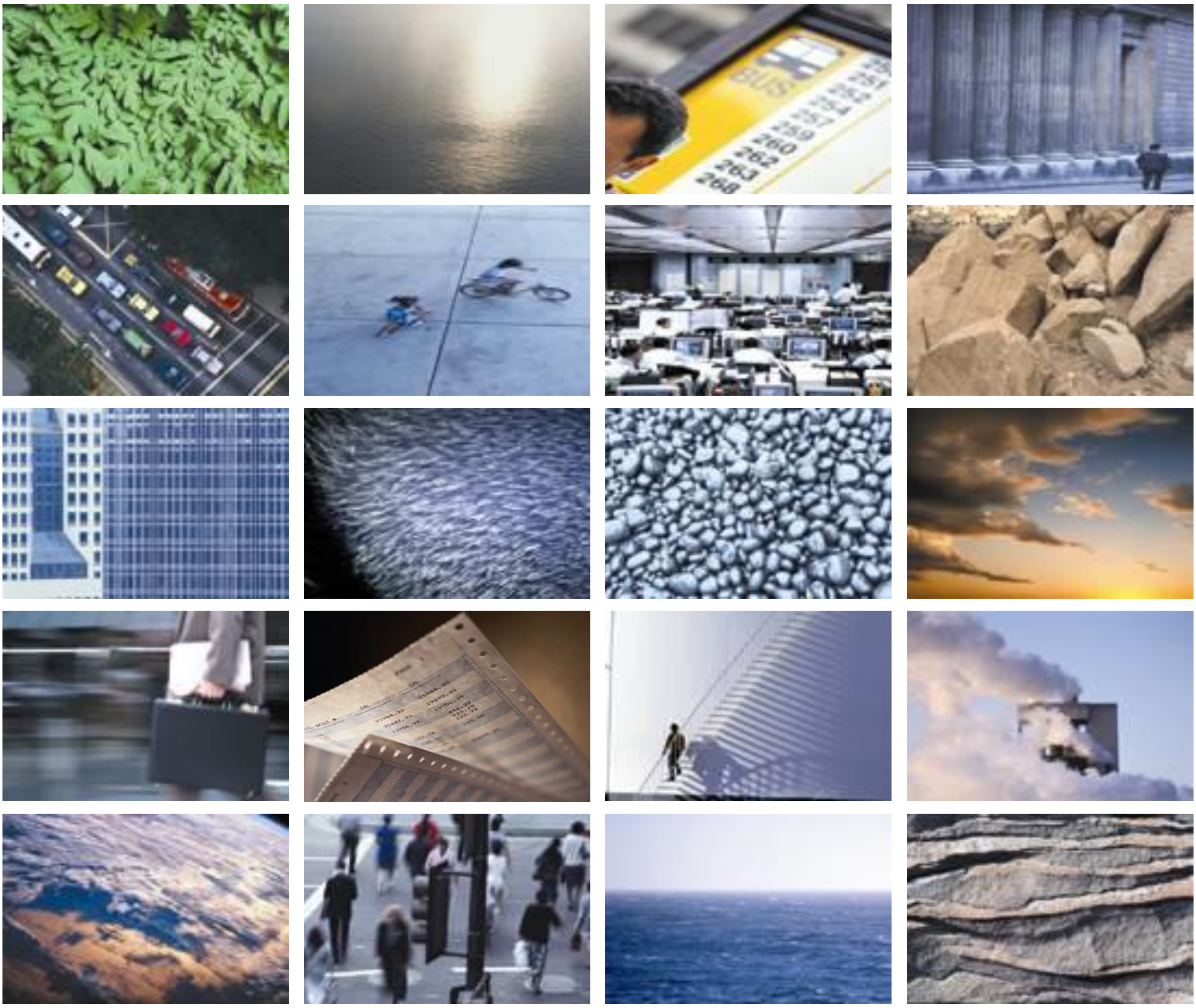

Eerste gefaseerd bodemsaneringsproject - Achtste tussentijds verslag bodemsaneringswerken

Periode juli 2016 - juli 2017

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

14 augustus 2018

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RAPPORT

Eerste gefaseerd bodemsaneringsproject - ***Achtste tussentijds verslag bodemsaneringswerken*** *Periode juli 2016 – juli 2017*

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

Projectnr.: 0398993

Ref.: R01-0398993_TTV8-v2

Voor en namens ERM nv

Opgemaakt door: Lieselotte Sorgeloos
Erik Boeckx
Mattias Verbeeck
Nanda Hermes

Goedgekeurd door: Dirk Nuyens
Functie: Principal Partner
Handtekening:

Datum: 14 augustus 2018

INHOUDSTAFEL

DEEL 1

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Bijlage 2 Milieukundig dagboeken (niet van toepassing)
Bijlage 3 Analysecertificaten
Bijlage 4 Overzicht veldmetingen en analyseresultaten
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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 -
Achtste tussentijds verslag bodemsaneringswerken
Periode juli 2016 – juli 2017

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

14 augustus 2018

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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 - Achtste tussentijds verslag bodemsaneringswerken: Periode juli 2016 – juli 2017 3M Belgium bvba Canadastraat 11 te 2070 Zwijndrecht
Referentie EBSD:	R01-0398993-TTV8 v2.docx
Rapportdatum:	14 augustus 2018
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:	-
Hoedanigheid:	☒ Eigenaar ☐ Gebruiker ☒ Exploitant ☐ Optredend in opdracht van de eigenaar/gebruiker/exploitant ☐ Andere: …………

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

Titel:	Eerste gefaseerd bodemsaneringsproject - Achtste tussentijds verslag bodemsaneringswerken: Periode juli 2016 – juli 2017 3M Belgium bvba Canadastraat 11 te 2070 Zwijndrecht
Referentie EBSD:	R01-0398993-TTV8 v2.docx
Rapportdatum:	14 augustus 2018
Naam contactpersoon:	Charlotte Tack
- Telefoon:	[redacted]
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- 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

Gemeentenummer	Sectie	Perceelnummer	Adres	Gemeente	Oppervlakte perceel (m^2)	Persoon (Eigenaar / gebruiker / exploitant) Periode Van	Tot	Type [3]	Naam	Adres	Letter [4]	Huidig bestemmingstype [1]	Toekomstig bestemmingstype [1]	Grondwaterkwetsbaarheid [2]
11056	A	467E	Canadastraat 11	Zwijndrecht	318499	1971	heden	E en Ex	3M Belgium bvba	Hermeslaan 7 1831 Machelen	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 -
Achtste tussentijds verslag bodemsaneringswerken
Periode juli 2016 – juli 2017

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

14 augustus 2018

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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 fluorochemicals (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) for these soil and groundwater impacts 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 wastewater 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. In the meantime, monitoring wells near the southern site boundary and the surface water of the Palingbeek are sampled.

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 2016 and July 2017.

In the period between July 2016 and July 2017, the following remedial and monitoring actions were performed at the site:

1. Operation and Maintenance (O&M) of the pump and treat (P&T) system installed in the two source areas (Building 16 and WWTP). These source removal systems were 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 FC's. Samples of the groundwater discharged from the extraction wells are collected quarterly to monitor the efficiency of the P&T system.

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

   - Groundwater wells 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
   - Storm water 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.

5. Statistical evaluation of the PFOS concentration trend in the groundwater and surface water of the Blokkersdijk and 3M pond.

In addition, the following related activity has been conducted during this period:

- Environmental follow up of the environmental incident that consisted of a release of hydraulic oil near building 32 in July 2017.

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 determine if sand and/or iron is accumulating in the extraction wells and to supervise the cleaning of the extraction wells, if needed. In addition, water samples are taken from the wells (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 local groundwater flow system.

The ERM inspections show that the extraction system requires regular maintenance to remove sand and/or iron deposits accumulating in the wells. The average uptime of the remedial system was estimated to be 88% for this reporting period (2015-2016: 53%, 2014-15: 75% and 2013-14: 71%), but the uptime varies from well to well: minimum 72% for well PP06 and maximum 97% for well PP01 as shown in the table below.

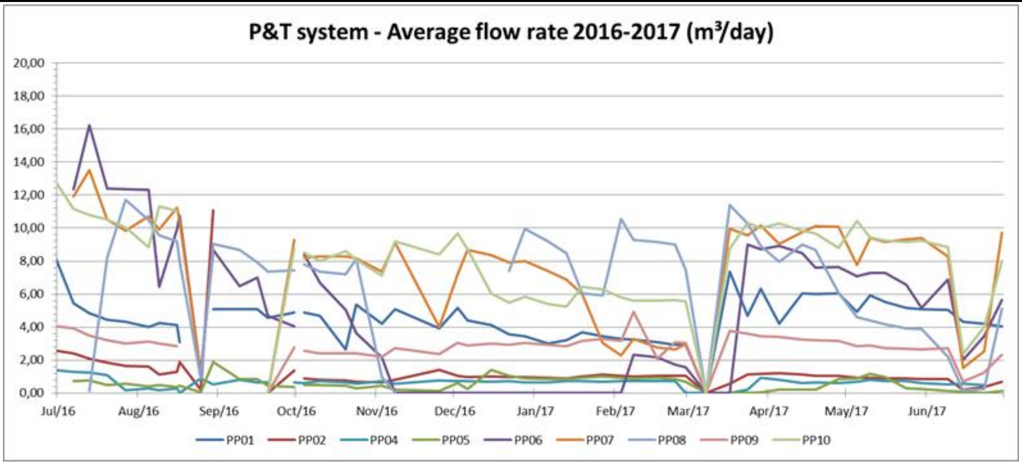
P&T system – Monthly summary of system uptime

	jul/16	aug/16	sep/16	okt/16	nov/16	dec/16	jan/17	feb/17	mrt/17	apr/17	mei/17	jun/17	uptime (%)
PP01													97
PP02													92
PP04													90
PP05													81
PP06													72
PP07													88
PP08													86
PP09													92
PP10													92

Pump operational whole month
Pump operational 1/2 month
pump not operational
pump operational but technical issues

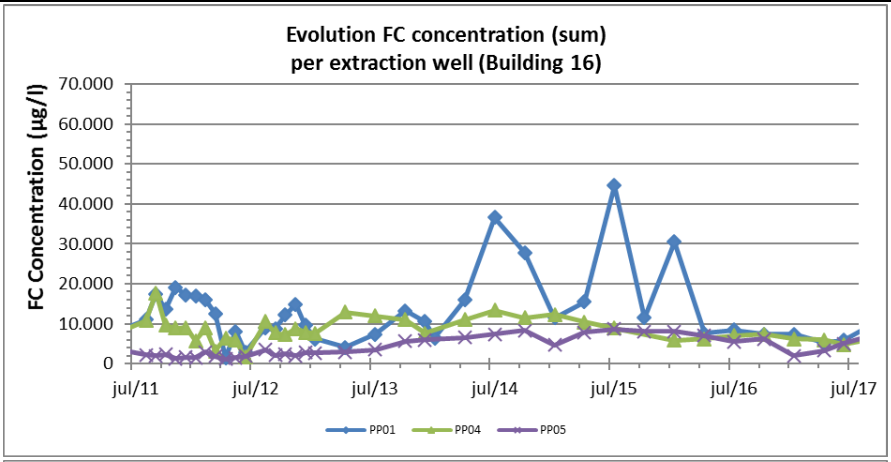
The average flow rate of each extraction well varies between a minimum of 0,40 m^3/day (well PP05) to a maximum of 7,44 m^3/day (well PP10) as shown in the following figure.

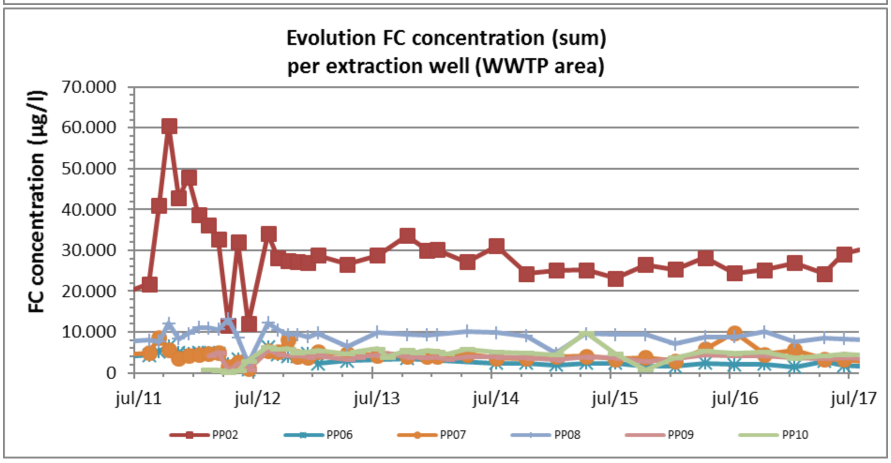
P&T system – Average flow P&T wells (July 2016 - July 2017)



The average groundwater FC concentration ranges between 2,0 mg/L (PP06) to 26,0 mg/L (PP02). As shown in the graph below, the long-term trend in FC concentrations in most of the extraction wells is stable, although fluctuations can occur. Additional monitoring is necessary to confirm the long-term trend in FC concentrations.

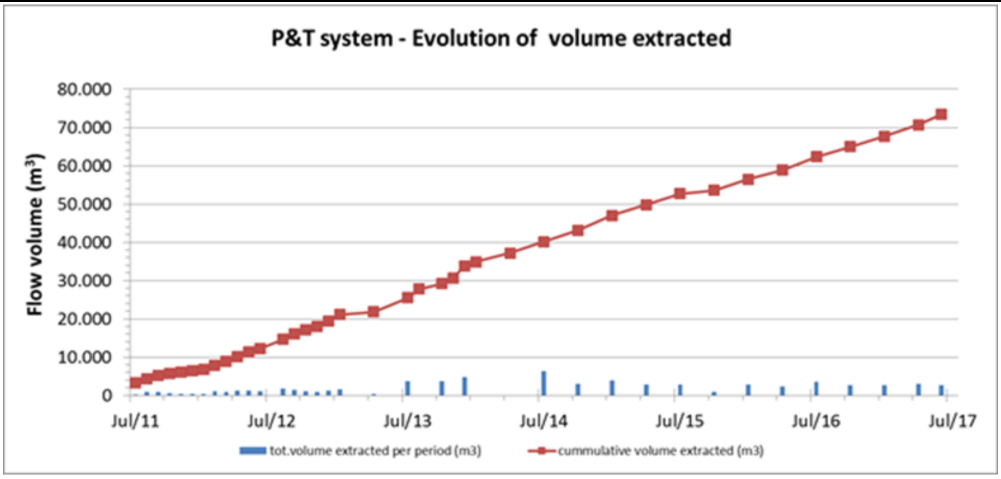
P&T system – Concentration Trends in Building 16 and WWTP area

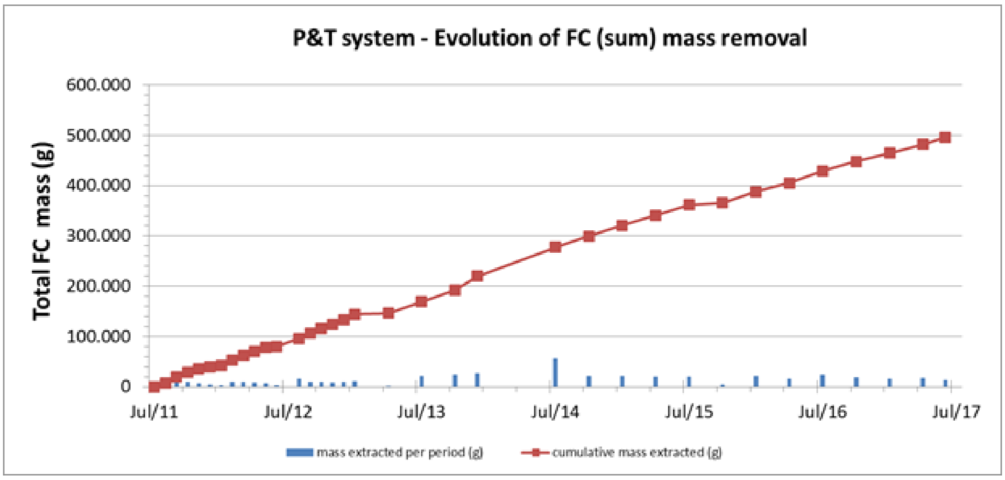




Between July 2011 and June 2017, approximately 73.339 m³ of contaminated groundwater was extracted and approximately 496 kg of FC's were removed, as shown in the graphs below.

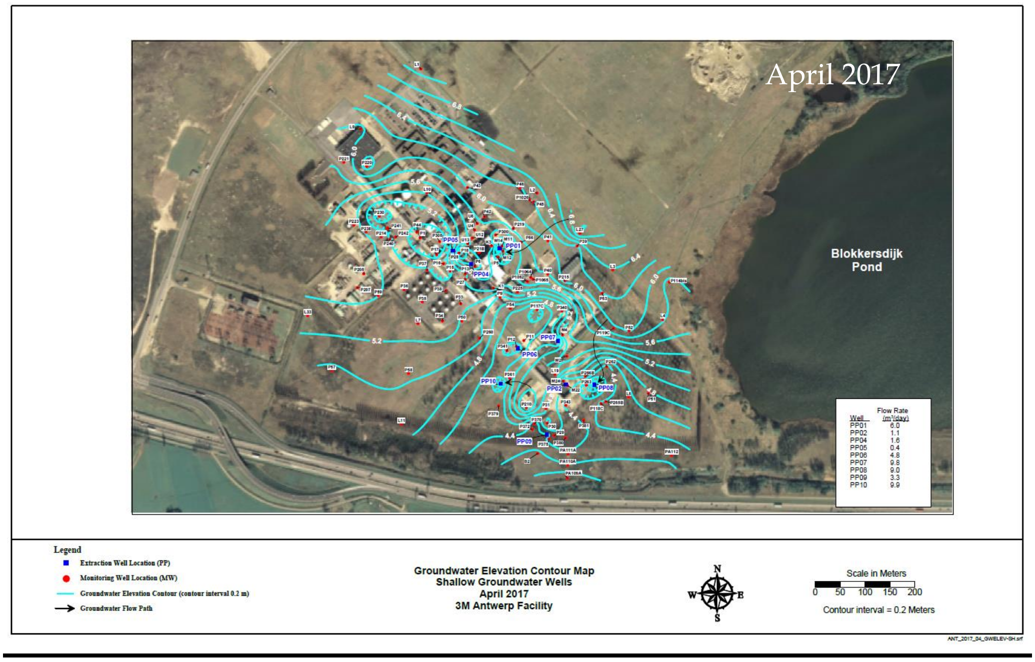
P&T system – Efficiency Trends





The system has a clear impact on the natural groundwater flow pattern of the first aquifer, as is shown by the depressions created in the groundwater surface near each extraction well presented 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 groundwater extraction system was within the predefined limits. Based on the recommendations of an independent geotechnical expert (Grontmij nv), a final measurement will be performed when all extraction wells are operational to verify the findings of 2013 and to evaluate if further monitoring is necessary.

In accordance with the RAP specifications, ERM recommends that regular inspections of the system (at a minimum monthly frequency) and quarterly sampling of the extraction wells be continued to assess long-term trends. 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 optimize contaminant capture and uptime.

Monitoring Groundwater, Surface Water and Storm Water Quality

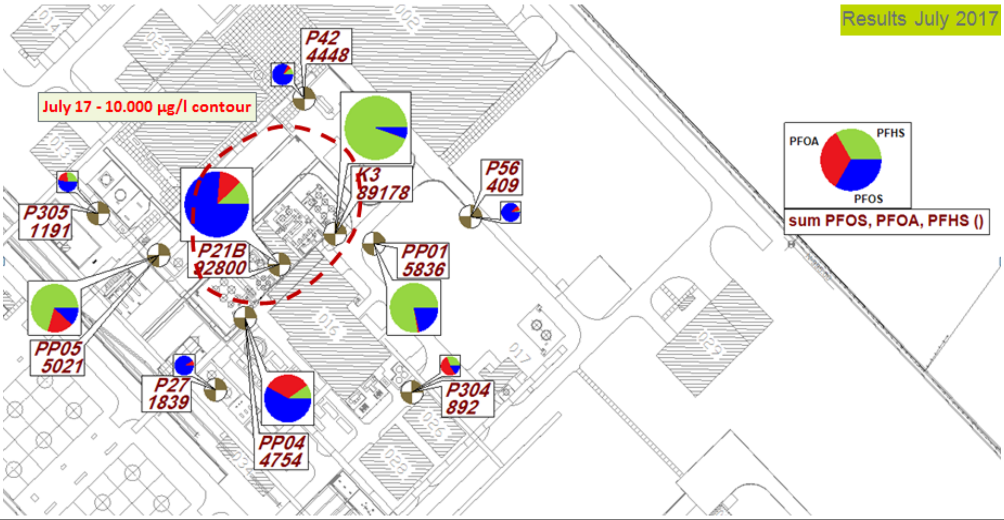
The remedial objectives of the monitoring program are to verify that the P&T system is mitigating the horizontal migration in the first aquifer in the areas of concern (Blokkersdijk nature reserve and the Paling ditch at the southern site boundary), as well as the vertical migration of contaminants from the first to the second aquifer. As part of this monitoring program, ground-, surface and storm water samples are collected 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:

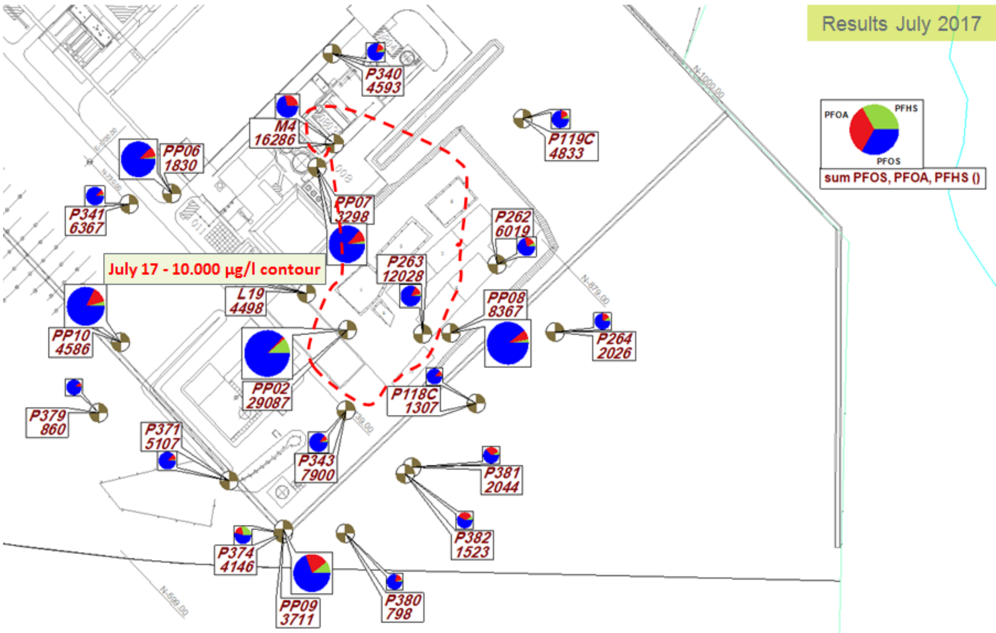
- In the Building 16 area: two groundwater monitor wells are sampled annually, four groundwater monitor wells are sampled semi-annually and one well is sampled quarterly, and;
- In the WWTP area: six groundwater monitor wells are sampled annually, ten monitor wells are sampled semi-annually and one monitor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

The results of the July 2017 groundwater monitoring event are shown in the figures below with the approximate extent of the sum 10.000 µg/L FC-concentration contour.

Building 16 source area



WWTP source area



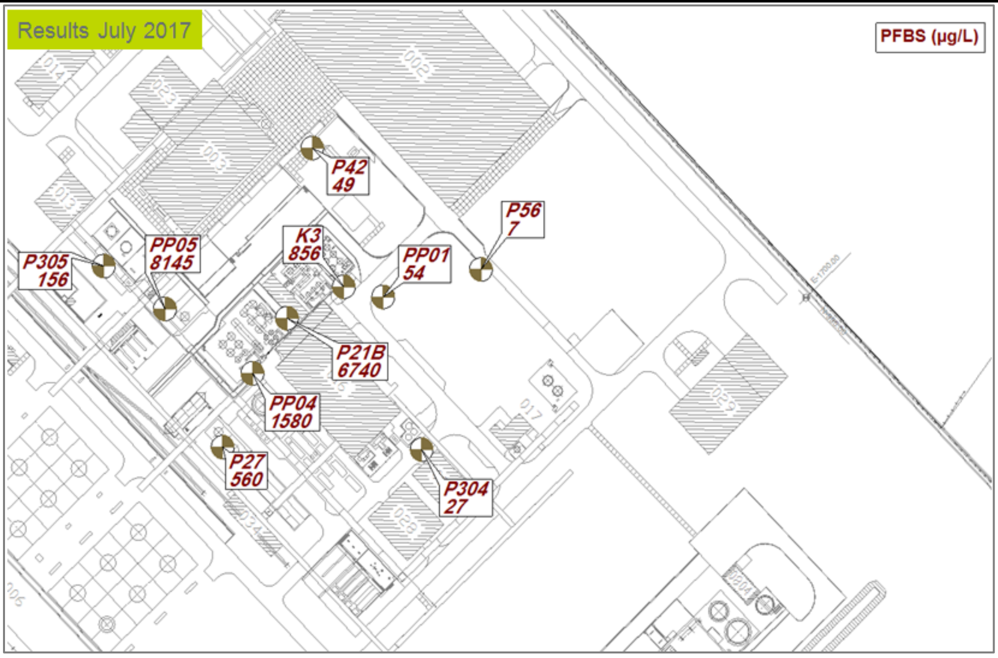
The general stable/decreasing trend in FC concentrations that has been observed, suggests that the shallow P&T system installed in the two source areas is reducing the concentrations of FC's in groundwater in the Building 16 and WWTP area.

Based on a review of the historical monitoring results, groundwater concentrations in the Building 16 area are stable or decreasing, except for the PFOS concentration in well P21B. The extent of the sum of groundwater FC concentrations contour appears to be equal to the contour based on the results of the previous monitoring round (2015-2016).

In the WWTP area, groundwater FC concentrations in most of the wells are stable or decreasing, except for wells P119C and P371. The PFOS concentration in these wells has increased. The extent of the sum of groundwater FC concentrations contour appears to be the same as the contour of the July 2016 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 higher PNEC value (10.040 µg/L) compared to PFOS. Recent groundwater analytical data indicates the presence of PFBS in the area of Building 16. The results of the July 2017 monitoring are shown in the following figure. The highest PFBS concentration in groundwater is measured in extraction well PP05 and monitoring well P21B. However, the PFBS concentration in extraction well PP05 has continued to decrease since 2014. The PFBS concentration in well P21B is stable.

