilent 1290
Analysis Method	ETS-8-044.3	ETS-8-044.3
Analysis Date	2/5/16, 2/12/16, 2/15/16	2/10/16, 2/11/16
Guard column	Betasil C18 (2.1 mm X 100 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 µL	5 µL
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 6500
Ion Source	Turbo Spray	Turbo Spray
Polarity	Negative	Negative
Software	Analyst 1.6.2	Analyst 1.6.2

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis				
Step Number	**Total Time (min)**	**Flow Rate (µL/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	4.00	300	70.0	30.0
3	6.00	300	70.0	30.0
4	11.0	300	20.0	80.0
5	13.0	300	20.0	80.0
6	13.5	300	10.0	90.0
7	16.0	300	10.0	90.0
8	16.5	300	90.0	10.0
9	19.0	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 surrogate	417/372	$[^{13}C_8]$-PFOA	421/376
$[^{13}C_4]$-PFOS surrogate	503/80	$[^{13}C_8]$-PFOS	507/80

NA = Not Applicable

Dwell time was between 10 and 50 msec for each transition. The individual transitions were summed to produce a “total ion chromatogram” (TIC), which was used for quantitation.

(1) Internal standards were used on 2/10/16, 2/12/16, and 2/15/16.

3 Data Analysis

3.1 Calibration

2/5/16 and 2/11/16 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 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.

2/10/16 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 mL of 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. Of these calibration standards, ten contained the 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.

2/12/16 and 2/15/16 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 25 mL of 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 were analyzed. Prior to analysis, the calibration standards were diluted 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±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.

Table 6. LOQ

Analyte	LOQ, ng/mL (1) 2/5/16 Analysis	LOQ, ng/mL (2) 2/10/16 Analysis	LOQ, ng/mL (1) 2/11/16 Analysis	LOQ, ng/mL (1) 2/12/16 Analysis	LOQ, ng/mL (1) 2/15/16 Analysis
PFOA	0.0192	0.0240	0.0192	NA	NA
PFBS	0.100	NA	0.0200	NA	NA
PFHS	0.0200	0.0250	0.0200	NA	NA
PFOS	0.0464	0.0464	0.0464	NA	NA
PFOSA	NA	NA	NA	0.0250	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% (100%±30% for LOQ).

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/solvent 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 [$^{13}C_4$]-PFOA and [$^{13}C_4$]-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:

- On 2/5/16, the high level LCSs were prepared above the resulting ULOQ (50 ng/mL) for PFBS and PFHS.

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.

$$\boxed{\text{LCS Percent Recovery} = \frac{\text{Calculated Concentration}}{\text{Spike Concentration}} * 100\%}$$

$$\boxed{\text{LCS\% RSD} = \frac{\text{standard deviation LCS replicates}}{\text{average LCS recovery}} * 100\%}$$

Table 7. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 2/5/16	**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-160205-1	100	126	126	100	115	115
LCS-160205-2	100	111	111	100	97.6	97.6
LCS-160205-3	100	96.4	96.4	100	93.5	93.5
Average ± %RSD	**111% ± 13%**			**102% ± 11%**		
LCS-160205-4	5000	5100	102	5000	5180	104
LCS-160205-5	5000	4920	98.4	5000	5030	101
LCS-160205-6	5000	4920	98.3	5000	4980	99.7
Average ± %RSD	**99.6% ± 2.1%**			**102% ± 2.2%**		
LCS-160205-7	35000	28800	82.3	35000	>ULOQ	NA
LCS-160205-8	35000	29000	82.8	35000	>ULOQ	NA
LCS-160205-9	35000	28500	81.4	35000	>ULOQ	NA
Average ± %RSD	**82.2% ± 0.86%**			**NA (1)**		

ETS-8-044.3 External Calibration Analyzed 2/5/16	**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-160205-1	100	128	128	100	102	102
LCS-160205-2	100	111	111	100	91.6	91.6
LCS-160205-3	100	97.7	97.7	100	84.7	84.7
Average ± %RSD	**112% ± 14%**			**92.8% ± 9.4%**		
LCS-160205-4	5000	5210	104	5000	5010	100
LCS-160205-5	5000	5190	104	5000	4920	98.5
LCS-160205-6	5000	5150	103	5000	4980	99.7
Average ± %RSD	**104% ± 0.56%**			**99.4% ± 0.80%**		
LCS-160205-7	35000	>ULOQ	NA	35000	37300	107
LCS-160205-8	35000	>ULOQ	NA	35000	36300	104
LCS-160205-9	35000	>ULOQ	NA	35000	35600	102
Average ± %RSD	**NA (1)**			**104% ± 2.4%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCSs prepared above the resulting ULOQ.
(2) LCS was spiked post dilution, but reported with the dilution factor applied.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 2/5/16	**[$^{13}C_4$]-PFOA** (2)			**[$^{13}C_4$]-PFOS** (2)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160205-1	99.8	105	105	95.6	100	105
LCS-160205-2	99.8	98.5	98.6	95.6	92.6	96.8
LCS-160205-3	99.8	99.9	100	95.6	95.0	99.3
Average ± %RSD	**101% ± 3.3%**			**100% ± 4.2%**		
LCS-160205-4	998	1000	101	956	973	102
LCS-160205-5	998	973	97.5	956	947	99.1
LCS-160205-6	998	993	99.5	956	979	102
Average ± %RSD	**99.3% ± 1.8%**			**101% ± 1.7%**		

ETS-8-044.3 Internal Calibration Analyzed 2/10/16	**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-160210-1	0.190	0.181	95.1	0.198	0.172	87.0
LCS-160210-2	0.190	0.187	98.2	0.198	0.176	88.9
LCS-160210-3	0.190	0.203	107	0.198	0.196	99.0
Average ± %RSD	**100% ± 6.2%**			**91.6% ± 7.0%**		
LCS-160210-4	1.90	1.79	94.1	1.98	1.98	99.8
LCS-160210-5	1.90	1.83	96.5	1.98	1.83	92.4
LCS-160210-6	1.90	1.74	91.7	1.98	1.90	96.2
Average ± %RSD	**94.1% ± 2.6%**			**96.1% ± 3.8%**		
LCS-160210-7	33.2	32.0	96.3	34.7	33.4	96.3
LCS-160210-8	33.2	29.6	89.1	34.7	32.8	94.5
LCS-160210-9	33.2	32.6	98.3	34.7	33.7	97.3
Average ± %RSD	**94.6% ± 5.1%**			**96.0% ± 1.5%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCSs prepared above the resulting ULOQ.
(2) LCS was spiked post dilution, but reported with the dilution factor applied.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 2/10/16	PFOS (Linear + Branched)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160210-1	0.184	0.206	112
LCS-160210-2	0.184	0.184	99.9
LCS-160210-3	0.184	0.176	95.6
Average ± %RSD	**103% ± 8.3%**		
LCS-160210-4	1.84	1.88	102
LCS-160210-5	1.84	1.82	99.0
LCS-160210-6	1.84	1.84	100
Average ± %RSD	**100% ± 1.5%**		
LCS-160210-7	32.2	31.5	97.9
LCS-160210-8	32.2	28.2	87.5
LCS-160210-9	32.2	31.1	96.7
Average ± %RSD	**94.0% ± 6.1%**		

ETS-8-044.3 Internal Calibration Analyzed 2/10/16	[$^{13}C_4$]-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-160210-1	0.198	0.177	89.2	0.189	0.170	89.8
LCS-160210-2	0.198	0.193	97.7	0.189	0.172	91.1
LCS-160210-3	0.198	0.206	104	0.189	0.163	86.4
Average ± %RSD	**97.0% ± 7.7%**			**89.1% ± 2.7%**		
LCS-160210-4	1.98	1.94	97.9	1.89	2.07	109
LCS-160210-5	1.98	1.93	97.6	1.89	1.98	105
LCS-160210-6	1.98	1.98	99.9	1.89	2.00	106
Average ± %RSD	**98.5% ± 1.3%**			**107% ± 2.0%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCSs prepared above the resulting ULOQ.
(2) LCS was spiked post dilution, but reported with the dilution factor applied.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 2/11/16	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-160210-1	200	210	105	200	210	105
LCS-160210-2	200	212	106	200	208	104
LCS-160210-3	200	212	106	200	208	104
Average ± %RSD	**106% ± 0.55%**			**104% ± 0.55%**		
LCS-160210-4	25000	25000	99.8	25000	26400	106
LCS-160210-5	25000	25000	99.8	25000	25500	102
LCS-160210-6	25000	25300	101	25000	25700	103
Average ± %RSD	**100% ± 0.69%**			**104% ± 2.0%**		
LCS-160210-7	70000	69400	99.1	70000	73800	105
LCS-160210-8	70000	70100	100	70000	72500	104
LCS-160210-9	70000	68900	98.4	70000	71900	103
Average ± %RSD	**99.2% ± 0.81%**			**104% ± 0.96%**		

ETS-8-044.3 External Calibration Analyzed 2/11/16	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-160210-1	200	203	102	200	181	90.4
LCS-160210-2	200	201	101	200	180	90.1
LCS-160210-3	200	195	97.7	200	182	90.9
Average ± %RSD	**100% ± 2.2%**			**90.5% ± 0.45%**		
LCS-160210-4	25000	27400	109	25000	23900	95.6
LCS-160210-5	25000	26600	106	25000	24000	96.1
LCS-160210-6	25000	26300	105	25000	23500	93.8
Average ± %RSD	**107% ± 2.0%**			**95.2% ± 1.3%**		
LCS-160210-7	70000	79200	113	70000	71400	102
LCS-160210-8	70000	77300	110	70000	70500	101
LCS-160210-9	70000	76900	110	70000	69800	99.7
Average ± %RSD	**111% ± 1.6%**			**101% ± 1.1%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCSs prepared above the resulting ULOQ.
(2) LCS was spiked post dilution, but reported with the dilution factor applied.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 2/11/16	**[$^{13}C_4$]-PFOA $^{(2)}$**			**[$^{13}C_4$]-PFOS $^{(2)}$**		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160210-1	998	1050	105	956	962	101
LCS-160210-2	998	1060	106	956	988	103
LCS-160210-3	998	1050	106	956	992	104
Average ± %RSD	**106% ± 0.55%**			**103% ± 1.5%**		
LCS-160210-7	9980	10100	101	9560	10000	105
LCS-160210-8	9980	10100	101	9560	10300	108
LCS-160210-9	9980	10200	102	9560	9950	104
Average ± %RSD	**101% ± 0.57%**			**106% ± 2.0%**		

ETS-8-044.3 Internal Calibration Analyzed 2/12/16	**PFOSA**		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160212-1	0.198	0.185	93.3
LCS-160212-2	0.198	0.218	110
LCS-160212-3	0.198	0.182	91.9
Average ± %RSD	**98.4% ± 10%**		
LCS-160212-4	19.8	18.7	94.3
LCS-160212-5	19.8	17.5	88.6
LCS-160212-6	19.8	19.1	96.3
Average ± %RSD	**93.1% ± 4.3%**		
LCS-160212-7	139	123	88.3
LCS-160212-8	139	120	86.1
LCS-160212-9	139	122	87.7
Average ± %RSD	**87.4% ± 1.3%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCSs prepared above the resulting ULOQ.
(2) LCS was spiked post dilution, but reported with the dilution factor applied.

3.7 Analytical Method 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-12-012.3. 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%.

Table 8. Analytical Method Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	9.46	±19%
PFBS	External	7.50	±15%
PFHS	External	8.58	±17%
PFOS	External	8.41	±17%
PFOA	Internal	7.94	±16%
PFHS	Internal	5.71	±11%
PFOS	Internal	8.15	±16%
PFOSA	Internal	8.51	±17%

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 contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. The stock solutions of PFOS and PFOA used to prepare the field matrix spikes were prepared from a different reference source than the one used to prepare the calibration standards. 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 $^{13}C_4$-PFOA and $^{13}C_4$-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles following sample collection. Samples analyzed using external calibration were spiked with surrogate recovery standards post dilution. The $^{13}C_4$- PFOA was selected to represent the C8 perfluorocarboxylic acid. The $^{13}C_4$-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids and PFOSA. 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.

$$\boxed{\text{FMS Recovery} = \frac{(\text{Sample Concentration of FMS} - \text{Average Concentration : Field Sample \& Field Sample Dup.})}{\text{Spike Concentration}} * 100\%}$$

Table 9. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
L21, P115	FMS	10.0	10.0	10.0	10.0	10.0
Effluent WWTP, Bemalingsstation	FMS	100	100	100	100	10.0
PP01	FMS	100	100	100	100	100
PP07, P42, L19, P265C, P372, PA112	FMS	1000	1000	1000	1000	100
BD24-3, P381	FMS	2010	10.0	2010	2010	10.0
Trip Blank	Low	10.0	10.0	10.0	10.0	10.0
	High	2000	10.0	2000	2000	100

4 Data Summary and Discussion

Tables 10-23 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. The field matrix spike concentrations were selected based on previous results for these sampling locations. The spike level, at times, 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. Surrogate recovery standards are summarized in Table 23. All surrogate recovery standards met method acceptance criteria.

Sample location P114bis: The initial sample (ISO16-0001-023) is not reportable for PFOSA. The area ratio (target analyte peak area counts over the internal standard area counts) for this sample was within the calibration range, however; the sample target analyte peak area counts for PFOSA were less than the peak area counts for the LOQ standard. Since the sample target analyte peak area counts were also less than twice the average area counts for the method blanks, the method specifies that the sample result is not reportable.

Sample location PP07: Field matrix spike recovery was 63.5% for PFOA, 62.5% for PFBS, and 60.1% for PFHS. Since the %bias of the FMS sample is greater than the overall method uncertainty for each analyte, the data uncertainty has been expanded to ±37% for PFOA, ±38% for PFBS, and ±40% for PFHS for location PP07. The method uncertainty was not expanded for other locations since another sample from the extraction wells (PP01) also contained a FMS sample which met acceptance criteria, where applicable. Also, the overall FMS recovery for all samples analyzed by external calibration had a pooled FMS recovery greater than 70%, for all analytes, which met method acceptance criteria.

Table 10. Location ID: BD24-3

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-001	BD24-3; Sample	69.2	NA	54.8	NA
ISO16-0001-002	BD24-3; Sample Dup	70.6	NA	55.6	NA
ISO16-0001-003	BD24-3; FMS	1910	91.5 (1)	2050	99.2 (1)
Average Concentration (ng/mL) ± %RPD		**69.9 ng/mL ± 2.0%**		**55.2 ng/mL ± 1.4%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-001	BD24-3; Sample	1270	NA	2.79	NA
ISO16-0001-002	BD24-3; Sample Dup	1270	NA	2.85	NA
ISO16-0001-003	BD24-3; FMS	3100	91.0	12.5	96.8
Average Concentration (ng/mL) ± %RPD		**1270 ng/mL ± 0.0%**		**2.82 ng/mL ± 2.1%**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.

Table 11. Location ID: L21

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-014	L21; Sample	0.446	NA	0.206	NA
ISO16-0001-015	L21; Sample Dup	0.462	NA	0.195	NA
ISO16-0001-016	L21; FMS	10.3	98.5 (1)	10.5	103 (1)
Average Concentration (ng/mL) ± %RPD		**0.454 ng/mL ± 3.5%**		**0.201 ng/mL ± 5.5%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-014	L21; Sample	5.56	NA	0.133	NA
ISO16-0001-015	L21; Sample Dup	5.73	NA	0.128	NA
ISO16-0001-016	L21; FMS	14.8	91.6	9.80	96.7 (1)
Average Concentration (ng/mL) ± %RPD		**5.65 ng/mL ± 3.0%**		**0.131 ng/mL ± 3.8%**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.

Table 12. Location ID: P115

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-025	P115; Sample	4.10	NA	2.35	NA
ISO16-0001-026	P115; Sample Dup	4.25	NA	2.25	NA
ISO16-0001-027	P115; FMS	14.8	106	12.4	101
Average Concentration (ng/mL) ± %RPD		**4.18 ng/mL ± 3.6%**		**2.30 ng/mL ± 4.3%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-025	P115; Sample	0.566	NA	<0.0250	NA
ISO16-0001-026	P115; Sample Dup	0.622	NA	<0.0250	NA
ISO16-0001-027	P115; FMS	10.2	96.1 (1)	9.77	97.7 (1)
Average Concentration (ng/mL) ± %RPD		**0.594 ng/mL ± 9.4%**		**<0.0250 ng/mL**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.

Table 13. Location ID: Effluent WWTP

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-032	Effluent WWTP; Sample	26.6	NA	32.7	NA
ISO16-0001-033	Effluent WWTP; Sample Dup	25.7	NA	32.4	NA
ISO16-0001-034	Effluent WWTP; FMS	116	89.9	122	89.5
Average Concentration (ng/mL) ± %RPD		**26.2 ng/mL ± 3.4%**		**32.6 ng/mL ± 0.92%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-032	Effluent WWTP; Sample	48.9	NA	1.43	NA
ISO16-0001-033	Effluent WWTP; Sample Dup	48.6	NA	1.39	NA
ISO16-0001-034	Effluent WWTP; FMS	135	86.3	11.1	96.9
Average Concentration (ng/mL) ± %RPD		**48.8 ng/mL ± 0.62%**		**1.41 ng/mL ± 2.8%**	

NA = Not Applicable

Table 14. Location ID: Bemalingsstation

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-061	Bemalingsstation; Sample	23.7	NA	17.5	NA
ISO16-0001-062	Bemalingsstation; Sample Dup	23.7	NA	18.1	NA
ISO16-0001-063	Bemalingsstation; FMS	113	89.3	111	93.2
Average Concentration (ng/mL) ± %RPD		**23.7 ng/mL ± 0.0%**		**17.8 ng/mL ± 3.4%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-061	Bemalingsstation; Sample	39.7	NA	0.137	NA
ISO16-0001-062	Bemalingsstation; Sample Dup	34.5	NA	0.132	NA
ISO16-0001-063	Bemalingsstation; FMS	108	70.9	9.68	95.5 (1)
Average Concentration (ng/mL) ± %RPD		**37.1 ng/mL ± 14%**		**0.135 ng/mL ± 3.7%**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.

Table 15. Location ID: L19

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-077	L19; Sample	207	NA	203	NA
ISO16-0001-078	L19; Sample Dup	209	NA	202	NA
ISO16-0001-079	L19; FMS	1160	95.2	1200	99.8
Average Concentration (ng/mL) ± %RPD		**208 ng/mL ± 0.96%**		**203 ng/mL ± 0.49%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-077	L19; Sample	3660	NA	45.2	NA
ISO16-0001-078	L19; Sample Dup	3590	NA	39.9	NA
ISO16-0001-079	L19; FMS	4560	NC	126	83.5
Average Concentration (ng/mL) ± %RPD		**3630 ng/mL ± 1.9%**		**42.6 ng/mL ± 12%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 16. Location ID: P381

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-095	P381; Sample	577	NA	167	NA
ISO16-0001-096	P381; Sample Dup	569	NA	167	NA
ISO16-0001-097	P381; FMS	2320	86.9	2060	94.2 (1)
Average Concentration (ng/mL) ± %RPD		**573 ng/mL ± 1.4%**		**167 ng/mL ± 0.0%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-095	P381; Sample	1200	NA	15.6	NA
ISO16-0001-096	P381; Sample Dup	1190	NA	15.6	NA
ISO16-0001-097	P381; FMS	2870	83.3	24.1	85.0
Average Concentration (ng/mL) ± %RPD		**1200 ng/mL ± 0.84%**		**15.6 ng/mL ± 0.0%**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.

Table 17. Location ID: P372

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-104	P372; Sample	69.7	NA	49.9	NA
ISO16-0001-105	P372; Sample Dup	68.5	NA	49.2	NA
ISO16-0001-106	P372; FMS	973	90.4 (1)	989	93.9 (1)
Average Concentration (ng/mL) ± %RPD		**69.1 ng/mL ± 1.7%**		**49.6 ng/mL ± 1.4%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-104	P372; Sample	1100	NA	64.3	NA
ISO16-0001-105	P372; Sample Dup	1080	NA	63.9	NA
ISO16-0001-106	P372; FMS	2010	92.0	149	84.9
Average Concentration (ng/mL) ± %RPD		**1090 ng/mL ± 1.8%**		**64.1 ng/mL ± 0.62%**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.

Table 18. Location ID: PA112

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-113	PA112; Sample	84.4	NA	20.9	NA
ISO16-0001-114	PA112; Sample Dup	84.4	NA	21.2	NA
ISO16-0001-115	PA112; FMS	952	86.8 (1)	947	92.6 (1)
Average Concentration (ng/mL) ± %RPD		**84.4 ng/mL ± 0.0%**		**21.1 ng/mL ± 1.4%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-113	PA112; Sample	326	NA	50.1	NA
ISO16-0001-114	PA112; Sample Dup	330	NA	49.5	NA
ISO16-0001-115	PA112; FMS	1130	80.2	137	87.2
Average Concentration (ng/mL) ± %RPD		**328 ng/mL ± 1.2%**		**49.8 ng/mL ± 1.2%**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.

Table 19. Location ID: PP01

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-035	PP01; Sample	80.2	NA	134	NA	24300	NA
ISO16-0001-036	PP01; Sample Dup	85.6	NA	132	NA	24400	NA
ISO16-0001-037	PP01; FMS	168	85.1	230	97.0	24400	NC
Average Concentration (ng/mL) ± %RPD		**82.9 ng/mL ± 6.5%**		**133 ng/mL ± 1.5%**		**24400 ng/mL ± 0.41%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-035	PP01; Sample	6150	NA	40.4	NA
ISO16-0001-036	PP01; Sample Dup	5960	NA	41.7	NA
ISO16-0001-037	PP01; FMS	5880	NC	128	87.0
Average Concentration (ng/mL) ± %RPD		**6060 ng/mL ± 3.1%**		**41.1 ng/mL ± 3.2%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 20. Location ID: PP07

		PFOA		**PFBS**		**PFHS**	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-046	PP07; Sample	403	NA	111	NA	116	NA
ISO16-0001-047	PP07; Sample Dup	388	NA	116	NA	112	NA
ISO16-0001-048	PP07; FMS	1030	63.5 (1)	176	62.5 (1)	715	60.1 (1)
Average Concentration (ng/mL) ± %RPD		**396 ng/mL ± 3.8% (2)**		**114 ng/mL ± 4.4%**		**114 ng/mL ± 3.5% (2)**	

		PFOS		**PFOSA**	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-046	PP07; Sample	2440	NA	64.0	NA
ISO16-0001-047	PP07; Sample Dup	2500	NA	61.8	NA
ISO16-0001-048	PP07; FMS	2980	NC	182	119
Average Concentration (ng/mL) ± %RPD		**2470 ng/mL ± 2.4%**		**62.9 ng/mL ± 3.5%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%.
(2) Data uncertainty has been expanded to ±37% for PFOA, ±38% for PFBS, and ±40% for PFHS.

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**
ISO16-0001-074	P42; Sample	340	NA	50.0	NA	479	NA
ISO16-0001-075	P42; Sample Dup	346	NA	47.9	NA	468	NA
ISO16-0001-076	P42; FMS	1270	92.7	151	102	1460	98.7
Average Concentration (ng/mL) ± %RPD		**343 ng/mL ± 1.7%**		**49.0 ng/mL ± 4.3%**		**474 ng/mL ± 2.3%**	

		PFOS		**PFOSA**	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-074	P42; Sample	3650	NA	14.7	NA
ISO16-0001-075	P42; Sample Dup	3700	NA	15.0	NA
ISO16-0001-076	P42; FMS	4560	NC	106	91.2
Average Concentration (ng/mL) ± %RPD		**3680 ng/mL ± 1.4%**		**14.9 ng/mL ± 2.0%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 22. Trip Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-116	Trip Blank; Sample	<0.0240	NA	<1.00	NA	<0.0250	NA
ISO16-0001-117	Trip Blank; FMS Low	9.53	95.3	10.3	103	9.77	97.7
ISO16-0001-118	Trip Blank; FMS High	1900	95.0	98.3	98.3	2020	101

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0001-116	Trip Blank; Sample	<0.0464	NA	<0.100	NA
ISO16-0001-117	Trip Blank; FMS Low	9.14	91.4	9.44	94.4
ISO16-0001-118	Trip Blank; FMS High	1930	96.5	94.3	94.3

NA = Not Applicable

Table 23. Surrogate Recovery Standard Results (1)

		Recovery (%)		Re-analysis Recovery (%)
3M LIMS ID	**Sample Description**	**[$^{13}C_4$]-PFOA**	**[$^{13}C_4$]-PFOS**	**[$^{13}C_4$]-PFOS**
ISO16-0001-001	BD24-3; Sample	104	102	NA
ISO16-0001-002	BD24-3; Sample Dup	102	99.5	NA
ISO16-0001-003	BD24-3; FMS	95.2	92.3	NA
ISO16-0001-004	D09; Sample	104	105	NA
ISO16-0001-005	D09; Sample Dup	88.5	88.8	NA
ISO16-0001-006	P121; Sample	105	104	NA
ISO16-0001-007	P121; Sample Dup	99.9	96.0	NA
ISO16-0001-008	P321; Sample	94.6	93.6	NA
ISO16-0001-009	P321; Sample Dup	88.6	87.2	NA
ISO16-0001-010	3M vijver; Sample	102	101	NA
ISO16-0001-011	3M vijver; Sample Dup	98.6	96.6	NA
ISO16-0001-012	Blokkersdijkvijver std; Sample	87.0 (2)	98.5 (2)	NA
ISO16-0001-013	Blokkersdijkvijver std; Sample Dup	85.1 (2)	79.2 (2)	NA
ISO16-0001-014	L21; Sample	89.5 (2)	84.3 (2)	NA
ISO16-0001-015	L21; Sample Dup	94.9 (2)	79.5 (2)	NA
ISO16-0001-016	L21; FMS	105 (2)	89.4 (2)	NA
ISO16-0001-017	L22; Sample	86.0 (2)	79.9 (2)	NA
ISO16-0001-018	L22; Sample Dup	94.1 (2)	76.5 (2)	NA
ISO16-0001-019	L31; Sample	85.2 (2)	90.1 (2)	NA
ISO16-0001-020	L31; Sample Dup	91.9 (2)	79.6 (2)	NA
ISO16-0001-021	L4; Sample	99.7	97.0	NA
ISO16-0001-022	L4; Sample Dup	105	102	NA
ISO16-0001-023	P114bis; Sample	102	103	NA
ISO16-0001-024	P114bis; Sample Dup	107	107	NA
ISO16-0001-025	P115; Sample	90.2 (2)	87.9 (2)	NA
ISO16-0001-026	P115; Sample Dup	93.9 (2)	92.1 (2)	NA
ISO16-0001-027	P115; FMS	78.6 (2)	91.9 (2)	NA
ISO16-0001-028	P116; Sample	97.8	94.8	NA
ISO16-0001-029	P116; Sample Dup	99.2	93.9	NA
ISO16-0001-030	P119C; Sample	97.6	93.4	NA
ISO16-0001-031	P119C; Sample Dup	100	94.7	NA
ISO16-0001-032	Effluent WWTP; Sample	93.1	90.2	NA
ISO16-0001-033	Effluent WWTP; Sample Dup	86.2	84.0	NA
ISO16-0001-034	Effluent WWTP; FMS	94.8	92.7	NA
ISO16-0001-035	PP01; Sample	99.3	91.3	111
ISO16-0001-036	PP01; Sample Dup	97.5	91.3	109
ISO16-0001-037	PP01; FMS	100	97.0	110
ISO16-0001-038	PP02; Sample	90.9	78.2	NA
ISO16-0001-039	PP02; Sample Dup	86.2	76.5	NA

NA = Not Applicable

(1) Unless noted otherwise, the surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS were added to the samples during sample preparation.

(2) The surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS were added to the sample bottles following sample receipt and prior to sample preparation.

Table 23 continued. Surrogate Recovery Standard Results [(1)]

		Recovery (%)		Re-analysis Recovery (%)
3M LIMS ID	**Sample Description**	**[$^{13}C_4$]-PFOA**	**[$^{13}C_4$]-PFOS**	**[$^{13}C_4$]-PFOS**
ISO16-0001-041	PP04; Sample Dup	95.3	89.3	NA
ISO16-0001-042	PP05; Sample	95.7	80.9	NA
ISO16-0001-043	PP05; Sample Dup	96.1	78.6	NA
ISO16-0001-044	PP06; Sample	100	91.5	NA
ISO16-0001-045	PP06; Sample Dup	96.2	89.3	NA
ISO16-0001-046	PP07; Sample	111	88.7	NA
ISO16-0001-047	PP07; Sample Dup	107	88.4	NA
ISO16-0001-048	PP07; FMS	107	89.4	NA
ISO16-0001-049	PP08; Sample	95.2	91.1	NA
ISO16-0001-050	PP08; Sample Dup	94.9	84.0	NA
ISO16-0001-051	PP09; Sample	91.8	85.9	NA
ISO16-0001-052	PP09; Sample Dup	98.4	89.1	NA
ISO16-0001-053	PP10; Sample	90.3	79.9	NA
ISO16-0001-054	PP10; Sample Dup	95.5	86.4	NA
ISO16-0001-055	12; Sample	94.2	87.7	NA
ISO16-0001-056	12; Sample Dup	93.8	87.5	NA
ISO16-0001-057	13; Sample	91.9	85.8	NA
ISO16-0001-058	13; Sample Dup	94.0	86.7	NA
ISO16-0001-059	5; Sample	93.4	91.6	NA
ISO16-0001-060	5; Sample Dup	99.2	94.8	NA
ISO16-0001-061	Bemalingsstation; Sample	95.7	91.9	NA
ISO16-0001-062	Bemalingsstation; Sample Dup	96.7	93.3	NA
ISO16-0001-063	Bemalingsstation; FMS	92.9	89.9	NA
ISO16-0001-064	Collector pit; Sample	100	90.8	NA
ISO16-0001-065	Collector pit; Sample Dup	92.6	84.8	NA
ISO16-0001-066	P27; Sample	101	93.5	NA
ISO16-0001-067	P27; Sample Dup	105	98.3	NA
ISO16-0001-068	P21B; Sample	94.1	90.5	105
ISO16-0001-069	P21B; Sample Dup	91.9	90.1	108
ISO16-0001-070	P304; Sample	106	103	NA
ISO16-0001-071	P304; Sample Dup	105	104	NA
ISO16-0001-072	P305; Sample	95.4	92.1	NA
ISO16-0001-073	P305; Sample Dup	104	100	NA
ISO16-0001-074	P42; Sample	98.4	88.9	NA
ISO16-0001-075	P42; Sample Dup	105	91.9	NA
ISO16-0001-076	P42; FMS	104	92.9	NA
ISO16-0001-077	L19; Sample	105	89.6	NA
ISO16-0001-078	L19; Sample Dup	104	88.1	NA
ISO16-0001-079	L19; FMS	93.5	82.2	NA
ISO16-0001-080	M4; Sample	77.2	109	NA
ISO16-0001-081	M4; Sample Dup	77.6	108	NA

NA = Not Applicable

(1) Unless noted otherwise, the surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS were added to the samples during sample preparation.

(2) The surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS were added to the sample bottles following sample receipt and prior to sample preparation.

Table 23 continued. Surrogate Recovery Standard Results (1)

		Recovery (%)		Re-analysis Recovery (%)
3M LIMS ID	**Sample Description**	**[$^{13}C_4$]-PFOA**	**[$^{13}C_4$]-PFOS**	**[$^{13}C_4$]-PFOS**
ISO16-0001-082	P118C; Sample	95.8	92.9	NA
ISO16-0001-083	P118C; Sample Dup	101	92.6	NA
ISO16-0001-084	P263; Sample	99.9	95.1	NA
ISO16-0001-085	P263; Sample Dup	99.5	96.4	NA
ISO16-0001-086	P264; Sample	98.3	93.2	NA
ISO16-0001-087	P264; Sample Dup	97.3	88.3	NA
ISO16-0001-091	P371; Sample	101	96.0	NA
ISO16-0001-092	P371; Sample Dup	95.4	90.5	NA
ISO16-0001-093	P374; Sample	92.8	87.0	NA
ISO16-0001-094	P374; Sample Dup	98.5	94.8	NA
ISO16-0001-095	P381; Sample	92.5	93.0	NA
ISO16-0001-096	P381; Sample Dup	97.5	95.8	NA
ISO16-0001-097	P381; FMS	88.0	84.0	NA
ISO16-0001-098	P382; Sample	99.9	94.7	NA
ISO16-0001-099	P382; Sample Dup	99.6	98.4	NA
ISO16-0001-100	B3; Sample	85.1 (2)	93.0	90.2 (2)
ISO16-0001-101	B3; Sample Dup	88.2 (2)	96.0	83.5 (2)
ISO16-0001-102	B7; Sample	95.3	91.7	NA
ISO16-0001-103	B7; Sample Dup	86.9	86.0	NA
ISO16-0001-104	P372; Sample	99.0	94.0	NA
ISO16-0001-105	P372; Sample Dup	93.1	84.9	NA
ISO16-0001-106	P372; FMS	94.4	88.4	NA
ISO16-0001-107	P378; Sample	103	99.4	NA
ISO16-0001-108	P378; Sample Dup	98.0	95.0	NA
ISO16-0001-109	PA109A; Sample	93.8	88.6	NA
ISO16-0001-110	PA109A; Sample Dup	92.8	91.2	NA
ISO16-0001-111	PA111A; Sample	95.2	92.0	NA
ISO16-0001-112	PA111A; Sample Dup	94.9	91.9	NA
ISO16-0001-113	PA112; Sample	96.8	93.3	NA
ISO16-0001-114	PA112; Sample Dup	96.7	94.1	NA
ISO16-0001-115	PA112; FMS	92.2	90.8	NA
ISO16-0001-116	Trip Blank; Sample	83.6 (2)	86.8 (2)	NA
ISO16-0001-117	Trip Blank; FMS Low	92.3 (2)	98.2 (2)	NA
ISO16-0001-118	Trip Blank; FMS High	100	101	NA

NA = Not Applicable

(1) Unless noted otherwise, the surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS were added to the samples during sample preparation.

(2) The surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS were added to the sample bottles following sample receipt and prior to 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 Environmental 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 10-23 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M Environmental Laboratory standard operating procedures.

7 Signatures

Chelsie Grochow, 3M Report Author

Susan T. Wolf, 3M Principal Analytical Investigator

William K. Reagen, Ph.D., 3M Environmental Laboratory Technical Director

The 3M Environmental 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 Environmental Laboratory.