FC Source Area near Building 16 – PFBS Results July 2017 Monitoring



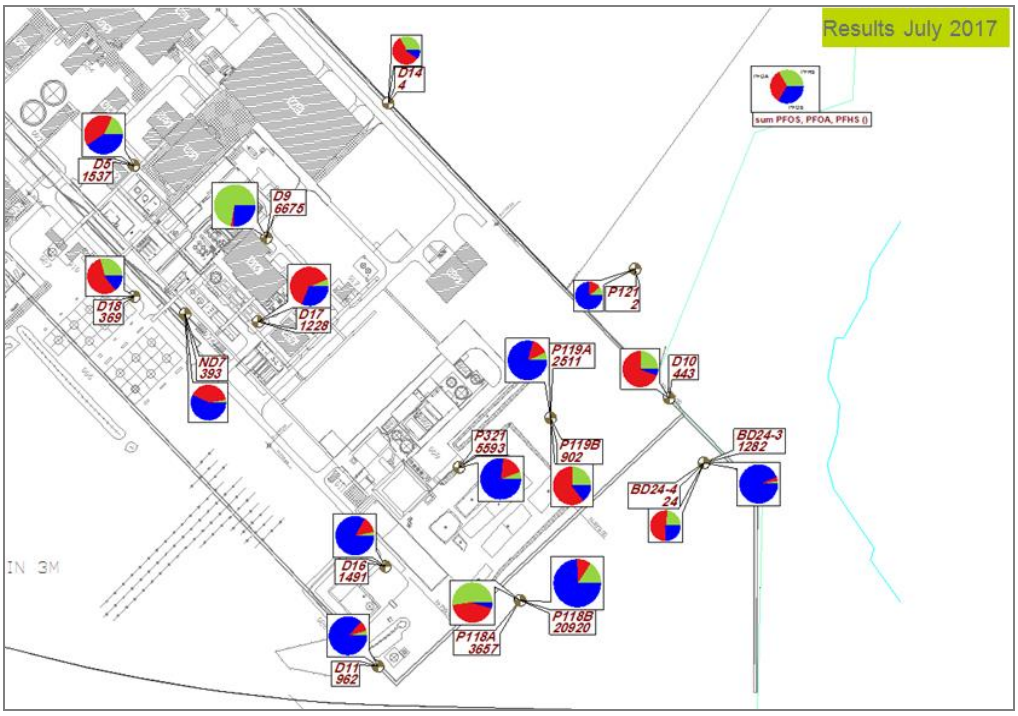
The cause of the PFBS detection and the observed increase in concentration is, at present, unknown. ERM recommends continuing the current groundwater monitoring program for wells completed in the shallow (1st) aquifer. This includes the continued monitoring of PFBS concentrations. In addition, ERM recommends assessing the need for further optimization of the P&T system in the shallow (1st)aquifer as some wells show increasing concentration trends.

To monitor the groundwater quality of the **second aquifer,** two wells are sampled annually, twelve wells are sampled on a semi-annual basis and four wells 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 2017 monitoring are shown in the following figure.

Second Aquifer - Results July 2017 Monitoring



The evaluation of the monitoring results shows that groundwater concentrations in the second aquifer fluctuate and are increasing in one well (P321) in the WWTP area. In 2016, an increasing concentration trend in several wells was observed. The July2017 groundwater concentrations show a decreasing or stable concentration trend for these monitoring wells.
The higher uptime of the groundwater extraction system in 2017 compared to 2016 may be a contributing factor to the decreasing or stable concentration trend in 2017. Additional efforts have been made to improve system uptime to enhance capture. Continued monitoring of these second aquifer 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.

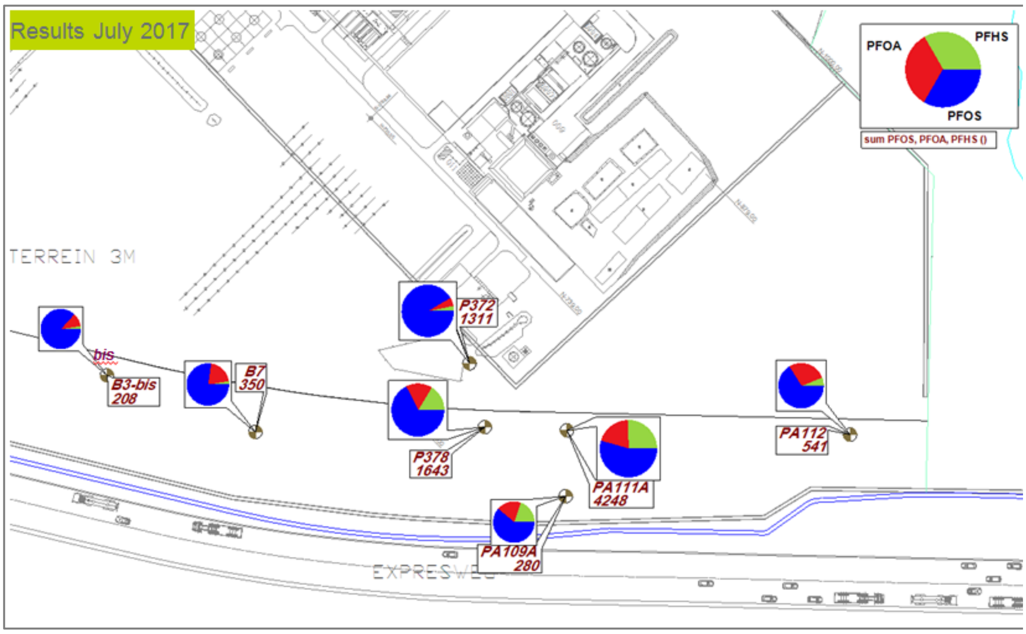
Seven of the eight additional wells that were sampled in July 2016, had higher concentrations than measured in the past (2002-2005). To assess the potential impact of lab changes and natural concentration fluctuations, further monitoring is recommended to assess concentration trends.

The FC concentrations are below the limit value of five-times the the FC 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 be assessed through continued monitoring. In addition, ERM recommends to optimize the operation of the pump and treat system in the first aquifer to effectively remove FC's.

The groundwater quality along the **southern site boundary** is currently being monitored semi-annually in seven wells. The results of the July 2017 monitoring are shown in the following figure.

Southern Site Boundary - Results July 2017 Monitoring



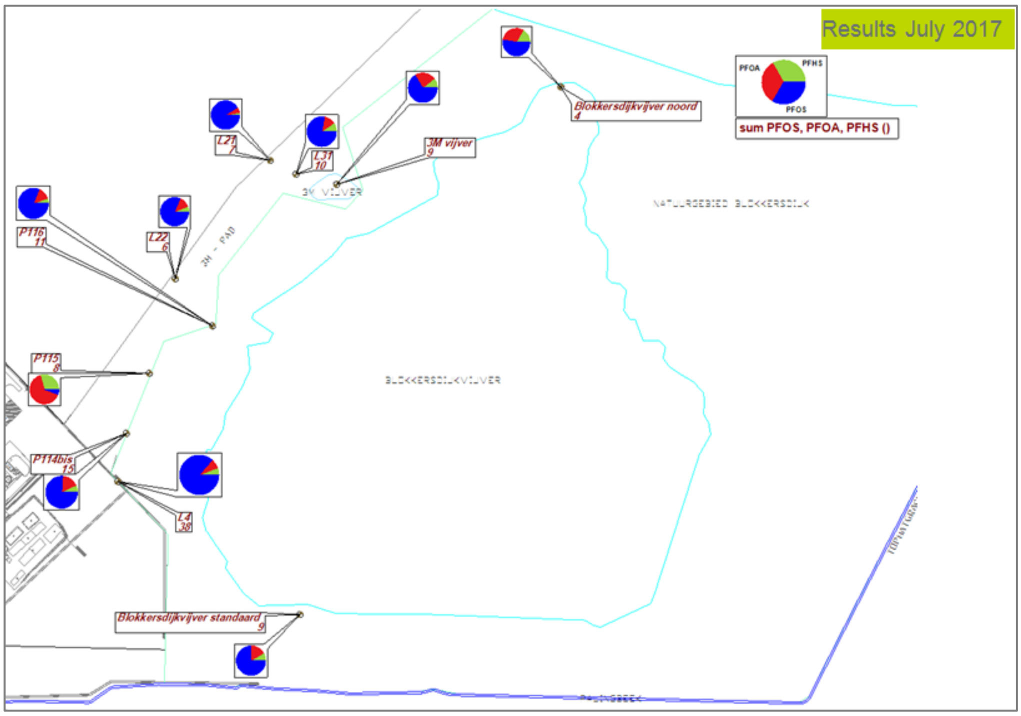
The evaluation of the historical monitoring results shows that, in general, the groundwater concentrations over time are decreasing or stable, but concentrations may fluctuate. The results suggest that the shallow P&T system installed at the WWTP source area is preventing contaminant migration to the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

The groundwater quality in the **tank farm area** (near Building 3) is currently monitored annually for TPH. 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 TPH concentration measured in well P28 in July 2017 has significantly decreased (<500 µg/L) compared to previous measurements (12.000 µg/L in 2016).

The source of the TPH is unknown but may be caused by interference from another source. The elevated pH and conductivity are likely related to (minor) spills from a 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 **<u>Blokkersdijk nature reserve</u>** is monitored via seven wells and two ponds. Seven groundwater wells and three surface water locations are monitored quarterly to statistically evaluate the trend of FC concentrations. The results of the July 2017 monitoring are shown in the figure below.

Blokkersdijk Nature Reserve - Results July 2017 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 data 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 affect 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. For this reason, only data analyzed by 3M is utilized for future trend analysis.

For the PFOS concentration dataset through August 2017 (3M data only), a statistically significant increasing trend for well P116 and a statistically significant decreasing trend for well L22 are observed. When the statistical evaluation is done only with the seven most recent groundwater concentrations, no significant trends are calculated.

It is recommended to continue quarterly sampling in all the sample locations in the Blokkersdijk area, to provide data to perform a meaningful statistical trend analysis. 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 following figure.

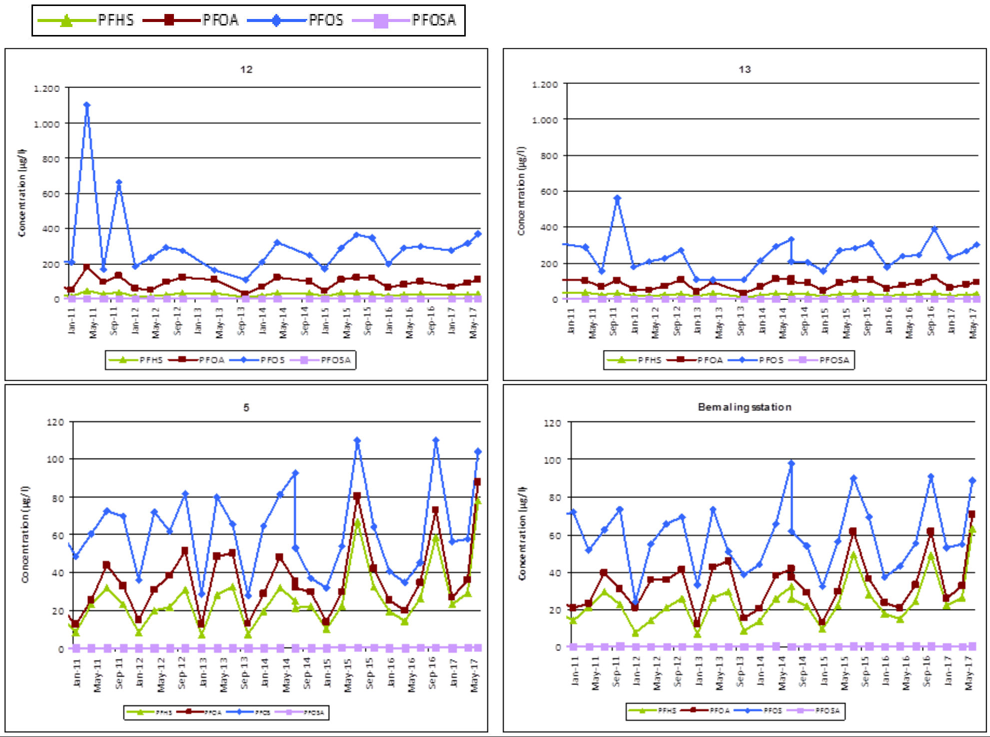
Location 3M site, Blokkersdijk nature reserve, 3M path and Schelde River

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.

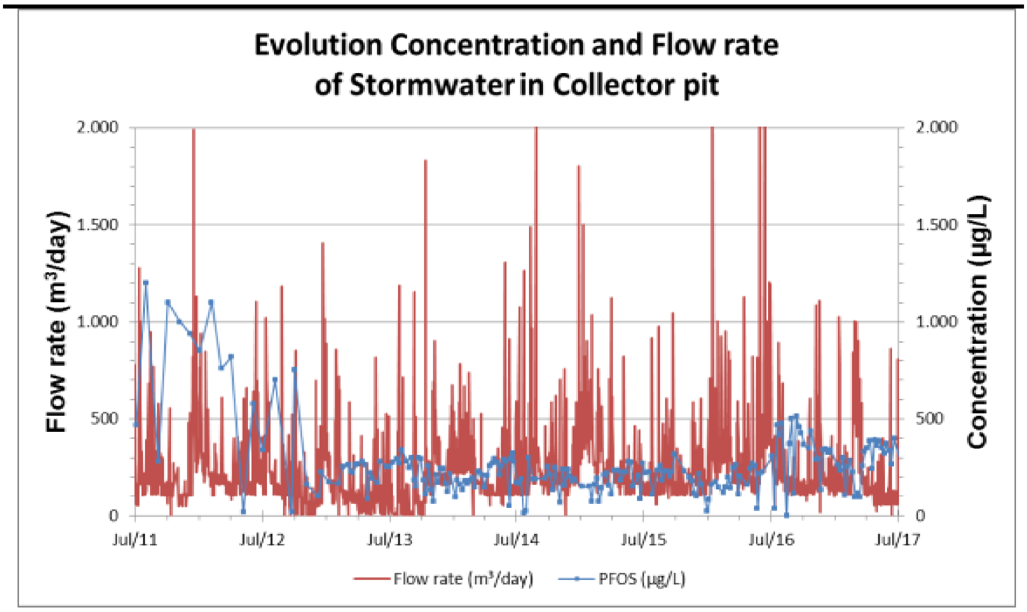
Based on the results of the monitoring, ERM recommends continuing the assessment of the FC concentrations in and close to the Blokkersdijk nature reserve through quarterly monitoring of the monitoring wells and the Blokkersdijk and 3M pond.

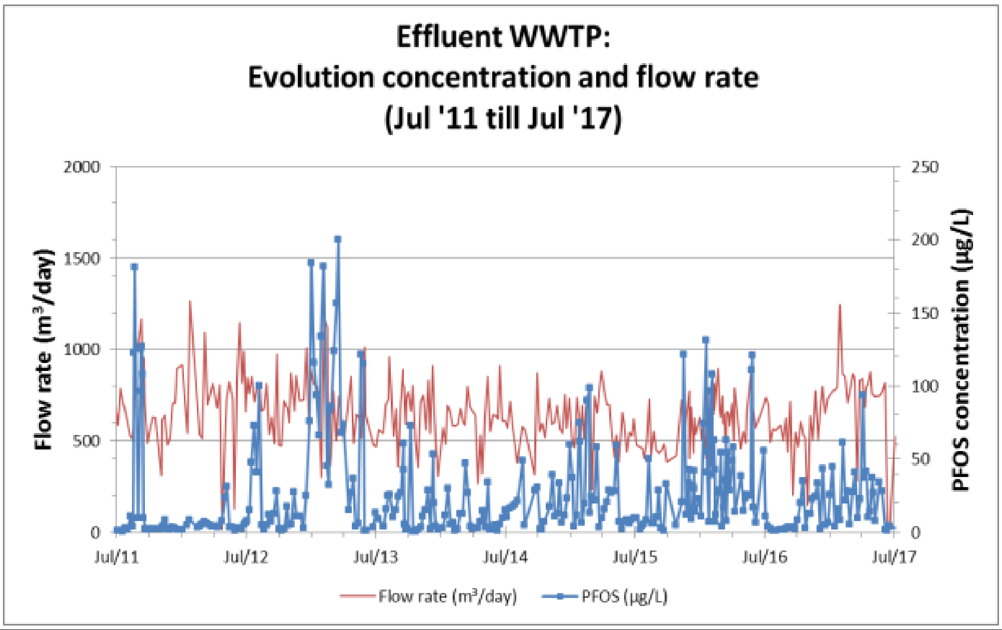
The quality of the surface water in the **Paling** and **Tophat ditches** is monitored quarterly for FC's at four different locations. The evaluation of the monitoring results shows that FC concentrations in the Paling ditch are stable at present, see graphs below, although concentrations can fluctuate.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

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.



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, weekly samples are taken from the **WWTP** and **storm water 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 storm water sewer system. The collector pit results are less sensitive to contaminant fluctuations caused by rainwater and therefore more reliable.





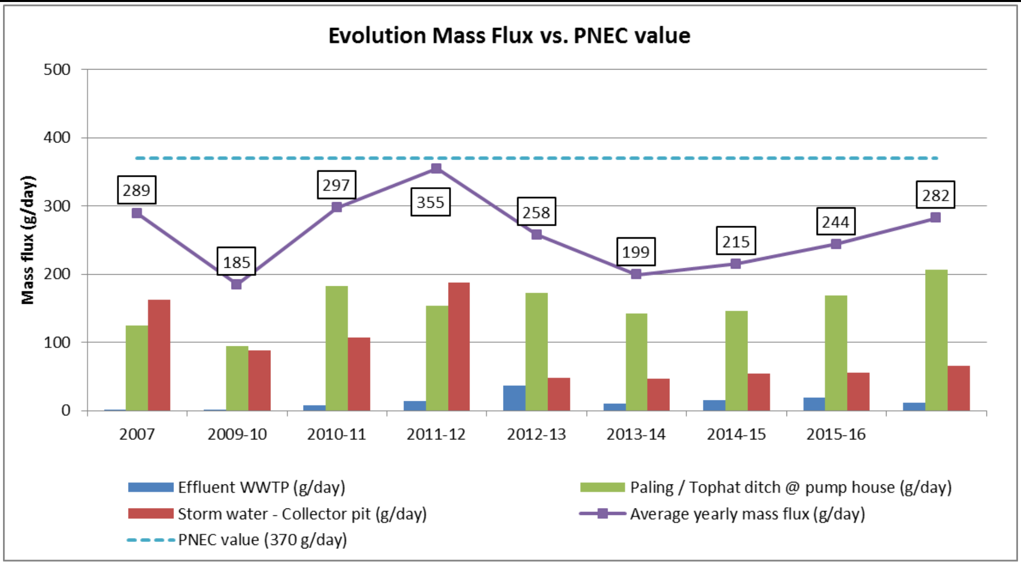
Since 2012-13, PFOS concentrations in the collector pit and wastewater treatment plant have fluctuated, but show a stable trend (see figure above). The cleaning of the collector pit in 2012 has significantly decreased the mass flux, and therefore there is, at present, no need to install an additional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

The average PFOS concentration between August 2016 and July 2017 is 298 µg/L for the collector pit and 17,2 µg/L for the wastewater treatment plant effluent discharged to the Schelde River. ERM recommends continuation of the weekly monitoring of the storm water collector pit and wastewater 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 using the analytical results of samples collected between August 2016 and July 2017 from the effluent of the WWTP, the storm water collector pit and the Tophat ditch near the pump station. The daily average mass flux of PFOS was compared to the maximum allowable mass flux value. The calculated mass flux value (282 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



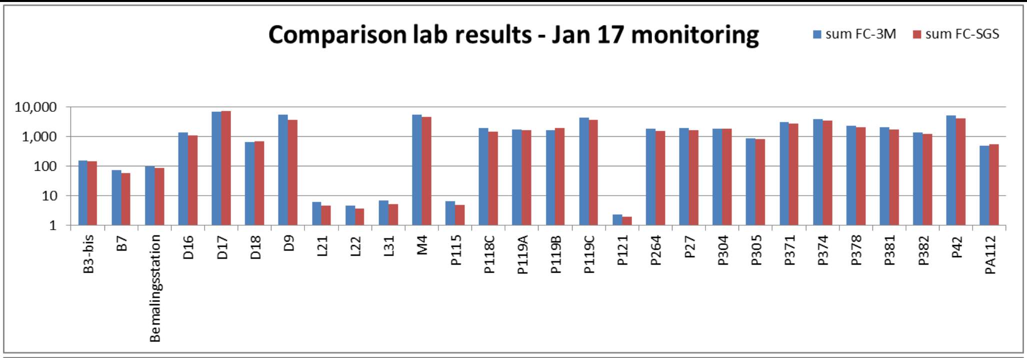
The average mass flux of PFOS shows an increase since 2013-2014, mainly due to an increasing mass flux in the Paling ditch and Tophat ditch. At present, there is no explanation for this increasing mass flux trend. The quarterly monitoring will be continued.

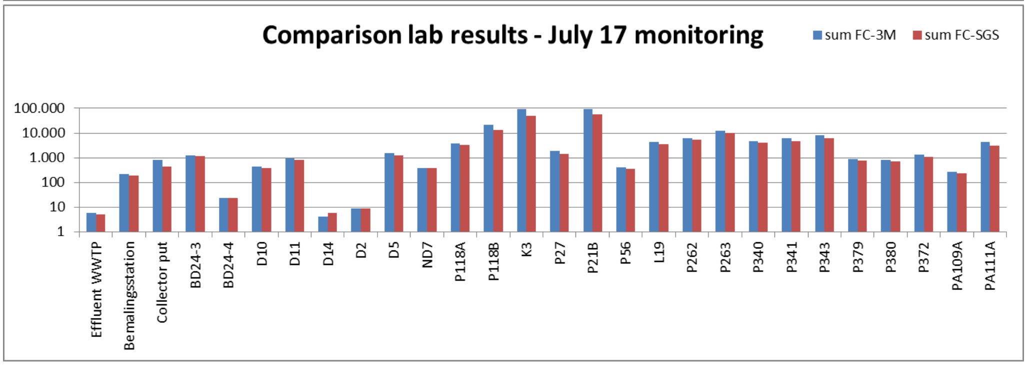
Evaluation of analytical results of water samples - SGS vs 3M Environmental Laboratory

Per the agreement reached with OVAM (meeting dd. 22 October 2013), all water samples taken in 2017 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 consistency, only the 3M results are shown in this report.

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

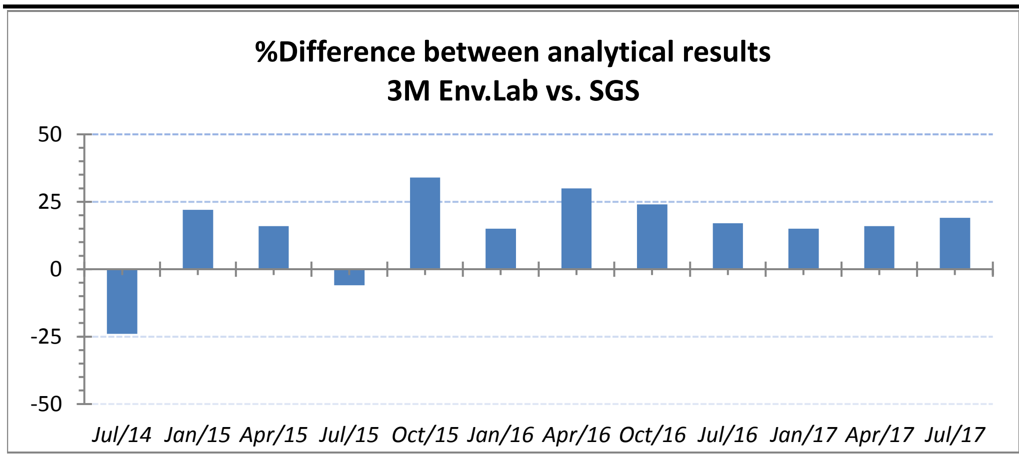
Lab results comparison (SGS vs 3M Environmental Laboratory)





The following graph shows that for all monitoring events since 2015, the average measured concentrations are higher for the 3M Environmental Laboratory compared to SGS.

Lab results comparison (SGS vs 3M Environmental Laboratory)



The difference is likely due to different sample preparation techniques and analytical methods used by each laboratory (instrument parameters and calibration).

ERM recommends that in July 2018 through July 2019 all water samples be analyzed by the 3M Environmental Laboratory and the duplicate sampling be spread out over the four monitoring events (similar to the monitoring in 2017). Duplicate water samples will be analyzed by the SGS laboratory during the monitoring period.

Monitoring of the Soil Pile Impacted with FCs and Mercury

As agreed with OVAM in 2013 the pile officially needs to be visually inspected once every five years, as a new cover was installed in 2012. The last visual inspection by ERM (at the request from OVAM) in April 2016 showed that the condition of the liner covering the soil piles was still intact. The next visual inspection is scheduled for 2018.

Environmental Follow-up of an Environmental Incident

On July 5, 2017, a minor spill of hydraulic oil occurred at the southern side of Building 32, on a gravel surface. The incident was reported to OVAM in accordance with local regulations.

The impacted area was excavated in order to limit the impact of hydraulic oil in soil. Five soil samples were taken to assess the impact of the spill: one sample of the excavated soil, two samples of the wall of the excavation pit and two sample of at the bottom of the excavation pit at different depths. The soil samples were analyzed by SGS.

The TPH concentration in one sample taken of the wall of the excavation pit (480 mg/kg dm) exceeded the target value of 300 mg/kg dm. As a result, an additional excavation was done on the August 25, 2017. Four new samples of the wall and two samples of the bottom of the excavation pit were submitted for analyses. After the second excavation, the TPH concentrations for the samples collected from the excavation pit wall and bottom were all lower than the detection limit of 50 mg/kg dm. In addition, the TPH concentration in groundwater (monitor well P221) is lower than the detection limit of 100 µg/L.

Because it was a minor impact and the measured concentration of TPH is below the remedial target value, ERM concludes that no further actions are needed.

As is agreed with OVAM, the results and conclusions will be reported to OVAM in the periodical orientative site investigation of 2017 (submittal to OVAM in 2018).

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 table below.

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 in groundwater. Between July 2011 and June 2017, approximately 73.339 m^3 of contaminated groundwater was extracted and approximately 496 kg of FC's has been removed.

In the period from July 2016 until July 2017, the system had an average uptime of approximately 88% (higher than previous periods), but the uptime varies from well to well (between 72% and 97%). 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 continued monitoring will be performed at these locations. At this moment, the remedial criteria specified in the RAP continue to be met.

The (potential) increasing trend in one of the Blokkersdijk monitoring wells needs to be verified via further monitoring and statistical evaluation. As there is no statistically significant increasing trend for the PFOS concentration in the Blokkersdijk pond, there is no need for additional remedial actions.

The yearly average mass flux of PFOS towards the Schelde River is well below the relevant PNEC value and is comparable to previous results.
The monitoring results of the surface water indicates that there is a slightly increasing concentration trend for FC's in the surface water of the Tophat ditch.

The potential groundwater impact by TPH in the tank farm area will continue to be evaluated.

One incident occurred at the 3M site on July 5, 2017. After an excavation of potentially impacted soils was completed, the sampling results identified no impact to soil and/or groundwater. Therefore, no further action is required. As agreed with OVAM, the incident will be officially reported in the 2017 periodic orientative site 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 expansion of the remedial system to optimize the contaminant capture. During this evaluation, options to improve the operation of the pump and treat system will also 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 in the first quarter of 2019, describing the remedial actions and monitoring results taken from August 2017 through July 2018.

■

Proposed Remedial Monitoring Program for 2018

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 (cancellation 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	-	Once every five years	Visual inspection of soil pile		From 2013 onwards, one monitoring event performed 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	-	
	4	Semi-annual	P27, P42, P304, P305	-	
	1	Quarterly	P21B		Increasing concentration trend at monitoring well P21B
WWTP	3	Annual	P341, P379, P380,		-
	2	Annual	P381, P382	P381 and P382: monitoring frequency changed from semi-annual to annual, because relative low and stable concentrations.	
	8	Semi-annual	P118C, P264, P265B, P371, P374, L19, M4, P119C	P265C (4,5-5,5 m bgl) is damaged and replaced by monitoring well P265B (2,8-3,8 m bgl).	
	3	Semi-annual	P262, P340, P343	No significant concentration trend, concentrations fluctuate. Monitoring wells to delineate the 10.000 µg/L contour; same monitoring frequency as other monitoring wells to delineate the 10.000 µg/l contour.	
	1	Semi-annual	P263	Monitoring well in source area, no significant concentration trend. Same monitoring frequency as other monitoring well in source area.	

Location	Number of Wells	Frequency	Well ID	Rationale	Remarks
Second Aquifer	1	Annual	D2		
	2	Semi-annual	D9, BD24-3	In 2017, D9 and BD24-3 are sampled quarterly. The groundwater concentrations decreased or stabilized compared to 2016, so monitoring frequency changed to semi-annual.	
	8	Semi-annual	D16, D17, D18, D5, ND7, P121, P119A en P119B		
	1	Semi-annual	BD24-4	Relative low concentrations; same monitoring frequency for other wells.	
	1	Quarterly	P321	-	-
Southern Site Boundary	7	Semi-annual	B3-bis, B7, P372, P378, PA109A, PA111A, PA112	-	
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	-	-
Surface water	3	Quarterly	Blokkersdijk standaard, 3M vijver, Blokkersdijk Noord	Blokkersdijk Oost is replaced by Blokkersdijk Noord because better accessibility	
Paling and Tophat Ditch	4	Quarterly	12, 13, 5, pumping house	-	-
Storm Water	1	Weekly	Collector put	Weekly samples analyzed by 3M internal lab	Quarterly verification samples by OVAM certified laboratory (eg. SGS)
Effluent WWTP	1	Weekly	Discharge line	Weekly 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 achtste tussentijds verslag (TTV8) van de saneringswerken, zoals beschreven in het door de OVAM conform verklaarde bodemsaneringsproject (1e fase) (2) en de zeven voorgaande tussentijdse verslagen (3), (4), (5), (6), (7), (8) en (9) . Het betreft de saneringswerken op het deel productiezone, voormalige slibbekkens/waterzuiveringsinstallatie, en het natuurgebied Blokkersdijk. De beschouwde saneringsperiode loopt van juli 2016 tot en met juli 2017.

De organofluorimpact ter hoogte van de zuidelijke perceelgrens en de Palingbeek wordt momenteel door middel van een grond- en oppervlaktewatermonitoring 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 studiewerken voor het deelproject Linkeroever zijn lopende 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
(9) "Zevende tussentijds verslag bodemsaneringswerken Eerste gefaseerd bodemsaneringsproject", opgesteld door ERM in September 2017

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 2017) 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 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 1 juli tot 30 juni. Vanaf voorliggend tussentijds rapport is ervoor gekozen om de monitoringsperiode van 1 augustus tot 31 juli te nemen, zodat deze samen valt met de monitoringsperiode voor grondwater.

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

Tabel 2.1 ***Gemiddelde PFOS vuilvracht richting de Schelde (2007 tem. 2016)***

Vuilvracht naar de Schelde	Debiet (m^3/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**
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

Vuilvracht naar de Schelde	Debiet (m^3/dag)	PFOS concentratie (µg/L)	Vuilvracht (g/dag)
Totale vuilvracht			**215**
juli 2015 – juni 2016 (TTV7)			
Effluent van het bedrijfsafvalwater (incl. opgepompt grondwater)	611	31	19
Regenwaterriolering	298	187	56
Palingbeek/Tophatgracht***	2.865	59	169
Totale vuilvracht			**244**

* 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, de regenwaterriolering en/of het 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 4.9).

3. ALGEMENE BESCHRIJVING SANERINGSWERKEN

3.1 ALGEMEEN

In het eerste gefaseerd bodemsaneringsproject (2) zijn de selectie en uitwerking van de saneringsaanpak voor de verontreinigingen met organofluorverbindingen (FC's) en vluchtige aromaten (BTEX) op de site van 3M beschreven, waarvoor een saneringsnoodzaak bestaat. De saneringswerken uitgevoerd tussen juli 2009 en juni 2016 zijn beschreven in de zeven voorgaande tussentijdse verslagen (3), (4), (5), (6), (7), (8) en (9).

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. Ter hoogte van de zuidelijke perceelsgrens van het 3M terrein zijn infrastructuurwerken voor het Oosterweelproject gepland. Daar deze werken een belangrijke impact kunnen hebben op de waterhuishouding op deze locatie, dient een saneringsaanpak afgestemd te worden op deze werken. De studiewerken voor het deelproject Linkeroever zijn in uitvoering 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, zal – indien noodzakelijk- een saneringsaanpak voor de FC impact uitgewerkt worden in een tweede gefaseerd bodemsaneringsproject.
In afwachting van een bodemsaneringsproject wordt de organofluorimpact ter hoogte van de zuidelijke perceelgrens halfjaarlijks en ter hoogte van de Palingbeek driemaandelijks gemonitord.

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 houden 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 voor:

- 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 (1^e^ aquifer)*

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 waterzuiveringsinstallatie 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 (1^e aquifer)

3.2.5.1 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.

3.2.5.2 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; en
- 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 waterzuiveringsinstallatie 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 2e aquifer*

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

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 individuele FC verbindingen in de bronzone in de 2e 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	-

De evolutie van de grondwaterconcentraties aan FC verbindingen in de 2e 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 is 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 de studiewerken in het kader van de Oosterweelverbinding in uitvoering en dient de impact hiervan op de verontreiniging en waterhuishouding in deze zone nog bestudeerd te worden.

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 hogervermelde saneringsaanpak wordt gevolgd, is samengevat in onderstaande tabel.

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

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), (8) en (9) een aantal wijzigingen voorgesteld. Deze wijzigingen zijn toegelicht in onderstaande paragrafen. In paragraaf 3.3.11 wordt een overzicht gegeven van het monitoringsprogramma voor 2017.

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. Op vraag van OVAM d.d. 13 april 2016 is nog een tussentijds controle van het afdek uitgevoerd op 26 april 2016. Aangezien de afdekking op dat moment in goede staat was, zal de volgende controle in 2018 worden uitgevoerd.

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 bleek dat de concentraties bleven dalen. De concentraties liggen onder de relevante richtwaarden 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;
- De werking van de grondwateronttrekking wordt jaarlijks geëvalueerd; en
- Installatie en opstart van de tweede stap van de grondwateronttrekking indien nodig.

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 hogervermelde 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	Debiet ingeschat in BSP		1e stap onttrekking		2e stap onttrekking	
Zone WWTP	10	12 m^3/dag	4	15 m^3/dag	14	17 m^3/dag

3.3.3.3 *Grondwatermonitoring*

De grondwatermonitoring ter hoogte van zone Gebouw 16 loopt intussen zeven jaar (sinds midden 2011).

In de loop van de jaren zijn verschillende aanpassingen aan het monitoringsprogramma gedaan, om bepaalde trends of concentratiepieken op te volgen:

- Peilbuis K4 is in 2011 uit het monitoringsprogramma geschrapt omdat deze beschadigd was en niet langer bruikbaar was;
- In 2012 zijn drie peilbuizen (P300, P5 en U11) geschrapt uit het monitoringsprogramma omdat de concentraties ter hoogte van deze peilbuizen stabiel waren of een dalende trend vertoonden;
- Om een beeld te krijgen van de verontreinigingstoestand stroomafwaarts van de bronzone zijn in 2012 twee nieuwe peilbuizen geplaatst (P381 en P382) en is peilbuis P264 toegevoegd aan het programma;
- In overeenstemming met het conform verklaarde bodemsaneringsproject, was in 2013 de monitoringsfrequentie verlaagd naar jaarlijks. De monitoringsfrequentie van de peilbuizen P118C, P264, P265C, P381 en P382 was in 2013 halfjaarlijks;
- De oorzaak en stijging van de PFBS concentraties ter hoogte van de bronzone Gebouw 16 diende verder onderzocht en jaarlijks opgevolgd te worden;
- Sinds 2014 is de monitoringsfrequentie van peilbuizen P371 en P374 eveneens halfjaarlijks om een stijgende trend voor bepaalde FC's beter te kunnen opvolgen. Peilbuis K1 is sinds 2014 vervangen door peilbuis P27, omdat de concentraties ter hoogte van K1 sinds 2008 stabiel en laag zijn;
- 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 2015 is de monitoringsfrequentie van de peilbuizen P27, P21B, P304, P305, P42, L19, M4 en P263 verhoogd naar halfjaarlijks; en
- In 2016 is de monitoringsfrequentie voor de peilbuizen P21B (zone Gebouw 16) en P263 (zone WWTP) verhoogd naar driemaandelijks om een stijgende trend voor bepaalde FC's beter te kunnen opvolgen.

3.3.4 *Monitoring 2e aquifer*

In de loop van de jaren zijn verschillende aanpassingen aan het monitoringsprogramma gedaan, om bepaalde trends of concentratiepieken op te volgen:

- Aangezien voor de meeste peilbuizen in de bronzone in de tweede aquifer aan de saneringsdoelstelling was voldaan in 2013, was de monitoringsfrequentie in 2013 verlaagd naar jaarlijks. Voor peilbuis P321 bleef de verhoogde monitoringsfrequentie (driemaandelijks) behouden om de afwijkende resultaten uit het verleden te verifiëren;
- De monitoringsfrequentie van de peilbuizen BD24-3, D10, P121 en P118A is in 2014 aangepast naar halfjaarlijks om de mogelijk stijgende trend te verifiëren;
- In 2015 is de monitoringsfrequentie voor de peilbuizen D10 en P118A verlaagd naar jaarlijks en voor peilbuis D09 verhoogd naar halfjaarlijks;
- In 2016 zijn de peilbuizen P121, BD24-3 en D09 driemaandelijks bemonsterd en peilbuizen P118B en D10 zijn halfjaarlijks bemonsterd;
- In juli 2016 zijn de peilbuizen D2, D5, ND7, D16, D17, D18, P119A, P119B aan het monitoringsprogramma toegevoegd, als bijkomende controle van de grondwaterkwaliteit van de 2e aquifer. Deze peilbuizen worden halfjaarlijks (D2 jaarlijks) bemonsterd; en
- In 2017 is de monitoringsfrequentie voor de peilbuizen D11, D14, P118A verhoogd naar halfjaarlijks.

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 een periodieke 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 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.

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 januari 2017 is ter vervanging van het ontoegankelijk staalnamepunt "Blokkersdijk oost" een nieuw staalnamepunt "Blokkersdijk noord" vastgelegd, dat driemaandelijk bemonsterd wordt.

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 worden in overleg met en akkoord van OVAM enkel nog de data van het 3M Environmental laboratorium in de Verenigde Staten gebruikt, met controle analyses door SGS nv.

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. Peilbuis B3 is in oktober 2015 vervangen door peilbuis B3bis.

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).

Alle peilbuizen in deze zone zijn na akkoord van OVAM definitief buitengebruik gesteld door Servaco conform de wettelijke voorschriften onder begeleiding van ERM op 8 december 2015 en 23 februari 2016.

3.3.10 Vuilvracht richting de Schelde

Geen wijzigingen.

3.3.11 Monitoringsprogramma 2017

Op basis van de resultaten uit de periode juli 2011 tot en met juli 2016 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 2017, met verantwoording. Voor meer details wordt verwezen naar het zevende TTV(9).

Tabel 3.4 *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 stabiliteits-deskundige)	-	-	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	-	
	4	Halfjaarlijks	P27, P42, P304, P305	-	
	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	-	
	1	Driemaandelijks	P263		-
Tweede aquifer	2	Jaarlijks	BD24-4, D2	-	
	3	Halfjaarlijks	D11, D14, P118A	-	
	2	Halfjaarlijks	P118B en D10	-	Potentieel stijgende concentratietrend voor FC flux naar 2e aquifer opvolgen door halfjaarlijkse monitoring
	7	Halfjaarlijks	D16, D17, D18, D5, ND7, P119A en P119B	Bijkomende staalnames om potentieel stijgende	Peilbuizen zijn in juli 2016 bemonsterd geweest. Bijkomende

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
				concentratietrend voor FC flux naar 2de aquifer beter te begrijpen.	monitoring nodig om concentratietrend te begrijpen.
	4	Driemaandelijks	P321, D9, BD24-3, P121	-	D9, BD24-3, P121 waren initieel voorzien om halfjaarlijks te bemonsteren, maar om de potentieel stijgende concentratietrend voor FC flux naar 2e aquifer op te volgen worden deze peilbuizen driemaandelijks bemonsterd.
Zuidelijke terreingrens	7	Halfjaarlijks	B3-bis, B7, P372, P378, PA109A, PA111A, PA112	-	
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 noord	Bemonsteringslocatie Oost is vervangen door het beter toegankelijke Blokkersdijk Noord	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	Wekelijks	Collector put	-	Driemaandelijkse verificatie van de stalen door SGS.
Effluent WWTP	1	Wekelijks	Lozingspijp	-	Driemaandelijkse verificatie van de stalen door SGS.

Legende

Verhoogde monitoringsfrequentie

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.

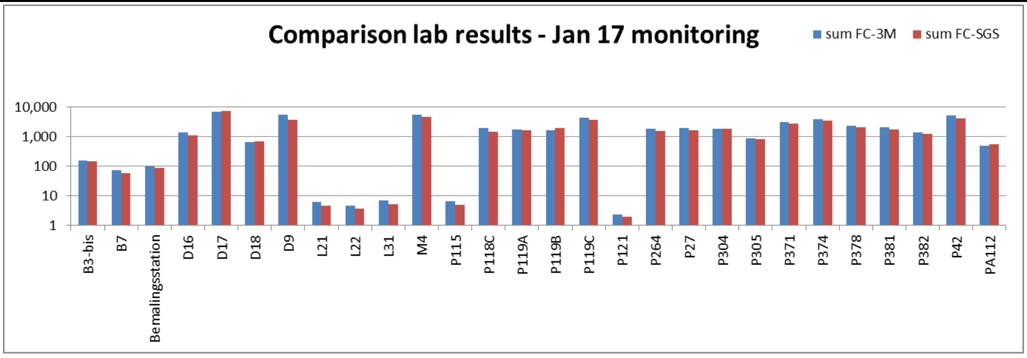
De stalen van alle monitoringsrondes zijn geanalyseerd door het 3M Environmental Laboratory en alle stalen zijn minstens éénmaal door het labo SGS nv geanalyseerd ter controle. De controle staalname en analyses zijn gespreid over de vier monitoringsperiodes (in plaats van één controle tijdens de monitoring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

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

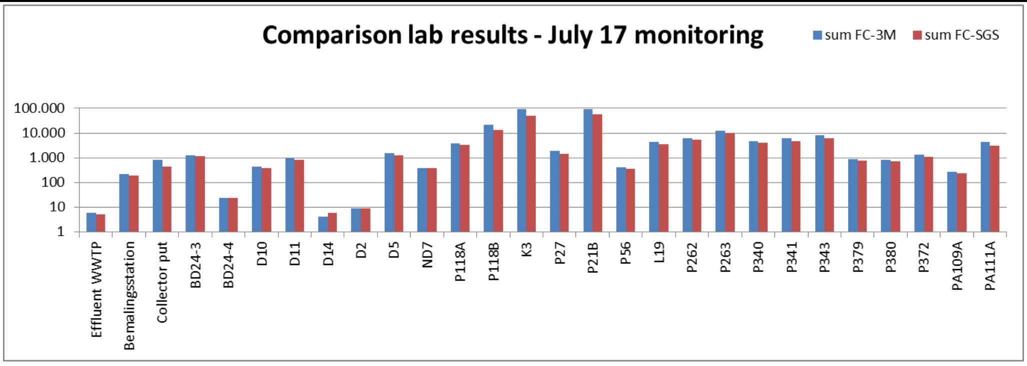
Uit een evaluatie van de datasets voor desbetreffende periode (SGS vs. 3M labo) volgt dat de resultaten van beide labo's in de meeste gevallen vergelijkbaar zijn.

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.

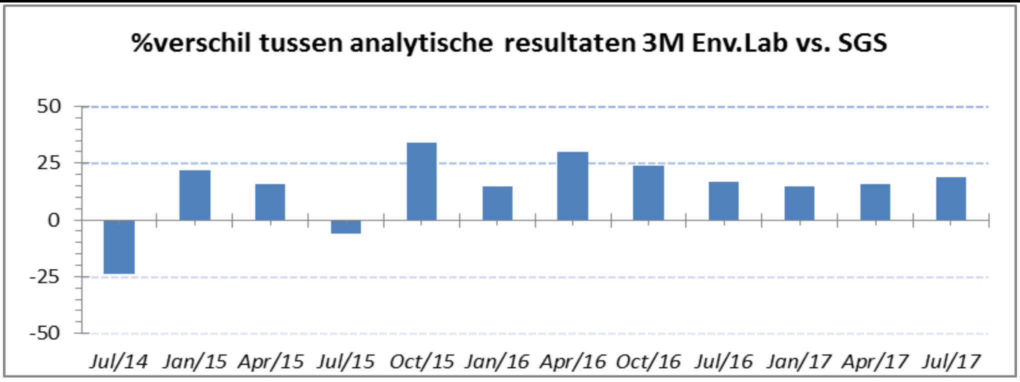
Grafiek 4.1 Vergelijking som FC resultaten SGS vs 3M Environmental Laboratory 2017



Vervolg Grafiek 4.1



Grafiek 4.2 *Vergelijking som FC resultaten SGS vs 3M Environmental Laboratory 2014-2017*



ERM stelt voor dat in de eerstvolgende periode (augustus 2017 – juli 2018) opnieuw alle waterstalen geanalyseerd worden door het 3M Environmental Laboratory, en dat de duplicaatstaalnames verdeeld worden 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 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.

Op vraag van OVAM d.d. 13 april 2016 is nog een tussentijds controle van het afdek uitgevoerd op 26 april 2016. Aangezien de afdekking op dat moment in goede staat was, zal de volgende controle in 2018 worden uitgevoerd.

4.2.2 *Uitgevoerde werken*

In de periode juli 2016 - juli 2017 heeft conform het laatste tussentijds verslag geen visuele inspectie van de afdekking plaatsgevonden.

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 16 en zone WWTP) met een zekere periodiciteit door ERM bemonsterd.

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

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

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum okt 2016	jan 2017	apr 2017	juli 2017	Analyse
Zone Gebouw 16						
K3	3,6-5,6				X	FC inclusief PFBS
P21B	3,5-5,5	X	X	X	X	FC inclusief PFBS
P27	0,9-2,9		X		X	FC inclusief PFBS
P304	4,0-6,0		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	X	FC
P264	5,0-5,5		X		X	FC
P265B	2,8-3,8				Niet*	FC
P265C	4,5-5,5		Niet *		Niet *	FC
P340	4,0-6,0				X	FC
P341	3,6-5,6				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 in juli 2017 bleek dat ook peilbuis P265B niet bemonsterd kon worden.

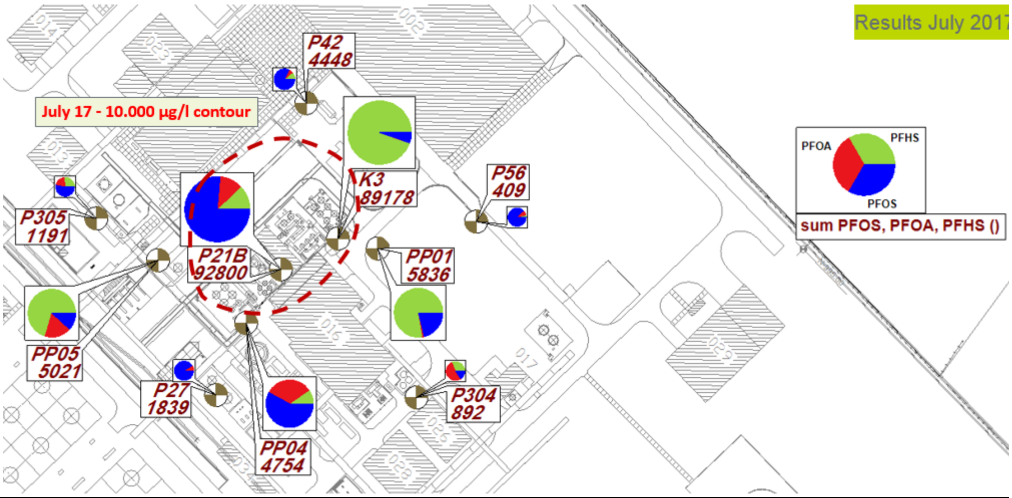
4.3.2 *Bespreking resultaten*

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

4.3.2.1 *Gebouw 16*

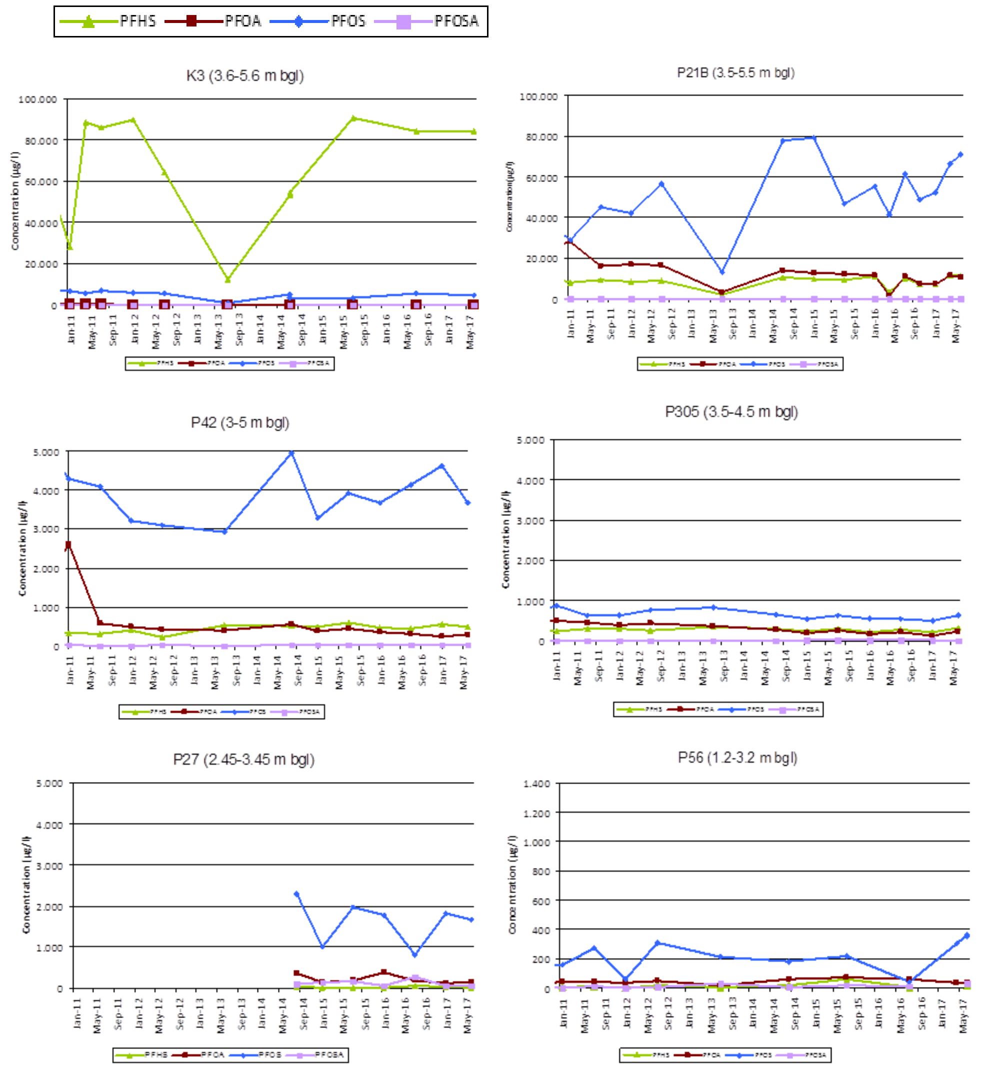
De grondwateranalyseresultaten voor de som van PFOS, PFHS en PFOA van juli 2017 ter hoogte van gebouw 16 zijn op de volgende figuur voorgesteld. De rode lijn stelt de contour van 10.000 µg/L voor de som van PFOS, PFHS en PFOA voor.

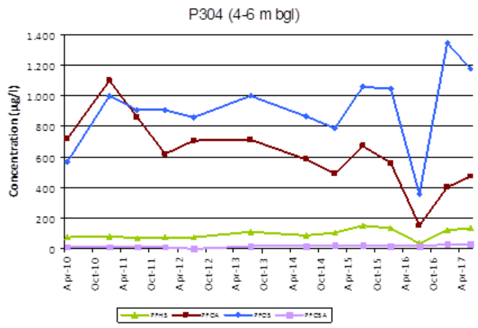
Figuur 4.1 *Schematische voorstelling FC-concentraties in grondwater thv. gebouw 16*



De evolutie van de concentraties aan FC's in het grondwater ter hoogte van gebouw 16 is voorgesteld in onderstaande grafieken.

Grafiek 4.3 *Evolutie FC concentraties t.h.v. gebouw 16*



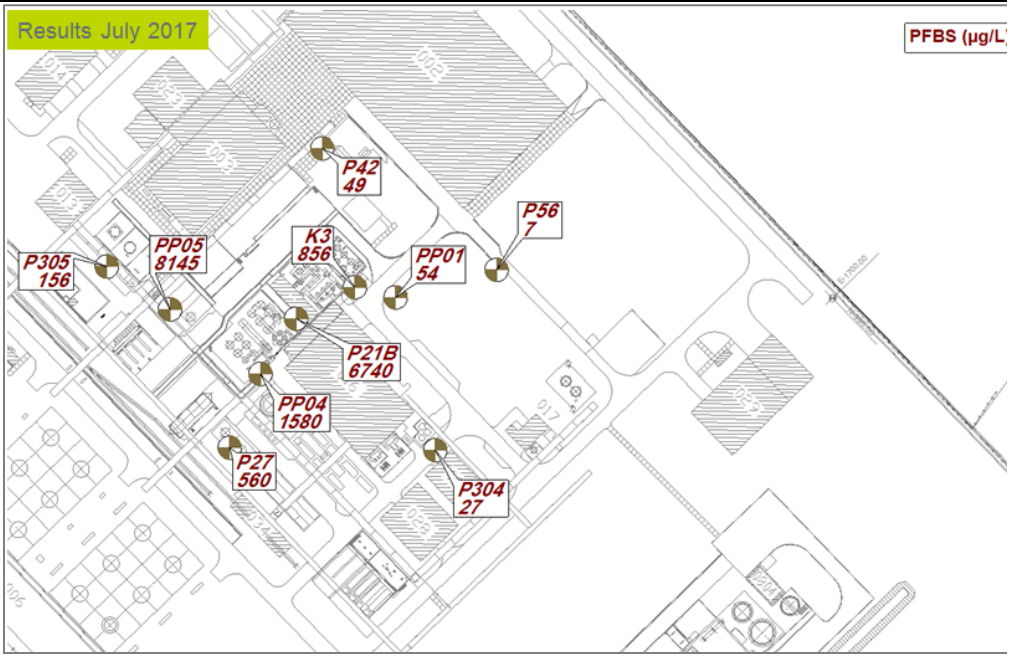


In het algemeen kan gesteld worden dat de FC concentraties in het grondwater ter hoogte van gebouw 16 dalen of stabiel zijn in de tijd, maar de concentraties fluctueren. De concentratie aan PFOS ter hoogte van peilbuis P21B lijkt over het algemeen een licht stijgende trend te vertonen. Deze peilbuis 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 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.