Final Report

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

April 2016 Sampling

Laboratory Request Number: ISO16-0004

Report Date – Date of Last Signature

Testing Laboratory

3M Environment, Health, Safety, and Sustainability
3M Environmental Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

Nicole Cauberghe
3M Belgium EHS Operations
3M Belgium; ZW018/0/33
Phone: [redacted]

ACCREDITED
Testing Cert. # 2052.01

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 Environmental Laboratory

3M Environmental Laboratory Technical Director: William K. Reagen, Ph.D.
3M Principal Analytical Investigator: Susan Wolf
Report Author: Chelsie Grochow

Analytical Report ISO16-0004

Water Sample Analysis at 3M Antwerp, Belgium
April 2016 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M Environmental Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. Samples were collected between April 5, 2016 and April 7, 2016. Samples were returned to the 3M Environmental Laboratory on April 13, 2016 at ambient temperature for the analysis of Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO16-0004.

The 3M Environmental Laboratory prepared sample containers for twenty-seven 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 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.

ACCREDITED
Testing Cert. # 2052.01

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)**
ISO16-0004-001	P321; Sample	2980	934	4940	0.399
ISO16-0004-001-DUP	P321; Sample Dup	3030	940	4970	0.384
Average		**3010**	**937**	**4960**	**0.392**
%RPD Sample/Sample Dup		**1.7**	**0.64**	**0.61**	**3.8**
ISO16-0004-002	3M vijver; Sample	1.47	0.743	9.21	0.231
ISO16-0004-002-DUP	3M vijver; Sample Dup	1.47	0.709	8.24	0.189
Average		**1.47**	**0.726**	**8.73**	**0.210**
%RPD Sample/Sample Dup		**0.0**	**4.7**	**11**	**20**
ISO16-0004-003	Blokkersdijkvijver std; Sample	1.25	0.942	16.0	0.624
ISO16-0004-003-DUP	Blokkersdijkvijver std; Sample Dup	1.15	0.991	15.1	0.546
Average		**1.20**	**0.967**	**15.6**	**0.585**
%RPD Sample/Sample Dup		**8.3**	**5.1**	**5.8**	**13**
ISO16-0004-004	L4; Sample	1.06	0.461	24.4	5.97
ISO16-0004-004-DUP	L4; Sample Dup	1.03	0.481	24.6	5.99
Average		**1.05**	**0.471**	**24.5**	**5.98**
%RPD Sample/Sample Dup		**2.9**	**4.2**	**0.82**	**0.33**
ISO16-0004-005	P114bis; Sample	3.39	1.32	13.3	0.0961
ISO16-0004-005-DUP	P114bis; Sample Dup	3.34	1.36	13.3	0.0696
Average		**3.37**	**1.34**	**13.3**	**0.0829**
%RPD Sample/Sample Dup		**1.5**	**3.0**	**0.0**	**32 (3)**
ISO16-0004-006	P116; Sample	1.74	0.800	8.96	0.0523
ISO16-0004-006-DUP	P116; Sample Dup	1.72	0.753	8.79	<0.0500
Average		**1.73**	**0.777**	**8.88**	**0.0523**
%RPD Sample/Sample Dup		**1.2**	**6.1**	**1.9**	**NA**
ISO16-0004-007	Effluent WWTP; Sample	23.6	24.5	77.4	1.77
ISO16-0004-007-DUP	Effluent WWTP; Sample Dup	23.4	24.2	77.9	1.76
Average		**23.5**	**24.4**	**77.7**	**1.77**
%RPD Sample/Sample Dup		**0.85**	**1.2**	**0.64**	**0.57**
ISO16-0004-017	12; Sample	79.5	20.8	288	0.609
ISO16-0004-017-DUP	12; Sample Dup	79.6	21.1	283	0.646
Average		**79.6**	**21.0**	**286**	**0.628**
%RPD Sample/Sample Dup		**0.13**	**1.4**	**1.8**	**5.9**
ISO16-0004-018	13; Sample	75.9	21.3	239	0.513
ISO16-0004-018-DUP	13; Sample Dup	74.3	21.3	234	0.574
Average		**75.1**	**21.3**	**237**	**0.544**
%RPD Sample/Sample Dup		**2.1**	**0.0**	**2.1**	**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 ± 14%, PFBS ± 9.2%, PFHS ± 15%, and PFOS ± 18%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOSA ± 14%.

(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**	**PFHS**	**PFOS**	**PFOSA (2)**
ISO16-0004-019	5; Sample	19.9	14.3	34.6	0.184
ISO16-0004-019-DUP	5; Sample Dup	20.0	14.4	34.7	0.184
Average		**20.0**	**14.4**	**34.7**	**0.184**
%RPD Sample/Sample Dup		**0.50**	**0.70**	**0.29**	**0.0**
ISO16-0004-020	Bemalingsstation; Sample	20.8	14.9	42.5	0.230
ISO16-0004-020-DUP	Bemalingsstation; Sample Dup	21.2	14.9	43.2	0.242
Average		**21.0**	**14.9**	**42.9**	**0.236**
%RPD Sample/Sample Dup		**1.9**	**0.0**	**1.6**	**5.1**
ISO16-0004-021	Collector put; Sample	74.4	23.4	247	12.6
ISO16-0004-021-DUP	Collector put; Sample Dup	74.3	23.5	250	12.4
Average		**74.4**	**23.5**	**249**	**12.5**
%RPD Sample/Sample Dup		**0.13**	**0.43**	**1.2**	**1.6**
ISO16-0004-023	P263; Sample	1920	586	10800	10.2
ISO16-0004-023-DUP	P263; Sample Dup	1900	595	11100	10.2
Average		**1910**	**591**	**11000**	**10.2**
%RPD Sample/Sample Dup		**1.0**	**1.5**	**2.7**	**0.0**
ISO16-0004-025	L21; Sample	0.404	0.152	8.27	0.203
ISO16-0004-025-DUP	L21; Sample Dup	0.451	0.152	8.47	0.185
Average		**0.428**	**0.152**	**8.37**	**0.194**
%RPD Sample/Sample Dup		**11**	**0.0**	**2.4**	**9.3**
ISO16-0004-026	L22; Sample	0.294	0.157	6.12	0.0682
ISO16-0004-026-DUP	L22; Sample Dup	0.323	0.170	5.87	0.0823
Average		**0.309**	**0.164**	**6.00**	**0.0753**
%RPD Sample/Sample Dup		**9.4**	**8.0**	**4.2**	**19**
ISO16-0004-027	L31; Sample	0.563	0.755	4.45	<0.0500
ISO16-0004-027-DUP	L31; Sample Dup	0.563	0.772	4.18	<0.0500
Average		**0.563**	**0.764**	**4.32**	**<0.0500**
%RPD Sample/Sample Dup		**0.0**	**2.2**	**6.3**	**NA**
ISO16-0004-028	P115; Sample	4.72	2.29	1.03	<0.0500
ISO16-0004-028-DUP	P115; Sample Dup	4.73	2.38	1.10	<0.0500
Average		**4.73**	**2.34**	**1.07**	**<0.0500**
%RPD Sample/Sample Dup		**0.21**	**3.9**	**6.6**	**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 ± 14%, PFBS ± 9.2%, PFHS ± 15%, and PFOS ± 18%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOSA ± 14%.

(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)**
ISO16-0004-008	PP01; Sample	71.3	106	5650	1670	18.2
ISO16-0004-008-DUP	PP01; Sample Dup	74.0	114	6020	1760	15.2
Average		**72.7**	**110**	**5840**	**1720**	**16.7**
%RPD Sample/Sample Dup		**3.7**	**7.3**	**6.3**	**5.2**	**18**
ISO16-0004-009	PP02; Sample	587	27.9	2560	24700	33.5
ISO16-0004-009-DUP	PP02; Sample Dup	563	28.4	2570	25200	33.4
Average		**575**	**28.2**	**2570**	**25000**	**33.5**
%RPD Sample/Sample Dup		**4.2**	**1.8**	**0.39**	**2.0**	**0.30**
ISO16-0004-010	PP04; Sample	665	982	265	5360	53.1
ISO16-0004-010-DUP	PP04; Sample Dup	674	945	263	5130	53.1
Average		**670**	**964**	**264**	**5250**	**53.1**
%RPD Sample/Sample Dup		**1.3**	**3.8**	**0.76**	**4.4**	**0.0**
ISO16-0004-011	PP05; Sample	1720	22000	5220	227	51.3
ISO16-0004-011-DUP	PP05; Sample Dup	1770	21000	4960	225	49.4
Average		**1750**	**21500**	**5090**	**226**	**50.4**
%RPD Sample/Sample Dup		**2.9**	**4.7**	**5.1**	**0.88**	**3.8**
ISO16-0004-012	PP06; Sample	235	18.7	92.5	2030	187
ISO16-0004-012-DUP	PP06; Sample Dup	235	18.3	89.9	2040	186
Average		**235**	**18.5**	**91.2**	**2040**	**187**
%RPD Sample/Sample Dup		**0.0**	**2.2**	**2.9**	**0.49**	**0.54**
ISO16-0004-013	PP07; Sample	461	77.1	187	5230	183
ISO16-0004-013-DUP	PP07; Sample Dup	459	77.5	190	5110	183
Average		**460**	**77.3**	**189**	**5170**	**183**
%RPD Sample/Sample Dup		**0.43**	**0.52**	**1.6**	**2.3**	**0.0**
ISO16-0004-014	PP08; Sample	577	41.5	262	7680	50.5
ISO16-0004-014-DUP	PP08; Sample Dup	588	40.3	260	8130	45.9
Average		**583**	**40.9**	**261**	**7910**	**48.2**
%RPD Sample/Sample Dup		**1.9**	**2.9**	**0.77**	**5.7**	**9.5**
ISO16-0004-015	PP09; Sample	746	30.5	445	3170	18.0
ISO16-0004-015-DUP	PP09; Sample Dup	704	28.9	457	3290	17.6
Average		**725**	**29.7**	**451**	**3230**	**17.8**
%RPD Sample/Sample Dup		**5.8**	**5.4**	**2.7**	**3.7**	**2.2**
ISO16-0004-016	PP10; Sample	616	18.2	196	4490	16.0
ISO16-0004-016-DUP	PP10; Sample Dup	611	19.0	200	4530	15.3
Average		**614**	**18.6**	**198**	**4510**	**15.7**
%RPD Sample/Sample Dup		**0.81**	**4.3**	**2.0**	**0.89**	**4.5**
ISO16-0004-022	P21B; Sample	1870	2990	3510	41000	83.4
ISO16-0004-022-DUP	P21B; Sample Dup	1860	3040	3490	41400	85.1
Average		**1870**	**3020**	**3500**	**41200**	**84.3**
%RPD Sample/Sample Dup		**0.54**	**1.7**	**0.57**	**0.97**	**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 ± 14%, PFBS ± 9.2%, PFHS ± 15%, and PFOS ± 18%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOSA ± 14%.

(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 Environmental 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 (1)	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

(1) The addition of PFBS was only requested for extraction wells PP01, PP02, PP04-PP10 and P21B.

2.2 Sample Collection

Samples were collected between April 5, 2016 and April 7, 2016 in Nalgene^{TM} (high-density polyethylene) bottles prepared at the 3M Environmental 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. In addition, all sample bottles were pre-spiked with a mass-labeled internal standard mix at a nominal concentration of 1 ng/mL. Collected sample bottles were returned to the laboratory at ambient temperature on April 13, 2016.

2.3 Sample Preparation

All samples were prepared for PFOA, PFBS, PFHS, and PFOS using a direct injection with methanol dilution method. Samples that required a 1:5 dilution were prepared by diluting 2 mL of a well-mixed sample with 8 mL of methanol. 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 9.9 mL of methanol. Diluted samples and LCSs were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

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 1.0 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. The calibration standards and method blanks were also 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).

2.4 Analysis

Samples and quality control samples were analyzed for the target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). 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-

010.1. 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 Kirk**	**ETS Rita**
Liquid Chromatograph	Agilent 1260	Agilent 1260
Analysis Method	ETS-8-044.3	ETS-8-044.3
Analysis Date	4/14/16, 4/20/16, 4/26/16 (External Standard)	4/20/16 (Internal Standard)
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	5 µL	2 µL
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 6500+
Ion Source	Turbo Spray	Turbo Spray
Polarity	Negative	Negative
Software	Analyst 1.6.2	Analyst 1.6.3

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis				
Step Number	**Total Time (min)**	**Flow Rate (µL/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	4.00	300	70.0	30.0
3	6.00	300	70.0	30.0
4	11.0	300	20.0	80.0
5	13.0	300	20.0	80.0
6	13.5	300	10.0	90.0
7	16.0	300	10.0	90.0
8	16.5	300	90.0	10.0
9	19.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard (1)	Mass Transition Q1/Q3
PFOA	413/369	NA	NA
	413/219		
	413/169		
PFBS	299/99	NA	NA
	299/80		
PFHS	399/99	NA	NA
	399/80		
PFOS	499/99	NA	NA
	499/80		
	499/130		
PFOSA	498/78	[$^{13}C_8$]-PFOSA	506/78
[$^{13}C_4$]-PFOA surrogate	417/372	NA	NA
[$^{13}C_4$]-PFOS surrogate	503/80	NA	NA

NA = Not Applicable

Dwell time was 20 or 60 msec for each transition. The individual transitions were summed to produce a “total ion chromatogram” (TIC), which was used for quantitation.

(1) Internal standards were not used for the analysis of PFOA, PFBS, PFHS, or PFOS.

3 Data Analysis

3.1 Calibration

External Standard Calibration: Samples were analyzed for PFOA, PFBS, PFHS, and PFOS 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. On 4/14/16 and 4/20/16, calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed, and on 4/26/16, calibration standards ranged from 0.02 ng/mL to 5.0 ng/mL (nominal). 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 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.

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 25 mL of 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 were analyzed. Prior to analysis, the calibration standards were diluted 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±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 except for PFOS and [$^{13}C_4$]-PFOS on 4/20/16 with area ratio RSDs of 10% and 11%, respectively. All other QC elements met method acceptance criteria for PFOS and [$^{13}C_4$]-PFOS including CCVs, LCSs and FMSs, therefore; the data are reported.

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.

Table 6. LOQ

Analyte	LOQ, ng/mL (1) 4/14/16 Analysis	LOQ, ng/mL (1) 4/20/16 Analysis	LOQ, ng/mL (1) 4/20/16 Analysis	LOQ, ng/mL (1) 4/26/16 Analysis
PFOA	0.0192	NA	NA	NA
PFBS	NA	0.0200	NA	NA
PFHS	NA	0.0200	NA	NA
PFOS	NA	0.0464	NA	0.0185
PFOSA	NA	NA	0.0500	NA

NA = Not Applicable
(1) 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% (100%±30% for LOQ).

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/solvent 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 $^{13}C_4$-PFOA and $^{13}C_4$-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:

- On 4/20/16, the high level LCSs were prepared above the resulting ULOQ (50 ng/mL) for PFHS.

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.

$$\boxed{\text{LCS Percent Recovery} = \frac{\text{Calculated Concentration}}{\text{Spike Concentration}} * 100\%}$$

$$\boxed{\text{LCS\% RSD} = \frac{\text{standard deviation LCS replicates}}{\text{average LCS recovery}} * 100\%}$$

Table 7. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 4/14/16	PFOA (Linear + Branched)			[$^{13}C_4$]-PFOA (1)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160414-1	100	99.0	99.0	99.8	113	113
LCS-160414-2	100	97.0	97.0	99.8	100	100
LCS-160414-3	100	96.7	96.7	99.8	107	107
Average ± %RSD	**97.6% ± 1.3%**			**107% ± 6.1%**		
LCS-160414-4	5000	5460	109	998	1170	117
LCS-160414-5	5000	5460	109	998	1170	117
LCS-160414-6	5000	5230	105	998	1100	111
Average ± %RSD	**108% ± 2.1%**			**115% ± 3.0%**		
LCS-160414-7	35000	33400	95.3			
LCS-160414-8	35000	32200	92.0			
LCS-160414-9	35000	32200	91.9			
Average ± %RSD	**93.1% ± 2.1%**					

ETS-8-044.3 External Calibration Analyzed 4/20/16	PFBS			PFHS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160420-1	100	93.5	93.5	100	95.4	95.4
LCS-160420-2	100	97.5	97.5	100	95.7	95.7
LCS-160420-3	100	102	102	100	96.7	96.7
Average ± %RSD	**97.7% ± 4.4%**			**95.9% ± 0.71%**		
LCS-160420-4	5000	5370	107	5000	5350	107
LCS-160420-5	5000	5430	109	5000	5440	109
LCS-160420-6	5000	5040	101	5000	5160	103
Average ± %RSD	**106% ± 3.9%**			**106% ± 2.9%**		
LCS-160420-7	35000	34200	97.8	35000	>ULOQ	NA
LCS-160420-8	35000	34300	98.0	35000	>ULOQ	NA
LCS-160420-9	35000	34700	99.3	35000	>ULOQ	NA
Average ± %RSD	**98.4% ± 0.83%**			**NA (2)**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCS was spiked post dilution, but reported with the dilution factor applied.
(2) LCSs prepared above the resulting ULOQ.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 4/20/16	PFOS (Linear + Branched)			$[^{13}C_4]$-PFOS (1)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160420-1	100	93.6	93.6	95.6	106	111
LCS-160420-2	100	91.0	91.0	95.6	108	113
LCS-160420-3	100	90.3	90.3	95.6	111	116
Average ± %RSD	**91.6% ± 1.9%**			**113% ± 2.2%**		
LCS-160420-4	5000	5120	102	955	991	104
LCS-160420-5	5000	5230	105	955	980	103
LCS-160420-6	5000	4930	98.6	955	957	100
Average ± %RSD	**102% ± 3.1%**			**102% ± 2.0%**		
LCS-160420-7	35000	37100	106			
LCS-160420-8	35000	36400	104			
LCS-160420-9	35000	37800	108			
Average ± %RSD	**106% ± 1.9%**					

ETS-8-044.3 Internal Calibration Analyzed 4/20/16	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160420-1	0.199	0.182	91.5
LCS-160420-2	0.199	0.173	86.8
LCS-160420-3	0.199	0.176	88.3
Average ± %RSD	**88.9% ± 2.7%**		
LCS-160420-4	19.9	16.9	84.8
LCS-160420-5	19.9	16.4	82.5
LCS-160420-6	19.9	17.0	85.2
Average ± %RSD	**84.2% ± 1.7%**		
LCS-160420-7	139	120	86.3
LCS-160420-8	139	117	84.1
LCS-160420-9	139	117	84.0
Average ± %RSD	**84.8% ± 1.5%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCS was spiked post dilution, but reported with the dilution factor applied.
(2) LCSs prepared above the resulting ULOQ.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 4/26/16	**PFOS (Linear + Branched)**			**$[^{13}C_4]$-PFOS (1)**		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160426-1	100	88.0	88.0	95.6	103	108
LCS-160426-2	100	82.8	82.8	95.6	104	109
LCS-160426-3	100	84.4	84.4	95.6	108	113
Average ± %RSD	**85.1% ± 3.1%**			**110% ± 2.4%**		
LCS-160426-4	374	325	86.9	955	919	96.2
LCS-160426-5	374	327	87.5	955	954	99.9
LCS-160426-6	374	336	89.9	955	955	100
Average ± %RSD	**88.1% ± 1.8%**			**98.7% ± 2.2%**		
LCS-160426-7	1740	1620	92.9			
LCS-160426-8	1740	1590	91.2			
LCS-160426-9	1740	1590	91.1			
Average ± %RSD	**91.7% ± 1.1%**					

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCS was spiked post dilution, but reported with the dilution factor applied.
(2) LCSs prepared above the resulting ULOQ.

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-12-012.3. 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 (%)	Data Uncertainty
PFOA	External	7.14	±14%
PFBS	External	4.61	±9.2%
PFHS	External	7.71	±15%
PFOS	External	9.08	±18%
PFOSA	Internal	6.75	±14%

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 contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. The stock solutions of PFOS and PFOA used to prepare the field matrix spikes were prepared from a different reference source than the one used to prepare the calibration standards. 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 [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles following sample collection. Samples analyzed using external calibration were spiked with surrogate recovery standards post dilution. The [$^{13}C_4$]-PFOA was selected to represent the C8 perfluorocarboxylic acid. The [$^{13}C_4$]-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids and PFOSA. 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.

$$\text{FMS Recovery} = \frac{(\text{Sample Concentration of FMS} - \text{Average Concentration : Field Sample \& Field Sample Dup.})}{\text{Spike Concentration}} * 100\%$$

Table 9. Field Matrix Spike Concentrations

		Final Concentration (ng/mL)				
Sampling Location	**Spike Level**	**PFOA**	**PFBS**	**PFHS**	**PFOS**	**PFOSA**
P321	FMS	1.92	2.00	2.00	1.85	2.00
L31	FMS	4.79	5.0	5.00	4.64	5.00
L4, P21B, P263	FMS	10.0	10.0	10.0	10.0	10.0
Effluent WWTP, 5	FMS	51.9	52.0	52.0	51.9	2.00
PP09	FMS	500	50.0	500	500	50.0
PP04 (1)	FMS	442	44.2	442	442	44.2
Trip Blank	Low	10.0	10.0	10.0	10.0	10.0
	High	500	50.0	500	500	50.0

(1) FMS concentration was adjusted due to sample bottle being overfilled by greater than 10%.

4 Data Summary and Discussion

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. The field matrix spike concentration was selected based on the expected concentration of PFOA, PFOS, and/or PFOSA, based on previous results for the Antwerp locations. As a result the spike level, at times, 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. Surrogate recovery standards are summarized in **Table 20**. All field matrix spikes and surrogate recovery standards met method acceptance criteria except where noted below.

Sample location L4: The L4-FMS sample had a [$^{13}C_4$]-PFOA surrogate recovery of 136%. Since the average [$^{13}C_4$]-PFOA surrogate recovery for all samples in the L4 sample set was within acceptance criteria, the data uncertainty was not expanded further.

Table 10. Location ID: P321

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-001	P321; Sample	2980	NA	934	NA
ISO16-0004-001-DUP	P321; Sample Dup	3030	NA	940	NA
ISO16-0004-001-FMS	P321; FMS	3000	NC	965	NC
Average Concentration (ng/mL) ± %RPD		**3010 ng/mL ± 1.7%**		**937 ng/mL ± 0.64%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-001	P321; Sample	4940	NA	0.399	NA
ISO16-0004-001-DUP	P321; Sample Dup	4970	NA	0.384	NA
ISO16-0004-001-FMS	P321; FMS	5170	NC	2.48	104
Average Concentration (ng/mL) ± %RPD		**4960 ng/mL ± 0.61%**		**0.392 ng/mL ± 3.8%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 11. Location ID: L4

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-004	L4; Sample	1.06	NA	0.461	NA
ISO16-0004-004-DUP	L4; Sample Dup	1.03	NA	0.481	NA
ISO16-0004-004-FMS	L4; FMS	11.0	99.6	10.5	100 (1)
Average Concentration (ng/mL) ± %RPD		**1.05 ng/mL ± 2.9%**		**0.471 ng/mL ± 4.2%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-004	ISO16-0004-004	24.4	NA	5.97	NA
ISO16-0004-004-DUP	ISO16-0004-004-DUP	24.6	NA	5.99	NA
ISO16-0004-004-FMS	ISO16-0004-004-FMS	34.1	NC	17.6	116
Average Concentration (ng/mL) ± %RPD		**24.5 ng/mL ± 0.82%**		**5.98 ng/mL ± 0.33%**	

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**
ISO16-0004-007	Effluent WWTP; Sample	23.6	NA	24.5	NA
ISO16-0004-007-DUP	Effluent WWTP; Sample Dup	23.4	NA	24.2	NA
ISO16-0004-007-FMS	Effluent WWTP; FMS	68.7	87.1	73.6	94.7
Average Concentration (ng/mL) ± %RPD		**23.5 ng/mL ± 0.85%**		**24.4 ng/mL ± 1.2%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-007	Effluent WWTP; Sample	77.4	NA	1.77	NA
ISO16-0004-007-DUP	Effluent WWTP; Sample Dup	77.9	NA	1.76	NA
ISO16-0004-007-FMS	Effluent WWTP; FMS	121	83.6	3.94	109
Average Concentration (ng/mL) ± %RPD		**77.7 ng/mL ± 0.64%**		**1.77 ng/mL ± 0.57%**	

NA = Not Applicable

Table 13. Location ID: 5

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-019	5; Sample	19.9	NA	14.3	NA
ISO16-0004-019-DUP	5; Sample Dup	20.0	NA	14.4	NA
ISO16-0004-019-FMS	5; FMS	61.7	80.4	62.8	93.2
Average Concentration (ng/mL) ± %RPD		**20.0 ng/mL ± 0.50%**		**14.4 ng/mL ± 0.70%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-019	5; Sample	34.6	NA	0.184	NA
ISO16-0004-019-DUP	5; Sample Dup	34.7	NA	0.184	NA
ISO16-0004-019-FMS	5; FMS	78.5	84.6	2.27	104 (1)
Average Concentration (ng/mL) ± %RPD		**34.7 ng/mL ± 0.29%**		**0.184 ng/mL ± 0.0%**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.

Table 14. Location ID: P263

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-023	P263; Sample	1920	NA	586	NA
ISO16-0004-023-DUP	P263; Sample Dup	1900	NA	595	NA
ISO16-0004-023-FMS	P263; FMS	2000	NC	613	NC
Average Concentration (ng/mL) ± %RPD		**1910 ng/mL ± 1.0%**		**591 ng/mL ± 1.5%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-023	P263; Sample	10800	NA	10.2	NA
ISO16-0004-023-DUP	P263; Sample Dup	11100	NA	10.2	NA
ISO16-0004-023-FMS	P263; FMS	11600	NC	21.1	109
Average Concentration (ng/mL) ± %RPD		**11000 ng/mL ± 2.7%**		**10.2 ng/mL ± 0.0%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 15. Location ID: L31

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-027	L31; Sample	0.563	NA	0.755	NA
ISO16-0004-027-DUP	L31; Sample Dup	0.563	NA	0.772	NA
ISO16-0004-027-FMS	L31; FMS	5.53	104	5.93	103
Average Concentration (ng/mL) ± %RPD		**0.563 ng/mL ± 0.0%**		**0.764 ng/mL ± 2.2%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-027	L31; Sample	4.45	NA	<0.0500	NA
ISO16-0004-027-DUP	L31; Sample Dup	4.18	NA	<0.0500	NA
ISO16-0004-027-FMS	L31; FMS	9.36	109	5.35	107 (1)
Average Concentration (ng/mL) ± %RPD		**4.32 ng/mL ± 6.3%**		**<0.0500 ng/mL**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.

Table 16. Location ID: PP04

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-010	PP04; Sample	665	NA	982	NA	265	NA
ISO16-0004-010-DUP	PP04; Sample Dup	674	NA	945	NA	263	NA
ISO16-0004-010-FMS	PP04; FMS	1070	90.5	975	NC	640	85.0
Average Concentration (ng/mL) ± %RPD		**670 ng/mL ± 1.3%**		**964 ng/mL ± 3.8%**		**264 ng/mL ± 0.76%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-010	PP04; Sample	5360	NA	53.1	NA
ISO16-0004-010-DUP	PP04; Sample Dup	5130	NA	53.1	NA
ISO16-0004-010-FMS	PP04; FMS	5540	NC	93.0	90.2
Average Concentration (ng/mL) ± %RPD		**5250 ng/mL ± 4.4%**		**53.1 ng/mL ± 0.0%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 17. Location ID: PP09

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-015	PP09; Sample	746	NA	30.5	NA	445	NA
ISO16-0004-015-DUP	PP09; Sample Dup	704	NA	28.9	NA	457	NA
ISO16-0004-015-FMS	PP09; FMS	1220	99.0	79.5	99.6	979	106
Average Concentration (ng/mL) ± %RPD		**725 ng/mL ± 5.8%**		**29.7 ng/mL ± 5.4%**		**451 ng/mL ± 2.7%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-015	PP09; Sample	3170	NA	18.0	NA
ISO16-0004-015-DUP	PP09; Sample Dup	3290	NA	17.6	NA
ISO16-0004-015-FMS	PP09; FMS	3750	NC	66.5	97.4
Average Concentration (ng/mL) ± %RPD		**3230 ng/mL ± 3.7%**		**17.8 ng/mL ± 2.2%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 18. Location ID: P21B

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-022	P21B; Sample	1870	NA	2990	NA	3510	NA
ISO16-0004-022-DUP	P21B; Sample Dup	1860	NA	3040	NA	3490	NA
ISO16-0004-022-FMS	P21B; FMS	1830	NC	3040	NC	3600	NC
Average Concentration (ng/mL) ± %RPD		**1870 ng/mL ± 0.54%**		**3020 ng/mL ± 1.7%**		**3500 ng/mL ± 0.57%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-022	P21B; Sample	41000	NA	83.4	NA
ISO16-0004-022-DUP	P21B; Sample Dup	41400	NA	85.1	NA
ISO16-0004-022-FMS	P21B; FMS	41800	NC	98.2	NC
Average Concentration (ng/mL) ± %RPD		**41200 ng/mL ± 0.97%**		**84.3 ng/mL ± 2.0%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 19. Trip Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-024	Trip Blank; Sample	<0.192	NA	<0.101	NA	<0.101	NA
ISO16-0004-024-FMS-LOW	Trip Blank; FMS Low	10.0	100	10.0	100	10.1	101
ISO16-0004-024-FMS-HIGH	Trip Blank; FMS High	502	100	49.9	99.8	482	96.4

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0004-024	Trip Blank; Sample	<0.233	NA	<0.0500	NA
ISO16-0004-024-FMS-LOW	Trip Blank; FMS Low	10.3	103	10.1	101
ISO16-0004-024-FMS-HIGH	Trip Blank; FMS High	476	95.2	48.8	97.6

NA = Not Applicable

Table 20. Surrogate Recovery Standard Results (1)

		Recovery (%)		
3M LIMS ID	**Sample Description**	**[$^{13}C_4$]-PFOA**	**[$^{13}C_4$]-PFOS**	**[$^{13}C_4$]-PFOS Re-Analysis**
ISO16-0004-001	P321; Sample	108	97.2	NA
ISO16-0004-001-DUP	P321; Sample Dup	110	97.1	NA
ISO16-0004-001-FMS	P321; FMS	100	102	NA
ISO16-0004-002	3M vijver; Sample	124	105	NA
ISO16-0004-002-DUP	3M vijver; Sample Dup	125	105	NA
ISO16-0004-003	Blokkersdijkvijver std; Sample	119	106	NA
ISO16-0004-003-DUP	Blokkersdijkvijver std; Sample Dup	119	105	NA
ISO16-0004-004	L4; Sample	118	107	NA
ISO16-0004-004-DUP	L4; Sample Dup	124	107	NA
ISO16-0004-004-FMS	L4; FMS	136 (2)	105	NA
ISO16-0004-005	P114bis; Sample	130	108	NA
ISO16-0004-005-DUP	P114bis; Sample Dup	123	106	NA
ISO16-0004-006	P116; Sample	120	106	NA
ISO16-0004-006-DUP	P116; Sample Dup	120	106	NA
ISO16-0004-007	Effluent WWTP; Sample	121	105	NA
ISO16-0004-007-DUP	Effluent WWTP; Sample Dup	118	105	NA
ISO16-0004-007-FMS	Effluent WWTP; FMS	121	104	NA
ISO16-0004-008	PP01; Sample	117	99.4	NA
ISO16-0004-008-DUP	PP01; Sample Dup	117	117	NA
ISO16-0004-009	PP02; Sample	119	102	NA
ISO16-0004-009-DUP	PP02; Sample Dup	129	102	NA
ISO16-0004-010	PP04; Sample	124	111	NA
ISO16-0004-010-DUP	PP04; Sample Dup	130	110	NA
ISO16-0004-010-FMS	PP04; FMS	119	111	NA
ISO16-0004-011	PP05; Sample	121	109	99.8
ISO16-0004-011-DUP	PP05; Sample Dup	105	109	101
ISO16-0004-012	PP06; Sample	128	109	NA
ISO16-0004-012-DUP	PP06; Sample Dup	122	108	NA
ISO16-0004-013	PP07; Sample	122	103	NA
ISO16-0004-013-DUP	PP07; Sample Dup	124	104	NA
ISO16-0004-014	PP08; Sample	124	110	NA
ISO16-0004-014-DUP	PP08; Sample Dup	124	109	NA
ISO16-0004-015	PP09; Sample	112	105	NA
ISO16-0004-015-DUP	PP09; Sample Dup	130	106	NA
ISO16-0004-015-FMS	PP09; FMS	119	106	NA
ISO16-0004-016	PP10; Sample	123	107	NA
ISO16-0004-016-DUP	PP10; Sample Dup	120	107	NA
ISO16-0004-017	12; Sample	123	103	NA
ISO16-0004-017-DUP	12; Sample Dup	123	104	NA

NA = Not Applicable
(1) The surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS were added to the samples during sample preparation.
(2) Surrogate recovery standard did not meet acceptance criteria of 100 ± 30%.

Table 20 continued. Surrogate Recovery Standard Results (1)

		Recovery (%)	
3M LIMS ID	**Sample Description**	**[$^{13}C_4$]-PFOA**	**[$^{13}C_4$]-PFOS**
ISO16-0004-018	13; Sample	121	104
ISO16-0004-018-DUP	13; Sample Dup	105	104
ISO16-0004-019	5; Sample	114	106
ISO16-0004-019-DUP	5; Sample Dup	122	104
ISO16-0004-019-FMS	5; FMS	122	105
ISO16-0004-020	Bemalingsstation; Sample	124	103
ISO16-0004-020-DUP	Bemalingsstation; Sample Dup	123	108
ISO16-0004-021	Collector put; Sample	121	102
ISO16-0004-021-DUP	Collector put; Sample Dup	129	105
ISO16-0004-022	P21B; Sample	125	103
ISO16-0004-022-DUP	P21B; Sample Dup	124	103
ISO16-0004-022-FMS	P21B; FMS	113	105
ISO16-0004-023	P263; Sample	124	112
ISO16-0004-023-DUP	P263; Sample Dup	125	103
ISO16-0004-023-FMS	P263; FMS	126	123
ISO16-0004-024	Trip Blank; Sample	129	107
ISO16-0004-024-FMS-LOW	Trip Blank; FMS Low	129	105
ISO16-0004-024-FMS-HIGH	Trip Blank; FMS High	123	102
ISO16-0004-025	L21; Sample	127	113
ISO16-0004-025-DUP	L21; Sample Dup	125	111
ISO16-0004-026	L22; Sample	126	109
ISO16-0004-026-DUP	L22; Sample Dup	128	109
ISO16-0004-027	L31; Sample	127	107
ISO16-0004-027-DUP	L31; Sample Dup	125	108
ISO16-0004-027-FMS	L31; FMS	109	108
ISO16-0004-028	P115; Sample	121	110
ISO16-0004-028-DUP	P115; Sample Dup	128	114

NA = Not Applicable
(1) The surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS were added to the samples during sample preparation.
(2) 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 Environmental 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 Environmental Laboratory standard operating procedures.

7 Signatures

Chelsie Grochow, 3M Report Author

Susan T. Wolf, 3M Principal Analytical Investigator

William K. Reagen, Ph.D., 3M Environmental Laboratory Technical Director

The 3M Environmental 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 Environmental Laboratory.

Final Report

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Antwerp, Belgium

July 2016 Sampling

Laboratory Request Number: ISO16-0008

Report Date – Date of Last Signature

Testing Laboratory

3M Environment, Health, Safety, and Sustainability
3M Environmental 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 Environmental Laboratory

3M Environmental Laboratory Technical Director: William K. Reagen, Ph.D.
3M Principal Analytical Investigator: Susan Wolf
Report Author: Chelsie Grochow

Analytical Report ISO16-0008

Water Sample Analysis at 3M Antwerp, Belgium
July 2016 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M Environmental 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 initially collected between July 20, 2016 and July 29, 2016 and returned to the 3M Environmental Laboratory on August 12, 2016 at ambient temperature. However, twenty sample bottles were not received which included sample locations P121, L4, WWTP Effluent, PP06, PP07, Collector pit, M4, P264, and P265C. These sample locations were recollected on September 19, 2016 and received on September 23, 2016 at ambient temperature. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO16-0008.

The 3M Environmental Laboratory prepared sample containers for seventy-three sampling locations. Each sample set consisted of a field sample and field sample duplicate. Twenty-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 [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS prior to being sent to the field for sample collection. Sample location B7 was not collected. According to the chain of custody, sample location P265C could not be sampled; therefore, P265B was collected in lieu of P265C. A GPO deviation is included with the raw data.

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.

ilac-MRA ACCREDITED Testing Cert. # 2052.01

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: 2nd Aquifer					
ISO16-0008-001	BD24-3; Sample	73.7	58.3	1230	4.66
ISO16-0008-001-DUP	BD24-3; Sample Dup	80.9	60.1	1260	4.54
Average		**77.3**	**59.2**	**1245**	**4.60 (2)**
%RPD Sample/Sample Dup		**9.3**	**3.0**	**2.4**	**2.6**
ISO16-0008-002	BD24-4; Sample	10.5	4.65	8.82	0.936
ISO16-0008-002-DUP	BD24-4; Sample Dup	10.2	4.54	9.03	0.736
Average		**10.4**	**4.60**	**8.93**	**0.836 (2)**
%RPD Sample/Sample Dup		**2.9**	**2.4**	**2.4**	**24 (3)**
ISO16-0008-003	D09; Sample	125	3260	1930	30.2
ISO16-0008-003-DUP	D09; Sample Dup	122	3230	1940	29.0
Average		**124**	**3250**	**1940**	**29.6**
%RPD Sample/Sample Dup		**2.4**	**0.92**	**0.52**	**4.1**
ISO16-0008-004	D10; Sample	485	144	36.0	0.527
ISO16-0008-004-DUP	D10; Sample Dup	478	141	36.5	0.514
Average		**482**	**143**	**36.3**	**0.521 (2)**
%RPD Sample/Sample Dup		**1.5**	**2.1**	**1.4**	**2.5**
ISO16-0008-005	D11; Sample	300	145	996	62.4
ISO16-0008-005-DUP	D11; Sample Dup	290	149	1010	64.3
Average		**295**	**147**	**1000**	**63.4**
%RPD Sample/Sample Dup		**3.4**	**2.7**	**1.4**	**3.0**
ISO16-0008-006	D14; Sample	2.44	1.38	7.18	0.647
ISO16-0008-006-DUP	D14; Sample Dup	2.60	1.48	7.26	0.753
Average		**2.52 (2)**	**1.43 (2)**	**7.22**	**0.700 (2)**
%RPD Sample/Sample Dup		**6.3**	**7.0**	**1.1**	**15**
ISO16-0008-007	D16; Sample	262	59.0	1650	75.7
ISO16-0008-007-DUP	D16; Sample Dup	263	58.1	1630	79.0
Average		**263**	**58.6**	**1640**	**77.4**
%RPD Sample/Sample Dup		**0.38**	**1.5**	**1.2**	**4.3**
ISO16-0008-008	D17; Sample	1910	121	437	26.6
ISO16-0008-008-DUP	D17; Sample Dup	1780	114	444	25.6
Average		**1850**	**118**	**441**	**26.1**
%RPD Sample/Sample Dup		**7.0**	**6.0**	**1.6**	**3.8**
ISO16-0008-009	D18; Sample	521	288	17.6	2.62
ISO16-0008-009-DUP	D18; Sample Dup	521	289	19.2	2.41
Average		**521**	**289**	**18.4**	**2.52 (2)**
%RPD Sample/Sample Dup		**0.0**	**0.35**	**8.7**	**8.3**

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 ± 21%, PFBS ± 12%, PFHS ± 13%, PFOS ± 21%, and PFOSA ± 36%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 11%, PFHS ± 13%, and PFOSA ± 12%.

(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(4) The data uncertainty for samples within the WWTP, Southern Site Boundary, and Building 16 locations has been expanded to ± 33% for PFOA based on field matrix spike recovery. See section 4 for details.

(5) The data uncertainty has been expanded based on field matrix spike or laboratory control spike recovery. See section 4 for details.

(6) The field matrix spike sample for location P305 did not meet method acceptance criteria and ***the report result for PFOA should be considered qualitative*** with an expanded analytical uncertainty of ± 52%. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
Zone: 2nd Aquifer					
ISO16-0008-010	D2; Sample	0.347	0.166	9.83	0.519
ISO16-0008-010-DUP	D2; Sample Dup	0.380	0.165	9.64	0.471
	Average	**0.364** (2)	**0.166** (2)	**9.74**	**0.495** (2)
	%RPD Sample/Sample Dup	**9.1**	**0.60**	**2.0**	**9.7**
ISO16-0008-011	D5; Sample	504	436	388	15.0
ISO16-0008-011-DUP	D5; Sample Dup	459	429	439	12.7
	Average	**482**	**433**	**414**	**13.9** (2)
	%RPD Sample/Sample Dup	**9.3**	**1.6**	**12**	**17**
ISO16-0008-012	ND7; Sample	169	6.84	282	384
ISO16-0008-012-DUP	ND7; Sample Dup	172	6.37	272	344
	Average	**171**	**6.61**	**277**	**364**
	%RPD Sample/Sample Dup	**1.8**	**7.1**	**3.6**	**11**
ISO16-0008-013	P118A; Sample	1300	780	5070	29.0
ISO16-0008-013-DUP	P118A; Sample Dup	1360	780	4980	29.9
	Average	**1330**	**780**	**5030**	**29.5**
	%RPD Sample/Sample Dup	**4.5**	**0.0**	**1.8**	**3.1**
ISO16-0008-014	P118B; Sample	2410	2280	10800	47.6
ISO16-0008-014-DUP	P118B; Sample Dup	2430	2270	10400	48.2
	Average	**2420**	**2280**	**10600**	**47.9**
	%RPD Sample/Sample Dup	**0.83**	**0.44**	**3.8**	**1.3**
ISO16-0008-015	P119A; Sample	144	37.2	71.0	13.6
ISO16-0008-015-DUP	P119A; Sample Dup	151	38.1	67.7	13.4
	Average	**148**	**37.7**	**69.4**	**13.5** (2)
	%RPD Sample/Sample Dup	**4.7**	**2.4**	**4.8**	**1.5**
ISO16-0008-016	P119B; Sample	2220	1090	436	10.6
ISO16-0008-016-DUP	P119B; Sample Dup	2240	1070	426	11.0
	Average	**2230**	**1080**	**431**	**10.8** (2)
	%RPD Sample/Sample Dup	**0.90**	**1.9**	**2.3**	**3.7**
ISO16-0008-075	P121; Sample	0.322	0.122	2.42	0.213
ISO16-0008-075-DUP	P121; Sample Dup	0.323	0.142	2.62	0.179
	Average	**0.323**	**0.132**	**2.52**	**0.196** (2)
	%RPD Sample/Sample Dup	**0.31**	**15**	**7.9**	**17**
ISO16-0008-018	P321; Sample	1170	376	4360	18.7
ISO16-0008-018-DUP	P321; Sample Dup	1200	372	4320	18.3
	Average	**1190**	**374**	**4340**	**18.5**
	%RPD Sample/Sample Dup	**2.5**	**1.1**	**0.92**	**2.2**

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 ± 21%, PFBS ± 12%, PFHS ± 13%, PFOS ± 21%, and PFOSA ± 36%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 11%, PFHS ± 13%, and PFOSA ± 12%.

(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(4) The data uncertainty for samples within the WWTP, Southern Site Boundary, and Building 16 locations has been expanded to ± 33% for PFOA based on field matrix spike recovery. See section 4 for details.

(5) The data uncertainty has been expanded based on field matrix spike or laboratory control spike recovery. See section 4 for details.

(6) The field matrix spike sample for location P305 did not meet method acceptance criteria and ***the report result for PFOA should be considered qualitative*** with an expanded analytical uncertainty of ± 52%. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
Zone: Blokkersdijk nature reserve					
ISO16-0008-019	3M vijver; Sample	2.18	2.54	12.8	1.13
ISO16-0008-019-DUP	3M vijver; Sample Dup	2.17	2.53	13.3	1.22
	Average	**2.18** (2)	**2.54** (2, 5)	**13.1**	**1.18** (2)
	%RPD Sample/Sample Dup	**0.46**	**0.39**	**3.8**	**7.7**
ISO16-0008-020	Blokkersdijkvijver standard; Sample	1.75	2.20	10.2	1.52
ISO16-0008-020-DUP	Blokkersdijkvijver standard; Sample Dup	1.73	2.16	11.3	1.54
	Average	**1.74** (2)	**2.18** (2)	**10.8**	**1.53** (2)
	%RPD Sample/Sample Dup	**1.1**	**1.8**	**10**	**1.3**
ISO16-0008-021	L21; Sample	0.436	0.194	5.82	0.333
ISO16-0008-021-DUP	L21; Sample Dup	0.403	0.191	6.52	0.307
	Average	**0.420** (2)	**0.193** (2)	**6.17**	**0.320** (2)
	%RPD Sample/Sample Dup	**7.9**	**1.6**	**11**	**8.1**
ISO16-0008-022	L22; Sample	0.843	0.170	4.68	0.347
ISO16-0008-022-DUP	L22; Sample Dup	0.830	0.166	5.19	0.333
	Average	**0.837** (2)	**0.168** (2)	**4.94**	**0.340** (2)
	%RPD Sample/Sample Dup	**1.6**	**2.4**	**10**	**4.1**
ISO16-0008-023	L31; Sample	0.685	0.370	10.2	0.530
ISO16-0008-023-DUP	L31; Sample Dup	0.787	0.402	9.68	0.691
	Average	**0.736** (2)	**0.386** (2)	**9.94**	**0.611** (2)
	%RPD Sample/Sample Dup	**14**	**8.3**	**5.2**	**26** (3)
ISO16-0008-076	L4; Sample	3.32	2.78	30.5	1.87
ISO16-0008-076-DUP	L4; Sample Dup	3.15	2.75	29.9	1.61
	Average	**3.24**	**2.77**	**30.2**	**1.74**
	%RPD Sample/Sample Dup	**5.3**	**1.1**	**2.0**	**15**
ISO16-0008-025	P114bis; Sample	2.67	1.51	16.2	0.198
ISO16-0008-025-DUP	P114bis; Sample Dup	2.69	1.53	15.4	0.189
	Average	**2.68** (2)	**1.52** (2)	**15.8**	**0.194** (2)
	%RPD Sample/Sample Dup	**0.75**	**1.3**	**5.1**	**4.7**
ISO16-0008-026	P115; Sample	4.12	2.42	0.495	0.0316
ISO16-0008-026-DUP	P115; Sample Dup	3.74	2.20	0.569	0.0379
	Average	**3.93**	**2.31**	**0.532**	**0.0348** (2)
	%RPD Sample/Sample Dup	**9.7**	**9.5**	**14**	**18**
ISO16-0008-027	P116; Sample	2.92	1.10	10.4	0.407
ISO16-0008-027-DUP	P116; Sample Dup	2.00	0.883	10.8	0.299
	Average	**2.46** (2)	**0.992** (2)	**10.6**	**0.353** (2)
	%RPD Sample/Sample Dup	**37** (3)	**22** (3)	**3.8**	**31** (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 ± 21%, PFBS ± 12%, PFHS ± 13%, PFOS ± 21%, and PFOSA ± 36%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 11%, PFHS ± 13%, and PFOSA ± 12%.

(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(4) The data uncertainty for samples within the WWTP, Southern Site Boundary, and Building 16 locations has been expanded to ± 33% for PFOA based on field matrix spike recovery. See section 4 for details.

(5) The data uncertainty has been expanded based on field matrix spike or laboratory control spike recovery. See section 4 for details.

(6) The field matrix spike sample for location P305 did not meet method acceptance criteria and ***the report result for PFOA should be considered qualitative*** with an expanded analytical uncertainty of ± 52%. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
Zone: Effluent WWTP					
ISO16-0008-077	Effluent WWTP; Sample	13.6	23.6	3.79	0.0661
ISO16-0008-077-DUP	Effluent WWTP; Sample Dup	13.3	23.2	3.73	0.0580
	Average	**13.5**	**23.4**	**3.76**	**0.0621**
	%RPD Sample/Sample Dup	**2.2**	**1.7**	**1.6**	**13**
Zone: Palingbeek & Tophatgracht					
ISO16-0008-038	12; Sample	97.7	25.5	325	1.52
ISO16-0008-038-DUP	12; Sample Dup	95.6	25.5	266	1.55
	Average	**96.7**	**25.5**	**296**	**1.54 (2)**
	%RPD Sample/Sample Dup	**2.2**	**0.0**	**20**	**2.0**
ISO16-0008-039	13; Sample	87.1	24.6	236	1.35
ISO16-0008-039-DUP	13; Sample Dup	87.3	24.0	251	1.06
	Average	**87.2**	**24.3**	**244**	**1.21 (2)**
	%RPD Sample/Sample Dup	**0.23**	**2.5**	**6.2**	**24 (3)**
ISO16-0008-040	5; Sample	34.2	26.0	44.4	0.610
ISO16-0008-040-DUP	5; Sample Dup	34.6	26.7	46.0	0.555
	Average	**34.4**	**26.4**	**45.2**	**0.583 (2)**
	%RPD Sample/Sample Dup	**1.2**	**2.7**	**3.5**	**9.4**
ISO16-0008-041	Bemalingsstation; Sample	33.3	24.5	55.1	0.416
ISO16-0008-041-DUP	Bemalingsstation; Sample Dup	32.7	24.4	55.1	0.525
	Average	**33.0**	**24.5**	**55.1**	**0.471 (2)**
	%RPD Sample/Sample Dup	**1.8**	**0.41**	**0.0**	**23 (3)**
Zone: Sewer					
ISO16-0008-080	Collector pit; Sample	106	34.2	469	22.5
ISO16-0008-080-DUP	Collector pit; Sample Dup	106	35.0	418	19.5
	Average	**106**	**34.6**	**444**	**21.0**
	%RPD Sample/Sample Dup	**0.0**	**2.3**	**11**	**14**
Zone: Source area – WWTP					
ISO16-0008-050	L19; Sample	237	82.0	5610	559
ISO16-0008-050-DUP	L19; Sample Dup	239	83.2	5750	543
	Average	**238 (4)**	**82.6**	**5680**	**551 (5)**
	%RPD Sample/Sample Dup	**0.84**	**1.5**	**2.5**	**2.9**
ISO16-0008-081	M4; Sample	576	168	11000	250
ISO16-0008-081-DUP	M4; Sample Dup	568	166	11200	258
	Average	**572 (4)**	**167**	**11100**	**254**
	%RPD Sample/Sample Dup	**1.4**	**1.2**	**1.8**	**3.1**

NA = Not Applicable

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(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 11%, PFHS ± 13%, and PFOSA ± 12%.

(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(4) The data uncertainty for samples within the WWTP, Southern Site Boundary, and Building 16 locations has been expanded to ± 33% for PFOA based on field matrix spike recovery. See section 4 for details.

(5) The data uncertainty has been expanded based on field matrix spike or laboratory control spike recovery. See section 4 for details.

(6) The field matrix spike sample for location P305 did not meet method acceptance criteria and ***the report result for PFOA should be considered qualitative*** with an expanded analytical uncertainty of ± 52%. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
Zone: Source area - WWTP					
ISO16-0008-052	P118C; Sample	303	103	2070	58.6
ISO16-0008-052-DUP	P118C; Sample Dup	308	105	2120	55.0
	Average	**306 (4)**	**104**	**2100**	**56.8**
	%RPD Sample/Sample Dup	**1.6**	**1.9**	**2.4**	**6.3**
ISO16-0008-053	P119C; Sample	542	285	3840	85.9
ISO16-0008-053-DUP	P119C; Sample Dup	541	287	3940	88.6
	Average	**542 (4)**	**286**	**3890**	**87.3**
	%RPD Sample/Sample Dup	**0.18**	**0.70**	**2.6**	**3.1**
ISO16-0008-054	P262; Sample	14.5	4.19	287	140
ISO16-0008-054-DUP	P262; Sample Dup	14.8	4.31	248	138
	Average	**14.7 (4)**	**4.25**	**268**	**139**
	%RPD Sample/Sample Dup	**2.0**	**2.8**	**15**	**1.4**
ISO16-0008-055	P263; Sample	1680	522	10200	16.9
ISO16-0008-055-DUP	P263; Sample Dup	1700	526	9900	17.1
	Average	**1690 (4)**	**524**	**10100**	**17.0**
	%RPD Sample/Sample Dup	**1.2**	**0.76**	**3.0**	**1.2**
ISO16-0008-082	P264; Sample	301	176	1530	91.5
ISO16-0008-082-DUP	P264; Sample Dup	299	176	1550	102
	Average	**300 (4)**	**176**	**1540**	**96.8**
	%RPD Sample/Sample Dup	**0.67**	**0.0**	**1.3**	**11**
ISO16-0008-083	P265B; Sample	138	36.0	1510	202
ISO16-0008-083-DUP	P265B; Sample Dup	137	36.1	1520	201
	Average	**138 (4)**	**36.1**	**1520**	**202**
	%RPD Sample/Sample Dup	**0.73**	**0.28**	**0.66**	**0.50**
ISO16-0008-058	P340; Sample	679	304	3500	79.2
ISO16-0008-058-DUP	P340; Sample Dup	687	302	3470	69.8
	Average	**683 (4)**	**303**	**3490**	**74.5**
	%RPD Sample/Sample Dup	**1.2**	**0.66**	**0.86**	**13**
ISO16-0008-059	P341; Sample	548	312	5720	116
ISO16-0008-059-DUP	P341; Sample Dup	540	314	5740	113
	Average	**544 (4)**	**313**	**5730**	**115**
	%RPD Sample/Sample Dup	**1.5**	**0.64**	**0.35**	**2.6**
ISO16-0008-060	P343; Sample	1020	759	7540	21.3
ISO16-0008-060-DUP	P343; Sample Dup	1020	749	7580	21.5
	Average	**1020 (4)**	**754**	**7560**	**21.4**
	%RPD Sample/Sample Dup	**0.0**	**1.3**	**0.53**	**0.93**

NA = Not Applicable

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(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 11%, PFHS ± 13%, and PFOSA ± 12%.

(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(4) The data uncertainty for samples within the WWTP, Southern Site Boundary, and Building 16 locations has been expanded to ± 33% for PFOA based on field matrix spike recovery. See section 4 for details.

(5) The data uncertainty has been expanded based on field matrix spike or laboratory control spike recovery. See section 4 for details.

(6) The field matrix spike sample for location P305 did not meet method acceptance criteria and ***the report result for PFOA should be considered qualitative*** with an expanded analytical uncertainty of ± 52%. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
Zone: Source area - WWTP					
ISO16-0008-061	P371; Sample	234	68.1	3790	83.7
ISO16-0008-061-DUP	P371; Sample Dup	232	68.8	3860	84.9
	Average	**233 (4)**	**68.5**	**3830**	**84.3**
	%RPD Sample/Sample Dup	**0.86**	**1.0**	**1.8**	**1.4**
ISO16-0008-062	P374; Sample	457	568	4930	22.8
ISO16-0008-062-DUP	P374; Sample Dup	468	556	4800	21.9
	Average	**463 (4)**	**562**	**4870**	**22.4**
	%RPD Sample/Sample Dup	**2.4**	**2.1**	**2.7**	**4.0**
ISO16-0008-063	P379; Sample	73.9	19.7	691	78.3
ISO16-0008-063-DUP	P379; Sample Dup	75.9	20.2	606	79.0
	Average	**74.9 (4)**	**20.0**	**649**	**78.7**
	%RPD Sample/Sample Dup	**2.7**	**2.5**	**13**	**0.89**
ISO16-0008-064	P380; Sample	82.1	48.7	915	114
ISO16-0008-064-DUP	P380; Sample Dup	81.8	47.3	893	118
	Average	**82.0 (4)**	**48.0**	**904**	**116**
	%RPD Sample/Sample Dup	**0.37**	**2.9**	**2.4**	**3.4**
ISO16-0008-065	P381; Sample	606	159	1320	16.9
ISO16-0008-065-DUP	P381; Sample Dup	603	161	1400	17.6
	Average	**605 (4)**	**160**	**1360**	**17.3 (2)**
	%RPD Sample/Sample Dup	**0.50**	**1.3**	**5.9**	**4.1**
ISO16-0008-066	P382; Sample	1100	334	1070	83.5
ISO16-0008-066-DUP	P382; Sample Dup	1080	332	1070	90.0
	Average	**1090 (4)**	**333**	**1070**	**86.8**
	%RPD Sample/Sample Dup	**1.8**	**0.60**	**0.0**	**7.5**
Zone: Southern site boundary					
ISO16-0008-067	B3-bis; Sample	6.91	1.63	177	1.09
ISO16-0008-067-DUP	B3-bis; Sample Dup	6.32	1.65	193	1.24
	Average	**6.62 (4)**	**1.64**	**185**	**1.17 (2)**
	%RPD Sample/Sample Dup	**8.9**	**1.2**	**8.6**	**13**
ISO16-0008-069	P372; Sample	71.1	45.6	1140	59.7
ISO16-0008-069-DUP	P372; Sample Dup	69.7	45.3	1290	61.0
	Average	**70.4 (4)**	**45.5**	**1220**	**60.4**
	%RPD Sample/Sample Dup	**2.0**	**0.66**	**12**	**2.2**

NA = Not Applicable

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(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 11%, PFHS ± 13%, and PFOSA ± 12%.

(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(4) The data uncertainty for samples within the WWTP, Southern Site Boundary, and Building 16 locations has been expanded to ± 33% for PFOA based on field matrix spike recovery. See section 4 for details.

(5) The data uncertainty has been expanded based on field matrix spike or laboratory control spike recovery. See section 4 for details.

(6) The field matrix spike sample for location P305 did not meet method acceptance criteria and ***the report result for PFOA should be considered qualitative*** with an expanded analytical uncertainty of ± 52%. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
Zone: Southern site boundary					
ISO16-0008-070	P378; Sample	370	430	1750	4.33
ISO16-0008-070-DUP	P378; Sample Dup	370	422	1670	5.55
	Average	**370 (4)**	**426**	**1710**	**4.94 (2)**
	%RPD Sample/Sample Dup	**0.0**	**1.9**	**4.7**	**25**
ISO16-0008-071	PA109A; Sample	40.1	40.1	135	2.40
ISO16-0008-071-DUP	PA109A; Sample Dup	40.2	39.6	126	2.01
	Average	**40.2 (4)**	**39.9**	**131**	**2.21 (2)**
	%RPD Sample/Sample Dup	**0.25**	**1.3**	**6.9**	**18**
ISO16-0008-072	PA111A; Sample	873	1100	2360	12.3
ISO16-0008-072-DUP	PA111A; Sample Dup	831	1070	2290	12.2
	Average	**852 (4)**	**1090**	**2330**	**12.3**
	%RPD Sample/Sample Dup	**4.9**	**2.8**	**3.0**	**0.82**
ISO16-0008-073	PA112; Sample	127	33.9	414	83.8
ISO16-0008-073-DUP	PA112; Sample Dup	130	33.9	419	84.4
	Average	**129 (4)**	**33.9**	**417**	**84.1**
	%RPD Sample/Sample Dup	**2.3**	**0.0**	**1.2**	**0.71**

		Concentration (ng/mL)				
3M LIMS ID	**Sample Description**	**PFOA**	**PFBS**	**PFHS**	**PFOS**	**PFOSA**
Zone: Extractions wells P&T						
ISO16-0008-029	PP01; Sample	65.3	58.9	5940	2430	63.1
ISO16-0008-029-DUP	PP01; Sample Dup	65.4	58.8	5930	2430	58.5
	Average	**65.4**	**58.9**	**5940**	**2430**	**60.8**
	%RPD Sample/Sample Dup	**0.15**	**0.17**	**0.17**	**0.0**	**7.6**
ISO16-0008-030	PP02; Sample	661	30.9	2380	21900	56.3
ISO16-0008-030-DUP	PP02; Sample Dup	632	28.6	2310	20900	56.7
	Average	**647**	**29.8**	**2350**	**21400**	**56.5**
	%RPD Sample/Sample Dup	**4.5**	**7.7**	**3.0**	**4.7**	**0.71**
ISO16-0008-031	PP04; Sample	2560	1690	713	3930	53.1
ISO16-0008-031-DUP	PP04; Sample Dup	2400	1640	677	3780	51.1
	Average	**2480**	**1670**	**695**	**3860**	**52.1**
	%RPD Sample/Sample Dup	**6.5**	**3.0**	**5.2**	**3.9**	**3.8**
ISO16-0008-032	PP05; Sample	750	9260	3840	1100	174
ISO16-0008-032-DUP	PP05; Sample Dup	672	8830	3640	936	185
	Average	**711**	**9050**	**3740**	**1020**	**180**
	%RPD Sample/Sample Dup	**11**	**4.8**	**5.3**	**16**	**6.1**

NA = Not Applicable

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(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 11%, PFHS ± 13%, and PFOSA ± 12%.

(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(4) The data uncertainty for samples within the WWTP, Southern Site Boundary, and Building 16 locations has been expanded to ± 33% for PFOA based on field matrix spike recovery. See section 4 for details.

(5) The data uncertainty has been expanded based on field matrix spike or laboratory control spike recovery. See section 4 for details.

(6) The field matrix spike sample for location P305 did not meet method acceptance criteria and ***the report result for PFOA should be considered qualitative*** with an expanded analytical uncertainty of ± 52%. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)				
3M LIMS ID	**Sample Description**	**PFOA**	**PFBS**	**PFHS**	**PFOS**	**PFOSA**
Zone: Extractions wells P&T						
ISO16-0008-078	PP06; Sample	224	18.7	54.0	1720	78.2
ISO16-0008-078-DUP	PP06; Sample Dup	214	17.8	52.4	1660	78.3
	Average	**219**	**18.3**	**53.2**	**1690**	**78.3**
	%RPD Sample/Sample Dup	**4.6**	**4.9**	**3.0**	**3.6**	**0.13**
ISO16-0008-079	PP07; Sample	646	65.4	308	8960	62.5
ISO16-0008-079-DUP	PP07; Sample Dup	629	64.7	303	8740	66.8
	Average	**638**	**65.1**	**306**	**8850**	**64.7**
	%RPD Sample/Sample Dup	**2.7**	**1.1**	**1.6**	**2.5**	**6.7**
ISO16-0008-035	PP08; Sample	541	23.7	203	8060	68.6
ISO16-0008-035-DUP	PP08; Sample Dup	536	21.1	203	8120	66.6
	Average	**539**	**22.4**	**203**	**8090**	**67.6**
	%RPD Sample/Sample Dup	**0.93**	**12**	**0.0**	**0.74**	**3.0**
ISO16-0008-036	PP09; Sample	759	28.8	434	3040	20.7
ISO16-0008-036-DUP	PP09; Sample Dup	773	28.1	440	3040	19.8
	Average	**766**	**28.5**	**437**	**3040**	**20.3**
	%RPD Sample/Sample Dup	**1.8**	**2.5**	**1.4**	**0.0**	**4.4**
ISO16-0008-037	PP10; Sample	590	16.4	185	4060	24.9
ISO16-0008-037-DUP	PP10; Sample Dup	591	18.5	227	3930	25.0
	Average	**591**	**17.5**	**206**	**4000**	**25.0**
	%RPD Sample/Sample Dup	**0.17**	**12**	**20**	**3.3**	**0.40**
Zone: Source area - Building 16						
ISO16-0008-043	K3; Sample	123	1490	83000	5660	17.7
ISO16-0008-043-DUP	K3; Sample Dup	118	1490	85300	5680	19.4
	Average	**121 (4)**	**1490**	**84200**	**5670**	**18.6 (2)**
	%RPD Sample/Sample Dup	**4.1**	**0.0**	**2.7**	**0.35**	**9.2**
ISO16-0008-044	P27; Sample	202	3230	58.7	820	278
ISO16-0008-044-DUP	P27; Sample Dup	199	3310	54.9	809	265
	Average	**201 (4)**	**3270**	**56.8**	**815**	**272**
	%RPD Sample/Sample Dup	**1.5**	**2.4**	**6.7**	**1.4**	**4.8**
ISO16-0008-045	P21B; Sample	10700	6060	9870	58200	6.24
ISO16-0008-045-DUP	P21B; Sample Dup	11100	6330	10300	64400	5.92
	Average	**10900 (4)**	**6200**	**10100**	**61300**	**6.08 (2)**
	%RPD Sample/Sample Dup	**3.7**	**4.4**	**4.3**	**10**	**5.3**

NA = Not Applicable

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(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 11%, PFHS ± 13%, and PFOSA ± 12%.

(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

(4) The data uncertainty for samples within the WWTP, Southern Site Boundary, and Building 16 locations has been expanded to ± 33% for PFOA based on field matrix spike recovery. See section 4 for details.

(5) The data uncertainty has been expanded based on field matrix spike or laboratory control spike recovery. See section 4 for details.

(6) The field matrix spike sample for location P305 did not meet method acceptance criteria and ***the report result for PFOA should be considered qualitative*** with an expanded analytical uncertainty of ± 52%. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)				
3M LIMS ID	**Sample Description**	**PFOA**	**PFBS**	**PFHS**	**PFOS**	**PFOSA**
Zone: Source area - Building 16						
ISO16-0008-046	P304; Sample	152	60.4	33.8	365	18.3
ISO16-0008-046-DUP	P304; Sample Dup	149	57.0	33.0	350	16.9
	Average	**151 (4)**	**58.7**	**33.4**	**358**	**17.6 (2)**
	%RPD Sample/Sample Dup	**2.0**	**5.8**	**2.4**	**4.2**	**8.0**
ISO16-0008-047	P305; Sample	211	134	279	533	20.6
ISO16-0008-047-DUP	P305; Sample Dup	224	133	293	567	22.3
	Average	**218 (6)**	**134**	**286**	**550**	**21.5**
	%RPD Sample/Sample Dup	**6.0**	**0.75**	**4.9**	**6.2**	**7.9**
ISO16-0008-048	P42; Sample	304	48.0	433	4120	23.6
ISO16-0008-048-DUP	P42; Sample Dup	308	49.4	439	4140	23.3
	Average	**306 (4)**	**48.7**	**436**	**4130**	**23.5**
	%RPD Sample/Sample Dup	**1.3**	**2.9**	**1.4**	**0.48**	**1.3**
ISO16-0008-049	P56; Sample	60.0	10.2	7.21	42.7	9.63
ISO16-0008-049-DUP	P56; Sample Dup	60.8	10.3	7.42	42.4	10.1
	Average	**60.4 (4)**	**10.3**	**7.32**	**42.6**	**9.87 (2)**
	%RPD Sample/Sample Dup	**1.3**	**0.98**	**2.9**	**0.71**	**4.8**

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 ± 21%, PFBS ± 12%, PFHS ± 13%, PFOS ± 21%, and PFOSA ± 36%.
(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 11%, PFHS ± 13%, and PFOSA ± 12%.
(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
(4) The data uncertainty for samples within the WWTP, Southern Site Boundary, and Building 16 locations has been expanded to ± 33% for PFOA based on field matrix spike recovery. See section 4 for details.
(5) The data uncertainty has been expanded based on field matrix spike or laboratory control spike recovery. See section 4 for details.
(6) The field matrix spike sample for location P305 did not meet method acceptance criteria and ***the report result for PFOA should be considered qualitative*** with an expanded analytical uncertainty of ± 52%. See section 4 for details.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M Environmental 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 between July 20, 2016 and July 29, 2016 in Nalgene^{TM} (high-density polyethylene) bottles prepared at the 3M Environmental Laboratory. However, nine locations were not included with the initial shipment received on August 12, 2016. The nine locations were recollected on September 19, 2016 and returned to the laboratory on September 23, 2016. 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.

2.3 Sample Preparation

All samples were prepared for PFOA, PFBS, PFHS, and PFOS using a direct injection by methanol dilution method. Samples that required a 1:2 dilution were prepared by diluting 5 mL of a well-mixed sample with 5 mL of methanol. 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 9.9 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 D14, D2, 3M vijver, Blokkersdijkvijver standard, L21, L22, L31, P114bis, P116, and the Trip Blank and Trip Blank FMS Low (set 1) were reanalyzed for PFOA and PFHS 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). Samples that required a 1:5 dilution were prepared by removing a 0.16 mL aliquot of the well mixed sample and diluting it with 0.64 mL of methanol. Samples that required a 1:10 dilution were prepared by removing a 0.08 mL aliquot of the well mixed sample and diluting it with 0.72 mL of methanol. 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. The calibration standards and method blanks were also 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).