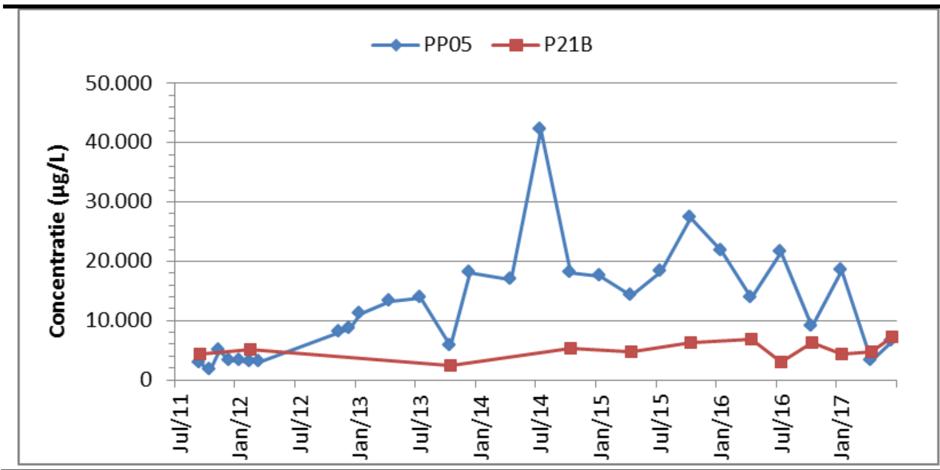
De resultaten voor PFBS van juli 2017 in het grondwater ter hoogte van gebouw 16 zijn op de volgende figuur voorgesteld. De hoogste concentraties worden gemeten ter hoogte van pompput PP05 en peilbuis P21B.

Figuur 4.2 ***Schematische voorstelling PFBSconcentraties 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 4.4 ***Evolutie PFBS concentraties t.h.v. PP05 en P21B (Gebouw 16)***

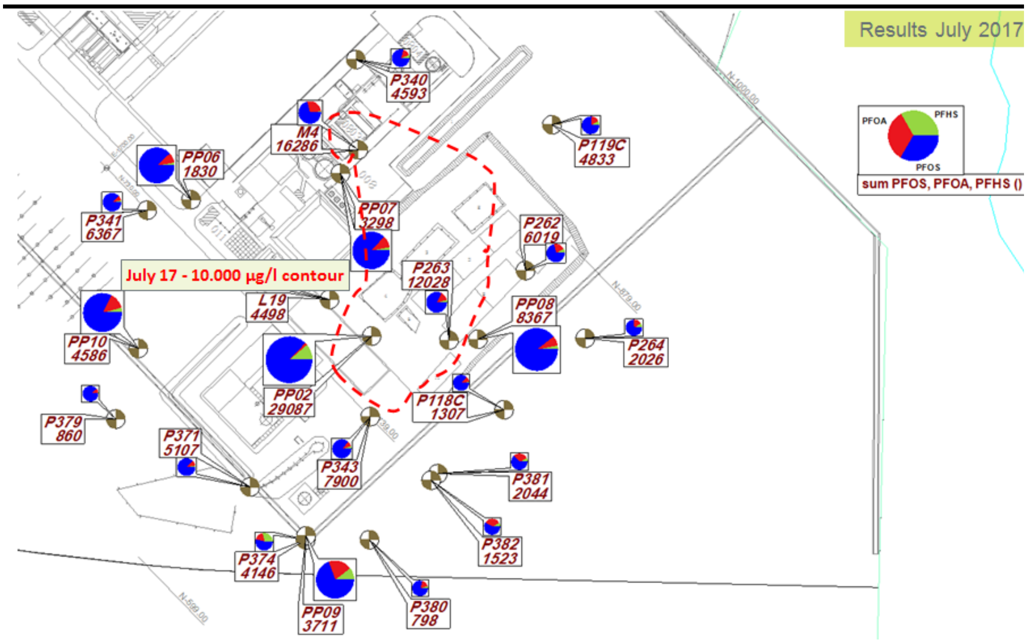


Uit de grafiek blijkt dat de concentratie in pompput PP05 opnieuw verder daalt. De oorzaak van de verhoogde PFBS detectie gemeten in 2014 is niet gekend en wordt verder opgevolgd.

4.3.2.2 *Zone WWTP*

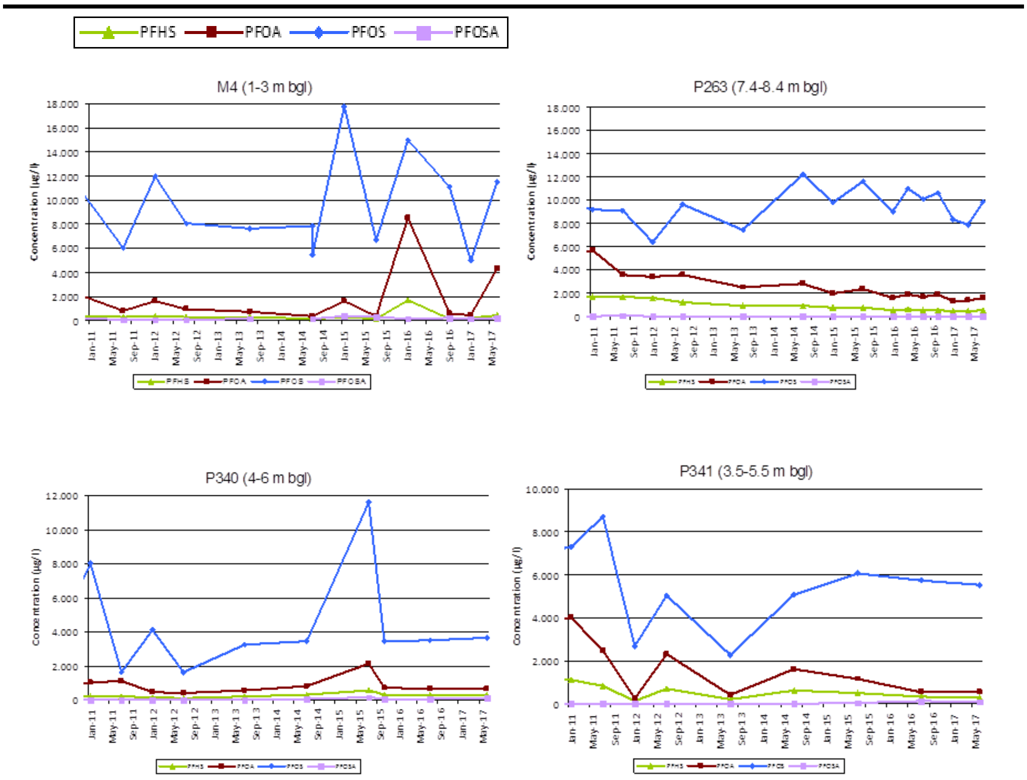
De grondwateranalyseresultaten voor de som van PFOS, PFHS en PFOA van juli 2017 ter hoogte van de WWTP zijn op de volgende figuur voorgesteld. De rode lijn stelt de concentratiecontour van 10.000 µg/L voor de som van PFOS, PFHS en PFOA voor.

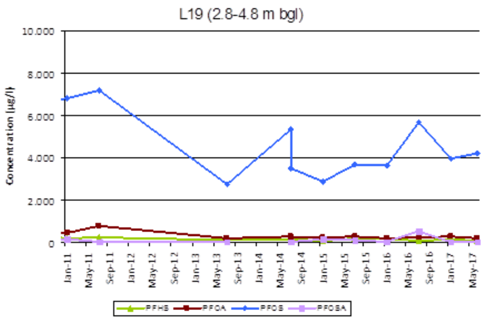
Figuur 4.3 Schematische voorstelling FC-concentraties in grondwater thv. WWTP

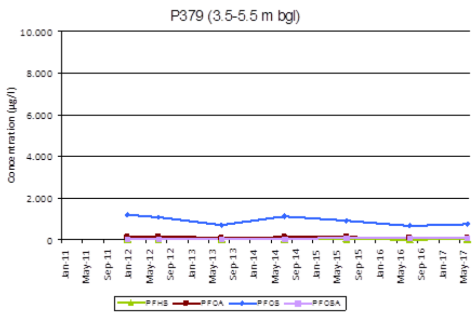


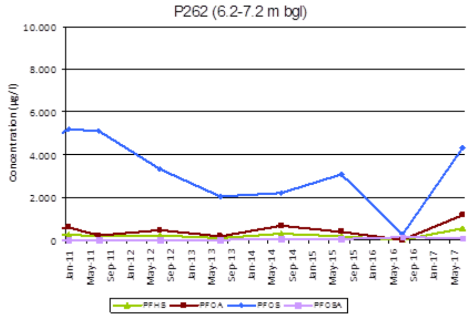
De evolutie van de concentraties aan PFOS, PFHS, PFOA en PFOSA in het grondwater ter hoogte van zone WWTP is voorgesteld in de volgende grafieken.

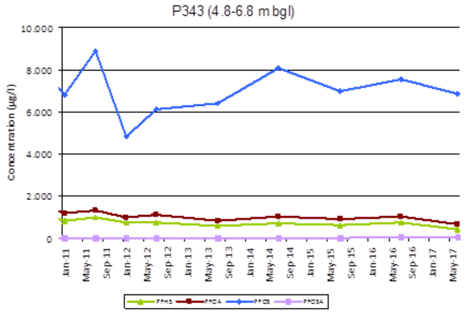
Grafiek 4.5 Evolutie FC concentraties t.h.v. zone WWTP

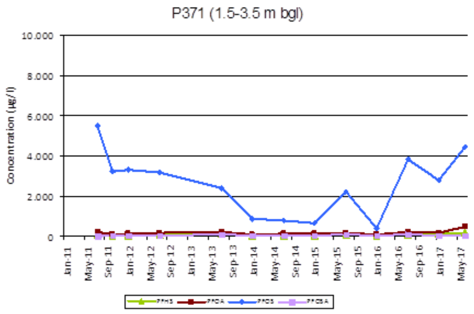


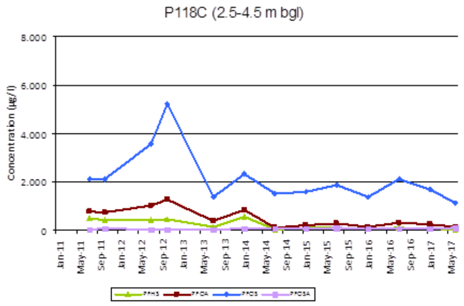

L19 (2.8-4.8 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA

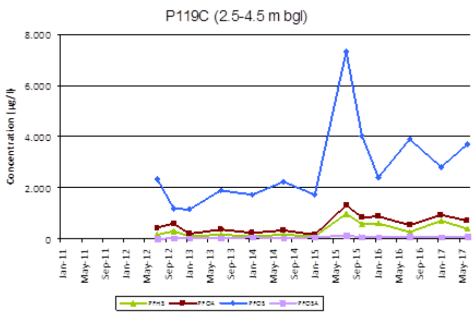

P379 (3.5-5.5 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA

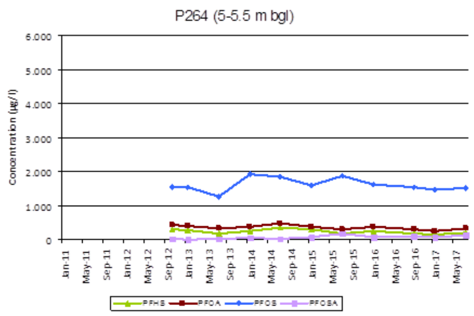

P262 (6.2-7.2 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA

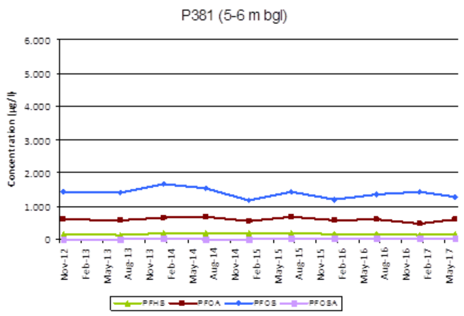

P343 (4.8-6.8 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA

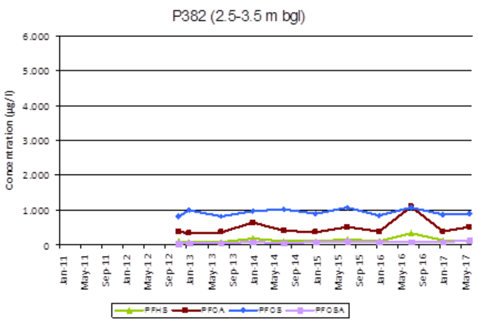

P371 (1.5-3.5 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA

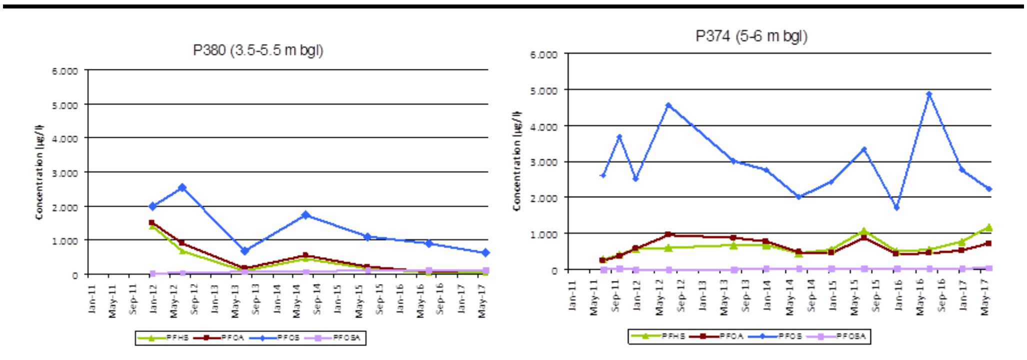

P118C (2.5-4.5 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA


P119C (2.5-4.5 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA


P264 (5-5.5 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA


P381 (5-6 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA


P382 (2.5-3.5 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA



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 P119C en P371 kan een stijgende PFOS concentratie worden waargenomen. De lichte concentratiestijging voor PFOS in het grondwater ter hoogte van peilbuis P263 die in het TTV7 besproken was, is niet bevestigd.

De som FC concentratiecontour (10.000 µg/L) is in omvang vergelijkbaar met de contour van de vorige monitoringsperiodes.

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 2018**.

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 Environmental lab en SGS geanalyseerd op FC verbindingen.

Tabel 4.2 *Overzicht bemonsterde peilbuizen tweede aquifer*

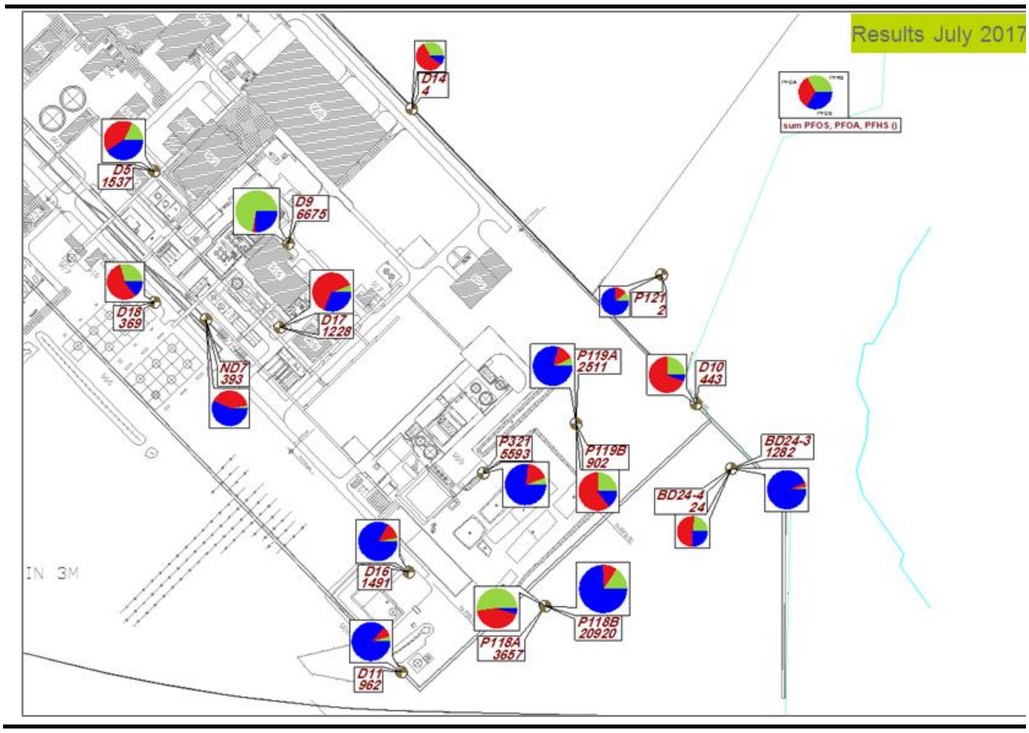
Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum Okt. 2016	Jan. 2017	Apr. 2017	Jul. 2017	Analyse
Zone Gebouw 16						
D9	13,0-15,0	X	X	X	X	FC
D14	15,5-16,5		X		X	FC
D17	15-16		X		X	FC
D18	14,0-15,0		X		X	FC
D5	15,0-16,0		X		X	FC
ND7	15,0-16,0		X		X	FC
Zone WWTP						
D10	15,0-16,0		X		X	FC
D11	14,9-15,9		X		X	FC
BD24-3	17,0-18,0	X	X	X	X	FC
BD24-4	22,0-24,0				X	FC
P121	23,5-24,5	X	X	X	X	FC
P321	15,5-15,6	X	X	X	X	FC
D16	14,0-15,0		X		X	FC
D2	24,3-25,3				X	FC
P118A	22,5-23,5		X		X	FC
P118B	13,0-14,0		X		X	FC
P119A	23,5-24,5		X		X	FC
P119B	13,0-14,0		X		X	FC

4.4.2 *Bespreking resultaten*

De analyseverslagen zijn toegevoegd in Bijlage 3. Een tabel 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 2017 in de tweede aquifer zijn op de onderstaande figuur voorgesteld.

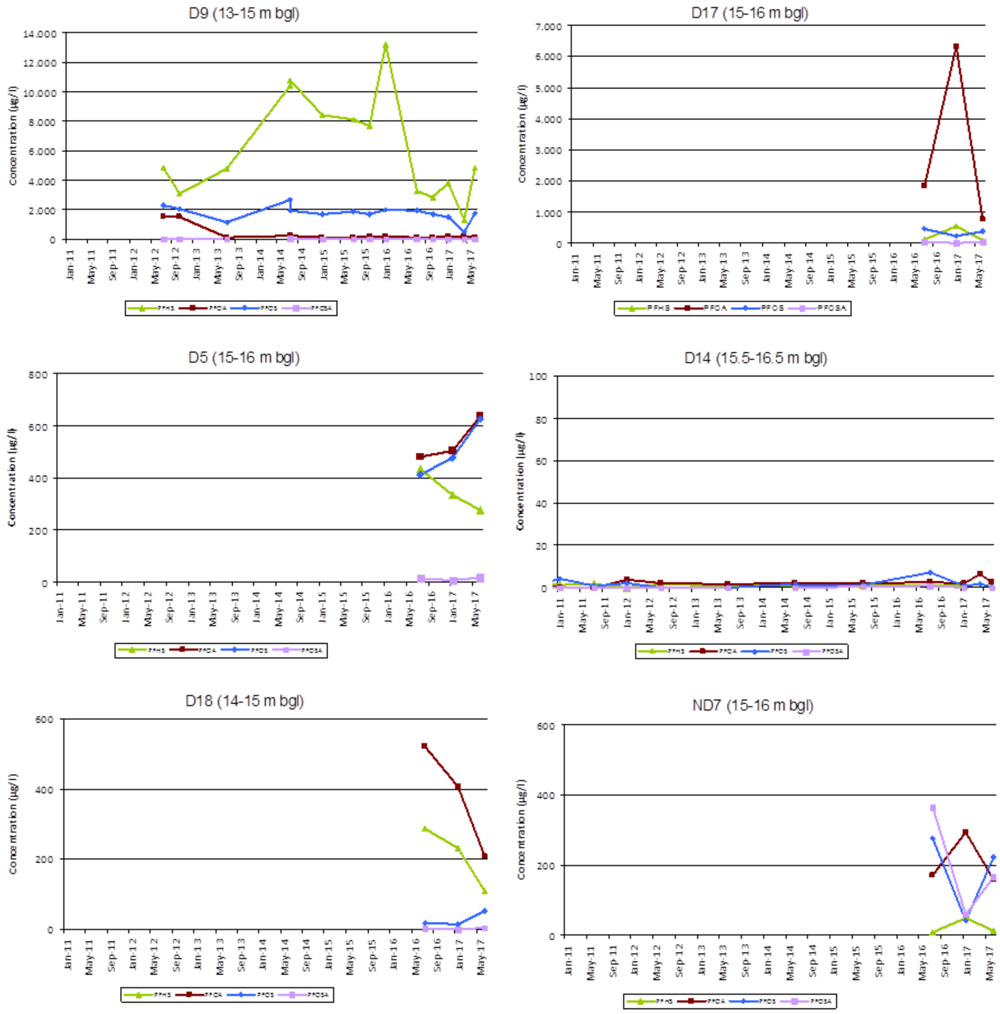
Figuur 4.4 Schematische voorstelling FC-concentraties in grondwater tweede aquifer



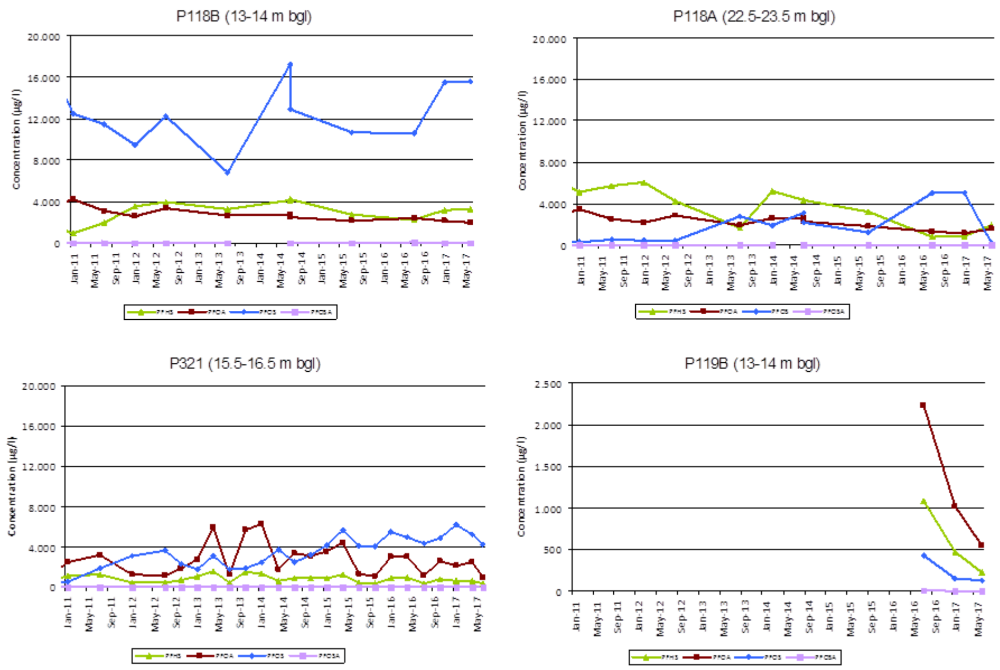
De evolutie van de concentraties aan PFOS, PFHS, PFOA en PFOSA in het grondwater van de tweede aquifer is voorgesteld in de volgende grafieken.

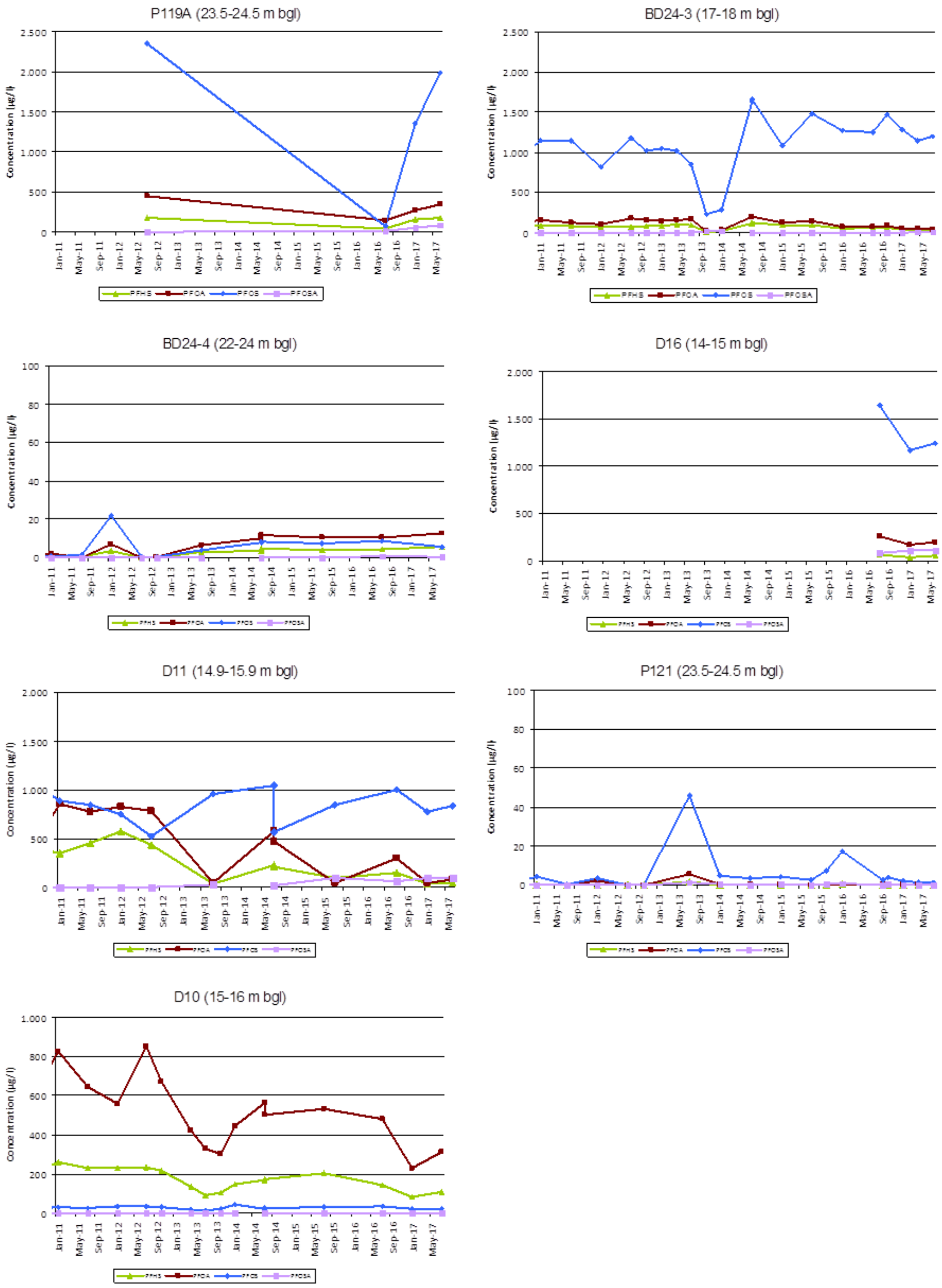
Grafiek 4.6 Evolutie FC concentraties in tweede aquifer t.h.v. Gebouw 16 en zone WWTP

Bronzone Gebouw 16



Bronzone WWTP





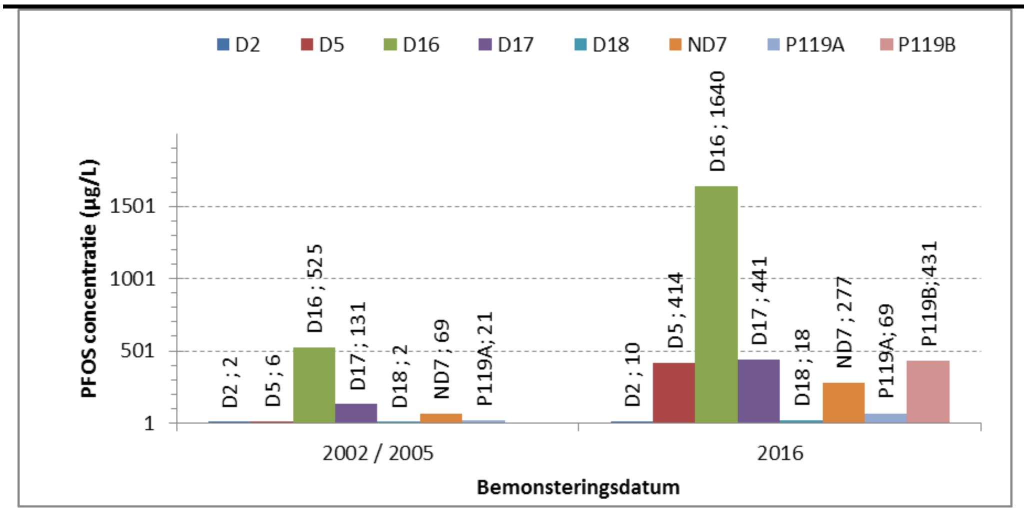
Ter hoogte van de WWTP toont de PFOS concentratie in het grondwater van peilbuis P321 een stijgende trend. De gemeten concentratie in juli 2017 (4.280 µg/l) is lager dan vijfmaal de concentratie die gemeten is bij opstart van de sanering (1.900 µg/l in juli 2011).

De concentratietrend ter hoogte van de peilbuizen BD24-3, D10 en P118A was in 2016 eerder als stijgend geëvalueerd. De meest recente analyseresultaten tonen terug een dalende tot stabiele concentratietrend. In de overige peilbuizen lijken de concentraties aan FC verbindingen in het grondwater stabiel te zijn in de tijd.

Ter hoogte van gebouw 16 is in januari 2016 een verhoogde concentratie aan PFHS in grondwater van peilbuis D9 gemeten. Deze verhoogde concentratie is niet bevestigd tijdens de volgende metingen.

In juli 2016 zijn een aantal bijkomende 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 in 2016 (beperkt) hogere concentraties gemeten voor een of meerdere organofluorverbindingen vergeleken met data uit de periode 2002-2005 (zie grafiek hieronder).

Grafiek 4.7 Evolutie FC concentraties in tweede aquifer t.h.v. Gebouw 16 en zone WWTP



Deze peilbuizen zijn opnieuw bemonsterd in januari en juli 2017. De dataset van de grondwaterconcentraties in deze peilbuizen met een filter in de tweede aquifer is nog relatief beperkt en zal tijdens volgende meetronden uitgebreid worden.

4.4.3 *Conclusies en aanbevelingen*

De gemeten concentraties in de onderzochte peilbuizen in de tweede aquifer zijn lager dan het vijfvoud van de concentraties bij de opstart van de sanering (juli 2011). 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 enkele 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.

Voor de peilbuizen die in juli 2016 aan het monitoringsprogramma zijn toegevoegd, wordt geadviseerd om deze ook in 2018 verder op te nemen in het monitoringsprogramma.

De eerstvolgende volledige monitoring zal plaatsvinden **in juli 2018**.

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 locatie van deze peilbuizen is aangeduid op Kaart 4D. De stalen zijn door 3M Environmental lab en SGS geanalyseerd op FC verbindingen.

Tabel 4.3 Overzicht bemonsterde peilbuizen zuidelijke terreingrens

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum januari 2017	Bemonsteringsdatum juli 2017	Analyse
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

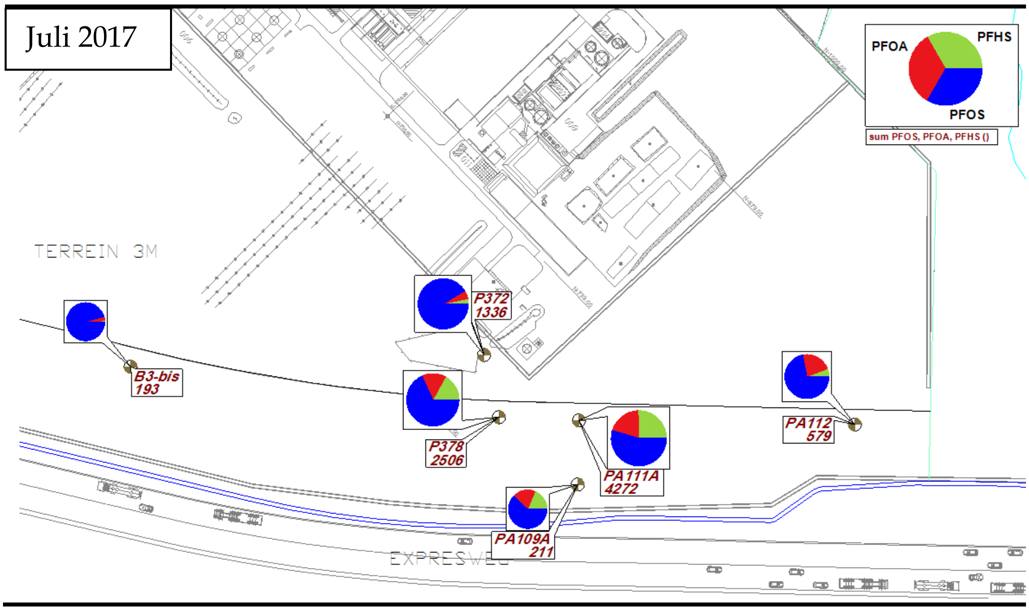
* Peilbuis B3 is vervangen door B3-bis in oktober 2015.
** Filterdiepte van B3-bis t.o.v. maaiveldniveau is aangepast in vergelijking met filterdiepte van voormalige peilbuis B3 (0,4-1,4 m-mv). Het maaiveldniveau ligt 2 meter hoger ter hoogte van de peilbuis B3-bis.

4.5.2 *Bespreking resultaten*

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

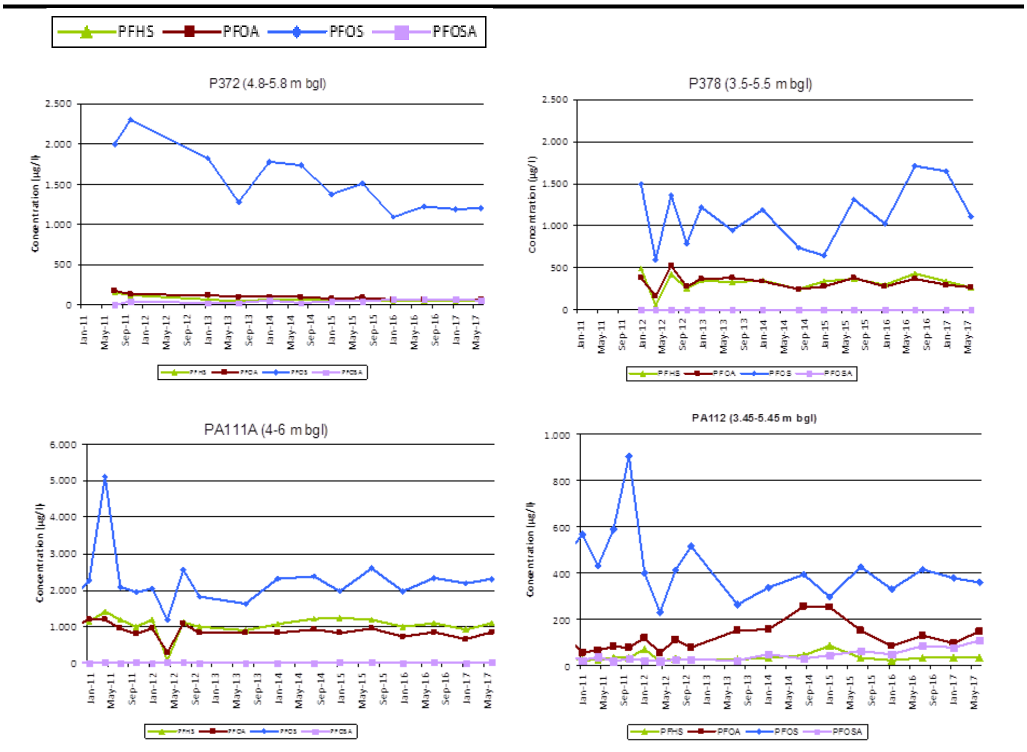
De grondwateranalyseresultaten voor de som van PFOS, PFHS en PFOA van juli 2017 ter hoogte van de zuidelijke perceelgrens zijn op de onderstaande figuur voorgesteld.

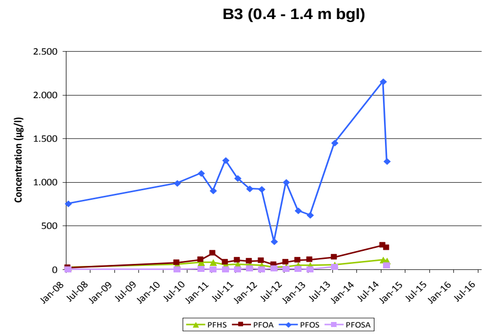
Figuur 4.5 Schematische voorstelling FC-concentraties in grondwater t.h.v. zuidelijke perceelgrens

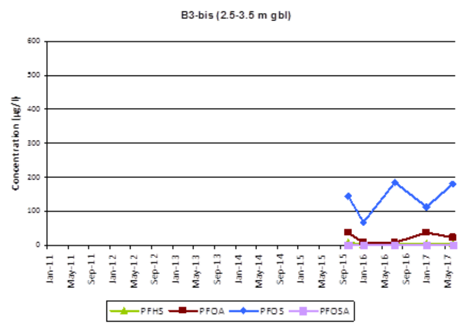


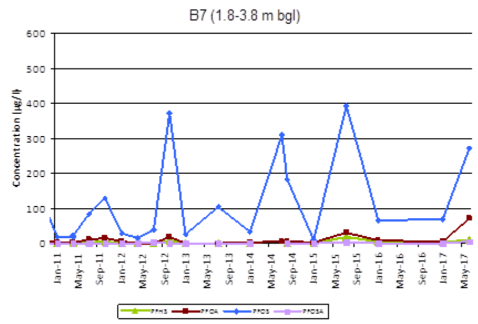
De evolutie van de FC concentraties in het grondwater ter hoogte van de zuidelijke perceelgrens is voorgesteld in onderstaande grafieken.

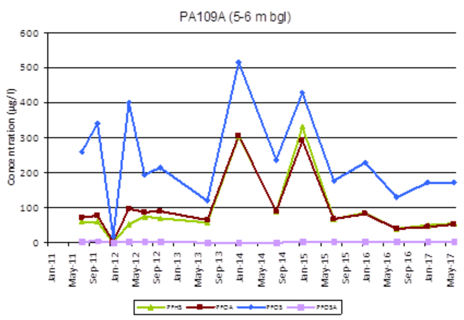
Grafiek 4.8 Evolutie FC concentraties t.h.v. zuidelijke perceelgrens











Ter hoogte van peilbuizen B7, P372, PA109A, PA111A, PA112, P378 en B3-bis vertonen de FC-concentraties in het grondwater ter hoogte van de zuidelijke perceelgrens een stabiele of dalende trend met sterke fluctuaties in de tijd.

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 2018**.

4.6 *TANKENPARK - MINERALE OLIE (P18 EN P28)*

Bij een periodieke grondwatermonitoring was ter hoogte van twee peilbuizen P18 en P28 een verhoogde concentratie aan minerale olie gemeten. De oorzaak hiervan is niet gekend. Op vraag van OVAM zijn beide peilbuizen mee opgenomen in de jaarlijkse monitoring voor analyse op minerale olie.

4.6.1 *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 2017	Analyses
P18	1,4-3,4	-*	-
P28	1,1-3,1	X	MO

*Peilbuis kon niet bemonsterd worden tijdens de monitoringsronde van juli 2017.

4.6.2 *Bespreking resultaten*

Het analyseverslag is toegevoegd in Bijlage 3.

4.6.2.1 *Veldparameters*

De pH en geleidbaarheid van het grondwater ter hoogte van peilbuis P28 zijn niet tijdens de bemonstering gemeten omdat er, omwille van sterk verhoogde waarden, een reële kans bestaat op beschadiging van de veldmeetapparatuur. De verhoogde pH en conductiviteit zijn mogelijk gerelateerd aan een schadegeval nabij de KOH tank.

4.6.2.2 *Minerale olie*

De concentratie aan minerale olie in het grondwater ter hoogte van peilbuis P28 was in juli 2017 kleiner dan 500 µg/L. Dit is lager dan de concentraties gemeten tijdens voorgaande monitoringsronden, alsook lager dan de bodemsaneringsnorm van 500 µg/L.

4.6.3 *Conclusies en aanbevelingen*

De verontreiniging met minerale olie, pH en geleidbaarheid is op basis van analyses afgeperkt en gelokaliseerd rond peilbuizen P18 en P28.
De concentratie aan minerale olie lijkt sterk te fluctueren doorheen de tijd. Momenteel kan geen verklaring voor de gemeten verhoogde waarden aan minerale olie in het grondwater tijdens de voorgaande monitoringsronden 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 2018**.

4.7 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.

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

4.7.1 *Monitoring grond- en oppervlaktewater*

4.7.1.1 *Uitgevoerd veldwerk*

De peilbuizen langs het 3M pad ten westen van het natuurgebied Blokkersdijk en het oppervlaktewater van de 3M vijver en de Blokkersdijkvijver worden driemaandelijks bemonsterd om de evolutie in FC concentraties in deze zones na te gaan. Sinds januari 2017 wordt ook een staal genomen in het noorden van de Blokkersdijkvijver 'Blokkersdijkvijver Noord'. Gezien de omvang van de Blokkersdijkvijver is het meer representatief om het oppervlaktewater op 2 locaties te bemonsteren, nu er weer meer toegang tot is.

In onderstaande tabel is een overzicht gegeven van de grondwater en oppervlaktewater monitoring. 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 het 3M Environmental lab en SGS.

Tabel 4.5 Overzicht staalnamelocaties natuurgebied Blokkersdijk

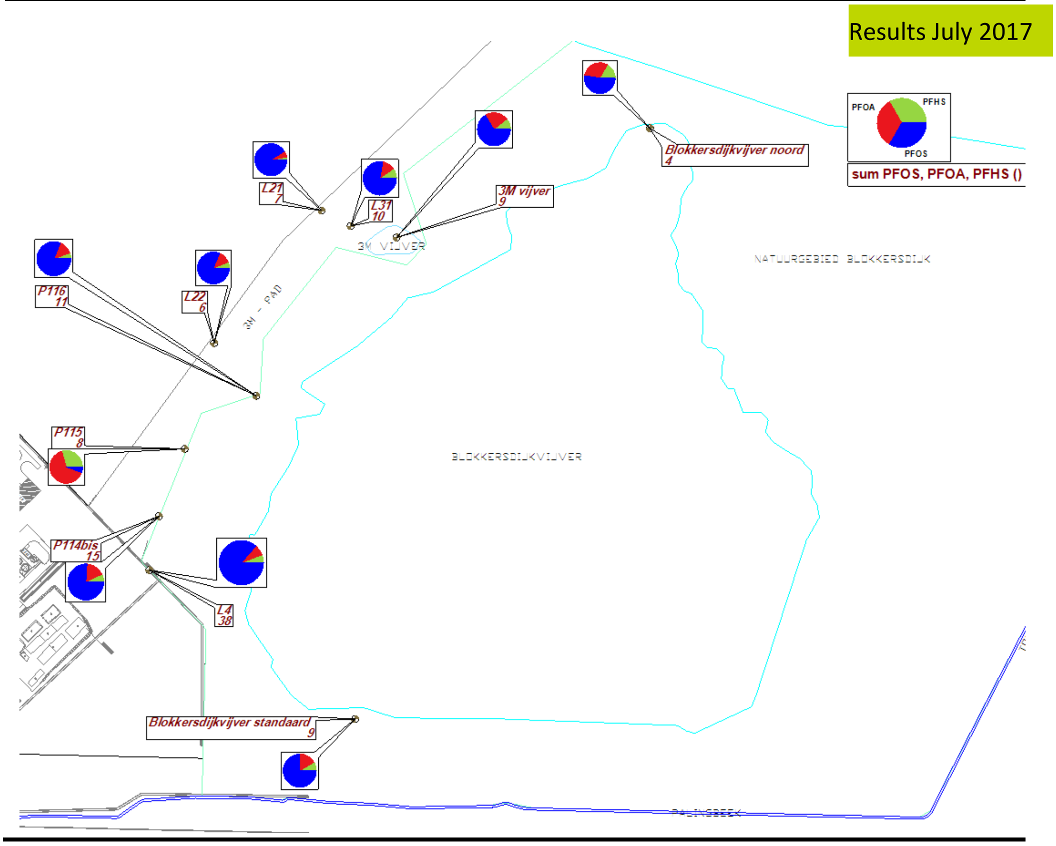
Locatie	Filterdiepte (m-mv)	Bemonsteringsdatum Okt. 2016	 Jan. 2017	 Apr. 2017	 Jul. 2017	Analyse
L21	3,0-5,0	X	X	X	X	FC
L22	2,0-4,0	X	X	X	X	FC
L31	1,2-3,2	X	X	X	X	FC
L4	2,0-4,0	X	X	X	X	FC
P114bis	3,6-4,6	X	X	X	X	FC
P115	3,3-4,3	X	X	X	X	FC
P116	2,5-3,5	X	X	X	X	FC
3M vijver	-	X	X	X	X	FC
Blokkersdijk standaard	-	X	X	X	X	FC
Blokkersdijk Noord	-		X	X	X	FC

4.7.1.2 *Resultaten*

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

Op onderstaande figuren zijn de meest recent (juli 2017) gemeten concentraties voor de som van PFOS, PFHS en PFOA in het grondwater langs de westkant van het natuurgebied Blokkersdijk en in het oppervlaktewater van de vijvers weergegeven.

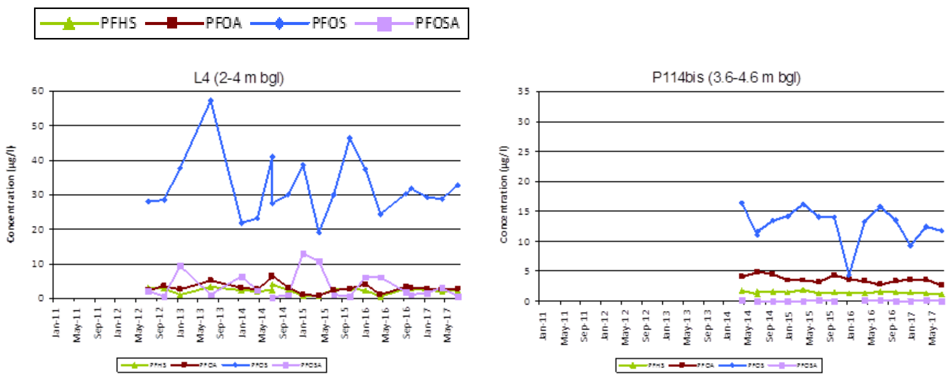
Figuur 4.6 *Schematische voorstelling FC-concentraties in grondwater en oppervlaktewater thv. natuurgebied Blokkersdijk*

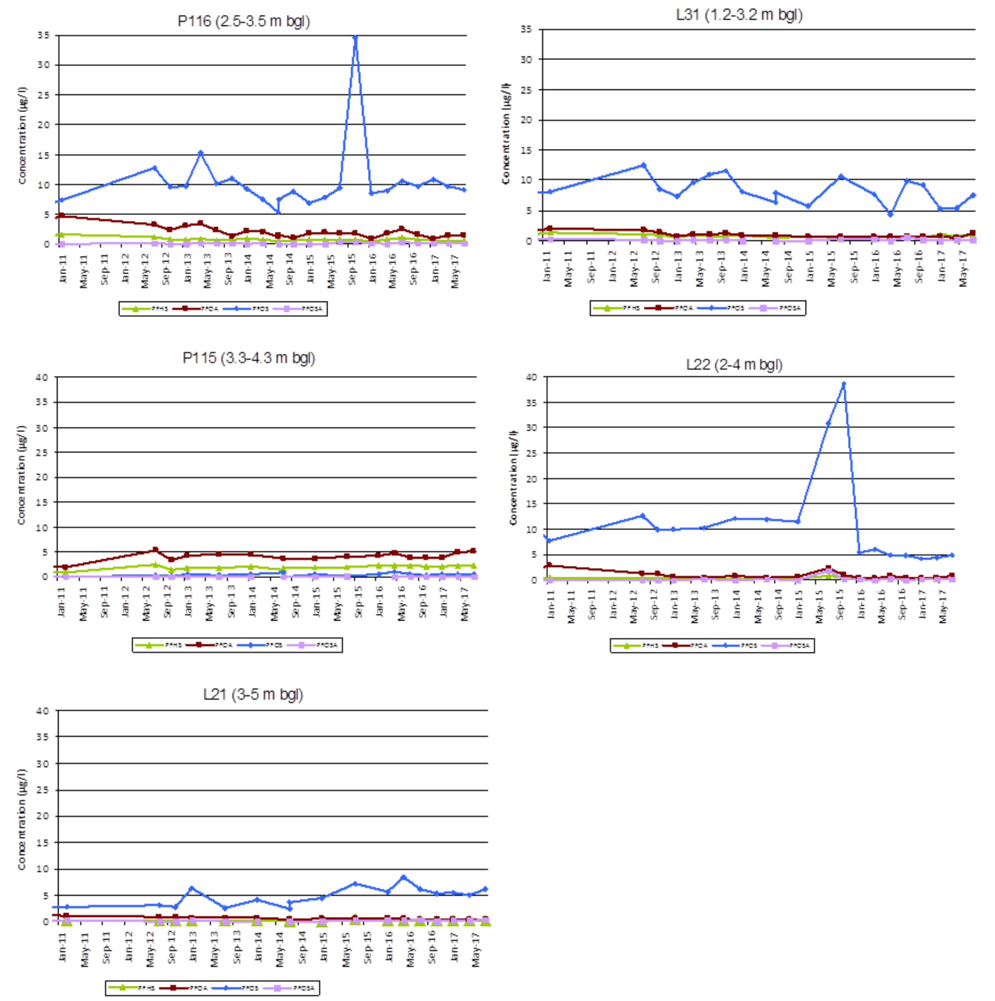


Algemeen kan worden gesteld dat de concentraties in grond- en oppervlaktewater relatief laag zijn.

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.

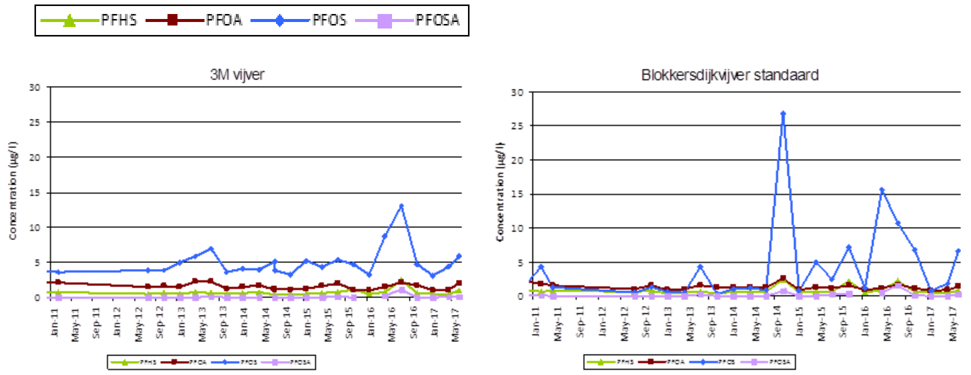
Grafiek 4.9 ***Evolutie FC concentraties in grondwater t.h.v. 3M pad (westelijk van Blokkersdijk natuurgebied)***

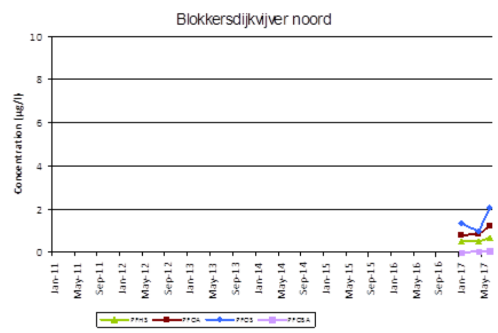




De evolutie van de FC concentraties in het oppervlaktewater van de Blokkersdijkvijver en de 3M vijver is in de onderstaande grafieken weergegeven.

Grafiek 4.10 Evolutie FC concentraties in oppervlaktewater Blokkersdijk natuurgebied





4.7.2 *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 P116 een significant stijgende trend wordt waargenomen en in peilbuis L22 een significant dalende trend. Wanneer voor beide peilbuizen enkel de laatste zeven concentraties die gemeten zijn, in beschouwing genomen worden (januari 2016-juli 2017), is er geen significante trend aangetoond.
De gemeten concentraties in de oppervlaktewaterstalen Blokkersdijk standaard geven geen statistisch significante trend weer. Voor het meetpunt Blokkersdijkvijver Noord is nog geen statische evaluatie uitgevoerd, gezien het beperkt aantal data.