2.4 Analysis

All samples and quality control samples were analyzed for five target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). The rinseate blank was not fortified with SRSs prior to sample collection; however, 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-12-010.1. 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:

9/1/16 (ETS Rita) External Standard Calibration Analysis:

- BD24-3, BD24-4, D09, D10, D18, D5, ND7, P118B, P119A, P115, PP02, PP04, PP05, PP08, and PP09 (PFOA, PFBS, PFHS, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- D11, D16, P118A, and P321 (PFOA, PFHS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- D14, D2, 3M vijver, Blokkersdijkvijver standard, L21, L22, L31, P114bis, and P116 (PFOS, [$^{13}C_4$]-PFOS)
- D17 and PP01 (PFHS, PFOS, [$^{13}C_4$]-PFOS)

9/6/16 (ETS Kirk) External Standard Calibration Analysis:

- PP10, 12, 13, 5, Bemalingsstation, Collector pit, P27, P304, P42, P56, P118C, P119C, P262, P340, P341, P343, P371, P374, P379, P380, P381, P382, P372, PA109A, PA111A, PA112, and Trip Blank FMS High (PFOA, PFBS, PFHS, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- K3 (PFOA, PFBS, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- P21B and P305 (PFOA, PFBS, PFHS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS).
- L19 (PFOA, PFHS, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- P263 and P378 (PFOA, PFHS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- L19 (PFOA, PFHS, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- P263 and P378 (PFOA, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- B3-bis (PFOA, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- Trip Blank and Trip Blank FMS Low (PFBS, PFOS, [$^{13}C_4$]-PFOS)

9/14/16 (ETS Kirk) Internal Standard Calibration Analysis:

- D14, D2, 3M vijver, Blokkersdijkvijver standard, L21, L22, L31, P114bis, P116, Trip Blank, and Trip Blank FMS Low (PFOA, PFHS, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)

10/3/16 (ETS Kirk) External Standard Calibration Analysis:

- P119B, L4, WWTP Effluent, PP06, PP07, Collector pit, M4, P264, P265B, Trip Blank 2; FMS Low, and Trip Blank 2; FMS High (PFOA, PFBS, PFHS, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- D11, D16, P118A, P321, P21B, and P263 (PFOS, [$^{13}C_4$]-PFOS)
- D17 and PP01 (PFOA, [$^{13}C_4$]-PFOA)
- B3-bis (PFHS, [$^{13}C_4$]-PFOS)
- Trip Blank 2; Sample (PFBS, [$^{13}C_4$]-PFOS)

10/5/16 (ETS Kirk) External Standard Calibration Analysis:

- P121 and Trip Blank 2; Sample (PFOA, PFHS, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- K3 (PFHS, [$^{13}C_4$]-PFOS)
- P305 (PFOS, [$^{13}C_4$]-PFOS)

9/12/16 (ETS Rita) Internal Standard Calibration Analysis for PFOSA for locations: BD24-3, BD24-4, D10, D14, D18, D2, D5, P119A, P119B, 3M vijver, Blokkersdijkvijver standard, L21, L22, L31, and P114bis.

9/15/16 (ETS Rita) Internal Standard Calibration Analysis for PFOSA for locations: 12, 13, K3, P21B, P304, P56, P381, B3-bis, P378, PA109A, and Trip Blank FMS Low.

9/19/16 (ETS Rita) External Standard Calibration Analysis for PFOSA for locations: D09, D11, D16, D17, P118A, P118B, P321, PP01, PP02, PP04, PP05, PP08, PP09, PP10, P305, P42, P118C, P119C, P262, P263, P340, P341, P343, P371, P374, P380, P382, P372, PA111A, PA112, and Trip Blank FMS High.

10/7/16 (ETS Rita) Internal Standard Calibration Analysis for PFOSA for locations: P121, L4, P116, 5, Bemalingsstation, Trip Blank 2; FMS Low, and Trip Blank 2; FMS High.

10/10/16 (ETS Rita) External Standard Calibration Analysis for PFOSA for locations: ND7, PP06, PP07, Collector pit, P27, M4, P264, P265B, and P379.

10/11/16 (ETS Rita) External Standard Calibration Analysis for PFOSA for location L19.

10/12/16 (ETS Kirk) Internal Standard Calibration Analysis for PFOSA for locations: P115, WWTP Effluent, Trip Blank, and Trip Blank 2.

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	9/6/16, 9/14/16, 10/3/16, 10/5/16, 10/12/16	9/1/16, 9/12/16, 9/15/16, 9/19/16, 10/7/16, 10/10/16, 10/11/16
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 μL	1, 2 or 5 μL
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 6500+
Ion Source	Turbo Spray	Turbo Spray
Polarity	Negative	Negative
Software	Analyst 1.6.2	Analyst 1.6.3

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis				
Step Number	**Total Time (min)**	**Flow Rate (μL/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	NA	NA
	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

The individual transitions were summed to produce a “total ion chromatogram” (TIC), which was used for quantitation.

3 Data Analysis

3.1 Calibration

9/1/16, 9/6/16, 10/3/16, 10/5/16 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 9/1/16, 9/6/16, and 10/3/16. On 10/5/16, calibration standards ranging from 0.02 ng/mL to 150 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.

9/14/16 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 ten calibration standards ranging from 0.0125 ng/mL to 10 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 standard of PFOA used to prepare the calibration standards consisted of both linear and branched isomers.

9/12/16, 9/15/16, 10/7/16, and 10/12/16 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 on 9/12/16, 9/15/16, and 10/7/16. Standards ranged from 0.025 ng/mL to 5 ng/mL (nominal) on 10/12/16. Prior to analysis, the calibration standards were diluted 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.

9/19/16, 10/10/16, and 10/11/16 Analysis of PFOSA (External Standard Calibration): Samples were analyzed for PFOSA against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 10 mL of laboratory reagent water. Calibration standards ranging from 5 ng/mL to 200 ng/mL (nominal) were analyzed on 9/19/16. Standards ranged from 0.025 ng/mL to 200 ng/mL (nominal) on 10/10/16 and 1 ng/mL to 150 ng/mL (nominal) on 10/11/16. Prior to analysis, the calibration standards were diluted 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 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±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) 9/1/16 Analysis**	**LOQ, ng/mL (1) 9/6/16 Analysis**	**LOQ, ng/mL (1) 9/12/16 Analysis**	**LOQ, ng/mL (2) 9/14/16 Analysis**	**LOQ, ng/mL (1) 9/15/16 Analysis**	**LOQ, ng/mL (1) 9/19/16 Analysis**
PFOA	0.240	0.0192	NA	0.0240	NA	NA
PFBS	0.0200	0.0200	NA	NA	NA	NA
PFHS	0.0200	0.0200	NA	0.0250	NA	NA
PFOS	0.0464	0.0185	NA	NA	NA	NA
PFOSA	NA	NA	0.100	NA	0.250	5.00
Analyte	**LOQ, ng/mL (1) 10/3/16 Analysis**	**LOQ, ng/mL (1) 10/5/16 Analysis**	**LOQ, ng/mL (1) 10/7/16 Analysis**	**LOQ, ng/ mL (1) 10/10/16 Analysis**	**LOQ, ng/ mL (3) 10/11/16 Analysis**	**LOQ, ng/mL (1) 10/12/16 Analysis**
PFOA	0.0192	0.0479	NA	NA	NA	NA
PFBS	0.0200	NA	NA	NA	NA	NA
PFHS	0.0200	0.0500	NA	NA	NA	NA
PFOS	0.0185	0.0464	NA	NA	NA	NA
PFOSA	NA	NA	0.0994	0.0994	5.00	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) A dilution factor of 5 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/solvent 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 [$^{13}C_4$]-PFOA and [$^{13}C_4$]-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:

- 9/1/16: Low-level LCSs were spiked below the resulting lower limit of quantitation (LLOQ) for PFOA (120 ng/mL). High-level LCSs were spiked above the resulting upper limit of quantitation (ULOQ) for PFHS (25,100 ng/mL) and PFOS (23,200 ng/mL).
- 9/6/16: High-level LCSs were spiked above the resulting ULOQ for PFBS and PFHS (25,100 ng/mL).
- 9/15/16: Low-level LCSs were spiked below the resulting LLOQ for PFOSA (0.25 ng/mL).
- 10/3/16: High-level LCSs had an average recovery of 121% for PFOS. High-level LCSs were spiked above the resulting ULOQ for PFHS (50,000 ng/mL).
- 10/5/16: Low-level LCSs had an average recovery of 121% for PFOA.
- 10/10/16: Low-level LCSs had an average recovery of 122% for PFOSA.
- 10/11/16: High-level LCSs had an average recovery of 140% for PFOSA.

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.

$$\boxed{\text{LCS Percent Recovery} = \frac{\text{Calculated Concentration}}{\text{Spike Concentration}} * 100\%}$$

$$\boxed{\text{LCS\% RSD} = \frac{\text{standard deviation LCS replicates}}{\text{average LCS recovery}} * 100\%}$$

Table 7. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 9/1/16	**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-160901-1	100	BLOQ	NA	100	114	114
LCS-160901-2	100	BLOQ	NA	100	111	111
LCS-160901-3	100	BLOQ	NA	100	109	109
Average ± %RSD	**NA (1)**			**111% ± 2.3%**		
LCS-160901-4	5000	6120	122	5000	5330	107
LCS-160901-5	5000	5920	118	5000	5230	105
LCS-160901-6	5000	5770	115	5000	5240	105
Average ± %RSD	**118% ± 3.0%**			**106% ± 1.1%**		
LCS-160901-7	35000	34700	99.2	35000	36000	103
LCS-160901-8	35000	34700	99.1	35000	36300	104
LCS-160901-9	35000	34400	98.4	35000	35100	100
Average ± %RSD	**98.9% ± 0.44%**			**102% ± 2.0%**		

ETS-8-044.3 External Calibration Analyzed 9/1/16	**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-160901-1	100	106	106	100	111	111
LCS-160901-2	100	110	110	100	106	106
LCS-160901-3	100	105	105	100	103	103
Average ± %RSD	**107% ± 2.5%**			**107% ± 3.8%**		
LCS-160901-4	5000	5270	105	5000	5700	114
LCS-160901-5	5000	5290	106	5000	5680	114
LCS-160901-6	5000	5220	104	5000	5700	114
Average ± %RSD	**105% ± 0.95%**			**114% ± 0.0%**		
LCS-160901-7	35000	>ULOQ	NA	35000	>ULOQ	NA
LCS-160901-8	35000	>ULOQ	NA	35000	>ULOQ	NA
LCS-160901-9	35000	>ULOQ	NA	35000	>ULOQ	NA
Average ± %RSD	**NA (1)**			**NA (1)**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 9/1/16	**$[^{13}C_4]$-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-160901-1	99.8	103	103	95.6	103	108
LCS-160901-2	99.8	114	114	95.6	102	106
LCS-160901-3	99.8	109	109	95.6	107	112
Average ± %RSD	**109% ± 5.1%**			**109% ± 2.8%**		
LCS-160901-4	998	1130	113	956	1070	112
LCS-160901-5	998	1110	111	956	1080	113
LCS-160901-6	998	1070	107	956	1080	113
Average ± %RSD	**110% ± 2.8%**			**113% ± 0.51%**		

ETS-8-044.3 External Calibration Analyzed 9/6/16	**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-160906-1	100	111	111	100	102	102
LCS-160906-2	100	107	107	100	99.6	99.6
LCS-160906-3	100	105	105	100	97.8	97.8
Average ± %RSD	**108% ± 2.8%**			**99.8% ± 2.1%**		
LCS-160906-4	5000	5470	109	5000	5130	103
LCS-160906-5	5000	5310	106	5000	4930	98.5
LCS-160906-6	5000	5230	105	5000	4930	98.7
Average ± %RSD	**107% ± 2.0%**			**100% ± 2.5%**		
LCS-160906-7	35000	30200	86.3	35000	>ULOQ	NA
LCS-160906-8	35000	30300	86.7	35000	>ULOQ	NA
LCS-160906-9	35000	29900	85.6	35000	>ULOQ	NA
Average ± %RSD	**86.2% ± 0.62%**			**NA (1)**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 9/6/16	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-160906-1	100	103	103	100	102	102
LCS-160906-2	100	98.4	98.4	100	103	103
LCS-160906-3	100	98.5	98.5	100	99.1	99.1
Average ± %RSD	**100% ± 2.6%**			**101% ± 2.0%**		
LCS-160906-4	5000	5340	107	5000	5670	113
LCS-160906-5	5000	5110	102	5000	5350	107
LCS-160906-6	5000	5020	100	5000	5270	105
Average ± %RSD	**103% ± 3.5%**			**108% ± 3.8%**		
LCS-160906-7	35000	>ULOQ	NA	35000	41700	119
LCS-160906-8	35000	>ULOQ	NA	35000	43100	123
LCS-160906-9	35000	>ULOQ	NA	35000	41300	118
Average ± %RSD	**NA (1)**			**120% ± 2.2%**		

ETS-8-044.3 External Calibration Analyzed 9/6/16	[$^{13}C_4$]-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-160906-1	99.8	105	105	95.6	99.8	104
LCS-160906-2	99.8	99.6	99.8	95.6	96.7	101
LCS-160906-3	99.8	99.5	99.7	95.6	95.8	100
Average ± %RSD	**102% ± 3.0%**			**102% ± 2.0%**		
LCS-160906-4	998	1140	114	956	1130	119
LCS-160906-5	998	1100	111	956	1110	116
LCS-160906-6	998	1120	112	956	1100	115
Average ± %RSD	**112% ± 1.4%**			**117% ± 1.8%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 9/8/16	**PFOSA**		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160908-1	0.199	0.212	107
LCS-160908-2	0.199	0.211	106
LCS-160908-3	0.199	0.202	102
Average ± %RSD	**105% ± 2.5%**		
LCS-160908-4	19.9	20.1	101
LCS-160908-5	19.9	19.2	96.5
LCS-160908-6	19.9	19.9	100
Average ± %RSD	**99.1% ± 2.4%**		
LCS-160908-7	139	128	92.0
LCS-160908-8	139	128	91.9
LCS-160908-9	139	127	91.7
Average ± %RSD	**91.9% ± 0.17%**		

ETS-8-044.3 Internal Calibration Analyzed 9/14/16	**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-160914-1	0.190	0.179	94.0	0.198	0.190	95.7
LCS-160914-2	0.190	0.185	97.5	0.198	0.191	96.5
LCS-160914-3	0.190	0.180	94.9	0.198	0.192	96.8
Average ± %RSD	**95.5% ± 1.9%**			**96.3% ± 0.59%**		
LCS-160914-4	1.90	1.92	101	1.98	2.05	103
LCS-160914-5	1.90	1.91	101	1.98	2.05	103
LCS-160914-6	1.90	1.95	103	1.98	2.04	103
Average ± %RSD	**102% ± 1.1%**			**103% ± 0.0%**		
LCS-160914-7	13.3	13.2	99.0	13.9	13.6	97.6
LCS-160914-8	13.3	12.8	95.9	13.9	13.2	95.3
LCS-160914-9	13.3	13.1	98.7	13.9	13.5	97.4
Average ± %RSD	**97.9% ± 1.7%**			**96.8% ± 1.3%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 9/14/16	$[^{13}C_4]$-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-160914-1	0.198	0.181	91.4	0.189	0.174	92.0
LCS-160914-2	0.198	0.189	95.5	0.189	0.176	93.0
LCS-160914-3	0.198	0.187	94.4	0.189	0.176	93.3
Average ± %RSD	**93.8% ± 2.3%**			**92.8% ± 0.73%**		
LCS-160914-4	1.98	1.96	98.9	1.89	1.86	98.3
LCS-160914-5	1.98	1.86	94.2	1.89	1.84	97.2
LCS-160914-6	1.98	1.95	98.7	1.89	1.82	96.1
Average ± %RSD	**97.3% ± 2.7%**			**97.2% ± 1.1%**		

ETS-8-044.3 Internal Calibration Analyzed 9/15/16	**PFOSA**		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160913-1	0.199	BLOQ	NA
LCS-160913-2	0.199	BLOQ	NA
LCS-160913-3	0.199	BLOQ	NA
Average ± %RSD	**NA (1)**		
LCS-160913-4	19.9	19.5	97.8
LCS-160913-5	19.9	20.0	100
LCS-160913-6	19.9	19.7	99.1
Average ± %RSD	**99.0% ± 1.1%**		
LCS-160913-7	139	127	91.2
LCS-160913-8	139	127	91.1
LCS-160913-9	139	126	90.3
Average ± %RSD	**90.9% ± 0.54%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 9/19/16	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-160919-1	19.9	20.4	102
LCS-160919-2	19.9	17.6	88.3
LCS-160919-3	19.9	18.9	94.8
Average ± %RPD	**95.0% ± 7.2%**		
LCS-160919-4	69.9	70.7	101
LCS-160919-5	69.9	69.0	98.7
LCS-160919-6	69.9	72.7	104
Average ± %RSD	**101% ± 2.6%**		
LCS-160919-7	139	163	118
LCS-160919-8	139	131	94.6
LCS-160919-9	139	132	94.9
Average ± %RSD	**103% ± 13%**		

ETS-8-044.3 External Calibration Analyzed 10/3/16	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-161003-1	200	233	116	200	223	112
LCS-161003-2	200	219	110	200	212	106
LCS-161003-3	200	206	103	200	205	102
Average ± %RSD	**110% ± 5.9%**			**107% ± 4.7%**		
LCS-161003-4	25000	26300	105	25000	26000	104
LCS-161003-5	25000	26300	105	25000	26300	105
LCS-161003-6	25000	26300	105	25000	26100	105
Average ± %RSD	**105% ± 0.0%**			**105% ± 0.55%**		
LCS-161003-7	70000	64800	92.6	70000	74100	106
LCS-161003-8	70000	63100	90.2	70000	72400	103
LCS-161003-9	70000	64700	92.4	70000	73700	105
Average ± %RSD	**91.7% ± 1.5%**			**105% ± 1.5%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 10/3/16	**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-161003-1	200	221	111	200	222	111
LCS-161003-2	200	206	103	200	201	100
LCS-161003-3	200	201	101	200	193	96.7
Average ± %RSD	**105% ± 5.0%**			**103% ± 7.3%**		
LCS-161003-4	25000	26200	105	25000	27800	111
LCS-161003-5	25000	26500	106	25000	28000	112
LCS-161003-6	25000	26600	106	25000	28300	113
Average ± %RSD	**106% ± 0.55%**			**112% ± 0.89%**		
LCS-161003-7	70000	>ULOQ	NA	70000	84900	121
LCS-161003-8	70000	>ULOQ	NA	70000	83200	119
LCS-161003-9	70000	>ULOQ	NA	70000	86700	124
Average ± %RSD	**NA (1)**			**121% ± 2.1% (2)**		

ETS-8-044.3 External Calibration Analyzed 10/3/16	**$[^{13}C_4]$-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-161003-1	199	210	105	191	202	106
LCS-161003-2	199	211	106	191	209	110
LCS-161003-3	199	213	107	191	208	109
Average ± %RSD	**106% ± 0.94%**			**108% ± 1.9%**		
LCS-161003-4	1990	2080	104	1910	2080	109
LCS-161003-5	1990	2110	106	1910	2140	112
LCS-161003-6	1990	2120	106	1910	2180	114
Average ± %RSD	**105% ± 1.1%**			**112% ± 2.3%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 10/5/16	**PFOA (Linear + Branched)**		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-161005-1	200	279	140
LCS-161005-2	200	223	111
LCS-161005-3	200	225	113
Average ± %RSD	**121% ± 13% (2)**		
LCS-161005-4	25000	26800	107
LCS-161005-5	25000	26500	106
LCS-161005-6	25000	25800	103
Average ± %RSD	**105% ± 2.0%**		
LCS-161005-7	100000	102000	102
LCS-161005-8	100000	100000	100
LCS-161005-9	100000	103000	103
Average ± %RSD	**102% ± 1.5%**		

ETS-8-044.3 External Calibration Analyzed 10/5/16	**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-161005-1	200	275	137	200	235	117
LCS-161005-2	200	220	110	200	197	98.5
LCS-161005-3	200	213	106	200	201	100
Average ± %RSD	**118% ± 14%**			**105% ± 9.8%**		
LCS-161005-4	25000	26700	107	25000	27800	111
LCS-161005-5	25000	25900	104	25000	26500	106
LCS-161005-6	25000	25300	101	25000	25900	104
Average ± %RSD	**104% ± 2.9%**			**107% ± 3.4%**		
LCS-161005-7	100000	106000	106	100000	108000	108
LCS-161005-8	100000	104000	104	100000	106000	106
LCS-161005-9	100000	104000	104	100000	109000	109
Average ± %RSD	**105% ± 1.1%**			**108% ± 1.4%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 10/5/16	**$[^{13}C_4]$-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-161005-1	199	229	115	191	226	119
LCS-161005-2	199	214	107	191	207	108
LCS-161005-3	199	222	112	191	214	112
Average ± %RSD	**111% ± 3.6%**			**113% ± 4.9%**		
LCS-161005-4	1990	2310	116	1910	2230	117
LCS-161005-5	1990	2250	113	1910	2230	117
LCS-161005-6	1990	2200	110	1910	2170	114
Average ± %RSD	**113% ± 2.7%**			**116% ± 1.5%**		

ETS-8-044.3 Internal Calibration Analyzed 10/7/16	**PFOSA**		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-161007-1	0.496	0.479	96.5
LCS-161007-2	0.496	0.543	109
LCS-161007-3	0.496	0.578	117
Average ± %RPD	**108% ± 9.6%**		
LCS-161007-4	49.6	48.5	97.7
LCS-161007-5	49.6	48.1	97.0
LCS-161007-6	49.6	48.2	97.3
Average ± %RSD	**97.3% ± 0.36%**		
LCS-161007-7	347	331	95.4
LCS-161007-8	347	334	96.2
LCS-161007-9	347	333	96.0
Average ± %RSD	**95.9% ± 0.43%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 10/10/16	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-161007-1	0.496	0.586	118
LCS-161007-2	0.496	0.623	126
LCS-161007-3	0.496	0.599	121
Average ± %RPD	**122% ± 3.3%** (2)		
LCS-161007-4	49.6	51.4	104
LCS-161007-5	49.6	50.5	102
LCS-161007-6	49.6	49.7	100
Average ± %RSD	**102% ± 2.0%**		
LCS-161007-7	347	373	108
LCS-161007-8	347	449	129
LCS-161007-9	347	400	115
Average ± %RSD	**117% ± 9.1%**		

ETS-8-044.3 External Calibration Analyzed 10/11/16	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-161011-1	20.0	18.3	91.3
LCS-161011-2	20.0	18.5	92.5
LCS-161011-3	20.0	17.2	86.1
Average ± %RPD	**90.0% ± 3.8%**		
LCS-161011-4	100	95.2	95.2
LCS-161011-5	100	85.0	85.0
LCS-161011-6	100	94.3	94.3
Average ± %RSD	**91.5% ± 6.2%**		
LCS-161011-7	500	557	111
LCS-161011-8	500	743	149
LCS-161011-9	500	806	161
Average ± %RSD	**140% ± 19%** (2)		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 10/12/16	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-161012-1	0.0994	0.0986	99.2
LCS-161012-2	0.0994	0.0995	100
LCS-161012-3	0.0994	0.0993	99.9
Average ± %RPD	**99.7% ± 0.44%**		
LCS-161012-4	1.99	1.98	99.6
LCS-161012-5	1.99	1.97	99.0
LCS-161012-6	1.99	1.94	97.3
Average ± %RSD	**98.6% ± 1.2%**		
LCS-161012-7	3.47	3.70	107
LCS-161012-8	3.47	3.64	105
LCS-161012-9	3.47	3.66	106
Average ± %RSD	**106% ± 0.94%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.
(2) LCSs do 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-12-012.3. 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 PFOA using external calibration was calculated at ±19% following ETS-12-012.3; however, the method uncertainty was expanded to ±21% based on the recovery of the low-level LCSs analyzed on 10/5/16.

- The data uncertainty for PFOS using external calibration was calculated at ±18% following ETS-12-012.3; however, the method uncertainty was expanded to ±21% based on the recovery of the high-level LCSs analyzed on 10/3/16.

- The low-level LCSs analyzed on 10/10/16 had an average recovery of 122% for PFOSA. However, the data uncertainty for PFOSA using external calibration was not expanded further since the %bias was below the calculated data uncertainty following ETS-12-012.3 (±36%). The high-level LCSs analyzed on 10/11/16 had an average recovery of 140% for PFOSA. Since only one sample location (L19) was analyzed with the LCSs and no other samples were prepared using a 10x dilution, the overall data uncertainty was not expanded. However, the data uncertainty was expanded for sample location L19.

Table 8. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	NA	±21%
PFBS	External	5.84	±12%
PFHS	External	6.39	±13%
PFOS	External	NA	±21%
PFOSA	External	17.8	±36%
PFOA	Internal	5.47	±11%
PFHS	Internal	6.32	±13%
PFOSA	Internal	5.87	±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 contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. The stock solutions of PFOS and PFOA used to prepare the field matrix spikes were prepared from a different reference source than the one used to prepare the calibration standards. 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 [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles prior to sample collection. The [$^{13}C_4$]-PFOA was selected to represent the C8 perfluorocarboxylic acid. The [$^{13}C_4$]-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids and PFOSA. 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.

$$\boxed{\text{FMS Recovery} = \frac{(\text{Sample Concentration of FMS} - \text{Average Concentration : Field Sample \& Field Sample Dup.})}{\text{Spike Concentration}} * 100\%}$$

Table 9. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
D14, 3M vijver, L22, P116, B7	FMS	4.79	5.00	5.00	4.64	5.00
D16, D17, D18, D5, ND7, P119A, P119B	FMS	100	100	100	100	10.0
D2 (1)	FMS	89.7	89.7	89.7	89.7	8.97
BD24-4 (1)	FMS	89.7	89.7	89.7	89.7	89.7
12, Collector pit	FMS	200	10.0	200	200	10.0
PA109A (1)	FMS	457	8.97	457	457	8.97
PP02, PP06, PP10, K3, P305, P118C, P262, P340, P379	FMS	500	50.0	500	500	50.0
P118B	FMS	2010	10.0	2010	2010	10.0
Trip Blank	Low	4.79	5.00	5.00	4.64	5.00
	High	500	50.0	500	500	50.0
Trip Blank 2	Low	4.79	5.00	5.00	4.64	5.00
	High	200	10.0	200	200	10.0

(1) FMS concentration was adjusted due to sample bottle being overfilled by greater than 10%.

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.

The field matrix spike and surrogate recovery standard recoveries met acceptance criteria except where noted below.

Sampling locations within zones Building 16, WWTP, and Southern site boundary: Three locations had noncompliant field matrix spike recoveries for PFOA. Sample location P305 (Source area – Building 16) had a recovery of 48.3%, location P340 (Source area – WWTP) had a recovery of 52.2%, and location PA109A (Southern site boundary) had a recovery of 56.6%. For this sampling round, field matrix spikes were only prepared at select sampling locations. For zones Building 16, WWTP, and Southern site boundary, the field matrix spike was appropriate for PFOA for locations K3, P305, P118C, P262, P340, P379, and PA109A. The analytical uncertainty was expanded for all samples within these three sampling zones, using the average recovery for all appropriate field matrix spike samples, which was calculated at 67.3%. Therefore, the analytical uncertainty was expanded to ±33% for PFOA for all sampling locations within these zones. Since location P305 had a field matrix spike recovery outside the acceptance criteria of 100 ± 50%, the results are flagged as qualitative with an expanded uncertainty of ±52% for PFOA.

BD24-3: Surrogate recovery standard [$^{13}C_4$]-PFOA had a recovery of 133% for BD24-3; Sample Dup. The average surrogate recovery for the BD24-3 sample set was within the acceptance criteria of 100 ± 30%. Therefore, the results are reported and the analytical uncertainty was not expanded.

D14: Surrogate recovery standard [$^{13}C_4$]-PFOS had a recovery of 132% for D14; Sample Dup. The average surrogate recovery for the D14 sample set was within the acceptance criteria of 100 ± 30%. Therefore, the results are reported and the analytical uncertainty was not expanded.

D18: Surrogate recovery standard [$^{13}C_4$]-PFOS had a recovery of 133% for D18; Sample Dup. The average surrogate recovery for the D18 sample set was within the acceptance criteria of 100 ± 30%. Therefore, the results are reported and the analytical uncertainty was not expanded.

3M vijver: The field matrix spike recovery was 139% for PFHS. Therefore, the analytical uncertainty was expanded to ±39% for PFHS.

P115: The surrogate recovery was 131% for [$^{13}C_4$]-PFOA and 134% for [$^{13}C_4$]-PFOS for sample P115; Sample Dup. The average surrogate recovery for the P115 sample set was within the acceptance criteria of 100 ± 30%. Therefore, the results are reported and the analytical uncertainty was not expanded.

P379: The field matrix spike recovery was 135% for PFOSA. Since the %bias was less than the overall data uncertainty (±36%), the data uncertainty was not expanded further.

L19: The analytical uncertainty for location L19 was expanded to ±40% due to the high-level LCSs having an average recovery of 140%. Location L19 was the only sample set analyzed on 10/11/16 using a dilution factor of 10; therefore, the analytical uncertainty was only expanded for this location.

Table 10. Location ID: BD24-4

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-002	BD24-4; Sample	10.5	NA	4.65	NA
ISO16-0008-002-DUP	BD24-4; Sample Dup	10.2	NA	4.54	NA
ISO16-0008-002-FMS	BD24-4; FMS	122	124	97.6	104 (1)
Average Concentration (ng/mL) ± %RPD		**10.4 ng/mL ± 2.9%**		**4.60 ng/mL ± 2.4%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-002	BD24-4; Sample	8.82	NA	0.936	NA
ISO16-0008-002-DUP	BD24-4; Sample Dup	9.03	NA	0.736	NA
ISO16-0008-002-FMS	BD24-4; FMS	108	110 (1)	85.4	94.3 (1)
Average Concentration (ng/mL) ± %RPD		**8.93 ng/mL ± 2.4%**		**0.836 ng/mL ± 24% (2)**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.
(2) Sample/sample duplicate RPD does not meet acceptance criteria of ≤20%.

Table 11. Location ID: D14

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-006	D14; Sample	2.44	NA	1.38	NA
ISO16-0008-006-DUP	D14; Sample Dup	2.60	NA	1.48	NA
ISO16-0008-006-FMS	D14; FMS	7.23	98.3	6.40	99.4
Average Concentration (ng/mL) ± %RPD		**2.52 ng/mL ± 6.3%**		**1.43 ng/mL ± 7.0%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-006	D14; Sample	7.18	NA	0.647	NA
ISO16-0008-006-DUP	D14; Sample Dup	7.26	NA	0.753	NA
ISO16-0008-006-FMS	D14; FMS	10.5	70.8	5.53	96.6
Average Concentration (ng/mL) ± %RPD		**7.22 ng/mL ± 1.1%**		**0.700 ng/mL ± 15%**	

NA = Not Applicable

Table 12. Location ID: D16

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-007	D16; Sample	262	NA	59.0	NA
ISO16-0008-007-DUP	D16; Sample Dup	263	NA	58.1	NA
ISO16-0008-007-FMS	D16; FMS	346	NC	160	101
Average Concentration (ng/mL) ± %RPD		**263 ng/mL ± 0.38%**		**58.6 ng/mL ± 1.5%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-007	D16; Sample	1650	NA	75.7	NA
ISO16-0008-007-DUP	D16; Sample Dup	1630	NA	79.0	NA
ISO16-0008-007-FMS	D16; FMS	1750	NC	90.3	NC
Average Concentration (ng/mL) ± %RPD		**1640 ng/mL ± 3.3%**		**77.4 ng/mL ± 4.3%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 13. Location ID: D17

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-008	D17; Sample	1910	NA	121	NA
ISO16-0008-008-DUP	D17; Sample Dup	1780	NA	114	NA
ISO16-0008-008-FMS	D17; FMS	1680	NC	205	87.5
Average Concentration (ng/mL) ± %RPD		**1850 ng/mL ± 7.0%**		**118 ng/mL ± 6.0%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-008	D17; Sample	437	NA	26.6	NA
ISO16-0008-008-DUP	D17; Sample Dup	444	NA	25.6	NA
ISO16-0008-008-FMS	D17; FMS	NA (1)	NA (1)	38.4	NC
Average Concentration (ng/mL) ± %RPD		**441 ng/mL ± 1.6%**		**26.1 ng/mL ± 3.8%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) The field matrix spike sample was not re-analyzed since it was not appropriate based on the endogenous sample concentration.

Table 14. Location ID: D18

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-009	D18; Sample	521	NA	288	NA
ISO16-0008-009-DUP	D18; Sample Dup	521	NA	289	NA
ISO16-0008-009-FMS	D18; FMS	589	NC	389	NC
Average Concentration (ng/mL) ± %RPD		**521 ng/mL ± 0.0%**		**289 ng/mL ± 0.35%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-009	D18; Sample	17.6	NA	2.62	NA
ISO16-0008-009-DUP	D18; Sample Dup	19.2	NA	2.41	NA
ISO16-0008-009-FMS	D18; FMS	117	98.6	11.8	92.9
Average Concentration (ng/mL) ± %RPD		**18.4 ng/mL ± 8.7%**		**2.52 ng/mL ± 8.3%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 15. Location ID: D2

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-010	D2; Sample	0.347	NA	0.166	NA
ISO16-0008-010-DUP	D2; Sample Dup	0.380	NA	0.165	NA
ISO16-0008-010-FMS	D2; FMS	NA (1)	NA (1)	NA (1)	NA (1)
Average Concentration (ng/mL) ± %RPD		**0.364 ng/mL ± 9.1%**		**0.166 ng/mL ± 0.60%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-010	D2; Sample	9.83	NA	0.519	NA
ISO16-0008-010-DUP	D2; Sample Dup	9.64	NA	0.471	NA
ISO16-0008-010-FMS	D2; FMS	112	114	9.35	98.7 (2)
Average Concentration (ng/mL) ± %RPD		**9.74 ng/mL ± 2.0%**		**0.495 ng/mL ± 9.7%**	

NA = Not Applicable
(1) The field matrix spike sample was not re-analyzed since it was not appropriate based on the endogenous sample concentration (spike level greater than 100x the endogenous concentration).
(2) FMS concentration greater than 10 times the sample concentration.

Table 16. Location ID: D5

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-011	D5; Sample	504	NA	436	NA
ISO16-0008-011-DUP	D5; Sample Dup	459	NA	429	NA
ISO16-0008-011-FMS	D5; FMS	565	NC	NA (1)	NA (1)
Average Concentration (ng/mL) ± %RPD		**482 ng/mL ± 9.3%**		**433 ng/mL ± 1.6%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-011	D5; Sample	388	NA	15.0	NA
ISO16-0008-011-DUP	D5; Sample Dup	439	NA	12.7	NA
ISO16-0008-011-FMS	D5; FMS	NA (1)	NA (1)	21.1	72.5
Average Concentration (ng/mL) ± %RPD		**414 ng/mL ± 12%**		**13.9 ng/mL ± 17%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) The field matrix spike sample was not re-analyzed since it was not appropriate based on the endogenous sample concentration.

Table 17. Location ID: ND7

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-012	ND7; Sample	169	NA	6.84	NA
ISO16-0008-012-DUP	ND7; Sample Dup	172	NA	6.37	NA
ISO16-0008-012-FMS	ND7; FMS	270	99.5	99.9	93.3 (1)
Average Concentration (ng/mL) ± %RPD		**171 ng/mL ± 1.8%**		**6.61 ng/mL ± 7.1%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-012	ND7; Sample	282	NA	384	NA
ISO16-0008-012-DUP	ND7; Sample Dup	272	NA	344	NA
ISO16-0008-012-FMS	ND7; FMS	376	NC	NA (2)	NA (2)
Average Concentration (ng/mL) ± %RPD		**277 ng/mL ± 3.6%**		**364 ng/mL ± 11%**	

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) The field matrix spike sample was not re-analyzed since it was not appropriate based on the endogenous sample concentration.

Table 18. Location ID: P118B

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-014	P118B; Sample	2410	NA	2280	NA
ISO16-0008-014-DUP	P118B; Sample Dup	2430	NA	2270	NA
ISO16-0008-014-FMS	P118B; FMS	4560	106	4290	100
Average Concentration (ng/mL) ± %RPD		**2420 ng/mL ± 0.83%**		**2280 ng/mL ± 0.44%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-014	P118B; Sample	10800	NA	47.6	NA
ISO16-0008-014-DUP	P118B; Sample Dup	10400	NA	48.2	NA
ISO16-0008-014-FMS	P118B; FMS	12500	NC	58.6	NC
Average Concentration (ng/mL) ± %RPD		**10600 ng/mL ± 3.8%**		**47.9 ng/mL ± 1.3%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 19. Location ID: P119A

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-015	P119A; Sample	144	NA	37.2	NA
ISO16-0008-015-DUP	P119A; Sample Dup	151	NA	38.1	NA
ISO16-0008-015-FMS	P119A; FMS	237	89.5	130	92.4
Average Concentration (ng/mL) ± %RPD		**148 ng/mL ± 4.7%**		**37.7 ng/mL ± 2.4%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-015	P119A; Sample	71.0	NA	13.6	NA
ISO16-0008-015-DUP	P119A; Sample Dup	67.7	NA	13.4	NA
ISO16-0008-015-FMS	P119A; FMS	160	90.7	21.4	79.0
Average Concentration (ng/mL) ± %RPD		**69.4 ng/mL ± 4.8%**		**13.5 ng/mL ± 1.5%**	

NA = Not Applicable

Table 20. Location ID: P119B

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-016	P119B; Sample	2220	NA	1090	NA
ISO16-0008-016-DUP	P119B; Sample Dup	2240	NA	1070	NA
ISO16-0008-016-FMS	P119B; FMS	2310	NC	1180	NC
Average Concentration (ng/mL) ± %RPD		**2230 ng/mL ± 0.90%**		**1080 ng/mL ± 1.9%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-016	P119B; Sample	436	NA	10.6	NA
ISO16-0008-016-DUP	P119B; Sample Dup	426	NA	11.0	NA
ISO16-0008-016-FMS	P119B; FMS	519	NC	19.8	90.0
Average Concentration (ng/mL) ± %RPD		**431 ng/mL ± 2.3%**		**10.8 ng/mL ± 3.7%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 21. Location ID: 3M vijver

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-019	3M vijver; Sample	2.18	NA	2.54	NA
ISO16-0008-019-DUP	3M vijver; Sample Dup	2.17	NA	2.53	NA
ISO16-0008-019-FMS	3M vijver; FMS	7.94	120	9.46	139 (1)
Average Concentration (ng/mL) ± %RPD		**2.18 ng/mL ± 0.46%**		**2.54 ng/mL ± 0.39% (2)**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-019	3M vijver; Sample	12.8	NA	1.13	NA
ISO16-0008-019-DUP	3M vijver; Sample Dup	13.3	NA	1.22	NA
ISO16-0008-019-FMS	3M vijver; FMS	17.2	NC	5.98	96.1
Average Concentration (ng/mL) ± %RPD		**13.1 ng/mL ± 3.8%**		**1.18 ng/mL ± 7.7%**	

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 analytical uncertainty was expanded to ± 39% for PFHS.

Table 22. Location ID: L22

		PFOA		**PFHS**	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-022	L22; Sample	0.843	NA	0.170	NA
ISO16-0008-022-DUP	L22; Sample Dup	0.830	NA	0.166	NA
ISO16-0008-022-FMS	L22; FMS	5.46	96.5	5.21	101 (1)
Average Concentration (ng/mL) ± %RPD		**0.837 ng/mL ± 1.6%**		**0.168 ng/mL ± 2.4%**	

		PFOS		**PFOSA**	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-022	L22; Sample	4.68	NA	0.347	NA
ISO16-0008-022-DUP	L22; Sample Dup	5.19	NA	0.333	NA
ISO16-0008-022-FMS	L22; FMS	9.70	103	5.20	97.2 (1)
Average Concentration (ng/mL) ± %RPD		**4.94 ng/mL ± 10%**		**0.340 ng/mL ± 4.1%**	

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 23. Location ID: P116

		PFOA		**PFHS**	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-027	P116; Sample	2.92	NA	1.10	NA
ISO16-0008-027-DUP	P116; Sample Dup	2.00	NA	0.883	NA
ISO16-0008-027-FMS	P116; FMS	7.03	95.4	5.95	99.2
Average Concentration (ng/mL) ± %RPD		**2.46 ng/mL ± 37% (1)**		**0.992 ng/mL ± 22% (1)**	

		PFOS		**PFOSA**	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-027	P116; Sample	10.4	NA	0.407	NA
ISO16-0008-027-DUP	P116; Sample Dup	10.8	NA	0.299	NA
ISO16-0008-027-FMS	P116; FMS	13.8	NC	5.28	98.5 (2)
Average Concentration (ng/mL) ± %RPD		**10.6 ng/mL ± 3.8%**		**0.353 ng/mL ± 31% (1)**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Sample/sample duplicate RPD does not meet acceptance criteria of ≤20%.
(2) FMS concentration greater than 10 times the sample concentration.

Table 24. Location ID: PP02

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-030	PP02; Sample	661	NA	30.9	NA	2380	NA
ISO16-0008-030-DUP	PP02; Sample Dup	632	NA	28.6	NA	2310	NA
ISO16-0008-030-FMS	PP02; FMS	1090	88.7	79.2	98.9	2920	NC
Average Concentration (ng/mL) ± %RPD		**647 ng/mL ± 4.5%**		**29.8 ng/mL ± 7.7%**		**2350 ng/mL ± 3.0%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-030	PP02; Sample	21900	NA	56.3	NA
ISO16-0008-030-DUP	PP02; Sample Dup	20900	NA	56.7	NA
ISO16-0008-030-FMS	PP02; FMS	NA (1)	NA (1)	97.6	82.2
Average Concentration (ng/mL) ± %RPD		**21400 ng/mL ± 4.7%**		**56.5 ng/mL ± 0.71%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) The field matrix spike sample was not re-analyzed since it was not appropriate based on the endogenous sample concentration.

Table 25. Location ID: PP06

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-078	PP06; Sample	224	NA	18.7	NA	54.0	NA
ISO16-0008-078-DUP	PP06; Sample Dup	214	NA	17.8	NA	52.4	NA
ISO16-0008-078-FMS	PP06; FMS	705	97.2	62.7	NC	531	95.6
Average Concentration (ng/mL) ± %RPD		**219 ng/mL ± 4.6%**		**18.3 ng/mL ± 4.9%**		**53.2 ng/mL ± 3.0%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-078	PP06; Sample	1720	NA	78.2	NA
ISO16-0008-078-DUP	PP06; Sample Dup	1660	NA	78.3	NA
ISO16-0008-078-FMS	PP06; FMS	2230	NC	143	130
Average Concentration (ng/mL) ± %RPD		**1690 ng/mL ± 3.6%**		**78.3 ng/mL ± 0.13%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 26. Location ID: PP10

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-037	PP10; Sample	590	NA	16.4	NA	185	NA
ISO16-0008-037-DUP	PP10; Sample Dup	591	NA	18.5	NA	227	NA
ISO16-0008-037-FMS	PP10; FMS	970	75.9	64.6	94.3	705	99.8
Average Concentration (ng/mL) ± %RPD		**591 ng/mL ± 0.17%**		**17.5 ng/mL ± 12%**		**206 ng/mL ± 20%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-037	PP10; Sample	4060	NA	24.9	NA
ISO16-0008-037-DUP	PP10; Sample Dup	3930	NA	25.0	NA
ISO16-0008-037-FMS	PP10; FMS	4320	NC	76.7	104
Average Concentration (ng/mL) ± %RPD		**4000 ng/mL ± 3.3%**		**25.0 ng/mL ± 0.40%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 27. Location ID: 12

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-038	12; Sample	97.7	NA	25.5	NA
ISO16-0008-038-DUP	12; Sample Dup	95.6	NA	25.5	NA
ISO16-0008-038-FMS	12; FMS	255	79.2	221	97.8
Average Concentration (ng/mL) ± %RPD		**96.7 ng/mL ± 2.2%**		**25.5 ng/mL ± 0.0%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-038	12; Sample	325	NA	1.52	NA
ISO16-0008-038-DUP	12; Sample Dup	266	NA	1.55	NA
ISO16-0008-038-FMS	12; FMS	551	128	11.7	102
Average Concentration (ng/mL) ± %RPD		**296 ng/mL ± 20%**		**1.54 ng/mL ± 2.0%**	

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 28. Location ID: Collector pit

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-080	Collector pit; Sample	106	NA	34.2	NA
ISO16-0008-080-DUP	Collector pit; Sample Dup	106	NA	35.0	NA
ISO16-0008-080-FMS	Collector pit; FMS	264	79.0	191	78.2
Average Concentration (ng/mL) ± %RPD		**106 ng/mL ± 0.0%**		**34.6 ng/mL ± 2.3%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-080	Collector pit; Sample	469	NA	22.5	NA
ISO16-0008-080-DUP	Collector pit; Sample Dup	418	NA	19.5	NA
ISO16-0008-080-FMS	Collector pit; FMS	655	NC	42.1	NC
Average Concentration (ng/mL) ± %RPD		**444 ng/mL ± 11%**		**21.0 ng/mL ± 14%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.

Table 29. Location ID: K3

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-043	K3; Sample	123	NA	1490	NA	83000	NA
ISO16-0008-043-DUP	K3; Sample Dup	118	NA	1490	NA	85300	NA
ISO16-0008-043-FMS	K3; FMS	468	69.5	1530	NC	NA (1)	NA (1)
Average Concentration (ng/mL) ± %RPD		**121 ng/mL ± 4.1% (2)**		**1490 ng/mL ± 0.0%**		**84200 ng/mL ± 2.7%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-043	K3; Sample	5660	NA	17.7	NA
ISO16-0008-043-DUP	K3; Sample Dup	5680	NA	19.4	NA
ISO16-0008-043-FMS	K3; FMS	6180	NC	62.1	87.1
Average Concentration (ng/mL) ± %RPD		**5670 ng/mL ± 0.35%**		**18.6 ng/mL ± 9.2%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) The field matrix spike sample was not re-analyzed since it was not appropriate based on the endogenous sample concentration.
(2) The analytical uncertainty was expanded to ± 33% for PFOA based on the average FMS recovery for all locations with the zones Building 16, WWTP, and Southern site boundary.

Table 30. Location ID: P305

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-047	P305; Sample	211	NA	134	NA	279	NA
ISO16-0008-047-DUP	P305; Sample Dup	224	NA	133	NA	293	NA
ISO16-0008-047-FMS	P305; FMS	459	48.3 (1)	169	NC	719	86.6
Average Concentration (ng/mL) ± %RPD		**218 ng/mL ± 6.0% (2)**		**134 ng/mL ± 0.75%**		**286 ng/mL ± 4.9%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-047	P305; Sample	533	NA	20.6	NA
ISO16-0008-047-DUP	P305; Sample Dup	567	NA	22.3	NA
ISO16-0008-047-FMS	P305; FMS	989	87.8	60.4	77.9
Average Concentration (ng/mL) ± %RPD		**550 ng/mL ± 6.2%**		**21.5 ng/mL ± 7.9%**	

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 ± 50%.
(2) The ***reported results for PFOA should be considered qualitative*** with an expanded uncertainty of ± 52%.

Table 31. Location ID: P118C

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-052	P118C; Sample	303	NA	103	NA
ISO16-0008-052-DUP	P118C; Sample Dup	308	NA	105	NA
ISO16-0008-052-FMS	P118C; FMS	738	86.5	594	98.0
Average Concentration (ng/mL) ± %RPD		**306 ng/mL ± 1.6% (1)**		**104 ng/mL ± 1.9%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-052	P118C; Sample	2070	NA	58.6	NA
ISO16-0008-052-DUP	P118C; Sample Dup	2120	NA	55.0	NA
ISO16-0008-052-FMS	P118C; FMS	2700	NC	107	100
Average Concentration (ng/mL) ± %RPD		**2100 ng/mL ± 2.4%**		**56.8 ng/mL ± 6.3%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) The analytical uncertainty was expanded to ± 33% for PFOA based on the average FMS recovery for all locations with the zones Building 16, WWTP, and Southern site boundary.

Table 32. Location ID: P262

		PFOA		**PFHS**	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-054	P262; Sample	14.5	NA	4.19	NA
ISO16-0008-054-DUP	P262; Sample Dup	14.8	NA	4.31	NA
ISO16-0008-054-FMS	P262; FMS	407	78.5 (1)	479	95.0 (1)
Average Concentration (ng/mL) ± %RPD		**14.7 ng/mL ± 2.0% (2)**		**4.25 ng/mL ± 2.8%**	

		PFOS		**PFOSA**	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-054	P262; Sample	287	NA	140	NA
ISO16-0008-054-DUP	P262; Sample Dup	248	NA	138	NA
ISO16-0008-054-FMS	P262; FMS	780	103	187	NC
Average Concentration (ng/mL) ± %RPD		**268 ng/mL ± 15%**		**139 ng/mL ± 1.4%**	

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) The analytical uncertainty was expanded to ± 33% for PFOA based on the average FMS recovery for all locations with the zones Building 16, WWTP, and Southern site boundary.

Table 33. Location ID: P340

		PFOA		**PFHS**	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-058	P340; Sample	679	NA	304	NA
ISO16-0008-058-DUP	P340; Sample Dup	687	NA	302	NA
ISO16-0008-058-FMS	P340; FMS	944	52.2 (1)	798	99.0
Average Concentration (ng/mL) ± %RPD		**683 ng/mL ± 1.2% (2)**		**303 ng/mL ± 0.66%**	

		PFOS		**PFOSA**	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-058	P340; Sample	3500	NA	79.2	NA
ISO16-0008-058-DUP	P340; Sample Dup	3470	NA	69.8	NA
ISO16-0008-058-FMS	P340; FMS	3950	NC	120	91.0
Average Concentration (ng/mL) ± %RPD		**3490 ng/mL ± 0.86%**		**74.5 ng/mL ± 13%**	

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 analytical uncertainty was expanded to ± 33% for PFOA based on the average FMS recovery for all locations with the zones Building 16, WWTP, and Southern site boundary.

Table 34. Location ID: P379

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-063	P379; Sample	73.9	NA	19.7	NA
ISO16-0008-063-DUP	P379; Sample Dup	75.9	NA	20.2	NA
ISO16-0008-063-FMS	P379; FMS	474	79.8	521	100 (1)
Average Concentration (ng/mL) ± %RPD		**74.9 ng/mL ± 2.7% (2)**		**20.0 ng/mL ± 2.5%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-063	P379; Sample	691	NA	78.3	NA
ISO16-0008-063-DUP	P379; Sample Dup	606	NA	79.0	NA
ISO16-0008-063-FMS	P379; FMS	1220	114	146	135 (3)
Average Concentration (ng/mL) ± %RPD		**649 ng/mL ± 13%**		**78.7 ng/mL ± 0.89%**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.
(2) The analytical uncertainty was expanded to ± 33% for PFOA based on the average FMS recovery for all locations with the zones Building 16, WWTP, and Southern site boundary.
(3) FMS recovery did not meet acceptance criteria of 100 ± 30%. The %bias was less than the overall data uncertainty (±36%) and the data uncertainty was not expanded further.

Table 35. Location ID: PA109A

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-071	PA109A; Sample	40.1	NA	40.1	NA
ISO16-0008-071-DUP	PA109A; Sample Dup	40.2	NA	39.6	NA
ISO16-0008-071-FMS	PA109A; FMS	299	56.6 (1)	562	114 (3)
Average Concentration (ng/mL) ± %RPD		**40.2 ng/mL ± 0.25% (2)**		**39.9 ng/mL ± 1.3%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-071	PA109A; Sample	135	NA	2.40	NA
ISO16-0008-071-DUP	PA109A; Sample Dup	126	NA	2.01	NA
ISO16-0008-071-FMS	PA109A; FMS	657	115	10.8	95.8
Average Concentration (ng/mL) ± %RPD		**131 ng/mL ± 6.9%**		**2.21 ng/mL ± 18%**	

NA = Not Applicable
(1) FMS recovery did not meet acceptance criteria of 100 ± 30%.
(2) The analytical uncertainty was expanded to ± 33% for PFOA based on the average FMS recovery for all locations with the zones Building 16, WWTP, and Southern site boundary.
(3) FMS concentration greater than 10 times the sample concentration.

Table 36. Trip Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-074	Trip Blank	<0.0240	NA	<0.200	NA	<0.0250	NA
ISO16-0008-074-FMS-LOW	Trip Blank FMS Low	4.53	94.6	4.50	90.0	4.85	97.0
ISO16-0008-074-FMS-HIGH	Trip Blank FMS High	468	93.6	44.1	88.2	448	89.6

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-074	Trip Blank	<0.185	NA	<0.0250	NA
ISO16-0008-074-FMS-LOW	Trip Blank FMS Low	4.42	95.4	5.12	102
ISO16-0008-074-FMS-HIGH	Trip Blank FMS High	471	94.2	47.2	94.4

NA = Not Applicable

Table 37. Trip Blank 2

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-084	Trip Blank 2; Sample	<0.0958	NA	<0.200	NA	<0.100	NA
ISO16-0008-084-FMS-LOW	Trip Blank 2; FMS Low	4.85	101	4.78	95.6	4.84	96.8
ISO16-0008-084-FMS-HIGH	Trip Blank 2; FMS High	206	103	9.74	97.4	201	101

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO16-0008-084	Trip Blank 2; Sample	<0.0928	NA	<0.0250	NA
ISO16-0008-084-FMS-LOW	Trip Blank 2; FMS Low	4.61	99.5	5.31	106
ISO16-0008-084-FMS-HIGH	Trip Blank 2; FMS High	210	105	11.0	110

NA = Not Applicable

Table 38. Surrogate Recovery Standard Results (1)

		Percent Recovery (%)		
3M LIMS ID	**Sample Description**	**[$^{13}C_4$]-PFOA**	**[$^{13}C_4$]-PFOS**	**[$^{13}C_4$]-PFOS Re-analysis**
ISO16-0008-001	BD24-3; Sample	121	118	NA
ISO16-0008-001-DUP	BD24-3; Sample Dup	133 (3)	123	NA
ISO16-0008-002	BD24-4; Sample	123	123	NA
ISO16-0008-002-DUP	BD24-4; Sample Dup	126	128	NA
ISO16-0008-002-FMS	BD24-4; FMS	117	121	NA
ISO16-0008-003	D09; Sample	128	116	NA
ISO16-0008-003-DUP	D09; Sample Dup	118	116	NA
ISO16-0008-004	D10; Sample	128	128	NA
ISO16-0008-004-DUP	D10; Sample Dup	121	119	NA
ISO16-0008-005	D11; Sample	103	85.7	117
ISO16-0008-005-DUP	D11; Sample Dup	104	87.7	120
ISO16-0008-006	D14; Sample	94.6 (2)	125	94.5 (2)
ISO16-0008-006-DUP	D14; Sample Dup	90.7 (2)	132 (3)	94.6 (2)
ISO16-0008-006-FMS	D14; FMS	88.7 (2)	125	94.7 (2)
ISO16-0008-007	D16; Sample	113	118	NA
ISO16-0008-007-DUP	D16; Sample Dup	106	121	NA
ISO16-0008-007-FMS	D16; FMS	93.7	120	NA
ISO16-0008-008	D17; Sample	111	99.1	NA
ISO16-0008-008-DUP	D17; Sample Dup	111	101	NA
ISO16-0008-008-FMS	D17; FMS	113	96.7	NA
ISO16-0008-009	D18; Sample	92.5	133 (3)	NA
ISO16-0008-009-DUP	D18; Sample Dup	82.2	128	NA
ISO16-0008-009-FMS	D18; FMS	82.2	120	NA
ISO16-0008-010	D2; Sample	90.3 (2)	126	92.9 (2)
ISO16-0008-010-DUP	D2; Sample Dup	90.3 (2)	128	100 (2)
ISO16-0008-010-FMS	D2; FMS	NA	118	NA
ISO16-0008-011	D5; Sample	100	117	NA
ISO16-0008-011-DUP	D5; Sample Dup	88.4	108	NA
ISO16-0008-011-FMS	D5; FMS	92.7	107	NA
ISO16-0008-012	ND7; Sample	105	111	NA
ISO16-0008-012-DUP	ND7; Sample Dup	111	108	NA
ISO16-0008-012-FMS	ND7; FMS	109	103	NA
ISO16-0008-013	P118A; Sample	110	107	121
ISO16-0008-013-DUP	P118A; Sample Dup	111	99.4	119
ISO16-0008-014	P118B; Sample	123	120	NA
ISO16-0008-014-DUP	P118B; Sample Dup	121	116	NA
ISO16-0008-014-FMS	P118B; FMS	104	109	NA
ISO16-0008-015	P119A; Sample	109	124	NA
ISO16-0008-015-DUP	P119A; Sample Dup	114	123	NA
ISO16-0008-015-FMS	P119A; FMS	107	115	NA

NA = Not Applicable

(1) The surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-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 38 continued. Surrogate Recovery Standard Results (1)

		Percent Recovery (%)		
3M LIMS ID	**Sample Description**	**[$^{13}C_4$]-PFOA**	**[$^{13}C_4$]-PFOS**	**[$^{13}C_4$]-PFOS Re-analysis**
ISO16-0008-016	P119B; Sample	103	114	NA
ISO16-0008-016-DUP	P119B; Sample Dup	109	120	NA
ISO16-0008-016-FMS	P119B; FMS	112	121	NA
ISO16-0008-075	P121; Sample	108	103	NA
ISO16-0008-075-DUP	P121; Sample Dup	113	114	NA
ISO16-0008-018	P321; Sample	98.6	95.4	113
ISO16-0008-018-DUP	P321; Sample Dup	105	99.8	115
ISO16-0008-019	3M vijver; Sample	90.9 (2)	121	88.1 (2)
ISO16-0008-019-DUP	3M vijver; Sample Dup	90.5 (2)	126	91.5 (2)
ISO16-0008-019-FMS	3M vijver; FMS	95.8 (2)	115	89.2 (2)
ISO16-0008-020	Blokkersdijkvijver standard; Sample	96.2 (2)	121	85.3 (2)
ISO16-0008-020-DUP	Blokkersdijkvijver standard; Sample Dup	89.6 (2)	123	92.1 (2)
ISO16-0008-021	L21; Sample	94.5 (2)	123	90.4 (2)
ISO16-0008-021-DUP	L21; Sample Dup	90.2 (2)	121	97.7 (2)
ISO16-0008-022	L22; Sample	94.4 (2)	121	90.6 (2)
ISO16-0008-022-DUP	L22; Sample Dup	89.2 (2)	118	91.5 (2)
ISO16-0008-022-FMS	L22; FMS	87.2 (2)	126	89.8 (2)
ISO16-0008-023	L31; Sample	90.1 (2)	128	90.3 (2)
ISO16-0008-023-DUP	L31; Sample Dup	96.5 (2)	127	92.8 (2)
ISO16-0008-076	L4; Sample	114	123	NA
ISO16-0008-076-DUP	L4; Sample Dup	107	112	NA
ISO16-0008-025	P114bis; Sample	94.0 (2)	117	93.0 (2)
ISO16-0008-025-DUP	P114bis; Sample Dup	95.0 (2)	122	95.3 (2)
ISO16-0008-026	P115; Sample	124	124	NA
ISO16-0008-026-DUP	P115; Sample Dup	131 (3)	134 (3)	NA
ISO16-0008-027	P116; Sample	92.0 (2)	118	91.5 (2)
ISO16-0008-027-DUP	P116; Sample Dup	90.8 (2)	126	90.8 (2)
ISO16-0008-027-FMS	P116; FMS	87.6 (2)	117	91.7 (2)
ISO16-0008-077	Effluent WWTP; Sample	111	120	NA
ISO16-0008-077-DUP	Effluent WWTP; Sample Dup	113	121	NA
ISO16-0008-029	PP01; Sample	106	109	NA
ISO16-0008-029-DUP	PP01; Sample Dup	103	116	NA
ISO16-0008-030	PP02; Sample	128	101	NA
ISO16-0008-030-DUP	PP02; Sample Dup	119	97.4	NA
ISO16-0008-030-FMS	PP02; FMS	124	99.1	NA
ISO16-0008-031	PP04; Sample	120	122	NA
ISO16-0008-031-DUP	PP04; Sample Dup	128	126	NA
ISO16-0008-032	PP05; Sample	125	119	NA
ISO16-0008-032-DUP	PP05; Sample Dup	127	123	NA

NA = Not Applicable

(1) The surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-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 38 continued. Surrogate Recovery Standard Results (1)

		Percent Recovery (%)		
3M LIMS ID	**Sample Description**	**[$^{13}C_4$]-PFOA**	**[$^{13}C_4$]-PFOS**	**[$^{13}C_4$]-PFOS Re-analysis**
ISO16-0008-078	PP06; Sample	108	114	NA
ISO16-0008-078-DUP	PP06; Sample Dup	104	113	NA
ISO16-0008-078-FMS	PP06; FMS	110	118	NA
ISO16-0008-079	PP07; Sample	106	105	NA
ISO16-0008-079-DUP	PP07; Sample Dup	113	112	NA
ISO16-0008-035	PP08; Sample	128	117	NA
ISO16-0008-035-DUP	PP08; Sample Dup	118	116	NA
ISO16-0008-036	PP09; Sample	116	104	NA
ISO16-0008-036-DUP	PP09; Sample Dup	113	111	NA
ISO16-0008-037	PP10; Sample	117	119	NA
ISO16-0008-037-DUP	PP10; Sample Dup	118	115	NA
ISO16-0008-037-FMS	PP10; FMS	119	116	NA
ISO16-0008-038	12; Sample	118	120	NA
ISO16-0008-038-DUP	12; Sample Dup	119	118	NA
ISO16-0008-038-FMS	12; FMS	115	115	NA
ISO16-0008-039	13; Sample	113	123	NA
ISO16-0008-039-DUP	13; Sample Dup	116	120	NA
ISO16-0008-040	5; Sample	116	120	NA
ISO16-0008-040-DUP	5; Sample Dup	115	122	NA
ISO16-0008-041	Bemalingsstation; Sample	117	123	NA
ISO16-0008-041-DUP	Bemalingsstation; Sample Dup	117	121	NA
ISO16-0008-080	Collector pit; Sample	106	116	NA
ISO16-0008-080-DUP	Collector pit; Sample Dup	111	124	NA
ISO16-0008-080-FMS	Collector pit; FMS	111	121	NA
ISO16-0008-043	K3; Sample	114	121	119
ISO16-0008-043-DUP	K3; Sample Dup	110	116	124
ISO16-0008-043-FMS	K3; FMS	117	124	NA
ISO16-0008-044	P27; Sample	113	117	NA
ISO16-0008-044-DUP	P27; Sample Dup	117	120	NA
ISO16-0008-045	P21B; Sample	113	116	105
ISO16-0008-045-DUP	P21B; Sample Dup	111	114	118
ISO16-0008-046	P304; Sample	113	115	NA
ISO16-0008-046-DUP	P304; Sample Dup	117	123	NA
ISO16-0008-047	P305; Sample	112	119	120
ISO16-0008-047-DUP	P305; Sample Dup	107	113	120
ISO16-0008-047-FMS	P305; FMS	114	117	121
ISO16-0008-048	P42; Sample	121	123	NA
ISO16-0008-048-DUP	P42; Sample Dup	116	118	NA
ISO16-0008-049	P56; Sample	114	123	NA
ISO16-0008-049-DUP	P56; Sample Dup	113	121	NA

NA = Not Applicable

(1) The surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-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 38 continued. Surrogate Recovery Standard Results (1)

		Percent Recovery (%)		
3M LIMS ID	**Sample Description**	**[$^{13}C_4$]-PFOA**	**[$^{13}C_4$]-PFOS**	**[$^{13}C_4$]-PFOS Re-analysis**
ISO16-0008-050	L19; Sample	117	109	NA
ISO16-0008-050-DUP	L19; Sample Dup	117	111	NA
ISO16-0008-081	M4; Sample	105	112	NA
ISO16-0008-081-DUP	M4; Sample Dup	112	117	NA
ISO16-0008-052	P118C; Sample	111	115	NA
ISO16-0008-052-DUP	P118C; Sample Dup	123	123	NA
ISO16-0008-052-FMS	P118C; FMS	112	116	NA
ISO16-0008-053	P119C; Sample	102	105	NA
ISO16-0008-053-DUP	P119C; Sample Dup	115	119	NA
ISO16-0008-054	P262; Sample	116	123	NA
ISO16-0008-054-DUP	P262; Sample Dup	87.0	89.9	NA
ISO16-0008-054-FMS	P262; FMS	118	127	NA
ISO16-0008-055	P263; Sample	104	103	113
ISO16-0008-055-DUP	P263; Sample Dup	120	118	113
ISO16-0008-082	P264; Sample	110	118	NA
ISO16-0008-082-DUP	P264; Sample Dup	110	118	NA
ISO16-0008-083	P265B; Sample	115	121	NA
ISO16-0008-083-DUP	P265B; Sample Dup	114	123	NA
ISO16-0008-058	P340; Sample	116	118	NA
ISO16-0008-058-DUP	P340; Sample Dup	116	119	NA
ISO16-0008-058-FMS	P340; FMS	109	112	NA
ISO16-0008-059	P341; Sample	120	117	NA
ISO16-0008-059-DUP	P341; Sample Dup	116	111	NA
ISO16-0008-060	P343; Sample	103	99.3	NA
ISO16-0008-060-DUP	P343; Sample Dup	114	110	NA
ISO16-0008-061	P371; Sample	114	109	NA
ISO16-0008-061-DUP	P371; Sample Dup	118	114	NA
ISO16-0008-062	P374; Sample	111	111	NA
ISO16-0008-062-DUP	P374; Sample Dup	125	123	NA
ISO16-0008-063	P379; Sample	118	120	NA
ISO16-0008-063-DUP	P379; Sample Dup	115	120	NA
ISO16-0008-063-FMS	P379; FMS	120	127	NA
ISO16-0008-064	P380; Sample	116	120	NA
ISO16-0008-064-DUP	P380; Sample Dup	115	119	NA
ISO16-0008-065	P381; Sample	119	123	NA
ISO16-0008-065-DUP	P381; Sample Dup	109	115	NA
ISO16-0008-066	P382; Sample	117	121	NA
ISO16-0008-066-DUP	P382; Sample Dup	113	121	NA
ISO16-0008-067	B3-bis; Sample	121	125	118
ISO16-0008-067-DUP	B3-bis; Sample Dup	120	126	106

NA = Not Applicable
(1) The surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-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) Standard recovery standard did not meet acceptance criteria of 100 ± 30%.

Table 38 continued. Surrogate Recovery Standard Results (1)

		Percent Recovery (%)		
3M LIMS ID	**Sample Description**	**[$^{13}C_4$]-PFOA**	**[$^{13}C_4$]-PFOS**	**[$^{13}C_4$]-PFOS Re-analysis**
ISO16-0008-069	P372; Sample	117	127	NA
ISO16-0008-069-DUP	P372; Sample Dup	114	119	NA
ISO16-0008-070	P378; Sample	111	101	116
ISO16-0008-070-DUP	P378; Sample Dup	111	99.0	117
ISO16-0008-071	PA109A; Sample	117	124	NA
ISO16-0008-071-DUP	PA109A; Sample Dup	116	125	NA
ISO16-0008-071-FMS	PA109A; FMS	109	114	NA
ISO16-0008-072	PA111A; Sample	121	126	NA
ISO16-0008-072-DUP	PA111A; Sample Dup	109	116	NA
ISO16-0008-073	PA112; Sample	106	114	NA
ISO16-0008-073-DUP	PA112; Sample Dup	109	116	NA
ISO16-0008-074	Trip Blank	98.1 (2)	125	101 (2)
ISO16-0008-074-FMS-LOW	Trip Blank FMS Low	93.2 (2)	126	92.3 (2)
ISO16-0008-074-FMS-HIGH	Trip Blank FMS High	120	121	NA
ISO16-0008-084	Trip Blank 2; Sample	112	124	117
ISO16-0008-084-FMS-LOW	Trip Blank 2; FMS Low	115	122	NA
ISO16-0008-084-FMS-HIGH	Trip Blank 2; FMS High	115	119	NA

NA = Not Applicable
(1) The surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-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) Standard 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 Environmental 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 10-38 of this report.

6 Data / Sample Retention

All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M Environmental Laboratory standard operating procedures.

7 Signatures

Chelsie Grochow, 3M Report Author

Susan T. Wolf, 3M Principal Analytical Investigator

William K. Reagen, Ph.D., 3M Environmental Laboratory Technical Director

The 3M Environmental Laboratory’s Quality Assurance Unit has audited the data and report for this project.