4.7.3 *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 een statistische evaluatie. Voor de statistische analyse wordt enkel gewerkt met 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 P116 een significant stijgende trend wordt waargenomen en in peilbuis L22 een significant dalende trend. Indien enkel de laatste zeven gemeten concentraties in rekening worden gebracht, wordt ook voor deze peilbuizen geen significante trend aangetoond.

Momenteel is geen noodzaak voor bijkomende saneringsacties in deze zone aangezien er geen stijgende concentratietrend is waargenomen in de Blokkersdijk vijver.

Uit de resultaten van het geohydrologisch onderzoek uitgevoerd in 2014 blijkt dat 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 monitoring zal plaatsvinden **in oktober 2017**.

4.8 PALINGBEEK EN TOPHAT GRACHT

4.8.1 *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 het 3M Environmental lab en SGS geanalyseerd op FC verbindingen.

Tabel 4.6 Overzicht staalnamelocaties Palingbeek en Tophatgracht

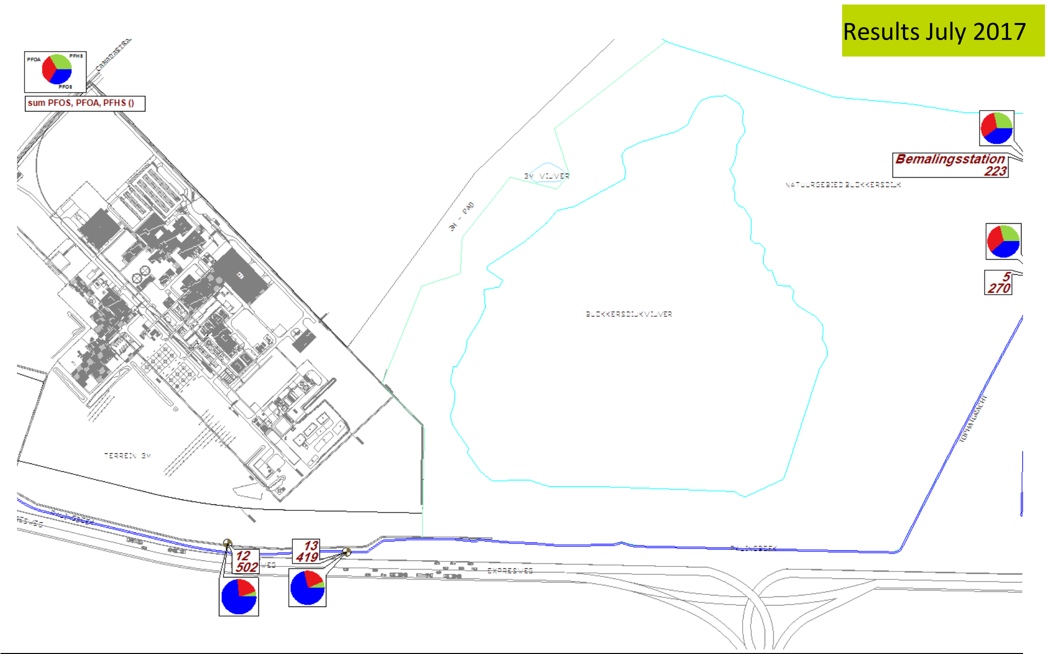
Locatie	Bemonsteringsdatum				Analyse
	Okt. 2016	Jan. 2017	Apr. 2017	Jul. 2017	
12	X	X	X	X	FC
13	X	X	X	X	FC
5	X	X	X	X	FC
Bemalingsstation	X	X	X	X	FC

4.8.2 *Bespreking resultaten*

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

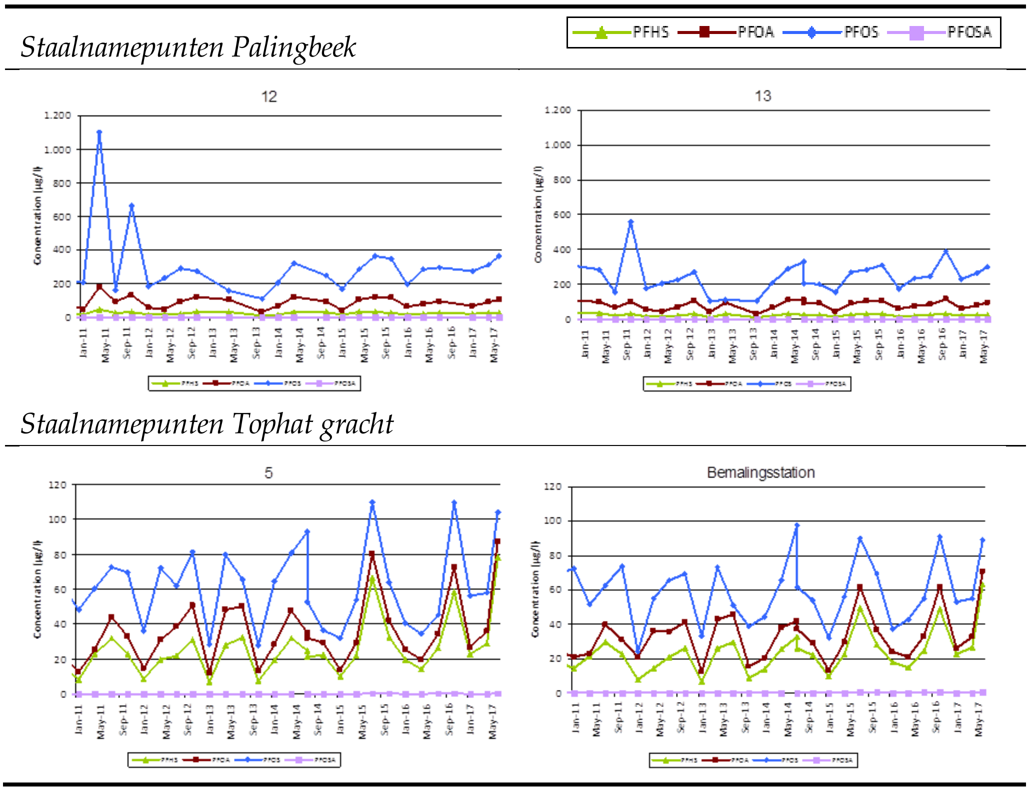
De analyseresultaten van het oppervlaktewater voor de som van PFOS, PFHS en PFOA van juli 2017 zijn op de volgende figuur voorgesteld.

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 4.11 Evolutie FC concentraties in oppervlaktewater Palingbeek en Tophat gracht

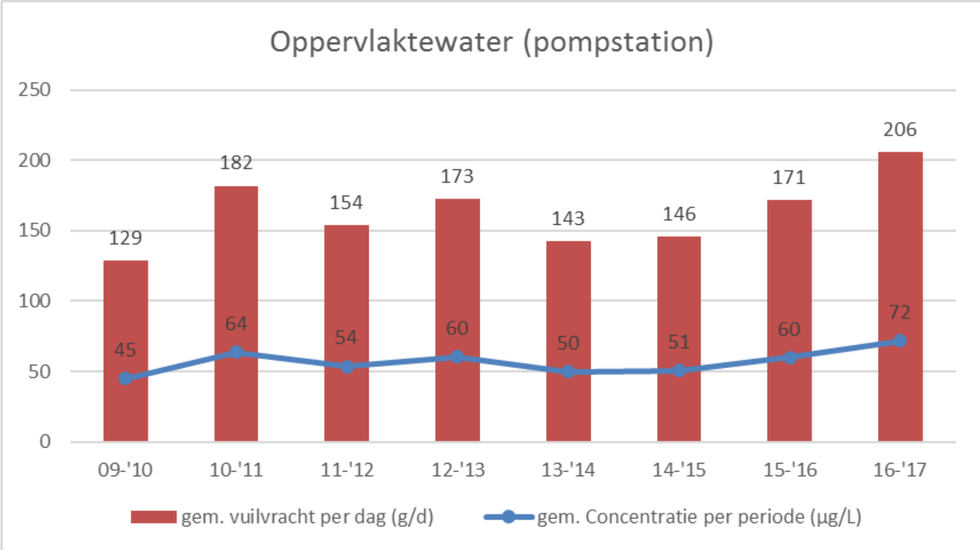


De FC-concentraties ter hoogte van de Palingbeek vertonen globaal gezien een stabiel verloop sinds 2012. De concentraties gemeten in de Tophat gracht zijn lager dan de concentraties in de Palingbeek, maar lijken licht te stijgen.

4.8.3 *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 206 g in de periode 1 augustus 2016 tot en met 31 juli 2017. In onderstaande grafiek is een overzicht gegeven van de berekende vuilvracht per jaar sinds de start van de monitoring in 2009.

Grafiek 4.12 PFOS Vuilvrachtberekening in oppervlaktewater



*Om de rapportageperiode van de grondwatermonitoring en de vuilvrachtberekening op elkaar af te stemmen, is ervoor gekozen om de periode van de vuilvrachtberekening vanaf voorliggend tussentijds rapport te laten lopen van augustus tem juli.

4.8.4 *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 stijgen licht. Momenteel is er geen verklaring voor deze lichte stijging.

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

De volgende monitoring zal plaats vinden **in oktober 2017**.

4.9 *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.

4.9.1 *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 in het labo van 3M Zwijndrecht. Ook van de bedrijfswaterzuivering worden wekelijks stalen genomen door 3M en geanalyseerd in het labo van 3M Zwijndrecht. Het debiet van de regenwatercollectorput en de bedrijfswaterzuivering wordt dagelijks door 3M opgevolgd.

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

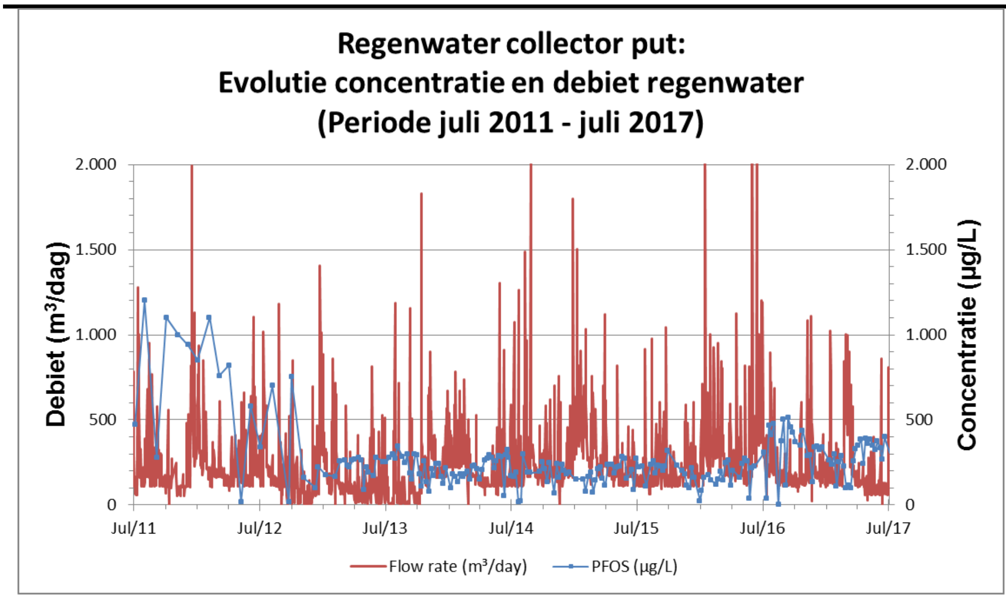
De locatie van de collectorput en de bedrijfswaterzuivering is aangeduid op Kaart 4G.

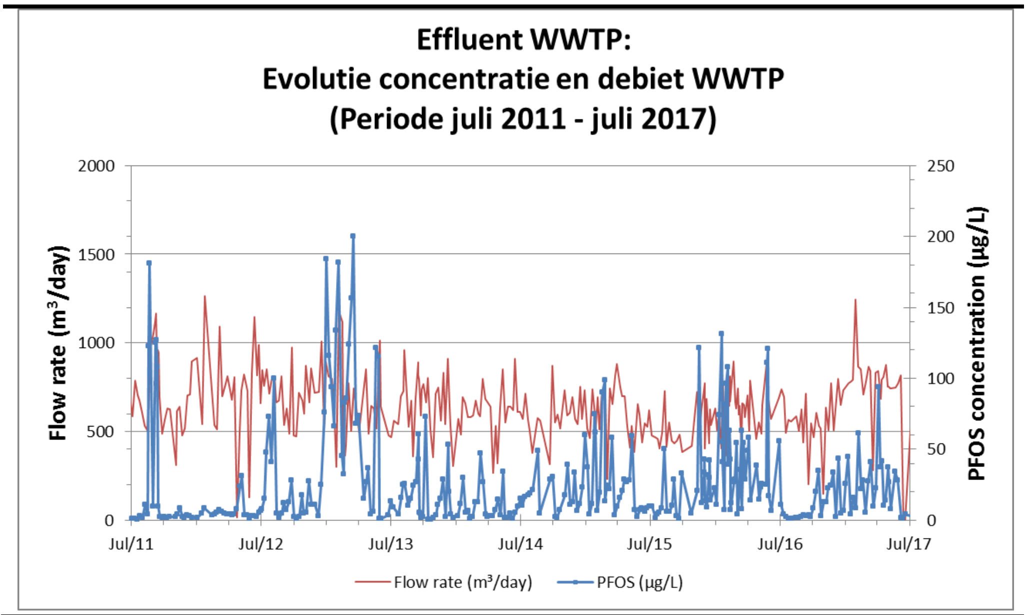
4.9.2 *Bespreking resultaten, conclusies en aanbevelingen*

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

In onderstaande grafieken is de evolutie van concentratie aan PFOS ter hoogte van de regenwatercollectorput en in het effluent van de bedrijfswaterzuivering weergegeven, in combinatie met het gemeten debiet. Onderstaande grafieken zijn gebaseerd op de wekelijkse analyseresultaten van 3M.

Grafiek 4.13 Evolutie PFOS concentratie in collectorput en bedrijfswaterzuivering

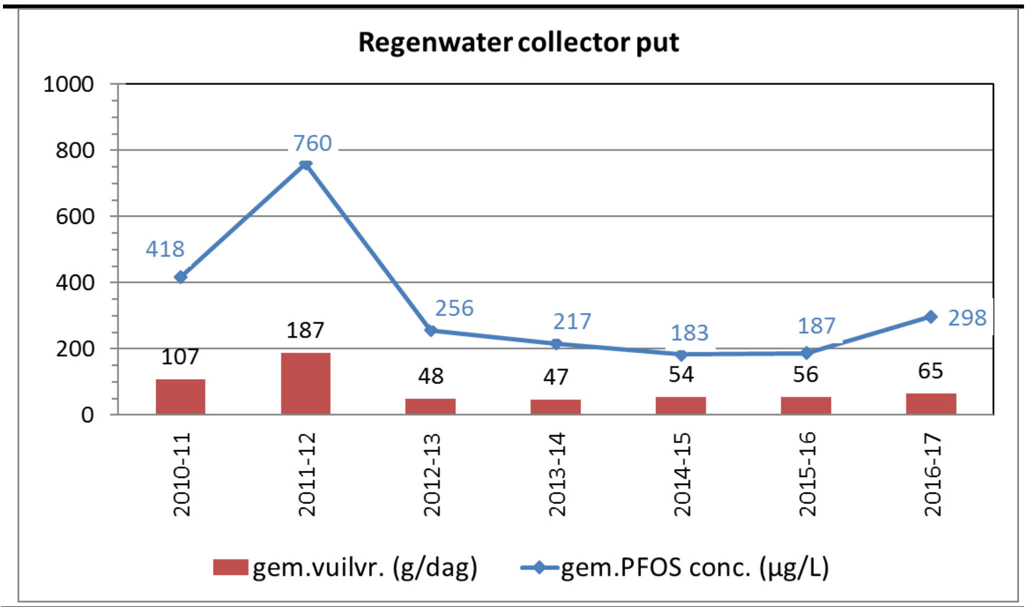


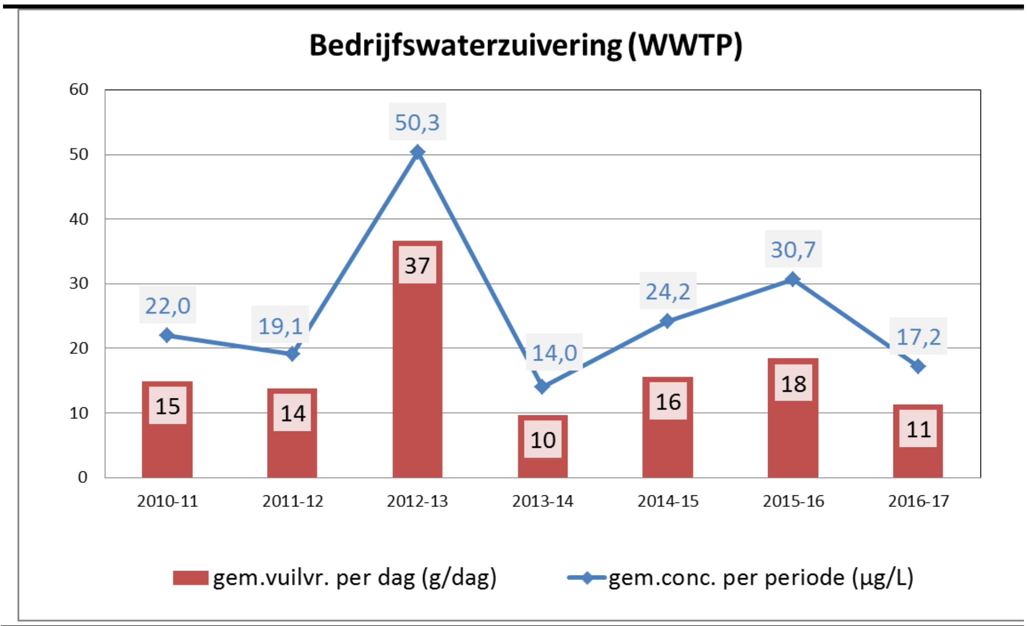


Sinds november 2012 vertonen de PFOS concentraties in de collectorput een stabiel verloop, maar kunnen echter fluctueren.
De gemiddelde PFOS concentratie in de collectorput voor de periode augustus 2016 – juli 2017 bedraagt 298 µg/L. De gemiddelde PFOS concentratie in het effluent van de bedrijfswaterzuivering in dezelfde periode bedraagt 17,2 µg/L.

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

Grafiek 4.14 PFOS Vuilvrachtberekening regenwatercollectorput en bedrijfswaterzuivering





Deze gegevens maken deel uit van de PFOS vuilvrachtberekening naar de Schelde (zie paragraaf 4.10). 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 wekelijkse opvolging van de concentraties in het effluent van de bedrijfswaterzuivering en in de collectorput door 3M te behouden, alsook de driemaandelijkse opvolging door ERM/SGS.

4.10 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 augustus 2016 tot en met 31 juli 2017 is de vuilvracht die dagelijks naar de Schelde stroomt berekend (zie Tabel 4.7).

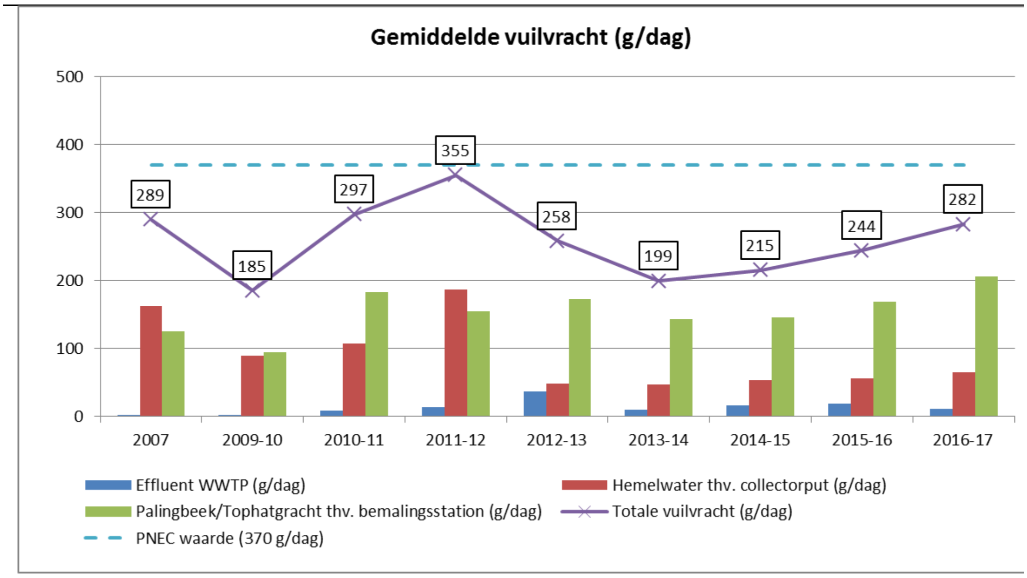
Tabel 4.7 ***Gemiddelde PFOS vuilvracht richting de Schelde (augustus 2016 tem juli 2017)***

Influx naar de Schelde	Gemiddeld debiet (m³/dag)	Gemiddelde PFOS concentratie (µg/L)	Gemiddelde vuilvracht (g/dag)
Effluent van het bedrijfsafvalwater (incl. opgepompt grondwater)	661	17	11
Regenwaterriolering	218	298	65
Palingbeek/Tophatgracht*	2865	72	206
Totale vuilvracht			**282**

* *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 4.15 ***Evolutie gemiddelde PFOS concentratie en vuilvracht naar de Schelde***



De gemiddelde vuilvracht is sinds 2013-2014 opnieuw aan het stijgen, voornamelijk door de stijgende vuilvracht in de Palingbeek en Tophat gracht. Momenteel is er geen verklaring voor de stijging. De driemaandelijkse monitoring wordt voortgezet.

Voor de periode augustus 2016 tot en met juli 2017 bedraagt de berekende totale PFOS vuilvracht naar de Schelde 282 g/dag. Dit betekent dat de berekende vuilvracht de PNEC waarde (370 g/dag) niet overschrijdt.

4.11 VOORSTEL MONITORINGSPROGRAMMA 2018

In de volgende tabel is een overzicht gegeven van het voorgestelde monitoringsprogramma voor 2018.

Tabel 4.8 Monitoringsprogramma 2018

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		Vanaf 2013 vijfjaarlijkse monitoring, aangezien bovenafdek in 2012 vervangen is; eerst volgende controle: 2018.
FC Bronzones					
Gebouw 16	2	Jaarlijks	K3, P56	-	
	4	Halfjaarlijks	P27, P42, P304, P305	-	
	1	Driemaandelijks	P21B		Stijgende concentratietrend ter hoogte van P21B.
WWTP	3	Jaarlijks	P341, P379, P380,		-
	2	Jaarlijks	P381, P382	P381 en P382: van halfjaarlijkse naar jaarlijkse monitoringsfrequentie, want relatief lage concentraties die stabiel zijn doorheen de tijd.	
	8	Halfjaarlijks	P118C, P264, P265B, P371, P374, , L19, M4, P119C	P265C (4,5-5,5 m-mv) is niet meer bruikbaar door knik in peilbuis en wordt vervangen door P265B (2,8-3,8 m-mv).	
	3	Halfjaarlijks	P262, P340, P343	Geen significante concentratietrend, fluctuerende concentraties. Peilbuizen ter afbakening van de 10.000 µg/l contour; zelfde	

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
				monitoringsfrequentie als de andere peilbuizen ter afbakening van deze contour.	
	1	Halfjaarlijks	P263	Peilbuis in kern van verontreiniging, geen significante concentratietrend. Zelfde monitoringsfrequentie als andere peilbuis in kern van verontreiniging.	
Tweede aquifer	1	Jaarlijks	D2		
	2	Halfjaarlijks	D9, BD24-3	D9 en BD24-3 zijn in 2017 driemaandelijks bemonsterd. De grondwaterconcentraties zijn tov 2016 terug gedaald of gestabiliseerd, waardoor de monitoringsfrequentie verlaagd wordt naar halfjaarlijks.	
	8	Halfjaarlijks	D16, D17, D18, D5, ND7, P121, P119A en P119B		
	1	Halfjaarlijks	BD24-4	Relatief lage concentraties; monitoringsfrequentie van peilbuizen op elkaar afstemmen	
	1	Driemaandelijks	P321	-	-
Zuidelijke terreingrens	7	Halfjaarlijks	B3-bis, B7, P372, P378, PA109A, PA111A, PA112	-	
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	-	-

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
Oppervlaktewater	3	Driemaandelijks	Blokkersdijk standaard, 3M vijver, Blokkersdijk Noord	Blokkersdijk Oost is vervangen door Blokkersdijk Noord owv betere bereikbaarheid	
Palingbeek en Tophat gracht	4	Driemaandelijks	12, 13, 5, Bemalingsstation	-	-
Regenwater	1	Wekelijks	Collector put		Driemaandelijkse verificatie van de stalen door SGS.
Effluent WWTP	1	Wekelijks	Lozingspijp		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 2016 tot juli 2017.

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 (locaties: zie Kaart 5). Er zijn drie pompputten in de zone "gebouw 16" 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	2,92
PP04	Gebouw 16	2,0-6,0	0,42
PP05	Gebouw 16	1,7-5,7	0,28
PP02	WWTP	3,2-6,2	0,70
PP06	WWTP	1,3-5,3	2,89
PP07	WWTP	2,0-6,0	5,12
PP08	WWTP	3,2-7,2	4,43
PP09	WWTP	3,0-6,0	1,77
PP10	WWTP	3,0-6,0	5,17

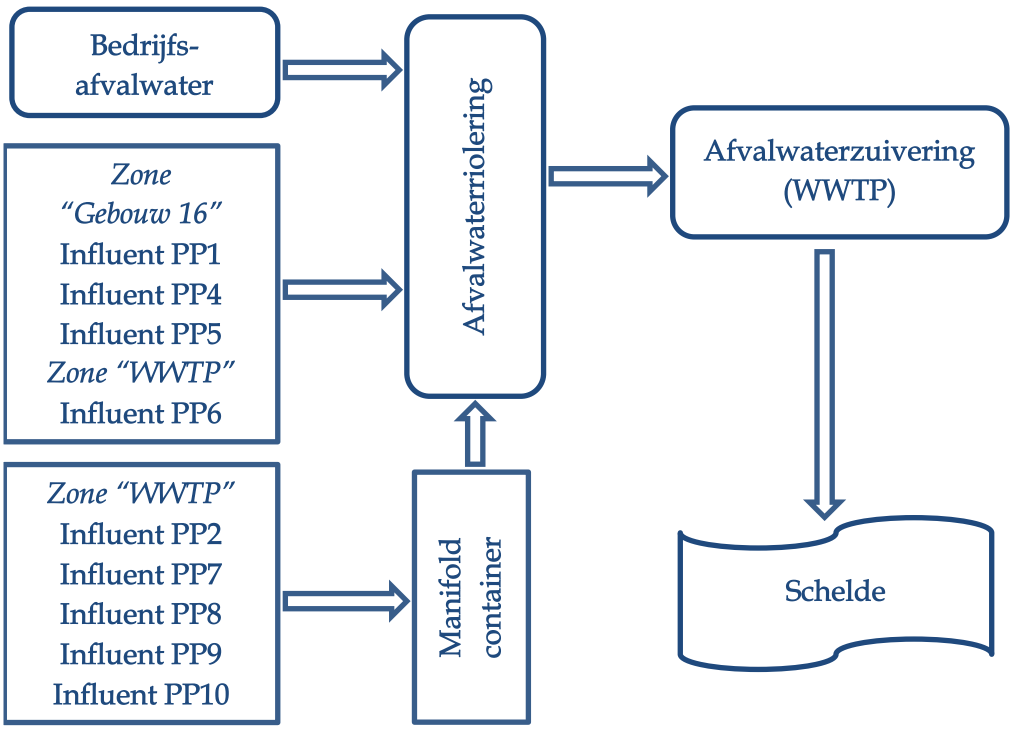
Elke pompput beschikt over een niveausensor (zie schematisch overzicht in Bijlage 6). De niveausensor bevindt zich net boven de inlaat van de pomp. De inlaat van de pomp mag nooit droog komen, daarom wordt steeds een minimale waterkolom van 20 cm aangehouden. De onttrekking in de betreffende pompput 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 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 16 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 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. De geregistreerde data worden op wekelijkse basis bezorgd aan ERM ter opvolging van de onttrekking.