Quality Assurance Representative

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Bijlage 4

Overzicht veldmetingen en analyseresultaten

Meetpunt		**12**	**12**	**12**	**12**	**13**	**13**	**13**	**13**	**3M vijver**	**3M vijver**
Filterstelling (m)											
Datum bemonstering	**Eenheid**	20/10/2015	12/01/2016	07/04/2016	28/07/2016	20/10/2015	12/01/2016	07/04/2016	28/07/2016	20/10/2015	12/01/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm										
Temperatuur	°C	10,61	6,77	8,5	17,23	10,58	6,79	9,64	17,18	10,64	5,19
pH		7,63	7,36	7,77	7,51	7,71	7,19	7,35	7,55	7,35	7,41
Conductiviteit	µS/cm	903	563	722	958	895	560	740	962	741	458
Redox	mV	116	60,9	267	156	112	70	269	140,6	110	58

Meetpunt		**3M vijver**	**3M vijver**	**5**	**5**	**5**	**5**	**B3-bis**	**B3-bis**	**B3-bis**	**B7**
Filterstelling (m)								**2,5-3,5**	**2,5-3,5**	**2,5-3,5**	**1,8-3,8**
Datum bemonstering	**Eenheid**	07/04/2016	28/07/2016	20/10/2015	12/01/2016	07/04/2016	28/07/2016	22/10/2015	19/01/2016	29/07/2016	19/01/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm							269	200	207	200
Temperatuur	°C	9,32	19,77	9,51	5,88	9,1	19,12	13,29	5,64	16,05	7,23
pH		7,68	7,79	7,74	7,24	7,76	7,51	7,36	7,24	7,39	7,09
Conductiviteit	µS/cm	578	756	1242	774	826	1156	419	211	370	204
Redox	mV	226	145,7	107	83	226	-70,5	-70	30	-120,8	36

Meetpunt		**BD24-4**	**BD24-3**	**BD24-3**	**Bemalingsstation**	**Bemalingsstation**	**Bemalingsstation**	**Bemalingsstation**	**Blokkersdijk - std**	**Blokkersdijk - std**	**Blokkersdijk - std**
Filterstelling (m)		**22,0-24,0**	**17,0-18,0**	**17,0-18,0**							
Datum bemonstering	**Eenheid**	25/07/2016	13/01/2016	25/07/2016	20/10/2015	12/01/2016	07/04/2016	28/07/2016	20/10/2015	12/01/2016	07/04/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm	296	318	296							
Temperatuur	°C	14,76	10,07	13,98	10,54	5,7	9,42	19,23	10,22	4,74	10,27
pH		7,12	7,22	7,45	7,58	7,15	7,74	7,52	7,11	7,39	7,37
Conductiviteit	µS/cm	1275	597	727	1363	797	870	1279	570	462	443
Redox	mV	-3,8	-25	-22,3	120	86	237	72	99	53,3	174

Meetpunt		**Blokkersdijk - std**	**Collector pit**	**Collector pit**	**Collector pit**	**Collector pit**	**D10**	**D11**	**D14**	**D16**	**D17**
Filterstelling (m)								**14,9-15,9**	**15,5-16,5**	**14,0-16,0**	**14,0-16,0**
Datum bemonstering	**Eenheid**	28/07/2016	22/10/2015	12/01/2016	06/04/2016	26/07/2016	25/07/2016	20/07/2016	27/07/2016	25/07/2016	27/07/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm						404	296	477	305	315
Temperatuur	°C	20,88	15,15	11,13	11,3	21,11	14,28	15,7	15,52	15,42	17,92
pH		8,55	7,68	7,86	7,9	8,6	7,23	7,3	6,92	6,98	7,15
Conductiviteit	µS/cm	408	500	577	560	1092	1390	904	3334	640	784
Redox	mV	83	-51	35,3	63,5	74,6	-96,3	-14,8	-107,4	35	45,8

Meetpunt		**D18**	**D2**	**D5**	**D9**	**D9**	**D9**	**Effluent WWTP**	**Effluent WWTP**	**Effluent WWTP**	**Effluent WWTP**
Filterstelling (m)		**14,0-16,0**	**24,0-26,2**	**14,0-16,0**	**13,0-15,0**	**13,0-15,0**	**13,0-15,0**				
Datum bemonstering	**Eenheid**	28/07/2016	27/07/2016	28/07/2016	19/10/2015	14/01/2016	27/07/2016	22/10/2015	12/01/2016	06/04/2016	26/07/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm	236	538	320	362	335	311				
Temperatuur	°C	15,81	15,33	15,08	16,3	13,49	18,58	15,26	10,65	14,62	26,7
pH		7,22	7,91	7	7,02	6,66	7,22	7,57	6,74	7,8	7,95
Conductiviteit	µS/cm	907	307	851	973	937	726	5276	4055	4810	5299
Redox	mV	-124,8	50,7	-47,3	-113	-126,6	-170	-93,4	89,9	84,2	-22,4

Meetpunt		**K3**	**L19**	**L19**	**L21**	**L21**	**L21**	**L22**	**L22**	**L22**	**L22**
Filterstelling (m)		**3,6-5,6**	**2,8-4,8**	**2,8-4,8**	**3,0-5,0**	**3,0-5,0**	**3,0-5,0**	**2,0-4,0**	**2,0-4,0**	**2,0-4,0**	**2,0-4,0**
Datum bemonstering	**Eenheid**	27/07/2016	13/01/2016	25/07/2016	14/01/2016	06/04/2016	26/07/2016	21/10/2015	14/01/2016	06/04/2016	26/07/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm	190	319	232	168	165	176	210	154	142	166
Temperatuur	°C	18,06	9,52	18,32	9,72	9,74	17,78	13,5	9,44	9,34	18,52
pH		7,42	7,28	7,38	7,53	6,85	7,56	7,43	7,48	6,97	7,43
Conductiviteit	µS/cm	577	648	497	467	651	409	385	510	485	395
Redox	mV	-134	-61,4	-18,4	44,2	2,9	-176,8	-54	43	17,6	-148

Meetpunt		**L31**	**L31**	**L31**	**L4**	**L4**	**L4**	**L4**	**M4**	**M4**	**ND7**
Filterstelling (m)		**1,2-3,2**	**1,2-3,2**	**1,2-3,2**	**2,0-4,0**	**2,0-4,0**	**2,0-4,0**	**2,0-4,0**	**1,0-3,0**	**1,0-3,0**	**14,0-16,0**
Datum bemonstering	**Eenheid**	14/01/2016	06/04/2016	26/07/2016	21/10/2015	13/01/2016	06/04/2016	25/07/2016	13/01/2016	26/07/2016	27/07/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm	203	210	219	239	177	133	173	192	105	258
Temperatuur	°C	6,93	10,17	19,12	13,35	9,25	9,49	17,78	11	22,76	16,27
pH		6,79	7,45	6,99	7,33	7,61	7,4	7,08	7,06	7,2	7,45
Conductiviteit	µS/cm	404	524	502	699	251	330	6,22	1219	1035	308
Redox	mV	-44	229	59,8	-148	-33	-13,5	-2	-49,9	48,2	33,1

Meetpunt		**P114bis**	**P114bis**	**P114bis**	**P114bis**	**P115**	**P115**	**P115**	**P116**	**P116**	**P116**
Filterstelling (m)		**3,6-4,6**	**3,6-4,6**	**3,6-4,6**	**3,6-4,6**	**3,3-4,3**	**3,3-4,3**	**3,3-4,3**	**2,5-3,5**	**2,5-3,5**	**2,5-3,5**
Datum bemonstering	**Eenheid**	21/10/2015	14/01/2016	06/04/2016	26/07/2016	14/01/2016	06/04/2016	26/07/2016	21/10/2015	14/01/2016	06/04/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm	161	118	118	146	140	137	161	257	210	217
Temperatuur	°C	13,82	8,17	10,94	16,75	10,13	10,1	16,13	13,54	7,62	10,79
pH		6,97	6,61	7,17	7,24	6,98	6,43	6,82	7,4	7,2	7,54
Conductiviteit	µS/cm	2628	3459	1891	751	2783	2418	3083	612	551	505
Redox	mV	-117	-83,4	-147	-176	25	-30,9	44,3	-77,9	-125	-150

Meetpunt		**P116**	**P118A**	**P118B**	**P118C**	**P118C**	**P119A**	**P119B**	**P119C**	**P119C**	**P119C**
Filterstelling (m)		**2,5-3,5**	**22,5-23,5**	**13,0-14,0**	**2,5-4,5**	**2,5-4,5**	**0,2-2,4**	**13,0-14,0**	**2,5-4,5**	**2,5-4,5**	**2,5-4,5**
Datum bemonstering	**Eenheid**	27/07/2016	20/07/2016	20/07/2016	11/01/2016	20/07/2016	25/07/2016	25/07/2016	22/10/2015	13/01/2016	25/07/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm	235	276	266	275	199	355	339	2,23	170	153
Temperatuur	°C	15,63	17,09	15,52	10,35	15,81	14	15,42	12,36	9,85	15,08
pH		6,97	7,19	7,67	6,85	7,2	6,92	6,91	7,19	7,28	7,53
Conductiviteit	µS/cm	623	1738	1869	747	1627	1186	1549	592	494	575
Redox	mV	17	-15,6	-108,8	-7,8	-151,8	-0,3	31,9	9	-42	-142

Meetpunt		**P121**	**P121**	**P121**	**P18**	**P18**	**P21B**	**P21B**	**P21B**	**P262**	**P263**
Filterstelling (m)		**23,5-24,5**	**23,5-24,5**	**23,5-24,5**	**1,4-3,4**	**1,4-3,4**	**3,5-5,5**	**3,5-5,5**	**3,5-5,5**	**6,2-7,2**	**7,4-8,4**
Datum bemonstering	**Eenheid**	22/10/2015	14/01/2016	26/07/2016	21/10/2015	27/07/2016	21/10/2015	14/01/2016	07/04/2016	25/07/2016	13/01/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm	481	4,48	448	schuim	schuim	92	183	83	304	516
Temperatuur	°C	13,22	10,52	16,36			12,38	8,91	10,4	16,77	10,22
pH		6,76	6,51	6,8			7,31	7,55	10,75	7,11	7,22
Conductiviteit	µS/cm	2537	2570	2804			2064	1765	1709	406	1243
Redox	mV	-50,4	-7,3	-50,1			-122	-25	-197	-2,9	-47,6

Meetpunt		**P263**	**P263**	**P264**	**P264**	**P265C**	**P265C**	**P27**	**P27**	**P28**	**P28**
Filterstelling (m)		**7,4-8,4**	**7,4-8,4**	**5,0-5,5**	**5,0-5,5**	**4,5-5,5**	**4,5-5,5**	**1,9-2,9**	**1,9-2,9**	**1,1-3,1**	**1,1-3,1**
Datum bemonstering	**Eenheid**	07/04/2016	25/07/2016	13/01/2016	20/07/2016	13/01/2016	20/07/2016	14/01/2016	27/07/2016	20/10/2015	27/07/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm	475	478	293	234		185	150	130	schuim	schuim
Temperatuur	°C	10,14	14,39	10,47	18,01		19,22	9	19,4		
pH		7,4	7,51	7	7,69		7,21	6,97	7,35		
Conductiviteit	µS/cm	1363	1378	597	723		713	761	660		
Redox	mV	-149	-25,6	2,1	-202		-122,8	-89	33,5		

Meetpunt		P304	P304	P305	P305	P321	P321	P321	P321	P340	P340
Filterstelling (m)		5,0-6,0	5,0-6,0	3,5-5,5	3,5-5,5	15,5-16,5	15,5-16,5	15,5-16,5	15,5-16,5	4,0-6,0	4,0-6,0
Datum bemonstering	Eenheid	14/01/2016	27/07/2016	14/01/2016	28/07/2016	22/10/2015	13/01/2016	06/04/2016	25/07/2016	20/10/2015	26/07/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm	231	211	179	163	412	400	354	375	273	195
Temperatuur	°C	10,5	18,47	12,07	17,87	13,5	11,08	12	15,3	16,21	19,5
pH		7,09	7,12	7,19	6,98	7,11	7,13	7,14	7,26	6,74	6,73
Conductiviteit	µS/cm	739	630	825	802	1068	1222	1372	1104	1626	1229
Redox	mV	8	-114,4	-28,9	-127,9	-73,4	-54	-85,2	-11,7	-74,4	-16,4

Meetpunt		**P341**	**P343**	**P371**	**P371**	**P372**	**P372**	**P374**	**P374**	**P378**	**P378**
Filterstelling (m)		**3,6-5,6**	**5,1-6,1**	**1,5-3,5**	**1,5-3,5**	**4,8-5,8**	**4,8-5,8**	**5,0-6,0**	**5,0-6,0**	**3,5-5,5**	**3,5-5,5**
Datum bemonstering	**Eenheid**	25/07/2016	25/07/2016	13/01/2016	20/07/2016	13/01/2016	20/07/2016	13/01/2016	20/07/2016	19/01/2016	29/07/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm	226	310	247	164	259	198	312	265	204	199
Temperatuur	°C	14,47	15,14	9,42	17,45	10,49	16,98	10,48	15,35	10,6	16,41
pH		6,85	6,95	6,8	7,28	6,81	7,17	6,48	6,94	6,57	6,86
Conductiviteit	µS/cm	931	1209	458	944	334	504	656	996	793	1003
Redox	mV	28,5	-14	96	225	49,7	-36,8	27,5	-26,8	33,8	-116

Meetpunt		**P379**	**P380**	**P381**	**P381**	**P382**	**P382**	**P42**	**P42**	**P56**	**PA109A**
Filterstelling (m)		**3,5-5,5**	**3,5-5,5**	**5,0-6,0**	**5,0-6,0**	**2,5-3,5**	**2,5-3,5**	**3,0-5,0**	**3,0-5,0**	**1,2-3,2**	**5,0-6,0**
Datum bemonstering	**Eenheid**	25/07/2016	20/07/2016	11/01/2016	20/07/2016	11/01/2016	20/07/2016	14/01/2016	27/07/2016	27/07/2016	19/01/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm	199	172	293	256	2,59	191	274	266	148	291
Temperatuur	°C	15,76	15,04	10,53	17,18	10,41	17,09	11,19	18,83	18,11	6,83
pH		7,11	7,43	6,63	7,05	7,02	7,08	6,45	6,92	7,64	6,89
Conductiviteit	µS/cm	545	524	675	878	864	1514	1261	1732	335	486
Redox	mV	-8,2	-83	29,1	-97,6	-154,6	-131,7	-103	-106	2,7	44,7

Meetpunt		**PA109A**	**PA111A**	**PA111A**	**PA112**	**PA112**	**PP01**	**PP01**	**PP01**	**PP01**	**PP02**
Filterstelling (m)		**5,0-6,0**	**4,0-6,0**	**4,0-6,0**	**3,5-5,5**	**3,5-5,5**	**2,6-5,6**	**2,6-5,6**	**2,6-5,6**	**2,6-5,6**	**3,2-6,2**
Datum bemonstering	**Eenheid**	29/07/2016	19/01/2016	29/07/2016	19/01/2016	29/07/2016	22/10/2015	12/01/2016	05/04/2016	02/08/2016	22/10/2015
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm	301	233	216	244	207	114				
Temperatuur	°C	15,77	10,47	15,84	9,34	15,8	13,81	10,82	11,35	17,62	14,61
pH		7,16	6,34	6,71	7,02	7,38	7,27	7,47	7,32	7,64	7,13
Conductiviteit	µS/cm	483	1147	1280	737	805	674	478	597	593	781
Redox	mV	-149	53,1	-72,7	67,3	-153,8	-29	28,5	-15,1	-66,5	-52,8

Meetpunt		**PP02**	**PP02**	**PP02**	**PP04**	**PP04**	**PP04**	**PP04**	**PP05**	**PP05**	**PP05**
Filterstelling (m)		**3,2-6,2**	**3,2-6,2**	**3,2-6,2**	**2,0-6,0**	**2,0-6,0**	**2,0-6,0**	**2,0-6,0**	**1,6-5,6**	**1,6-5,6**	**1,6-5,6**
Datum bemonstering	**Eenheid**	12/01/2016	05/04/2016	26/07/2016	22/10/2015	12/01/2016	05/04/2016	26/07/2016	20/10/2015	12/01/2016	05/04/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm										
Temperatuur	°C	12,16	12,01	18,45		12,6	11,74	17,6		extreme waarden	
pH		7,03	7,15	7,41		7,43	6,84	7,12			
Conductiviteit	µS/cm	661	775	859		596	830	1230			
Redox	mV	58,6	-44,4	-40,7		23,7	57,8	-59,5			

Meetpunt		**PP06**	**PP06**	**PP06**	**PP06**	**PP07**	**PP07**	**PP07**	**PP07**	**PP08**	**PP08**
Filterstelling (m)		**1,3-5,3**	**1,3-5,3**	**1,3-5,3**	**1,3-5,3**	**2,0-6,0**	**2,0-6,0**	**2,0-6,0**	**2,0-6,0**	**3,2-7,2**	**3,2-7,2**
Datum bemonstering	**Eenheid**	22/10/2015	12/01/2016	08/04/2016	26/07/2016	29/10/2015	12/01/2016	05/04/2016	26/07/2016	22/10/2015	12/01/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm										
Temperatuur	°C	15,83	12,3	10,23	18,26	16,57		11,46	19,59	13,47	10,5
pH		8,04	7,61	7,25	7,47	7,24	7,07	7,12	7,29	6,93	6,8
Conductiviteit	µS/cm	763	511	665	730	717	621	838	891	908	835
Redox	mV	-83,1	32	30,6	-65,8	-41,3	84,1	-41,8	-29	-26,2	92,9

Meetpunt		**PP08**	**PP08**	**PP09**	**PP09**	**PP09**	**PP09**	**PP10**	**PP10**	**PP10**	**PP10**
Filterstelling (m)		**3,2-7,2**	**3,2-7,2**	**3,0-6,0**	**3,0-6,0**	**3,0-6,0**	**3,0-6,0**	**3,0-6,0**	**3,0-6,0**	**3,0-6,0**	**3,0-6,0**
Datum bemonstering	**Eenheid**	05/04/2016	26/07/2016	22/10/2015	12/01/2016	05/04/2016	26/07/2016	22/10/2015	12/01/2016	05/04/2016	26/07/2016
Grondwaterstand											
Grondwaterstand (bovenkant peilbuis)	cm										
Temperatuur	°C	11,96	17,06	12,67	9,45	12,01	19,63	13,57	11,08	11,53	16,5
pH		7,11	7,37	7,19	7,22	7,04	7,21	7,36	7,18	7,17	7,35
Conductiviteit	µS/cm	1071	1153	605	639	764	849	361	663	800	882
Redox	mV	-40,9	-85,6	-24,1	67	-31,6	-41,1	-51,6	73	-70,5	-67,9

Locatie	**B3-bis**	**D9**	**L4**	**P114bis**	**P116**	**P119C**	**P121**	**P321**	**P340**	**L22**
Bemonsteringsdatum	*22/10/2015*	*19/10/2015*	*21/10/2015*	*21/10/2015*	*21/10/2015*	*22/10/2015*	*22/10/2015*	*22/10/2015*	*20/10/2015*	*21/10/2015*
FOSA (µg/l)	<1,09		<0,598		<0,201	14,8962				
PFBS (µg/l)										
PFHS (µg/l)	7,57	7700	2,77	1,45	0,73	570	<0,189	358	304	0,479
PFOA (µg/l)	36,4	168	2,77	4,38	1,72	855	0,283	1070	698	1,09
PFOS (µg/l)	146	1680	46,2	14	34,6	4050	7,46	4110	3450	38,6
PFOSA (µg/l)	0,555	19	0,618	0,0677	0,252	55,4	0,31	15	31,9	0,216

Locatie	**BD24-3**	**B3-bis**	**B7**	**D9**	**L19**	**L21**	**L31**	**L4**	**L22**	**M4**
Bemonsteringsdatum	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*
FOSA (µg/l)	<4,33	<2,16	<0,400	<23,8		0,129	0,222		0,113	56,166
PFBS (µg/l)										
PFHS (µg/l)	55,2	<0,833	4,2	13200	203	0,201	0,762	2,4	0,213	1680
PFOA (µg/l)	69,9	3,3078	7,58	161	208	0,454	0,531	3,98	0,471	8530
PFOS (µg/l)	1270	56,0772	65	1980	3630	5,65	7,61	37,3	5,39	15000
PFOSA (µg/l)	2,82	0,125	0,33	21,3	42,6	0,131	0,226	6,13	0,116	142

Locatie	**P114bis**	**P115**	**P116**	**P118C**	**P119C**	**P121**	**P21B**	**P263**	**P264**	**P27**
Bemonsteringsdatum	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*
FOSA (µg/l)		0,025		25,4856	29,2068	<0,400			38,6724	14,7852
PFBS (µg/l)							6850			3700
PFHS (µg/l)	1,28	2,3	0,498	69,2	600	0,377	11000	524	262	31,6
PFOA (µg/l)	3,62	4,18	0,9	153	888	0,64	11700	1600	369	385
PFOS (µg/l)	4,29	0,594	8,51	1370	2410	17,4	55400	9070	1610	1790
PFOSA (µg/l)			0,0898	62,3	53,2	0,763	2,02	10,9	73,8	53,9

Locatie	**P304**	**P305**	**P321**	**P371**	**P372**	**P374**	**P378**	**P381**	**P382**	**P42**
Bemonsteringsdatum	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*	*16/01/2016*
FOSA (µg/l)	6,3048	<4,33		9,3684		8,103	<4,33	<4,33	37,0518	<13,0
PFBS (µg/l)	221	93,4								49
PFHS (µg/l)	136	228	863	57,7	49,6	514	295	167	114	474
PFOA (µg/l)	563	183	2970	90,7	69,1	424	280	573	378	343
PFOS (µg/l)	1050	537	5470	396	1090	1700	1020	1200	841	3680
PFOSA (µg/l)	19,1	14,6	0,459	27,3	64,1	35,5	1,6	15,6	74,8	14,9

Locatie	**PA109A**	**PA111A**	**PA112**	**L4**	**L31**	**L21**	**L22**	**P114bis**	**P115**	**P116**
Bemonsteringsdatum	*16/01/2016*	*16/01/2016*	*16/01/2016*	*6/04/2016*	*6/04/2016*	*6/04/2016*	*6/04/2016*	*6/04/2016*	*6/04/2016*	*6/04/2016*
FOSA (µg/l)			25,53					0,229		
PFBS (µg/l)										
PFHS (µg/l)	84,4	997	21,1	0,471	0,764	0,152	0,164	1,34	2,34	0,777
PFOA (µg/l)	81,2	730	84,4	1,05	0,563	0,428	0,309	3,37	4,73	1,73
PFOS (µg/l)	229	1960	328	24,5	4,32	8,37	6	13,3	1,07	8,88
PFOSA (µg/l)	2,08	9,86	49,8	5,98	<0,0500	0,194	0,0753	0,0829	<0,0500	0,0523

Locatie	**P21B**	**P263**	**P321**	**B3-bis**	**BD24-3**	**BD24-4**	**D10**	**D11**	**D14**	**D16**
Bemonsteringsdatum	*7/04/2016*	*7/04/2016*	*6/04/2016*	*29/07/2016*	*25/07/2016*	*25/07/2016*	*25/07/2016*	*20/07/2016*	*27/07/2016*	*25/07/2016*
FOSA (µg/l)			<31,0					28,0386		
PFBS (µg/l)	3020									
PFHS (µg/l)	3500	591	937	1,64	59,2	4,595	142,5	147	1,43	58,55
PFOA (µg/l)	1870	1910	3010	6,615	77,3	10,35	481,5	295	2,52	262,5
PFOS (µg/l)	41200	11000	4960	185	1250	8,925	36,25	1000	7,22	1640
PFOSA (µg/l)	84,3	10,2	0,392	1,165	4,6	0,836	0,5205	63,35	0,7	77,35

Locatie	**D17**	**D18**	**D2**	**D5**	**D9**	**K3**	**L4**	**M4**	**L19**	**L21**
Bemonsteringsdatum	27/07/2016	28/07/2016	27/07/2016	28/07/2016	27/07/2016	27/07/2016	27/07/2016	27/07/2016	25/07/2016	26/07/2016
FOSA (µg/l)									173,382	
PFBS (µg/l)						1490				
PFHS (µg/l)	117,5	288,5	0,1655	432,5	3250	84200	2,765	167	82,6	0,1925
PFOA (µg/l)	1850	521	0,3635	481,5	123,5	120,5	3,235	572	238	0,4195
PFOS (µg/l)	440,5	18,4	9,735	413,5	1940	5670	30,2	11100	5680	6,17
PFOSA (µg/l)	26,1	2,515	0,495	13,85	29,6	18,55	1,74	254	551	0,32

Locatie	**L22**	**L31**	**ND7**	**P114bis**	**P115**	**P116**	**P118A**	**P118B**	**P118C**	**P119A**
Bemonsteringsdatum	26/07/2016	26/07/2016	27/07/2016	26/07/2016	26/07/2016	27/07/2016	20/07/2016	20/07/2016	20/07/2016	25/07/2016
FOSA (µg/l)										
PFBS (µg/l)										
PFHS (µg/l)	0,168	0,386	6,605	1,52	2,31	0,9915	780	2280	104	37,65
PFOA (µg/l)	0,8365	0,736	170,5	2,68	3,93	2,46	1330	2420	305,5	147,5
PFOS (µg/l)	4,935	9,94	277	15,8	0,532	10,6	5030	10600	2100	69,35
PFOSA (µg/l)	0,34	0,6105	364	0,1935	0,03475	0,353	29,45	47,9	56,8	13,5

Locatie	**P119B**	**P119C**	**P121**	**P21B**	**P262**	**P263**	**P264**	**P27**	**P304**	**P305**
Bemonsteringsdatum	25/07/2016	25/07/2016	25/07/2016	27/07/2016	25/07/2016	25/07/2016	25/07/2016	27/07/2016	27/07/2016	28/07/2016
FOSA (µg/l)					39,1941					
PFBS (µg/l)				6200				3270	58,7	133,5
PFHS (µg/l)	1080	286	0,132	10100	4,25	524	176	56,8	33,4	286
PFOA (µg/l)	2230	541,5	0,3225	10900	14,65	1690	300	200,5	150,5	217,5
PFOS (µg/l)	431	3890	2,52	61300	267,5	10100	1540	814,5	357,5	550
PFOSA (µg/l)	10,8	87,25	0,196	6,08	139	17	96,75	271,5	17,6	21,45

Locatie	**P321**	**P340**	**P341**	**P343**	**P371**	**P372**	**P374**	**P378**	**P379**	**P380**
Bemonsteringsdatum	25/07/2016	26/07/2016	25/07/2016	25/07/2016	20/07/2016	20/07/2016	20/07/2016	29/07/2016	25/07/2016	20/07/2016
FOSA (µg/l)		31,968	26,9841			23,8428			30,1032	52,4808
PFBS (µg/l)										
PFHS (µg/l)	374	303	313	754	68,45	45,45	562	426	19,95	48
PFOA (µg/l)	1190	683	544	1020	233	70,4	462,5	370	74,9	81,95
PFOS (µg/l)	4340	3490	5730	7560	3830	1220	4870	1710	648,5	904
PFOSA (µg/l)	18,5	74,5	114,5	21,4	84,3	60,35	22,35	4,94	78,65	116

Locatie	**P381**	**P382**	**P42**	**P56**	**PA109A**	**PA111A**	**PA112**	**P18**	**P28**
Bemonsteringsdatum	20/07/2016	20/07/2016	27/07/2016	27/07/2016	29/07/2016	29/07/2016	29/07/2016	18/07/16	18/07/16
FOSA (µg/l)				3,1746					
PFBS (µg/l)			48,7	10,25					
PFHS (µg/l)	160	333	436	7,315	39,85	1090	33,9		
PFOA (µg/l)	604,5	1090	306	60,4	40,15	852	128,5		
PFOS (µg/l)	1360	1070	4130	42,55	130,5	2330	416,5		
PFOSA (µg/l)	17,25	86,75	23,45	9,865	2,205	12,25	84,1		
minerale olie C10-C12 (µg/l)								<25	1100
minerale olie C12-C20 (µg/l)								140	8000
minerale olie C20-C30 (µg/l)								79	2000
minerale olie C30-C40 (µg/l)								<25	510
minerale olie C10-C40 (µg/l)								250	12000

Bijlage 5

Statistische evaluatie analyseresultaten Blokkersdijk

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From	ERM: Stephen Geiger; Nanda Hermes
Ref/Project number	M03-0341743-v1
Subject	Re-evaluation of the statistical trend analysis of the ground- and surface water concentrations collected in the Blokkersdijk nature reserve
Date	3-January-2017

Introduction

This memo presents an update to the statistical trend analysis of the perfluorooctane sulfonate (PFOS) concentrations in the Blokkerdijk nature reserve using data that are supplemental to the 31-March -2016 memo which presented an alternate 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 Blokkerdijk 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 18-March-2016 memo (ref. M01-0341743-v1, dd. 18/-03-16) 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 three quarters of data, and analyzed by the 3M lab.

The results of the revised evaluation will be included in the seventh annual interim report (“*Eerste gefaseerd bodemsaneringsproject - Zevende tussentijds verslag bodemsaneringswerken,*

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(1)“Eerste gefaseerd bodemsaneringsproject 3M Belgium NV, haven 1005 – Canadastraat 11, 2070 Zwijndrecht”, opgesteld door Arcadis op oktober 2008

Periode juli 2015 - juli 2016", dd. 25 augustus 2017) that will submitted to the Flemish environmental agency OVAM.

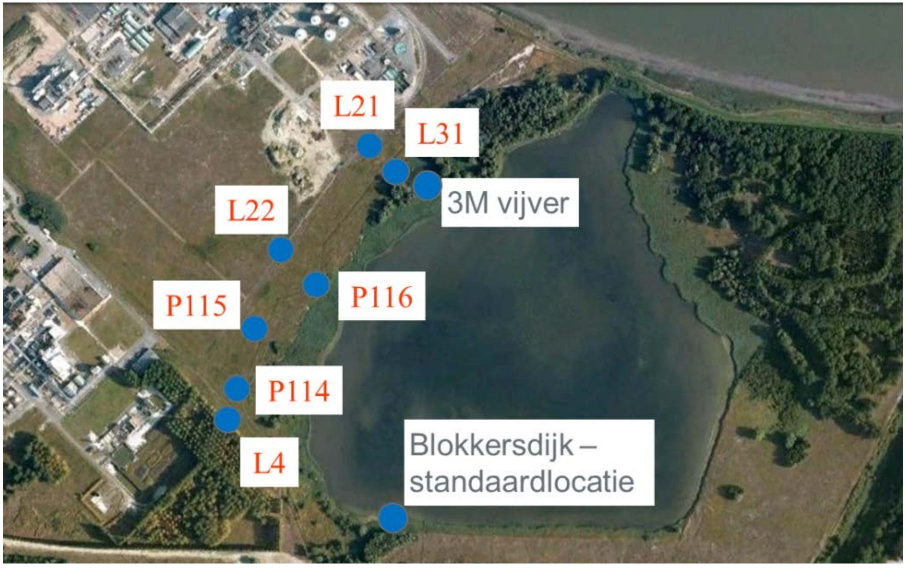
Background

The Blokkerdijk 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 Blokkerdijk 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, P114, P115, P116, and the 3M vijver and Blokkersdijk vijver – standardlocatie samples.

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 October 2016.

The trend analysis was conducted as follows:

1. Data distribution was determined using the Shapiro-Wilks and Lillifors tests for the entire data set and only the 3M lab data for each location;
2. Q-Q plots were developed for each location using the entire data set and the 3M data set, to evaluate if data outliers were present in the data;
3. Time series figures were created for each location showing the pre-3M and the 3M PFOS concentrations over time; and
4. Trend analyses were conducted using ordinary least-square (OLS) regression on both 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.

Table 1 (included in 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.

All statistical analyses were conducted using the U.S. EPA statistical software program ProUCL version 5.0.00.

Results

Figure 1 (included as Annex 2) shows time series graphs of PFOS concentrations for samples collected from each of the sampling locations. The results of the trend analyses are presented in Table 2 (included as Annex 3). The data was variously normally distributed, log-normally distributed, or distribution free (non-parametric). The trend analysis results show that well L21 has a significantly increasing trend in PFOS, and that well L21 has a significantly decreasing trend in PFOS. None of the other wells showed any significant trend.

Well L21

Figure 1 (see Annex 2) shows well L21 with an apparent upward trend in the 3M lab data. The Q-Q plot for the 3M lab data set (Figure 3) does not show any potential outliers. The data set is normally distributed, and the OLS regression for this data showed a significant upward trend in PFOS (Table 2; see Annex 3). However, note that PFOS concentrations in the last two quarters of data collection are decreasing relative to the April 2016 sample.

Well L22

Figure 1 (see Annex 2) shows well L22 with no apparent upward trend in the 3M lab data set. However, two intermediate values are substantially higher than the norm for this well. The Q-Q plots for the 3M lab data set show that these two values might be from a different population (i.e., are potential outliers), but evidence of outliers are not apparent when the data are log-transformed (Figures 2 and 3; see Annex 4). The 3M data set is log-normally distributed, and the OLS regression for the log-transformed data showed no significant trend in PFOS; however, the non-parametric Mann-Kendall test indicated a significant downward trend in PFOS concentration (Table 2; see Annex 3).

Well L31

Figure 1 (see Annex 2) shows well L31 with no apparent upward trend in the 3M lab data set, and the Q-Q plots did not indicate any data outliers. The 3M lab data was normally distributed and did not show any significant trend (Table 2; see Annex 3).

Well L4

Figure 1 (see Annex 2) shows well L4 with no apparent in the 3M lab data set. The data set was normally distributed and the Q-Q plot indicated no outliers. The OLS regression showed no significant trend in PFOS (Table 2; see Annex 3).

Well P114

Figure 1 (see Annex 2) shows well P114 with no apparent trend in the 3M lab data. The Q-Q plots did not indicate any potential outliers. The data were non-parametric, and Mann-Kendall regression did not show any significant trend (Table 2; see Annex 3).

Well P115

Figure 1 (see Annex 2) shows well P115 with no apparent trend in the 3M lab data set, although there was much variability in PFOS concentrations. The Q-Q plots did not indicate outliers. The data was normally distributed, and there was no significant trend in PFOS (Table 2; see Annex 3).

Well P116

Figure 1 (see Annex 2) shows well P116 with no apparent trend in the 3M lab data sets. Although there appears to be a data outlier in October 2015, the Q-Q plot (log-transformed data) does not indicate this (Figure 2; see Annex 4). The data set is non-parametric, and the Mann-Kendall trend showed no significant trend in PFOS (Table 2; see Annex 3).

3M vijver

Figure 1 (see Annex 2) shows the 3M vijver PFOS concentrations with no apparent trend in the 3M lab data set, although there was some variability in PFOS concentrations. The Q-Q plots (log-transformed) do not indicate data outliers. The data is non-parametric, and the Mann-Kendall test indicates no significant trend in PFOS (Table 2; see Annex 3).

Blokkersdijk vijver standard

Figure 1 (see Annex 2) shows the Blokkersdijk vijver standard_PFOS concentrations with a slightly upward trend in the 3M lab data set. However, the last two sampling periods showed a decrease in PFOS concentration in this well. The Q-Q plot (log-transformed) did not indicate data outliers (Figure 2; see Annex 4). The data set was normally distributed, and the OLS regression showed no significant trend in PFOS (Table 2; 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. One of the locations showed a statistically significant upward trend (L21) and one location showed a statistically significant downward trend (L22). None of the other wells evaluated showed a statistically significant trend in PFOS.