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 2016 tot 1 juli 2017 heeft ERM bezoeken uitgevoerd op de data vermeld in onderstaande tabel.
ERM komt ook steeds ter plaatse tijdens en 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
07/07/16	20/10/16	28/02/17
13/07/16	24/10/16	8/03/17
20/07/16	3/11/16	17/03/17
27/07/16	8/11/16	24/03/17
5/08/16	25/11/16	29/03/17
9/08/16	2/12/16	5/04/17
16/08/16	6/12/16	14/04/17
17/08/16	15/12/16	19/04/17
25/08/16	22/12/16	28/04/17
30/08/16	28/12/16	5/05/17
9/09/16	6/01/17	10/05/17
16/09/16	13/01/17	16/05/17
20/09/16	19/01/17	24/05/17
20/09/16	27/01/17	30/05/17
30/09/16	3/02/17	9/06/17
30/09/16	8/02/17	15/06/17
4/10/16	17/02/17	23/06/17
10/10/16	24/02/17	

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

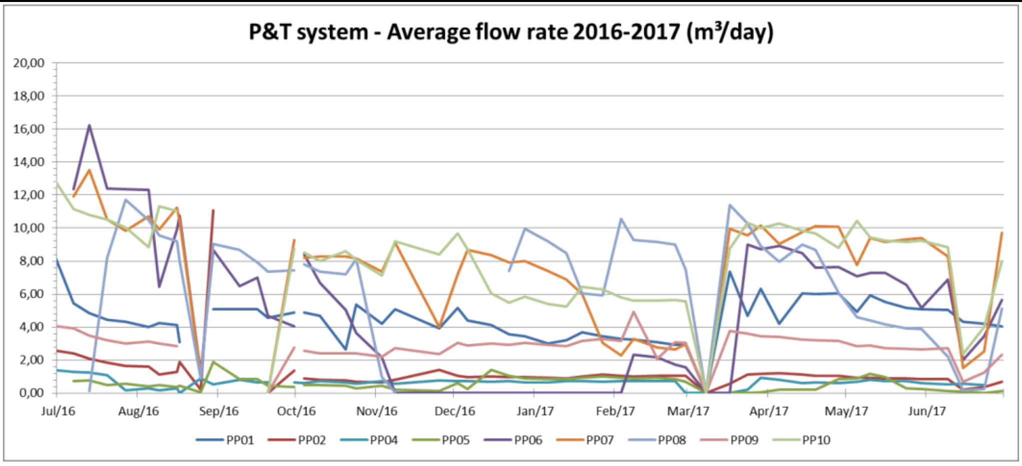
Datum	Bemonsterde pompputten
Juli 2016	PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 & PP10
Oktober 2016	PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 & PP10
Januari 2017	PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 & PP10
April 2017	PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 & PP10

5.1.3 Resultaten

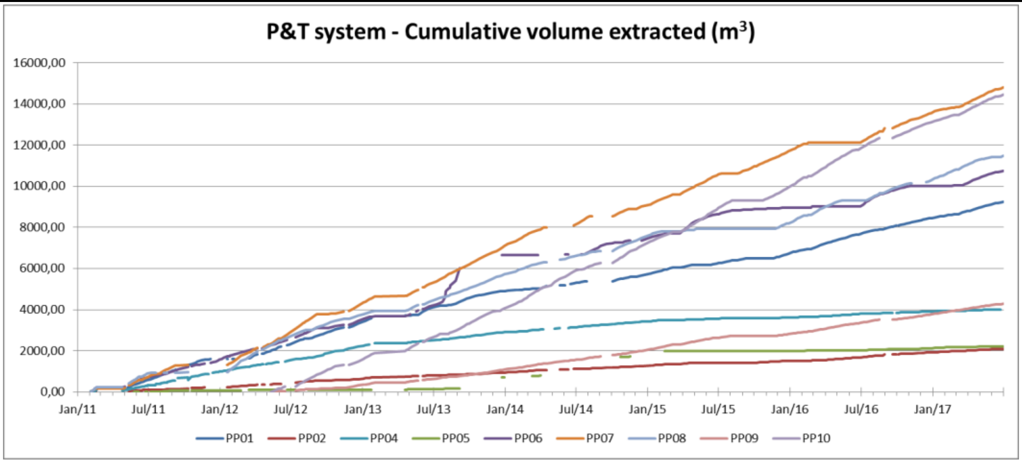
5.1.3.1 De onttrokken debieten

In onderstaande grafieken wordt getoond wat de gemiddelde en cumulatieve verpompte debieten zijn in de periode van 1 juli 2016 tot 1 juli 2017.

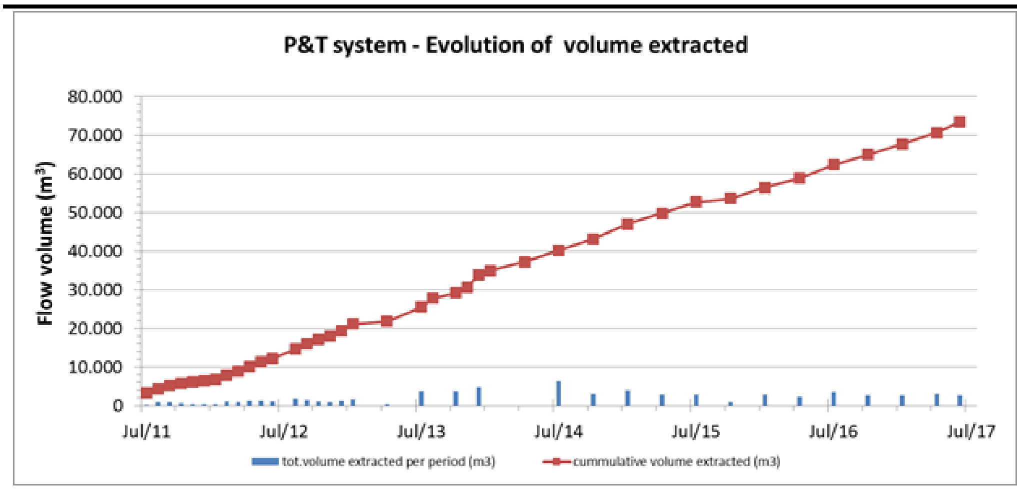
Grafiek 5.1 Gemiddeld debiet per onttrekkingsput (juli 2016 tot juli 2017)



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 voor wat betreft de periode juli 2016 – juli 2017:

- Het onttrokken debiet per put verschilt aanzienlijk en varieert tussen minimaal 0,4 m^3/dag (PP05) en maximaal 7,4 m^3/dag (PP10);

- De onttrekking in PP05 (debiet < 1 m^3/dag) werkt niet goed om verschillende redenen:
  - De toestroming in de pompput is zeer gering; en
  - Er is een harde afzetting geconstateerd in de pomp en debietmeter dat voor herhaaldelijke schade aan respectievelijk het pomphuis en de registratie in de debietmeter zorgt. De oorsprong en samenstelling van deze afzetting zal in de toekomst nader onderzocht worden.

- 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 uptime heeft van gemiddeld circa 88% (‘15-‘16: 53%; ‘14-‘15: 73%; ‘13-‘14: 71%), wat een sterke toename betekent ten opzichte van vorige jaren. Echter, zoals uit onderstaande Tabel 5.5 blijkt is er een groot verschil tussen de werking van de verschillende pompen. Pompput 6 heeft slechts een uptime van 72%, terwijl pompput PP01 een uptime heeft gerealiseerd van 97%.

Tabel 5.5 ***Werking pompputten***

	jul/16	aug/16	sep/16	okt/16	nov/16	dec/16	jan/17	feb/17	mrt/17	apr/17	mei/17	jun/17	uptime (%)
PP01													97
PP02													92
PP04													90
PP05													81
PP06													72
PP07													88
PP08													86
PP09													92
PP10													92

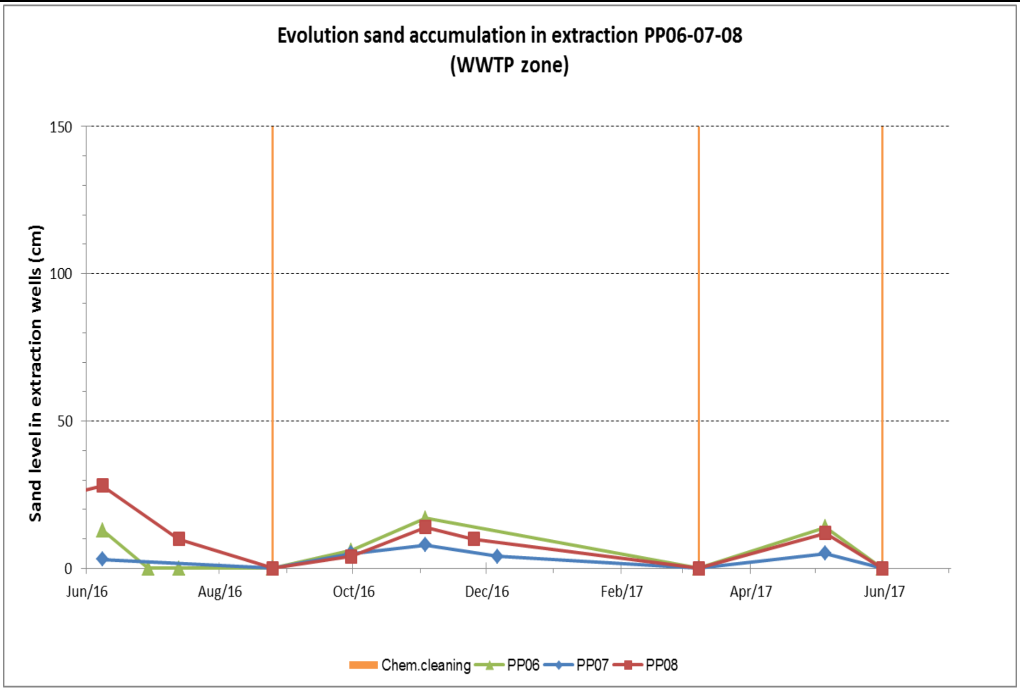
- pomp heel de maand operationeel
- pomp een halve maand operationeel
- pomp niet operationeel
- pomp operationeel maar techniche issues

- Tussen juli 2016 en juli 2017 is ongeveer 12.320 m^3 verontreinigd grondwater onttrokken (tegen circa 7.691 m^3 (2015-16) en 6241 m^3 (2014-15)). In totaal is sinds juli 2011 circa 73.339 m^3 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 vacuumtruck tijdens regelmatige reinigingswerken. Pompputten PP06, PP07 en PP08 blijken het meest gevoelig te zijn aan zandophoping. In volgende grafiek is de evolutie van de zandniveaus in de hogervermelde 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.

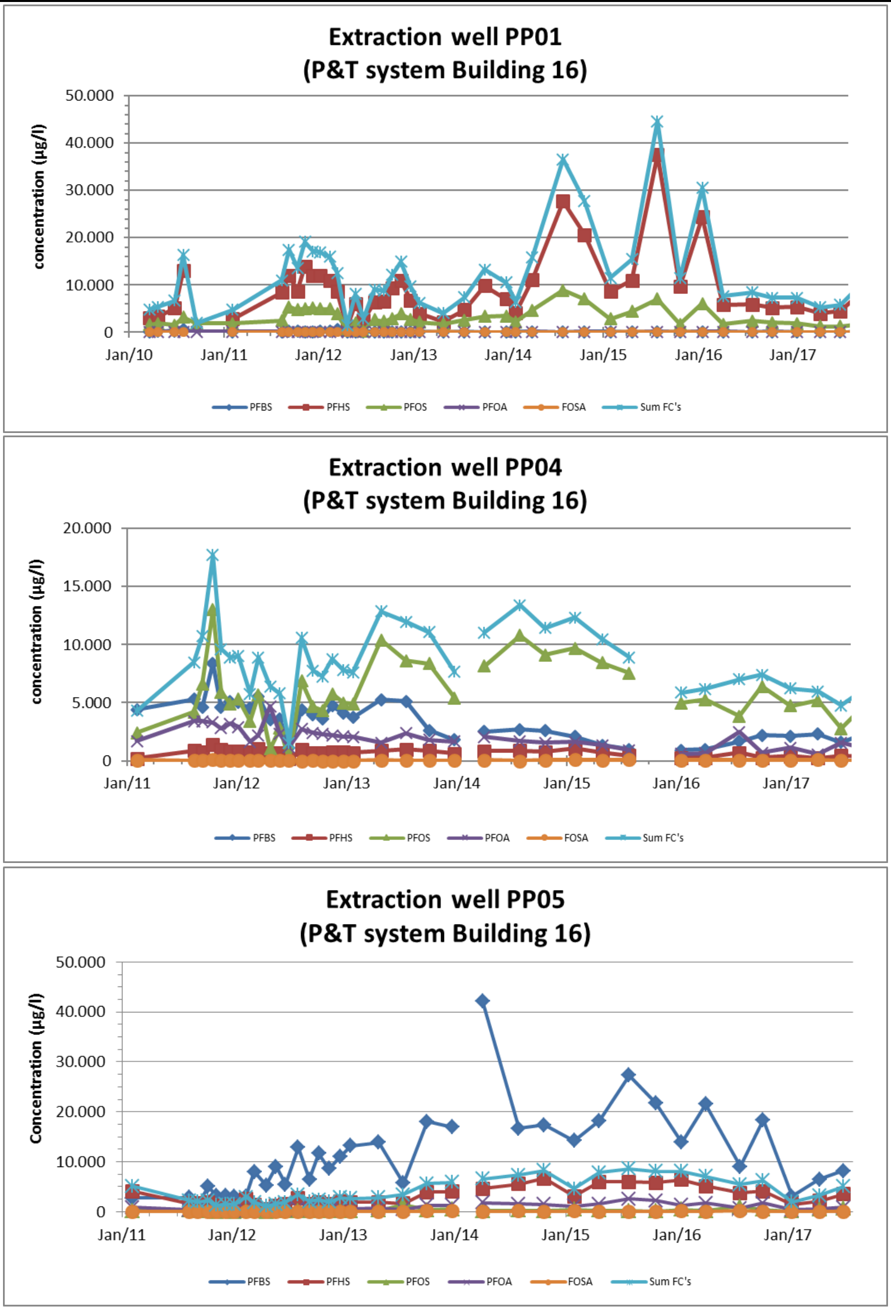
- Bij elektrische problemen of software problemen wordt de technische dienst van 3M gecontacteerd en gevraagd om de betreffende problemen op te lossen. Bij 3M is een vaste contactpersoon/technieker aangesteld om eventuele problemen op te lossen.

- Indien blijkt uit de data-opvolging dat het rendement van een bepaalde onttrekkingsput daalt zonder een aantoonbare technische reden, wordt ervan 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. Een dergelijke chemische reiniging vond in desbetreffende periode plaats in augustus 2016 en februari 2017.

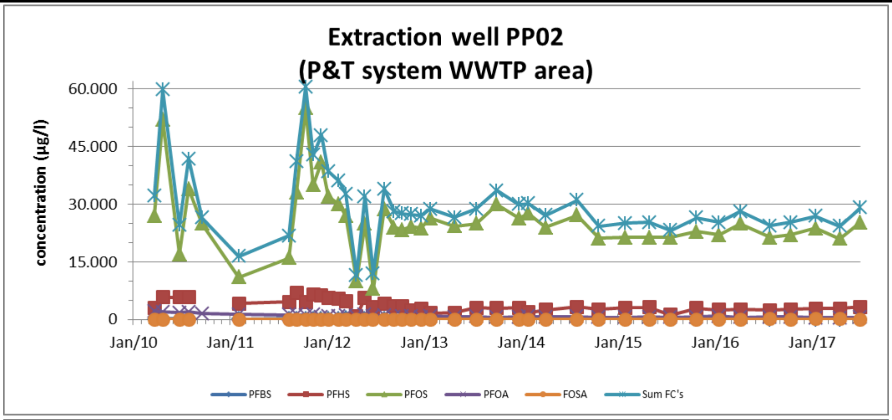
5.1.3.2 De concentraties in het onttrokken grondwater

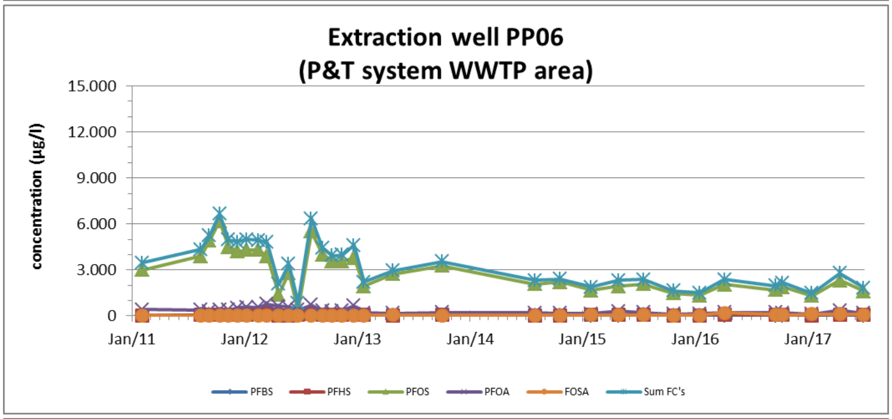
In onderstaande grafieken is per pompput weergegeven wat de verpompte concentraties zijn. In de laatste grafiek is de som van FC's (PFOS, PFOA en PFHS) weergegeven per pompput.

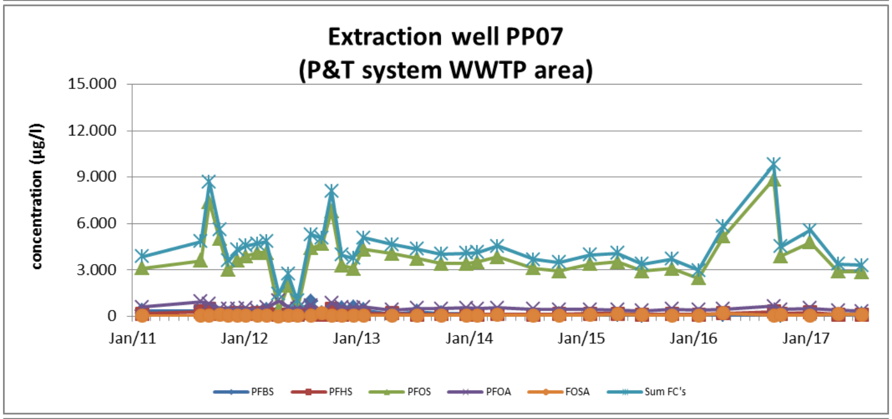
Grafiek 5.5 Evolutie FC concentraties t.h.v. gebouw 16 (pompput PP01, PP04 en PP05)

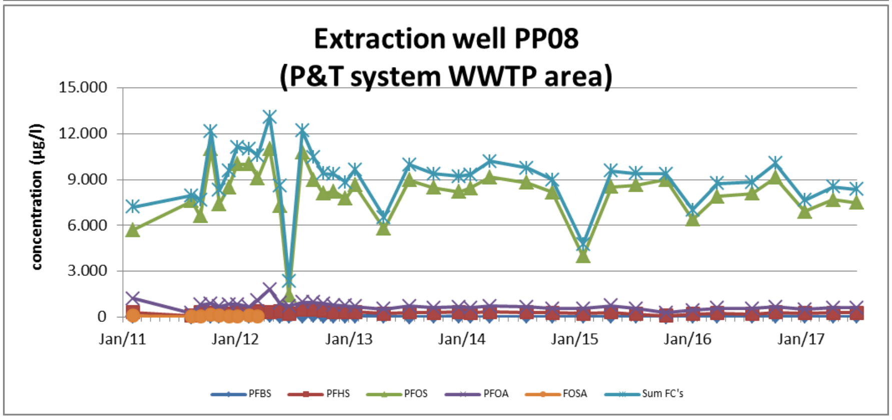


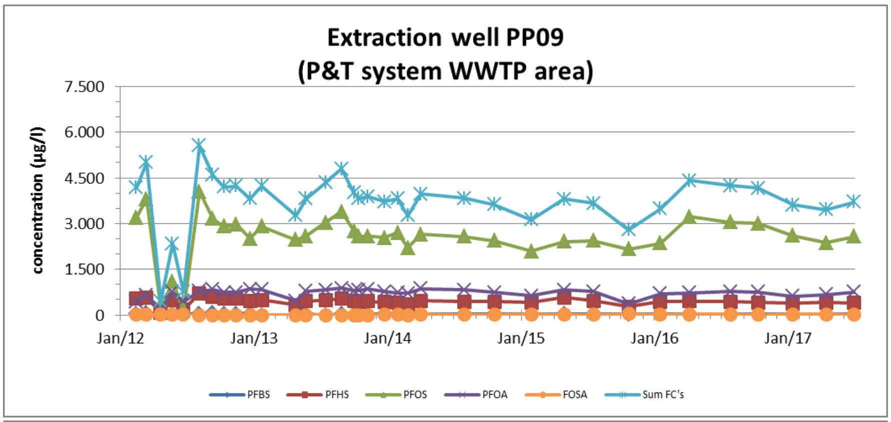
Grafiek 5.6 Evolutie FC concentraties t.h.v. WWTP zone (pompput PP02, PP06, PP07, PP08, PP09 en PP10)

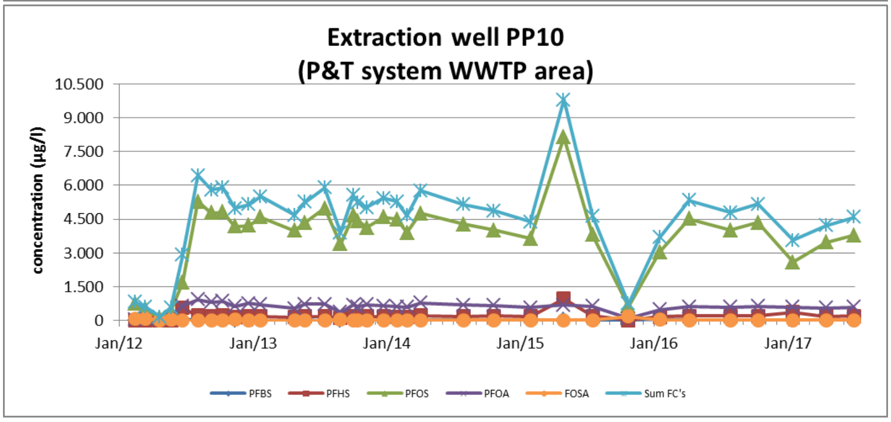




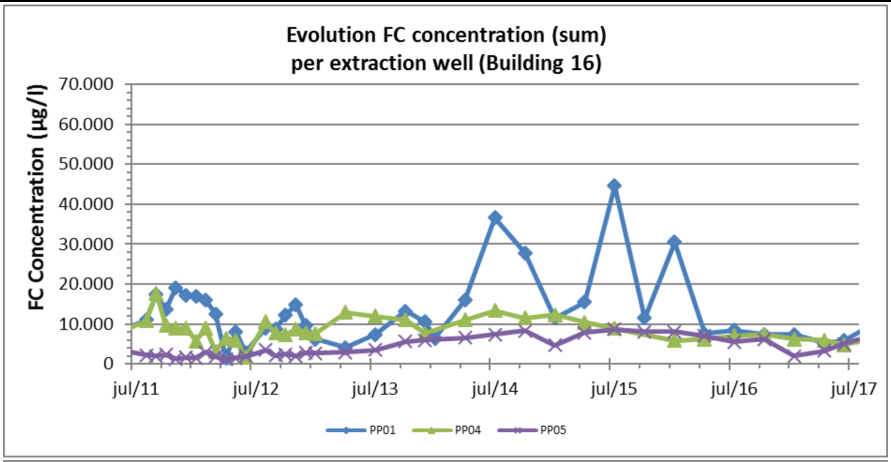


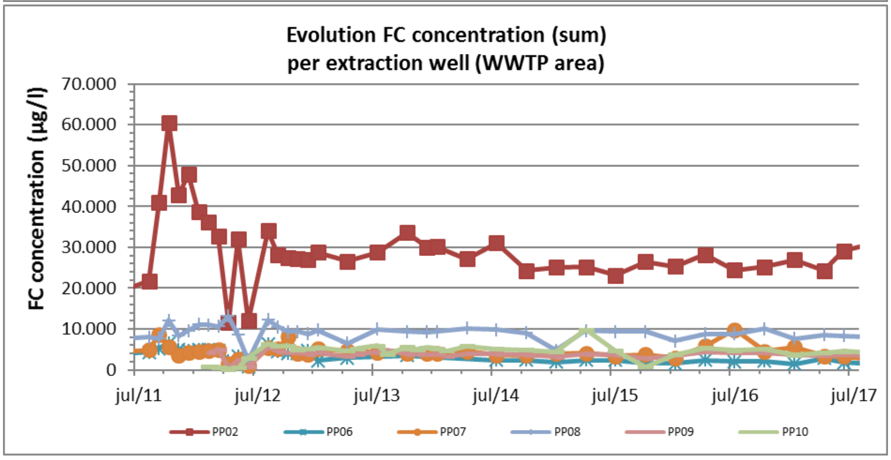




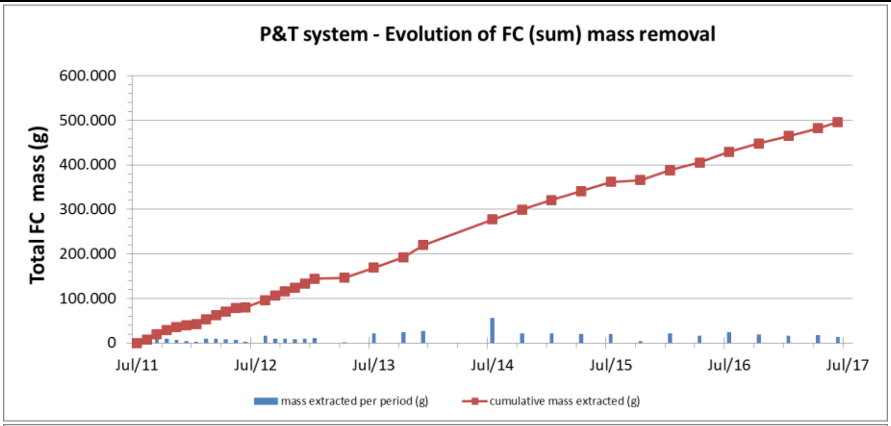


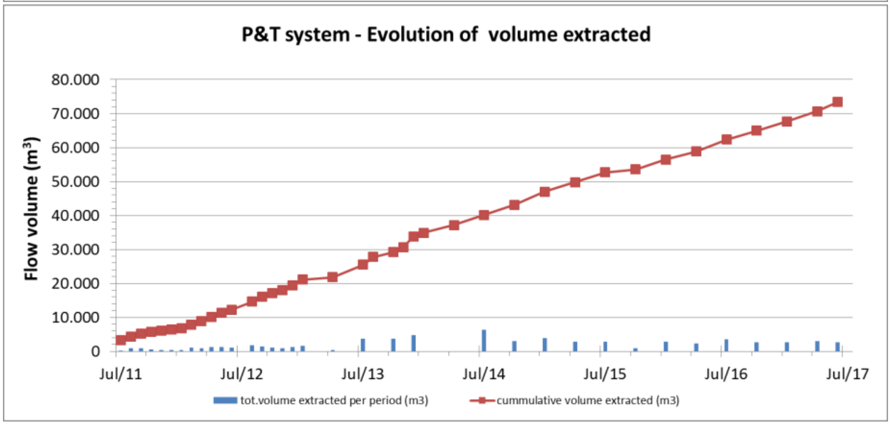
Grafiek 5.7 Overzicht evolutie som FC concentraties (PFOS, PFOA en PFHS) t.h.v. gebouw 16 en WWTP zone





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 16 (PP01, PP04 en PP05) en van het grondwater in de zone WWTP (overige pompputten):
  - In PP01 is PFHS de component met de hoogste concentraties;
  - In pompput PP05 wordt naast de "standaard" FC's (PFOS, PFOA, PFHS en PFOSA) ook een verhoogde concentratie PFBS (perfluorbutaansulfonzuur) gemeten; en
  - In de overige pompputten wordt voornamelijk PFOS gemeten.