■

Annexes:

Annex 1 - Table 1:	Data used in the trend analysis
Annex 2 - Figure 1:	Time series graphs of PFOS
Annex 3 - Table 2:	Results of the trend analyses
Annex 4 - Figure 2:	Q-Q plots

Annex 1

Table 1: Data used in the trend analysis

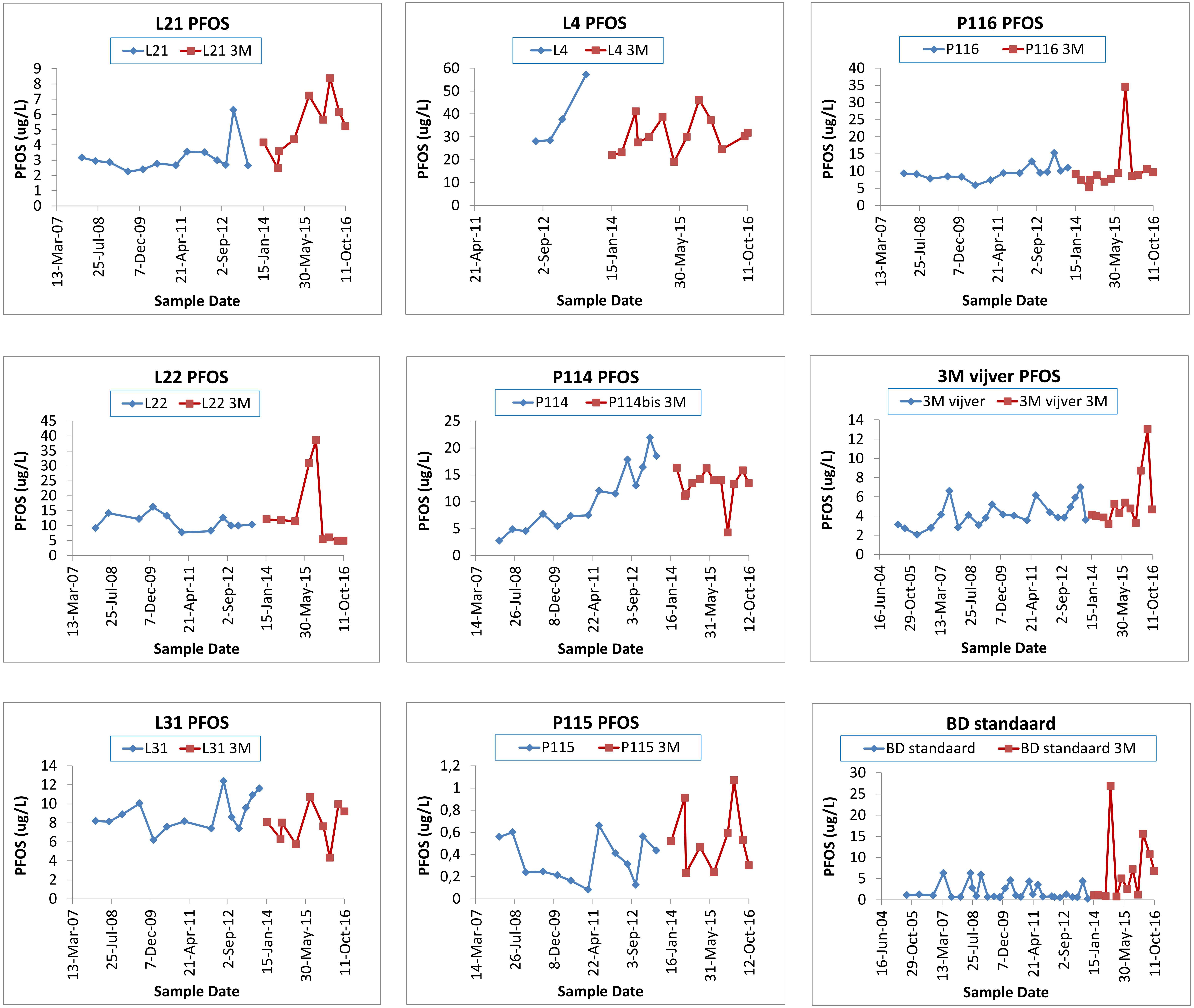
Table 1. PFOS Data from Selected Site Wells
Highlighted dates (red) are analyses conducted by 3M
P114 highlighted wells are labelled P114bis

L21		**L22**		L31		L4		**P114**		**P115**		**P116**		**3M vijver**		**BD standaard**	
Date	**PFOS (ug/L)**	**Date**	**PFOS (ug/L)**	**Date**	**PFOS (ug/L)**	**Date**	**PFOS (ug/L)**	**Date**	**PFOS (ug/L)**	**Date**	**PFOS (ug/L)**	**Date**	**PFOS (ug/L)**	**Date**	**PFOS (ug/L)**	**Date**	**PFOS (ug/L)**
10-Jan-08	3,16	10-Jan-08	9,2	10-Jan-08	8,2	12-Jul-12	28	10-Jan-08	2,75	10-Jan-08	0,56	10-Jan-08	9,28	26-Apr-05	3,1	10-Aug-05	1,1
26-Jun-08	2,95	26-Jun-08	14,2	26-Jun-08	8,13	24-Oct-12	28,5	26-Jun-08	4,84	26-Jun-08	0,6	26-Jun-08	9,12	10-Aug-05	2,7	1-Mar-06	1,26
15-Dec-08	2,86	24-Jul-09	12,2	15-Dec-08	8,88	22-Jan-13	37,6	15-Dec-08	4,54	15-Dec-08	0,24	15-Dec-08	7,8	1-Mar-06	2,05	18-Oct-06	1,07
23-Jul-09	2,25	20-Jan-10	16,2	24-Jul-09	10,03	15-Jul-13	57,1	24-Jul-09	7,71	24-Jul-09	0,246	24-Jul-09	8,41	18-Oct-06	2,76	5-Apr-07	6,33
24-Jul-09	2,25	14-Jul-10	13,3	20-Jan-10	6,21	21-jan-14	21,9	20-Jan-10	5,45	20-Jan-10	0,213	20-Jan-10	8,35	5-Apr-07	4,13	20-Aug-07	0,62
20-Jan-10	2,39	26-Jan-11	7,73	14-Jul-10	7,56	02-apr-14	23,2	14-Jul-10	7,33	14-Jul-10	0,164	14-Jul-10	5,86	20-Aug-07	6,62	10-Jan-08	0,67
14-Jul-10	2,77	6-Feb-12	8,22	22-Feb-11	8,14	14-jul-14	41,04	22-Feb-11	7,5	22-Feb-11	0,083	26-Jan-11	7,38	10-Jan-08	2,81	26-Jun-08	6,23
22-Feb-11	2,66	9-Jul-12	12,7	6-Feb-12	7,39	30-jul-14	27,5	12-Jul-11	12	12-Jul-11	0,661	12-Jul-11	9,42	26-Jun-08	4,1	28-Jul-08	2,83
12-Jul-11	3,56	25-Oct-12	10	9-Jul-12	12,4	20-okt-14	29,9	6-Feb-12	11,5	6-Feb-12	0,409	6-Feb-12	9,39	15-Dec-08	3,05	2-Oct-08	0,82
6-Feb-12	3,51	23-Jan-13	10	23-Oct-12	8,6	27-jan-15	38,6	9-Jul-12	17,8	9-Jul-12	0,313	9-Jul-12	12,8	7-Apr-09	3,82	15-Dec-08	5,9
9-Jul-12	3	19-Jul-13	10,3	23-Jan-13	7,39	21-apr-15	19	24-Oct-12	13	24-Oct-12	0,126	24-Oct-12	9,48	24-Jul-09	5,21	7-Apr-09	0,67
23-Oct-12	2,69	21-Jan-14	12,1	24-Apr-13	9,57	23-jul-15	30	25-Jan-13	16,4	25-Jan-13	0,564	23-Jan-13	9,73	20-Jan-10	4,14	23-Jul-09	0,799
23-Jan-13	6,31	30-Jul-14	11,9	19-Jul-13	10,9	21-okt-15	46,2	24-Apr-13	21,9	15-Jul-13	0,436	24-Apr-13	15,3	14-Jul-10	4,04	14-Oct-09	0,62
19-Jul-13	2,64	27-Jan-15	11,4	14-Oct-13	11,6	16-jan-16	37,3	15-Jul-13	18,5	21-jan-14	0,518	15-Jul-13	10,1	18-Feb-11	3,55	27-Oct-09	0,62
21-Jan-14	4,16	23-Jul-15	30,9	21-jan-14	8,07	06-apr-16	24,5	02-apr-14	16,3	14-jul-14	0,911	14-Oct-13	11	12-Jul-11	6,16	20-Jan-10	2,65
30-Jul-14	3,58	21-Oct-15	38,6	14-jul-14	6,3	19-sep-16	30,2	14-jul-14	11,05	30-jul-14	<0.464	21-jan-14	9,17	28-Feb-12	4,38	15-Apr-10	4,6
27-Jan-15	4,36	16-jan-16	5,39	30-jul-14	8,03	11-okt-16	31,8	30-jul-14	11,5	27-jan-15	0,467	02-apr-14	7,46	10-Jul-12	3,84	14-Jul-10	1,08
28-Jul-15	7,23	06-apr-16	6	27-jan-15	5,72			20-okt-14	13,4	23-jul-15	0,238	14-jul-14	5,25	23-Oct-12	3,83	7-Oct-10	0,65
16-jan-16	5,65	26-jul-16	4,935	28-jul-15	10,7			27-jan-15	14,2	16-jan-16	0,594	30-jul-14	7,42	31-Jan-13	4,93	18-Feb-11	4,34
06-apr-16	8,37	11-okt-16	4,94	16-jan-16	7,61			21-apr-15	16,2	06-apr-16	1,07	20-okt-14	8,77	25-Apr-13	5,91	19-Apr-11	1,3
26-jul-16	6,17			06-apr-16	4,32			23-jul-15	14	26-jul-16	0,532	27-jan-15	6,87	19-Jul-13	6,97	12-Jul-11	3,51
11-okt-16	5,22			26-jul-16	9,94			21-okt-15	14	12-okt-16	0,304	21-apr-15	7,71	14-Oct-13	3,59	4-Oct-11	0,74
				11-okt-16	9,2			16-jan-16	4,29			23-jul-15	9,43	23-jan-14	4,13	28-Feb-12	0,813
								06-apr-16	13,3			21-okt-15	34,6	02-apr-14	4	10-Apr-12	0,652
								26-jul-16	15,8			16-jan-16	8,51	31-jul-14	3,85	10-Jul-12	0,516
								12-okt-16	13,4			06-apr-16	8,88	21-okt-14	3,18	23-Oct-12	1,27
												27-jul-16	10,6	27-jan-15	5,27	31-Jan-13	0,617
												11-okt-16	9,66	21-apr-15	4,3	25-Apr-13	0,533
														27-jul-15	5,39	19-Jul-13	4,35
														20-okt-15	4,78	14-Oct-13	0,198
														16-jan-16	3,29	23-jan-14	1,06
														07-apr-16	8,73	02-apr-14	1,17
														28-jul-16	13,05	31-jul-14	0,86
														11-okt-16	4,68	21-okt-14	26,9
																27-jan-15	0,792
																21-apr-15	5,01
																27-jul-15	2,54
																20-okt-15	7,18
																16-jan-16	1,23
																07-apr-16	15,6
																28-jul-16	10,75
																11-okt-16	6,84

Annex 2

Figure 1: Time series graphs of PFOS

Figure 1. Time series plots of PFOS data with 3M-analyzed data demarcated.



Annex 3

Table 2: Results of the trend analyses

Table 2. PFOS Trend Analysis p-values for the 3M-analyzed data.

Well	**L21**	**L22**	**L31**	**L4**	**P114**	**P115**	**P116**	**3M vijver**	**Blokkersdijkvijver standaard**
3M PFOS data									
Data Distribution	N	LN	N	N	NP	N	NP	NP	N
OLS trend	0,0431	0,6092	0,6027	0,9192	0,7681	0,925	0,92	0,0983	0,4737
Ln-OLS trend	0,0346	0,2448	0,7405	0,9469	0,6902	0,92	0,2164	0,0969	0,5963
Mann-Kendall Trend	0,0382	0,0382	0,4585	0,1232	0,4725	0,4585	0,4585	0,1518	0,3156

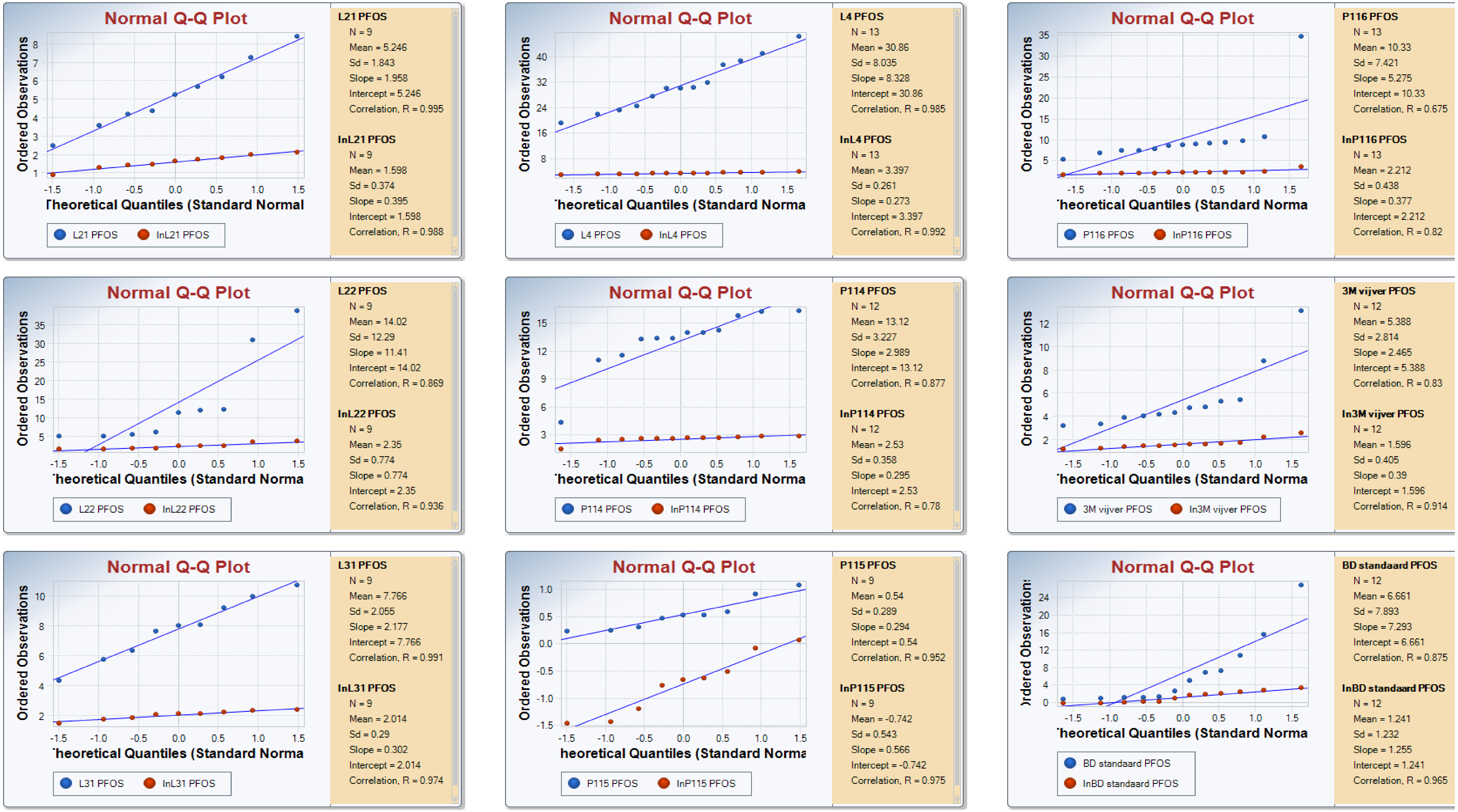
N = normal
LN = lognormal
NP = non-parametric

🟨 indicates significant trend

Annex 4

Figure 2: Q-Q plots

Figure 2. Q-Q plots for the 3M data sets.



L21 PFOS
N = 9
Mean = 5.246
Sd = 1.843
Slope = 1.958
Intercept = 5.246
Correlation, R = 0.995

lnL21 PFOS
N = 9
Mean = 1.598
Sd = 0.374
Slope = 0.395
Intercept = 1.598
Correlation, R = 0.988

L4 PFOS
N = 13
Mean = 30.86
Sd = 8.035
Slope = 8.328
Intercept = 30.86
Correlation, R = 0.985

lnL4 PFOS
N = 13
Mean = 3.397
Sd = 0.261
Slope = 0.273
Intercept = 3.397
Correlation, R = 0.992

P116 PFOS
N = 13
Mean = 10.33
Sd = 7.421
Slope = 5.275
Intercept = 10.33
Correlation, R = 0.675

lnP116 PFOS
N = 13
Mean = 2.212
Sd = 0.438
Slope = 0.377
Intercept = 2.212
Correlation, R = 0.82

L22 PFOS
N = 9
Mean = 14.02
Sd = 12.29
Slope = 11.41
Intercept = 14.02
Correlation, R = 0.869

lnL22 PFOS
N = 9
Mean = 2.35
Sd = 0.774
Slope = 0.774
Intercept = 2.35
Correlation, R = 0.936

P114 PFOS
N = 12
Mean = 13.12
Sd = 3.227
Slope = 2.989
Intercept = 13.12
Correlation, R = 0.877

lnP114 PFOS
N = 12
Mean = 2.53
Sd = 0.358
Slope = 0.295
Intercept = 2.53
Correlation, R = 0.78

3M vijver PFOS
N = 12
Mean = 5.388
Sd = 2.814
Slope = 2.465
Intercept = 5.388
Correlation, R = 0.83

ln3M vijver PFOS
N = 12
Mean = 1.596
Sd = 0.405
Slope = 0.39
Intercept = 1.596
Correlation, R = 0.914

L31 PFOS
N = 9
Mean = 7.766
Sd = 2.055
Slope = 2.177
Intercept = 7.766
Correlation, R = 0.991

lnL31 PFOS
N = 9
Mean = 2.014
Sd = 0.29
Slope = 0.302
Intercept = 2.014
Correlation, R = 0.974

P115 PFOS
N = 9
Mean = 0.54
Sd = 0.289
Slope = 0.294
Intercept = 0.54
Correlation, R = 0.952

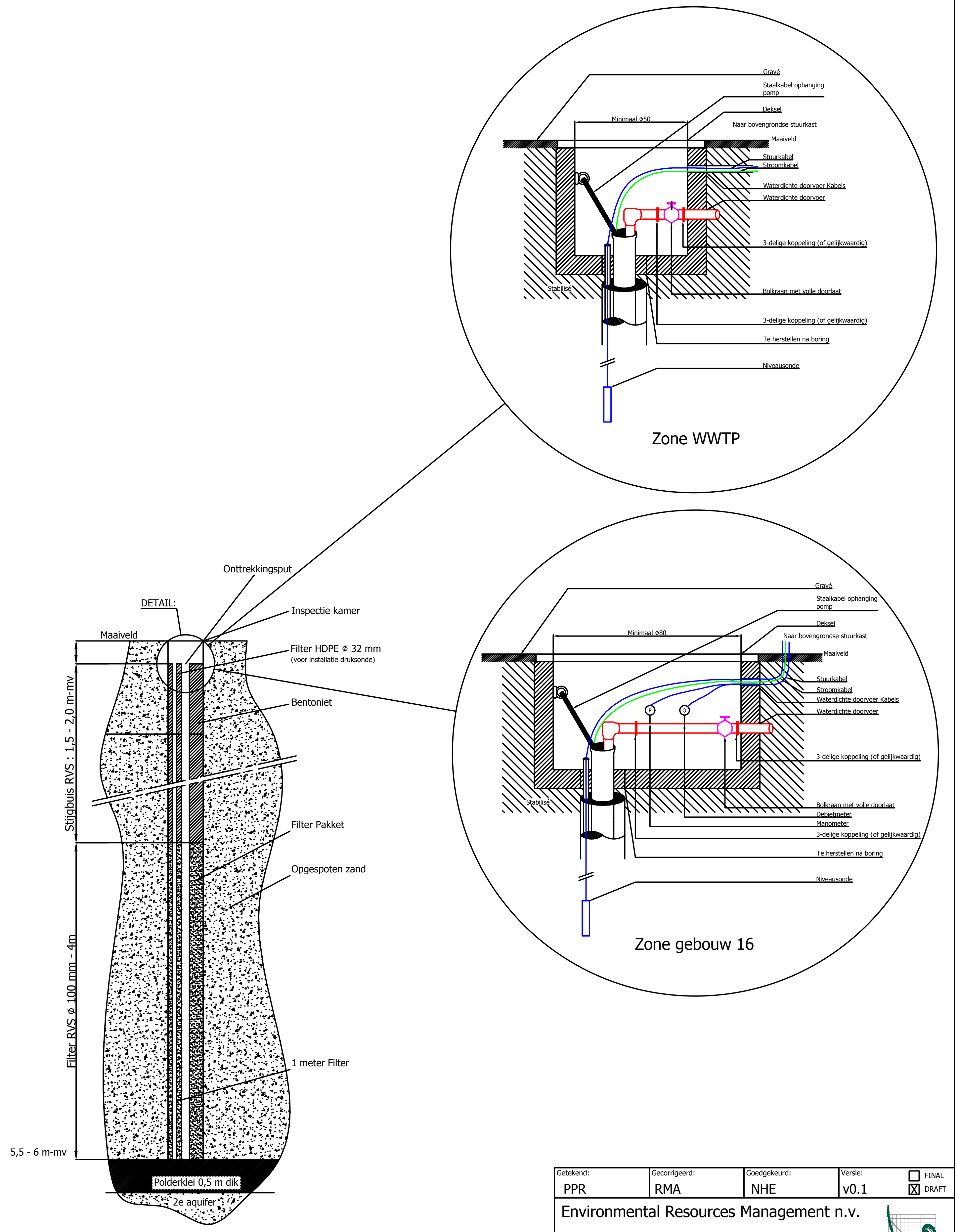
lnP115 PFOS
N = 9
Mean = -0.742
Sd = 0.543
Slope = 0.566
Intercept = -0.742
Correlation, R = 0.975

BD standaard PFOS
N = 12
Mean = 6.661
Sd = 7.893
Slope = 7.293
Intercept = 6.661
Correlation, R = 0.875

lnBD standaard PFOS
N = 12
Mean = 1.241
Sd = 1.232
Slope = 1.255
Intercept = 1.241
Correlation, R = 0.965

Bijlage 6

Schematische weergave pompput en inspectiekamer



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Goedgekeurd: NHE
Versie: v0.1
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Fax.: 010/48.35.36

ERM

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-Doorsnedes.dwg
Datum: 22/12/2016

DEEL 4: KAARTMATERIAAL

Eerste gefaseerd bodemsaneringsproject -
Zevende tussentijds verslag bodemsaneringswerken
Periode juli 2015 - juli 2016

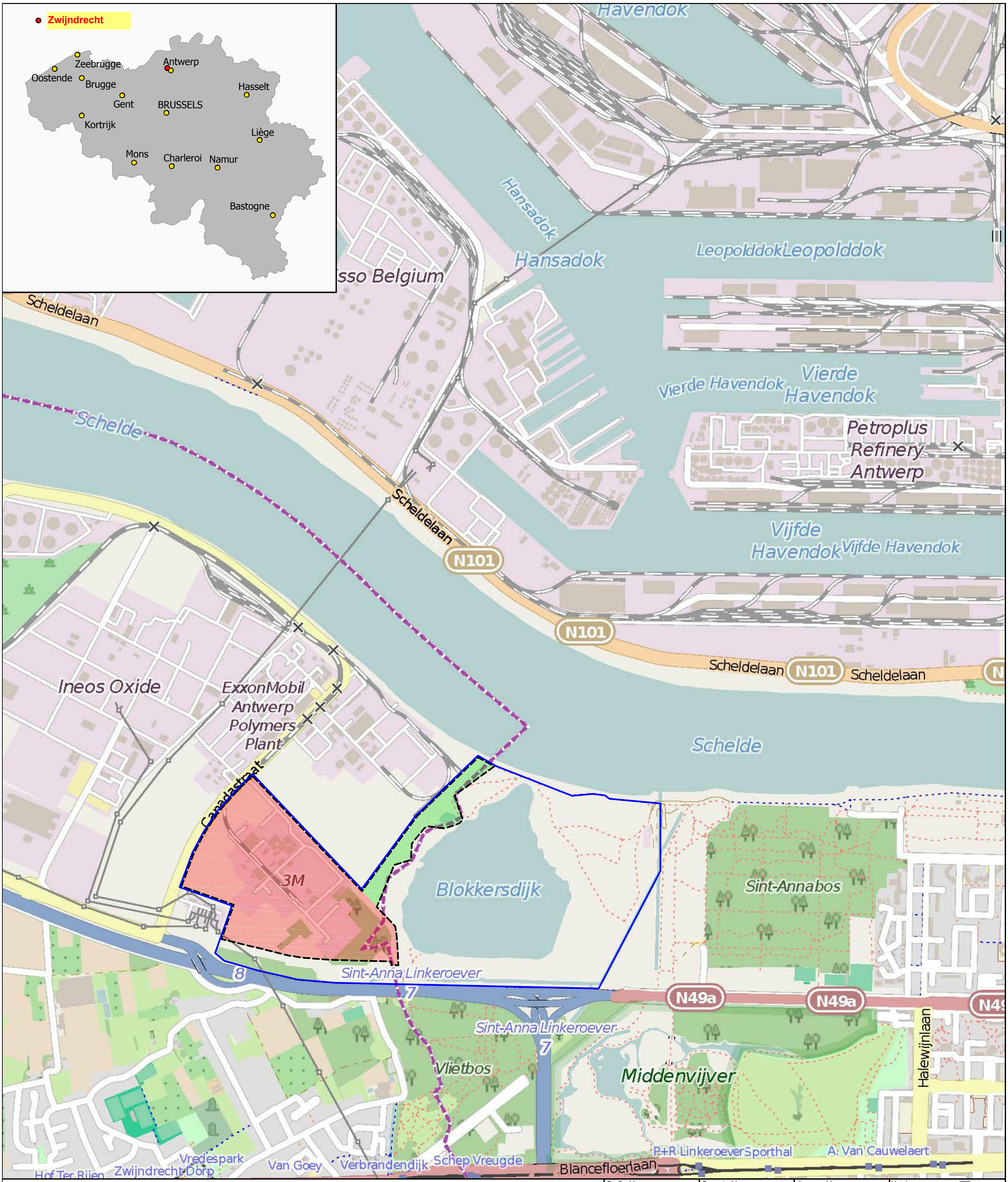
3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

25 augustus 2017

www.erm.com

Kaart 1

Situering onderzoekslocatie op topografische kaart



Legende:

- ━━ Onderzoekslocatie
- ━ ━ 3M terreingrens
- 🟥 3M Productiesite
- 🟩 3M Pad

0 250.0 500.0 750.0 1000.0 1250.0 (m)

N

Drafted by: PPR	Corrected by: RMA	Approved by: NHE	Version: v0.1	☐ FINAL ☒ DRAFT

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ERM

Client / Project Name: 3M Belgium bvba	Location: Zwijndrecht
Project Number: 0341743	Title: 7de Tussentijds Verslag Bodemsaneringswerken
Kaart: 2	Description: Topografische situering © OpenStreetMap-contributors
Phase: TTV-7	

Scale: 1:25 000	Paper Size: A4	File Name: 3M Zwijndrecht_Topo.dwg	Date: 22/12/2016

Kaart 2

Overzichtsplan onderzoekslocatie

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Fax.: 010/48.35.36

ERM

Klant / Projectnaam: 3M Belgium bvba

Locatie: Zwijndrecht

Projectnr.: 0341743

Titel: 7de Tussentijds Verslag Bodemsaneringswerken

Kaart: 3

Fase: TTV-7

Beschrijving: Overzicht van de onderzoekslocatie met aanduiding van Peilbuizen

Schaal: 1:3000

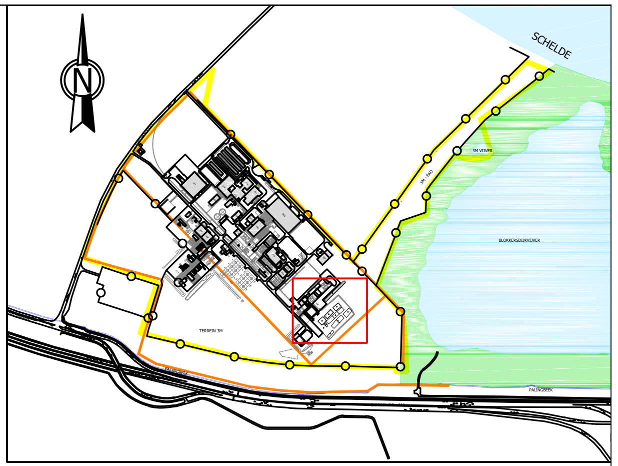
Formaat: A1

Bestandsnaam: 3M Zwijndrecht.dwg

Datum: 22/12/2016

Kaart 3

Detailplan ligging grondhopen



Legende:

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-·-·-	Kadastrale grens
267v2	Kadastrale nummer
029	Gebouw
③	Nummer grondhoop
═	Ondergrondse dijk
─o─	Hek
▦	Stelcon plaat

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
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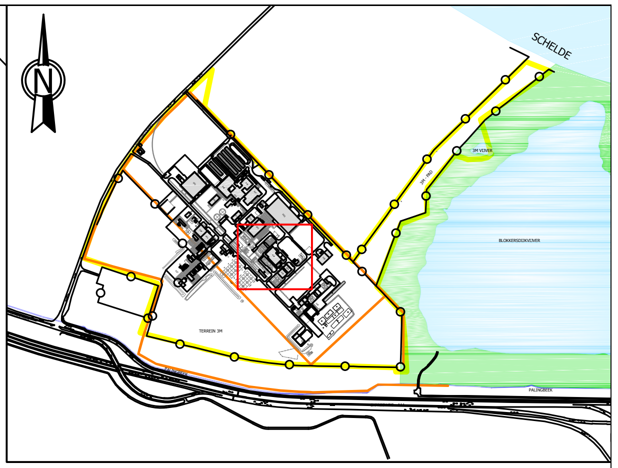
ERM

Klant / Projectnaam: 3M Belgium bvba
Locatie: Zwijndrecht
Projectnr.: 0341743
Titel: 7de Tussentijds Verslag Bodemsaneringswerken
Kaart: 4
Beschrijving: Overzicht Grondhopen
Fase: TTV-7
Schaal: 1:750
Formaat: A3
Bestandsnaam: 3M Zwijndrecht.dwg
Datum: 22/12/2016

Kaart 4

Detailplannen monsternamelocaties

A. Monsternamelocaties Gebouw 16
B. Monsternamelocaties Gebouw WWTP
C. Monsternamelocaties 2de aquifer
D. Monsternamelocaties Zuidelijke grens
E. Monsternamelocaties Blokkersdijk
F. Monsternamelocaties Paling- en Tophatbeek
G. Monsternamelocaties Collector put en lozingspunt WWTP



Legende:

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267v2	Kadastrale nummer
029	Gebouw
③	Nummer grondhoop
🟧	Ondergrondse dijk
—o—	Hek
▦	Stelcon plaat
●	Peilbuis
✕	Peilbuis verdwenen
⊗	Onttrekkingsput
⊠	Oppervlaktewaterstaalnamepunt
◎	Regenwaterrioleringsput

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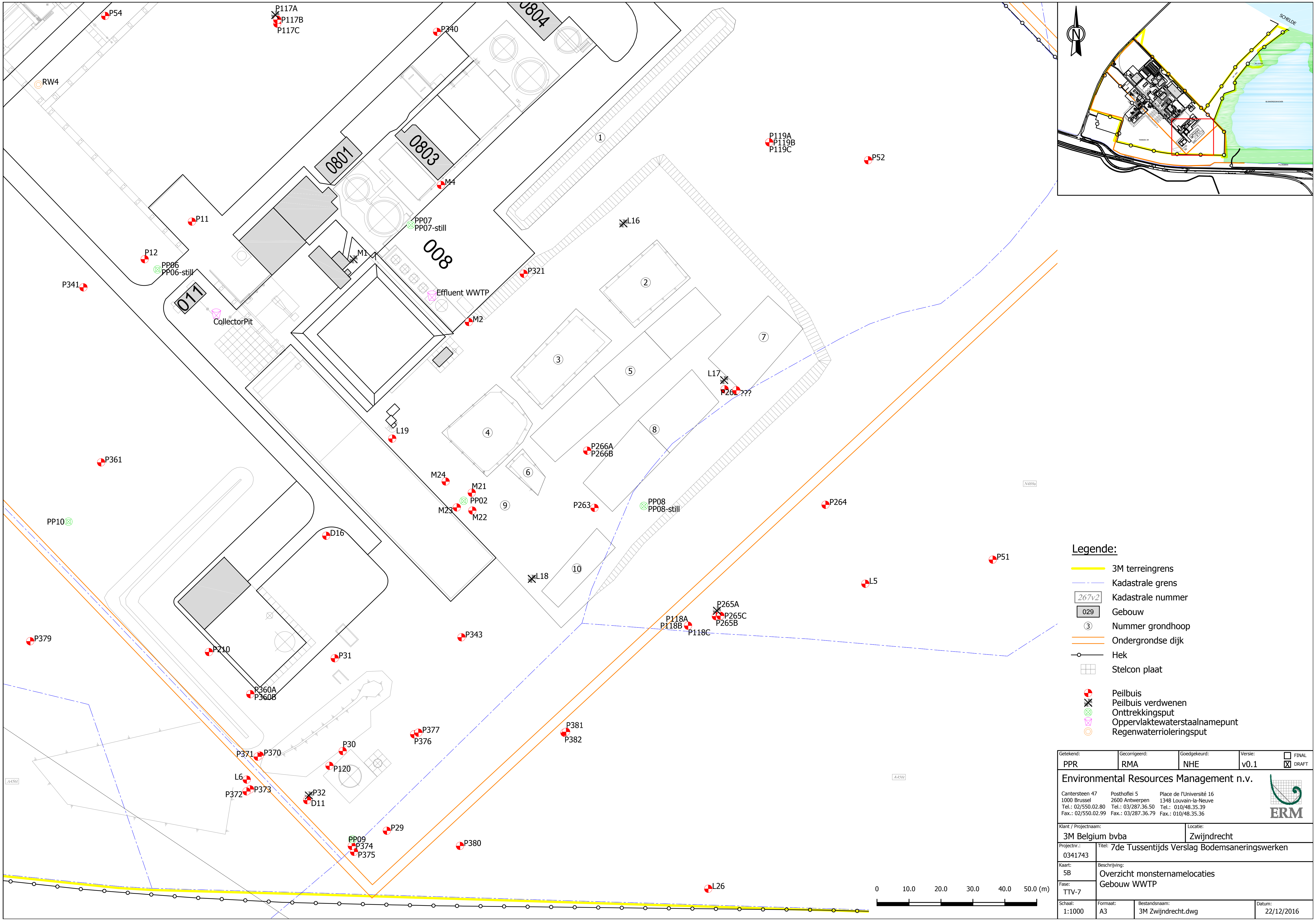
ERM

Klant / Projectnaam: 3M Belgium bvba
Locatie: Zwijndrecht
Projectnr.: 0341743
Titel: 7de Tussentijds Verslag Bodemsaneringswerken
Kaart: 5A
Fase: TTV-7
Beschrijving: Overzicht monsternamelocaties
Bronzone Gebouw 16 en zone ondergrondse tanken
Schaal: 1:750
Formaat: A3
Bestandsnaam: 3M Zwijndrecht.dwg
Datum: 22/12/2016



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1348 Louvain-la-Neuve
Tel.: 010/48.35.39
Fax.: 010/48.35.36

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Klant / Projectnaam: 3M Belgium bvba	Locatie: Zwijndrecht
Projectnr.: 0341743	Titel: 7de Tussentijds Verslag Bodemsaneringswerken
Kaart: 5B	Beschrijving: Overzicht monsternamelocaties Gebouw WWTP
Fase: TTV-7	

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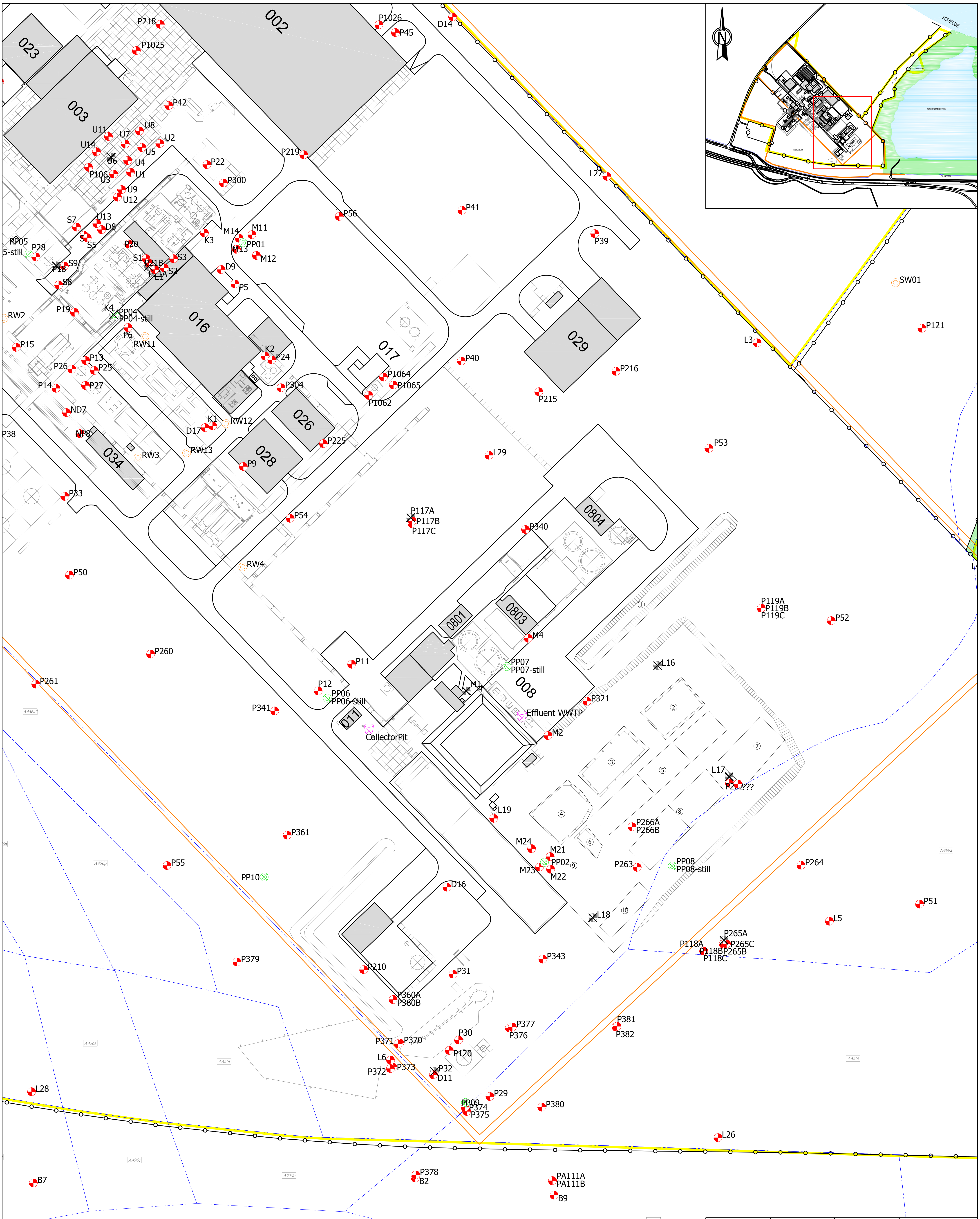

0 15.0 30.0 45.0 60.0 75.0 (m)

Legende:

🟨	3M terreingrens
— · —	Kadastrale grens
267v2	Kadastrale nummer
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—o—	Hek
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◐	Peilbuis
⊗	Peilbuis verdwenen
⊗	Onttrekkingsput
▽	Oppervlaktewaterstaalnamepunt
◎	Regenwaterrioleringsput

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:
PPR	RMA	NHE	v0.1 ☐ FINAL ☒ DRAFT

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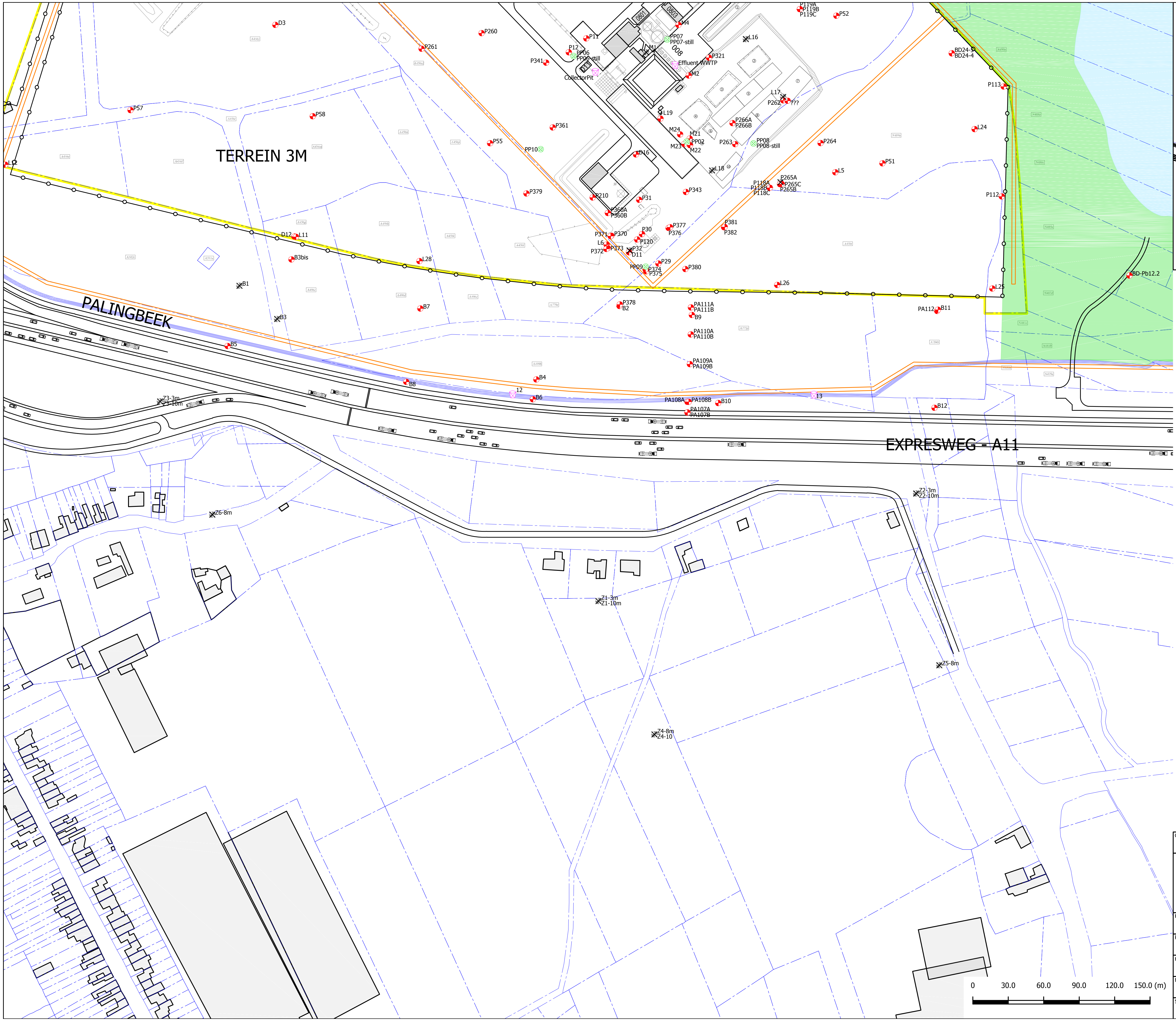
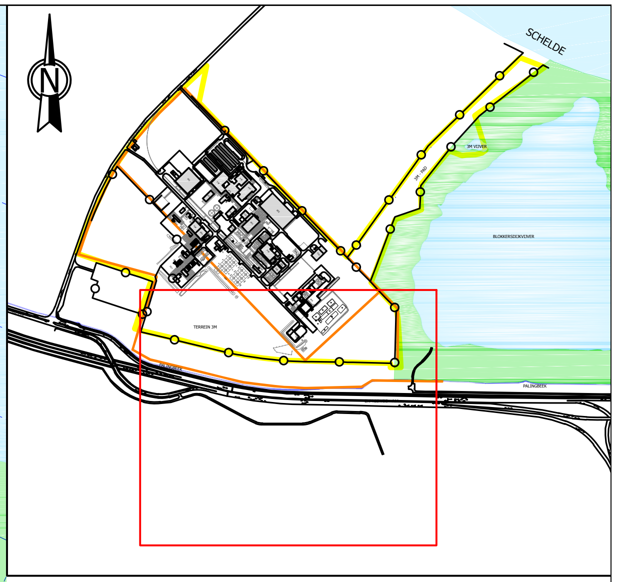
Tel.: 010/48.35.39
Fax.: 010/48.35.36

ERM

Klant / Projectnaam: 3M Belgium bvba
Locatie: Zwijndrecht
Projectnr.: 0341743
Titel: 7de Tussentijds Verslag Bodemsaneringswerken
Kaart: 5C
Fase: TTV-7
Beschrijving: Overzicht monsternamelocaties 2de Aquifer
Schaal: 1:1500
Formaat: A3
Bestandsnaam: 3M Zwijndrecht.dwg
Datum: 22/12/2016





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- · -	Kadastrale grens
267v2	Kadastrale nummer
029	Gebouw
③	Nummer grondhoop
🟧	Ondergrondse dijk
-o-	Hek
▦	Stelcon plaat
●	Peilbuis
✕	Peilbuis verdwenen
⊗	Onttrekkingsput
⊠	Oppervlaktewaterstaalnamepunt
◎	Regenwaterrioleringsput

Getekend: PPR	Gecorrigeerd: RMA	Goedgekeurd: NHE	Versie: v0.1 ☐ FINAL ☒ DRAFT

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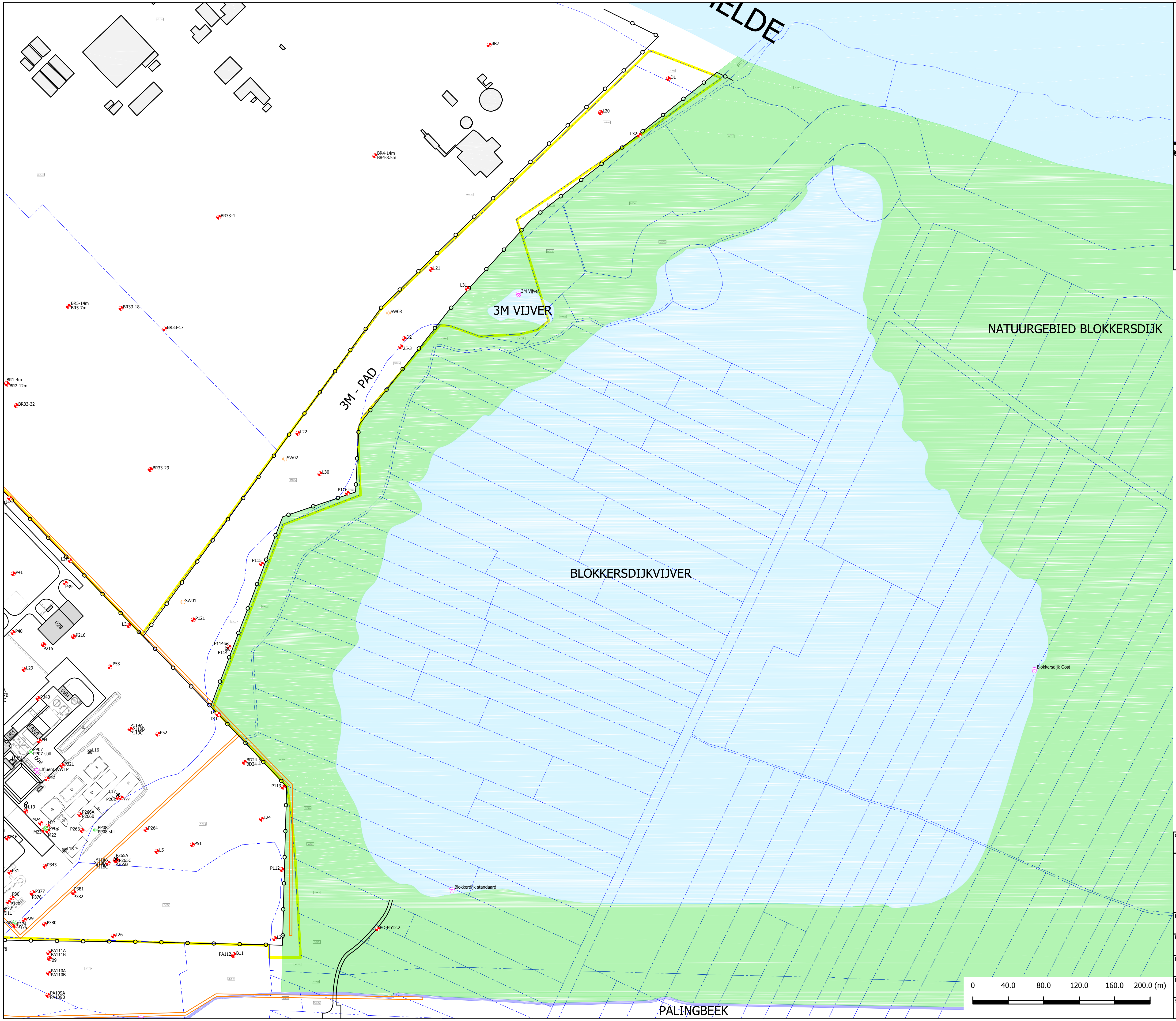
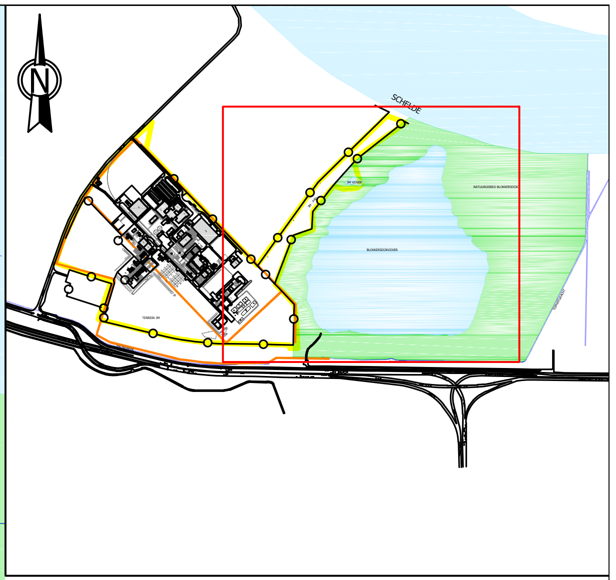
Fax.: 010/48.35.36

ERM

Klant / Projectnaam: 3M Belgium bvba	Locatie: Zwijndrecht
Projectnr.: 0341743	Titel: 7de Tussentijds Verslag Bodemsaneringswerken
Kaart: 5D	Beschrijving: Overzicht monsternamelocaties Zuidelijke grens en Zwijndrecht wells
Fase: TTV-7	

Schaal: 1:3000	Formaat: A3	Bestandsnaam: 3M Zwijndrecht.dwg	Datum: 22/12/2016





Legende:

- 🟨 3M terreingrens
- Kadastrale grens
- 267v2 Kadastrale nummer
- 029 Gebouw
- ③ Nummer grondhoop
- Ondergrondse dijk
- Hek
- Stelcon plaat

- Peilbuis
- Peilbuis verdwenen
- Onttrekkingsput
- Oppervlaktewaterstaalnamepunt
- Regenwaterrioleringsput

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
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ERM

Klant / Projectnaam: 3M Belgium bvba
Locatie: Zwijndrecht
Projectnr.: 0341743
Titel: 7de Tussentijds Verslag Bodemsaneringswerken
Kaart: 5E
Fase: TTV-7
Beschrijving: Overzicht monsternamelocaties Blokkersdijk
Schaal: 3" = 1'-0"_XREF
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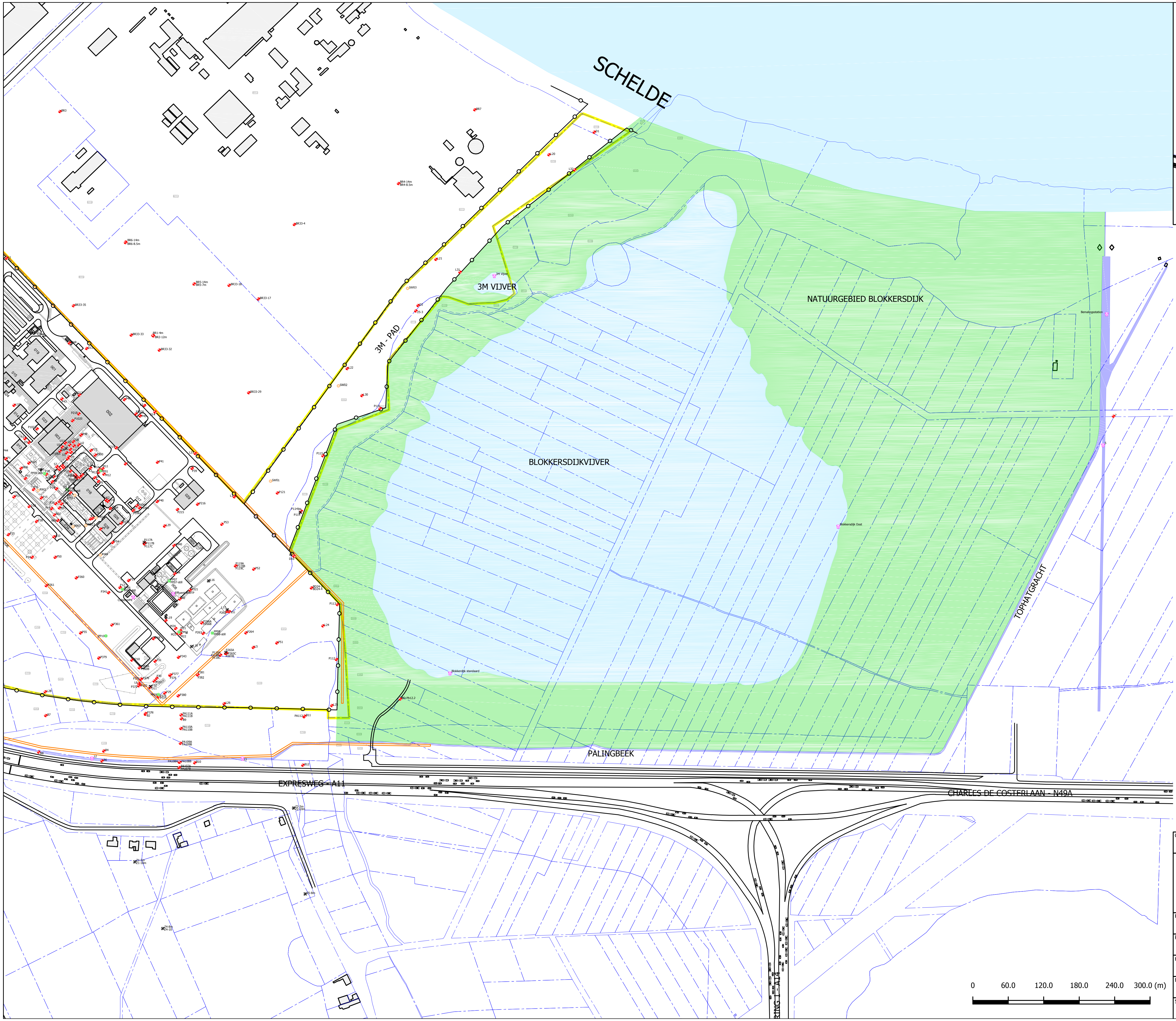
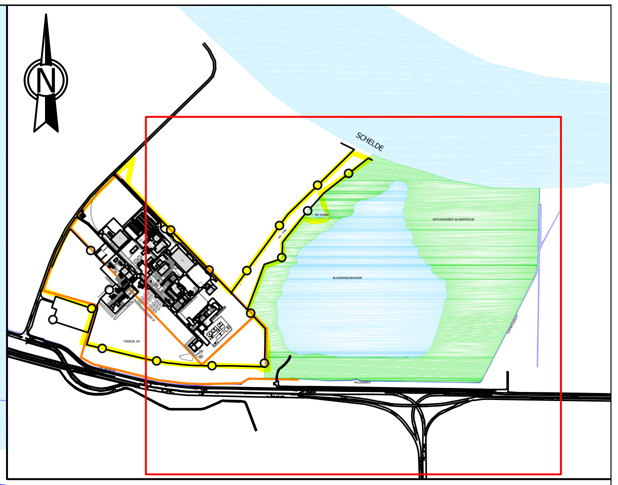
Bestandsnaam: 3M Zwijndrecht.dwg
Datum: 22/12/2016





Legende:

🟨	3M terreingrens
- · -	Kadastrale grens
267v2	Kadastrale nummer
029	Gebouw
③	Nummer grondhoop
🟧	Ondergrondse dijk
—o—	Hek
▦	Stelcon plaat
●	Peilbuis
✕	Peilbuis verdwenen
⊗	Onttrekkingsput
⊠	Oppervlaktewaterstaalnamepunt
◎	Regenwaterrioleringsput

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Gecorrigeerd: RMA
Goedgekeurd: NHE
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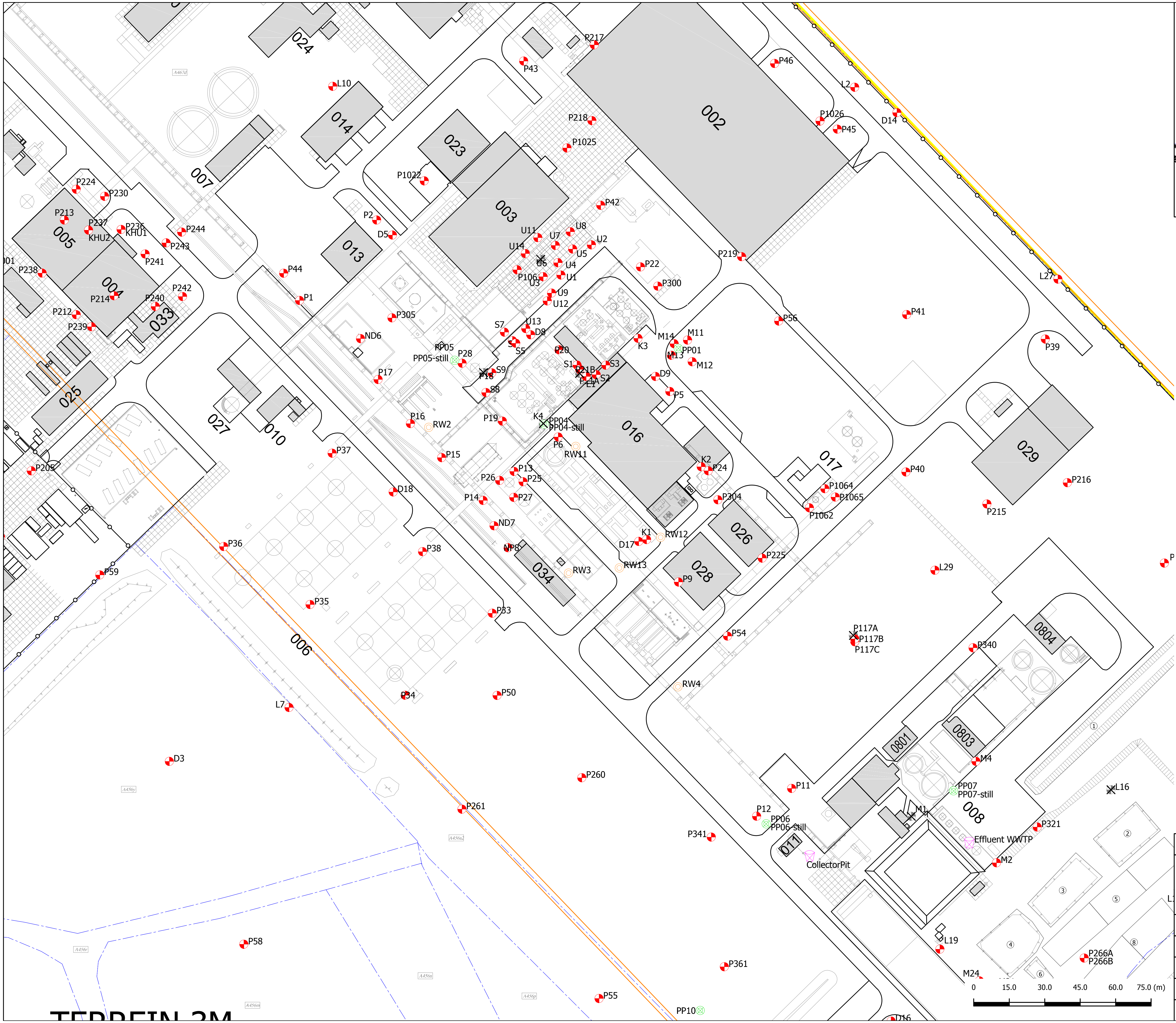
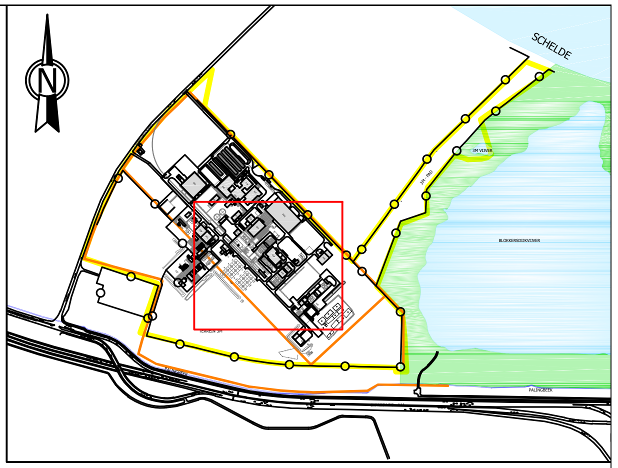
Klant / Projectnaam: 3M Belgium bvba
Locatie: Zwijndrecht
Projectnr.: 0341743
Titel: 7de Tussentijds Verslag Bodemsaneringswerken
Kaart: 5F
Fase: TTV-7
Beschrijving: Overzicht monsternamelocaties Palingbeek
Schaal: 1:6000
Formaat: A3
Bestandsnaam: 3M Zwijndrecht.dwg
Datum: 22/12/2016





Legende:

	3M terreingrens
	Kadastrale grens
267v2	Kadastrale nummer
029	Gebouw
③	Nummer grondhoop
	Ondergrondse dijk
	Hek
▦	Stelcon plaat
●	Peilbuis
✕	Peilbuis verdwenen
⊗	Onttrekkingsput
⊠	Oppervlaktewaterstaalnamepunt
◎	Regenwaterrioleringsput

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
PPR	RMA	NHE	v0.1	☐ FINAL ☒ DRAFT

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Fax.: 010/48.35.36

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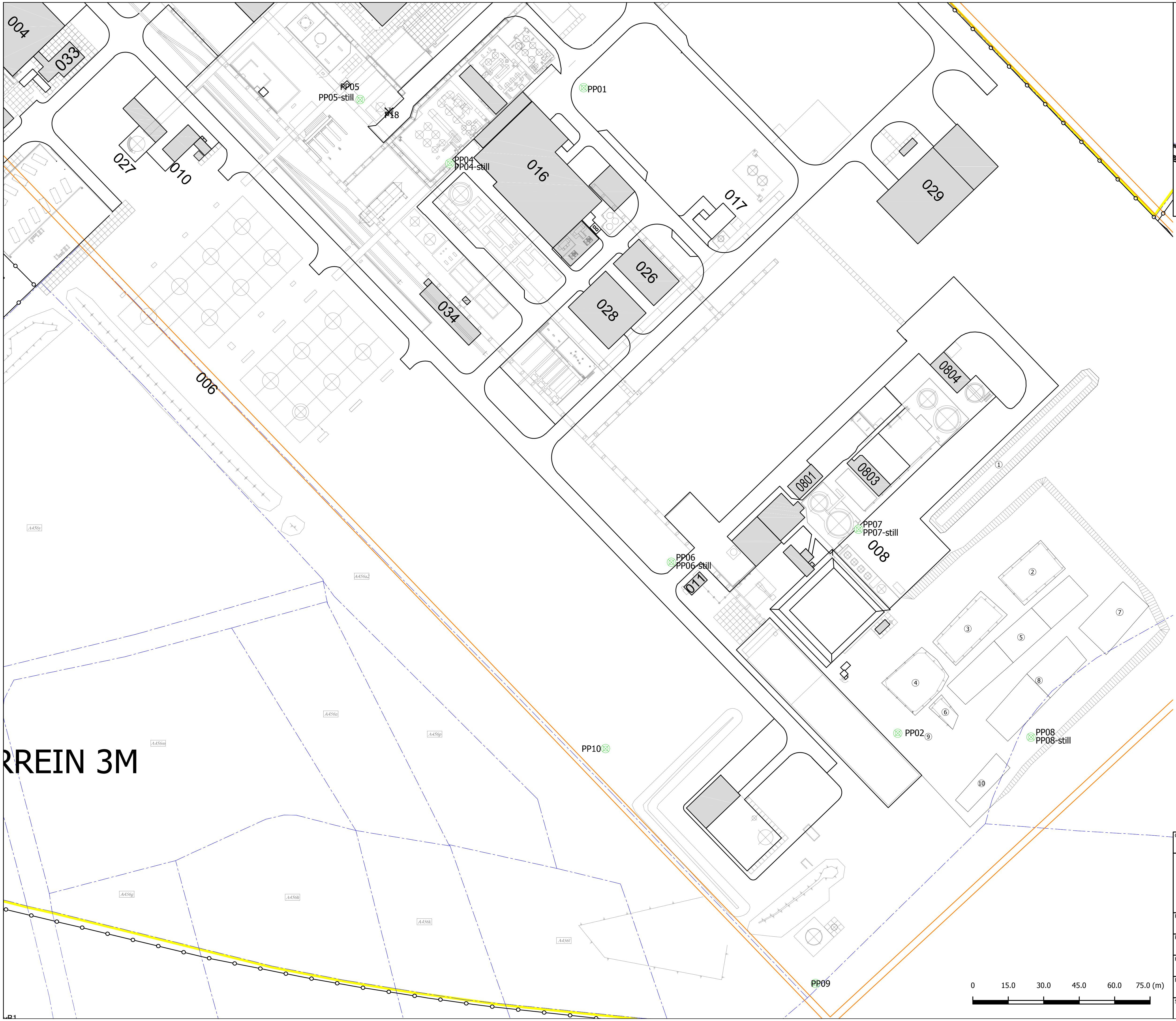
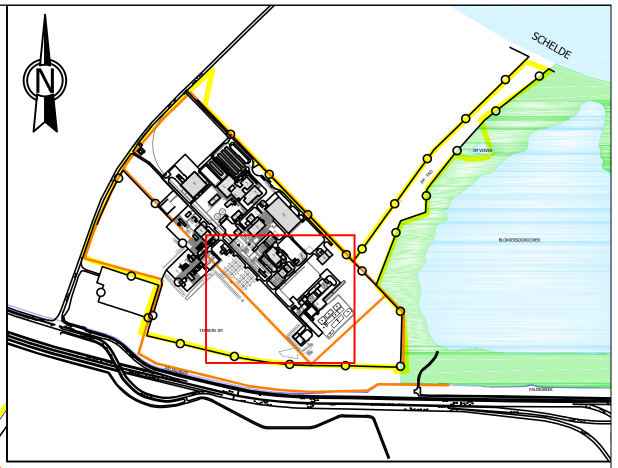
Klant / Projectnaam: 3M Belgium bvba	Locatie: Zwijndrecht
Projectnr.: 0341743	Titel: 7de Tussentijds Verslag Bodemsaneringswerken
Kaart: 5G	Beschrijving: Overzicht monsternamelocaties Regenwaterriolering
Fase: TTV-7	

Schaal:	Formaat:	Bestandsnaam:	Datum:
1:1500	A3	3M Zwijndrecht.dwg	22/12/2016

Kaart 5

Onttrekkingsfilters grondwateronttrekking





Legende:

🟨	3M terreingrens
-·-	Kadastrale grens
267v2	Kadastrale nummer
029	Gebouw
③	Nummer grondhoop
🟧	Ondergrondse dijk
—○—	Hek
▦	Stelcon plaat
⊗	Onttrekkingsput

Getekend: PPR	Gecorrigeerd: RMA	Goedgekeurd: NHE	Versie: v0.1	☐ FINAL ☒ DRAFT

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Klant / Projectnaam: 3M Belgium bvba	Locatie: Zwijndrecht
Projectnr.: 0341743	Titel: 7de Tussentijds Verslag Bodemsaneringswerken
Kaart: 9	Beschrijving: Onttrekkingsfilters grondwateronttrekking
Fase: TTV-7	

Schaal: 1:1500	Formaat: A3	Bestandsnaam: 3M Zwijndrecht.dwg	Datum: 22/12/2016