- De hoogste concentraties aan som FC's (PFOS, PFOA en PFHS) worden gemeten 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 PP04, PP06, PP09 en PP10 liggen doorgaans rond 5.000 µg/L.
  - Voor 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.

- In desbetreffende periode is ongeveer 67 kg FC's verwijderd. In totaal is sinds juli 2011 circa 496 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 2016 tot juli 2017 ongeveer 12.320 m^3 verontreinigd grondwater onttrokken en circa 67 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 het verleden werden in enkele pompputten stijgende concentraties gemeten wat erop kan duiden dat de captatie lokaal toch niet volledig is. Een mogelijke uitbreiding van het saneringssysteem om de captatie te optimaliseren zal worden geëvalueerd.

Tijdens de meetcampagne in juli 2015 en januari 2016 was ter hoogte van pompput PP01 een sterk verhoogde concentratie aan PFHS gemeten. Deze verhoogde concentratie is verder opgevolgd maar wordt niet bevestigd tijdens de meetcampagnes uitgevoerd in de periode juli 2016 – juli 2017. De concentratie aan PFHS lijkt voorlopig gestabiliseerd. Ter hoogte van PP07 is sterke schommeling in PFOS concentraties gemeten tussen januari 2016 en januari 2017. De concentraties lijken terug te stabiliseren bij de hieropvolgende meetcampagnes.

Het saneringssysteem is storingsgevoelig wat een negatief effect kan hebben op de gerealiseerde 'uptime'. 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 uptime van het saneringssysteem gemiddeld 88% bedraagt in de periode juli 2016 – juli 2017. Er is echter een sterk verschil tussen de onttrekkingsputten onderling met een minimaal waargenomen uptime van 72% ter hoogte van PP06 en een maximaal geregistreerde gemiddelde uptime van 97% ter hoogte van PP01. De up-time ligt behoorlijk hoger dan in de voorbije jaren (15-16: 53%; 14-15: 73%; 13-14: 71%) als een rechtreeks gevolg van de intensievere reiniging van het onttrekkingssysteem in desbetreffende periode.

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:
- 19 juli 2016;
- 10 oktober 2016;
- 9 januari 2017; en
- 13 april 2017

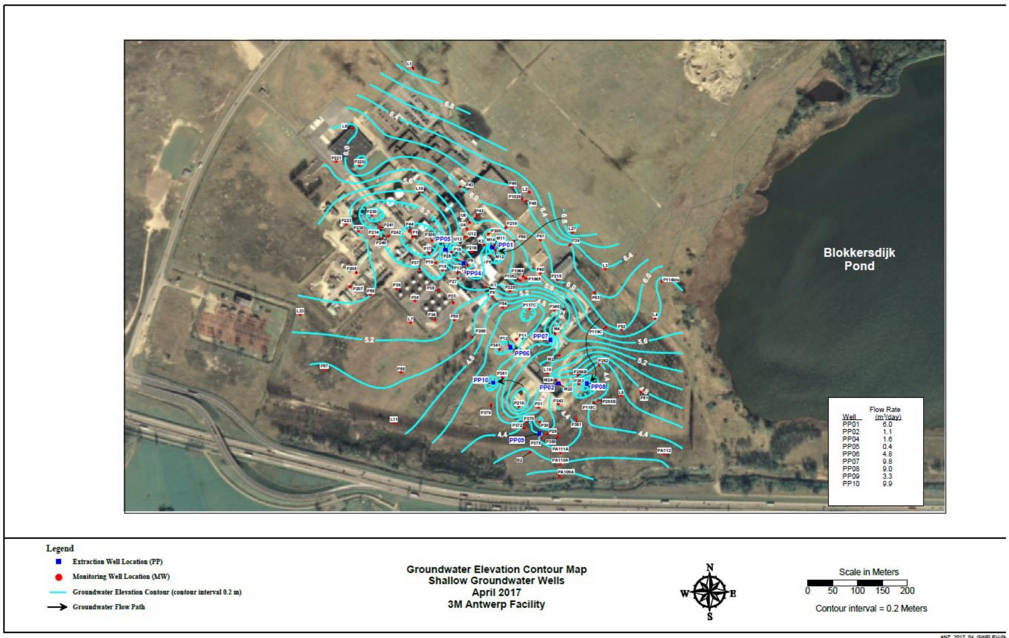
Op 13 april 2017 is een algemene dipronde uitgevoerd waarbij de grondwaterstand in een groot gedeelte van de peilbuizen op en in de onmiddellijke nabijheid van de site is opgemeten. De peilbuizen hebben een filter in de eerste freatische aquifer of de tweede aquifer.

In juli 2016, oktober 2016 en januari 2017 is een beperkte ronde uitgevoerd waarbij enkel de grondwaterstand in peilbuizen in de eerste freatische aquifer is opgemeten.

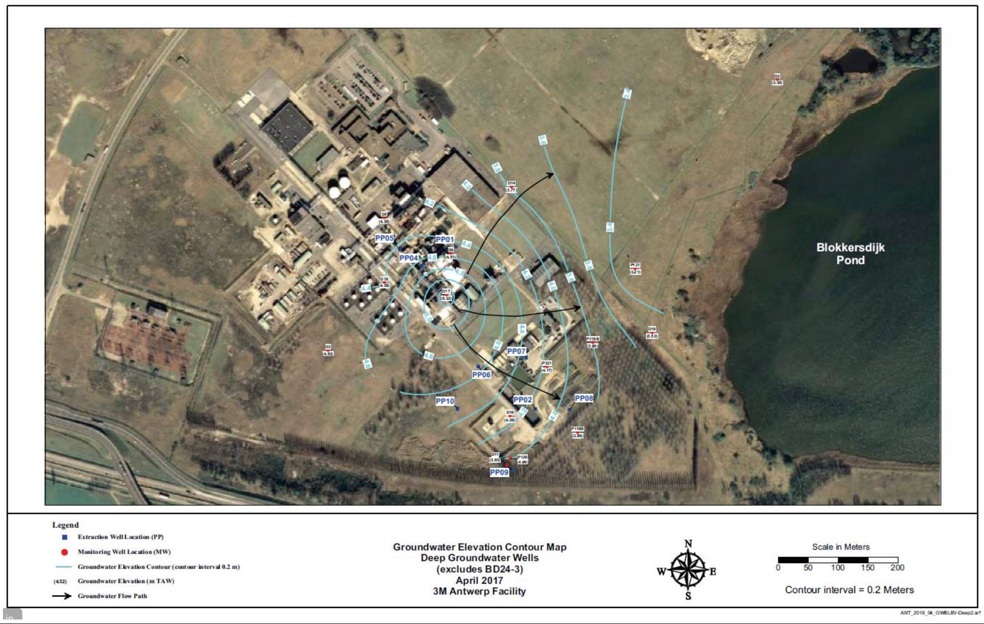
De grondwatercontouren die opgemaakt werden op basis van de meetresultaten van de meest recente algemene diprondes (april 2017) zijn weergegeven in Figuur 5.2.

Figuur 5.2 ***Grondwatercontouren april 2017***

Grondwatercontour eerste freatische aquifer april 2017



Grondwatercontour tweede aquifer april 2017



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 viermaal 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;

- In stand houden van de digitale data-transfer tussen de 3M systemen en ERM met registratie van:
  - Instantaan debiet;
  - Cumulatief debiet;
  - Waterkolom boven de pomp;
  - Druk op de afvoerleiding; en
  - Uptime 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.

- 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 uptime).

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 4.8 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.

- Vervolg 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 4.8;
- Driemaandelijkse opvolging van de grondwaterpeilen;
- Buitengebruik stellen van peilbuizen ter hoogte van de toekomstige ecocorridor;
- Verplaatsen van pompputten in het kader van de Oosterweelwerken; en
- Eerste kwartaal van 2019: Opstellen en indienen 9de tussentijds verslag.

7. *WIJZIGINGEN 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 2016) wordt verwezen naar paragraaf 3.3.

De wijzigingen in de saneringsaanpak van juli 2016 tot en met juli 2017 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 is 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 4.8.

Gezien de sterk beperkte bijdrage van de component PFOSA aan de somparameter FC, zal de component PFOSA vanaf 2018 niet meer geëvalueerd worden.

Om de monitoringsperiode voor de berekening van de totale vuilvracht naar de Schelde gelijk te laten lopen met de grondwatermonitoringsperiode, is ervoor gekozen om vanaf voorliggend tussentijds rapport de monitoringscampagne voor de berekening van de vuilvracht van augustus tot en met juli te laten lopen, zoals ook voor de grondwatermonitoring voorzien is.

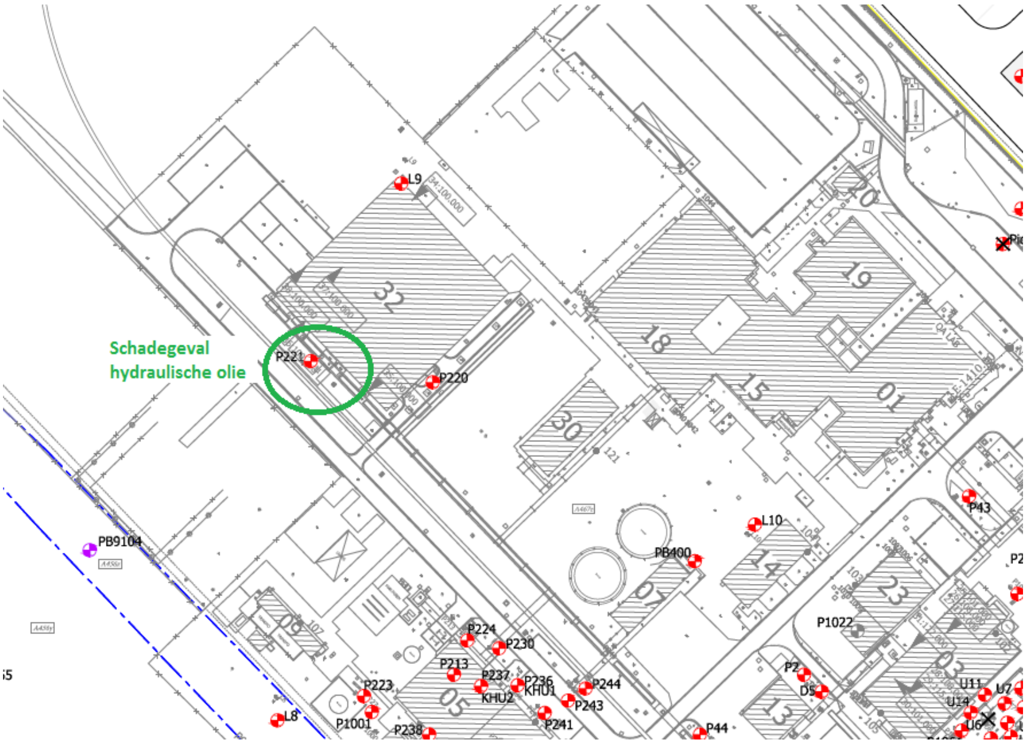
De beschreven wijzigingen betreffen elk een **kleine wijziging** van het eerste gefaseerd bodemsaneringsproject.

8. SCHADEGEVAL

8.1 HYDRAULISCHE OLIE TER HOOGTE VAN GEBOUW 32

Op 5 juli 2017 is ten zuiden van gebouw 32 een slang van een graafmachine met hydraulische olie gesprongen (zie Figuur 8.1). Hierdoor is circa 10 tot 20 liter hydraulische olie op een onverharde ondergrond (grind) en stelconplaten terecht gekomen.

Figuur 8.1 Locatie schadeval hydraulische olie



Het incident is op 7 juli 2017 (binnen de 14 dagen na vaststelling) via het standaardformulier gemeld aan OVAM. Op 16 oktober 2017 is de OVAM akkoord gegaan om het schadegeval met hydraulische olie op te nemen in het periodiek orienterend bodemonderzoek dat in 2017 op de site dient uitgevoerd te worden (mail in Bijlage 7). Er dient dus geen afzonderlijk evaluatierapport voor dit schadegeval opgemaakt te worden.

Op 6 juli 2017 is ter hoogte van de geïmpacteerde zone, die visueel waarnaambaar was door een donkere verkleuring en specifieke geur, de halfverharding verwijderd en een beperkte hoeveelheid grond tot ca. 20 cm diep afgegraven. Het betreft een zone van circa 2m bij 2m.
Vervolgens zijn door ERM twee wandstalen (W1 en W2) en twee bodemstalen op verschillende dieptes (B1 en B2) in het ontgravingsvlak genomen. De wanden langs de stelconplaten zijn niet bemonsterd. ERM heeft ook een mengstaal van de ontgraven grond genomen. De stalen zijn geanalyseerd door het erkende laboratorium van SGS.

In de bodemstalen is geen concentratie aan minerale olie gemeten hoger dan de detectielimiet van 50 mg/kg ds. In het meest westelijke wandstaal (W1) is een concentratie aan minerale olie gemeten (60 mg/kg ds) lager dan de richtwaarde van 300 mg/kg ds. De concentratie aan minerale olie in wandstaal W2 is 480 mg/kgd ds. Aangezien deze concentratie hoger is dan het saneringsdoel bij een nieuwe bodemverontreiniging (richtwaarde), is in een tweede fase een bijkomende ontgraving uitgevoerd.

Op 25 augustus 2017 heeft de 2de fase van de ontgraving plaatsgevonden. Tijdens deze werken zijn visueel ook nog enkele donker verkleurde spots waargenomen in de nabije omgeving van het initiele ontgravingsvlak. Deze spots zijn op dat moment mee ontgraven. In totaal betreft het een zone van 5m bij 2,5 m (inclusief eerste ontgravingsvlak) en een zone van 3,8 m op 2 m (extra verkleurde spots). Er zijn vier wandstalen (W3 –W6) en twee bodemstalen (B2 en B3) genomen ter controle. De stalen zijn geanalyseerd door het erkende laboratorium van SGS.

Zowel in de bodemstalen als in de wandstalen die tijdens de tweede fase van ontgraving genomen zijn, zijn concentraties aan minerale olie gemeten lager dan de detectielimiet van 50 mg/kg ds. In het grondwaterstaal uit peilbuis P221, dat in het kader van het periodiek oriënterend bodemonderzoek van 2017 geanalyseerd is op minerale olie, is een concentraties aan minerale olie gemeten lager dan 100 µg/l.
Bijgevolg is geoordeeld dat de verontreinigde grond ten gevolge van het schadegeval met hydraulische olie voldoende ontgraven is en er geen verdere maatregelen of een beschrijvend onderzoek nodig zijn.

De afgegraven grond was tijdelijk gestockeerd op een grote folie en afgedekt met een zeil om verspreiding naar de grond en/of het grondwater te voorkomen. In augustus 2017 is deze grond afgevoerd naar het erkende grondverwerkingscentrum Bremcom nv (zie verwerkingscertifiaat in Bijlage 7).

In het mengstaal van de ontgraven grond is een concentratie aan minerale olie gemeten van 90.000 mg/kg ds.

De analysecertificaten en de getoetste analyseresultaten zijn opgenomen in

Bijlage 7.

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 bodemsaneringsproject wordt de organofluorimpact ter hoogte van de zuidelijke perceelgrens en de Palingbeek opgevolgd door middel van een periodieke monitoring.

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 Achtste tussentijds verslag (TTV8) van de saneringswerken, zoals beschreven in het door de OVAM conform verklaarde bodemsaneringsproject (1e fase) en de zeven 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 2016 tot en met juli 2017.

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 2016 tot en met juli 2017 de volgende zaken uitgevoerd:

- Opvolging en onderhoud van het grondwatersaneringssysteem in twee bronzones (Gebouw 16 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 16 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 16 en WWTP), de 2de aquifer, de zuidelijke perceelgrens, de zone met peilbuizen P18 en P28 en het natuurgebied Blokkersdijk; de periodieke monitoring van de kwaliteit van het oppervlaktewater van de Palingbeek, Tophatgracht, 3M vijver en Blokkersdijkvijver; periodieke monitoring van de collectorput en het effluent van de waterzuivering;

- Het berekenen van gemiddelde PFOS vuilvracht die in de Schelde geloosd wordt via de 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;

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

- Herevaluatie statistische analyse op basis van meest recente analyseresultaten; en

- Begeleiden van de mitigerende maatregelen genomen naar aanleiding van het incident waarbij op 5 juli 2017 hydraulische olie op de bodem is terecht gekomen door het springen van een slang van een graafmachine ten zuiden van gebouw 32. De rapportage hiervan zal ook in het periodiek oriënterend bodemonderzoek van 2017-2018 opgenomen worden.

Uit de onderzoeksverrichtingen en saneringswerken uitgevoerd in de voorbije periode (juli 2016- juli 2017) 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 de periode juli 2016 tot juli 2017 is ongeveer 12.320 m^3 verontreinigd grondwater onttrokken en circa 67 kg FC verwijderd. In totaal is sinds juli 2011 circa 496 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. In de periode juli 2016 - juli 2017 werd een gemiddelde uptime verwezenlijkt van 88% dewelke hoger is dan voorgaande jaren.

- ERM raadt aan om de saneringsacties en regelmatige monitoring voort te zetten zoals samengevat in Tabel 4.8 (p.61).
  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 onttrekkingssysteem (i.c. uptime) verbeterd kan worden.

- De PFOS concentratie trend in de Blokkersdijk peilbuizen, de 3M vijver en de Blokkersdijk vijver worden verder opgevolgd via regelmatige monitoring. Uit de evaluatie blijkt dat in peilbuis P116 een significant stijgende trend wordt waargenomen en in peilbuis L22 een significant dalende trend. Wanneer voor beide peilbuizen enkel de laatste zeven concentraties die gemeten zijn, in beschouwing genomen worden (januari 2016-juli 2017), is er geen significante trend.
  De gemeten concentraties in het oppervlaktewater van de Blokkersdijkvijver geven geen statistisch significante trend weer.

- Momenteel is geen noodzaak voor bijkomende saneringsacties in deze zone aangezien er geen stijgende concentratietrend is waargenomen in de Blokkersdijk vijver.

- Uit de resultaten van de grondwatermonitoring ter hoogte van de twee kernzones (gebouw 16 en WWTP) blijkt dat de FC concentraties in het grondwater fluctueren, maar momenteel geen aanleiding geven tot verdere maatregelen.

- De gemiddelde berekende vuilvracht van PFOS naar de Schelde voor de periode 2016 – 2017 ligt in lijn met eerder berekende vuilvrachten. De berekende gemiddelde vuilvrachten zijn steeds onder de maximaal toegelaten vuilvracht van 370 g/dag gebleven.

- In de periode juli 2016 - juli 2017 heeft zich één milieu-incident voorgedaan op de site van 3M. De analyseresultaten toonden geen impact naar de bodem in deze zone. Bijgevolg zijn er geen verdere acties vereist. De volledige rapportering hiervan zal in het periodiek oriënterend bodemonderzoek 2017-2018 opgenomen worden, zoals overeengekomen met OVAM.

- 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.

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 augustus 2017 tot en met juli 2018 zal in het eerste kwartaal van 2019 aan OVAM overgemaakt worden.

■

DEEL 3: BIJLAGEN

Eerste gefaseerd bodemsaneringsproject -
Achtste tussentijds verslag bodemsaneringswerken
Periode juli 2016 – juli 2017

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

14 augustus 2018

www.erm.com

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 vollediggefaseerd bodemsaneringsproject

De OVAM heeft het gefaseerd bodemsaneringsproject ontvankelijk en volledig bevonden.

Kennisgeving

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

Advies en openbaar onderzoek

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

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

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

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

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

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

De onderstaande overheden of besturen hebben gunstig advies verleend:

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

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

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

- Alle wettelijke maatregelen om de milieuveiligheid en de arbeidsveiligheid te verzekeren bij de uitvoering van de bodemsaneringswerken, en de maatregelen zoals beschreven in Achilles (veiligheid, gezondheid en milieupreventiesysteem voor on-site bodemsaneringswerken) moeten strikt worden 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 pilootesten 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, 09 FEB. 2009

Eddy Van Dyck
Afdelingshoofd

Bijlage 2

Milieukundig dagboeken (niet van toepassing)

Bijlage 3

Analysecertificaten

Final Report

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

October 2016 Sampling

Laboratory Request Number: ISO16-0010

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 ISO16-0010

Water Sample Analysis at 3M Antwerp, Belgium
October 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 October 7, 2016 and October 12, 2016. Samples were returned to the 3M Environmental Laboratory on October 27, 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 ISO16-0010.

The 3M Environmental Laboratory prepared sample containers for thirty sampling locations. Each sample set consisted of a field sample and field sample duplicate. Seven locations also included a target analyte field matrix spike. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with a mass-labeled internal standard mix and select sample bottles were fortified with surrogate recovery standards (SRS) [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS prior to being sent to the field for sample collection. Sample location 12 was not accessible, and therefore, 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.

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)]**
ISO16-0010-030	BD24-3 Sample	82.6	59.1	1460	3.62
ISO16-0010-030-DUP	BD24-3 Sample Duplicate	83.5	60.0	1480	3.69
	Average	**83.1**	**59.6**	**1470**	**3.66**
	%RPD Sample/Sample Dup	**1.1**	**1.5**	**1.4**	**1.9**
ISO16-0010-031	D09 Sample	122	2900	1710	25.7
ISO16-0010-031-DUP	D09 Sample Duplicate	117	2840	1630	25.8
	Average	**120**	**2870**	**1670**	**25.8**
	%RPD Sample/Sample Dup	**4.2**	**2.1**	**4.8**	**0.39**
ISO16-0010-001	P121 Sample	0.285	0.111	3.90	0.272
ISO16-0010-001-DUP	P121 Sample Duplicate	0.285	0.114	3.82	0.196
	Average	**0.285 [(2)]**	**0.113 [(2)]**	**3.86 [(2)]**	**0.234**
	%RPD Sample/Sample Dup	**0.0**	**2.7**	**2.1**	**32 [(3)]**
ISO16-0010-002	P321 Sample	2610	753	4880	0.208
ISO16-0010-002-DUP	P321 Sample Duplicate	2600	762	4840	0.249
	Average	**2610**	**758**	**4860**	**0.229**
	%RPD Sample/Sample Dup	**0.38**	**1.2**	**0.82**	**18**
ISO16-0010-003	3M vijver Sample	1.67	0.660	4.61	0.0294
ISO16-0010-003-DUP	3M vijver Sample Duplicate	1.77	0.692	4.74	0.0360
	Average	**1.72 [(2)]**	**0.676 [(2)]**	**4.68 [(2)]**	**0.0327**
	%RPD Sample/Sample Dup	**5.8**	**4.7**	**2.8**	**20**
ISO16-0010-004	Blokkersdijkvijver standard Sample	1.14	0.603	6.79	0.144
ISO16-0010-004-DUP	Blokkersdijkvijver standard Sample Duplicate	1.11	0.625	6.88	0.144
	Average	**1.13 [(2)]**	**0.614 [(2)]**	**6.84 [(2)]**	**0.144**
	%RPD Sample/Sample Dup	**2.7**	**3.6**	**1.3**	**0.0**
ISO16-0010-005	L21 Sample	0.320	0.161	5.32	0.0557
ISO16-0010-005-DUP	L21 Sample Duplicate	0.315	0.155	5.11	0.0455
	Average	**0.318 [(2)]**	**0.158 [(2)]**	**5.22 [(2)]**	**0.0506**
	%RPD Sample/Sample Dup	**1.6**	**3.8**	**4.0**	**20**
ISO16-0010-006	L22 Sample	0.494	0.120	4.92	0.0794
ISO16-0010-006-DUP	L22 Sample Duplicate	0.482	0.119	4.96	0.0787
	Average	**0.488 [(2)]**	**0.120 [(2)]**	**4.94 [(2)]**	**0.0791**
	%RPD Sample/Sample Dup	**2.5**	**0.84**	**0.81**	**0.89**
ISO16-0010-007	L31 Sample	0.729	0.401	9.17	0.131
ISO16-0010-007-DUP	L31 Sample Duplicate	0.717	0.389	9.22	0.135
	Average	**0.723 [(2)]**	**0.395 [(2)]**	**9.20 [(2)]**	**0.133**
	%RPD Sample/Sample Dup	**1.7**	**3.0**	**0.54**	**3.0**
ISO16-0010-008	L4 Sample	3.09	2.73	30.9	1.10
ISO16-0010-008-DUP	L4 Sample Duplicate	3.21	2.87	32.6	1.11
	Average	**3.15 [(2)]**	**2.80 [(2)]**	**31.8 [(2)]**	**1.11**
	%RPD Sample/Sample Dup	**3.8**	**5.0**	**5.4**	**0.90**

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 ± 23%, PFBS ± 9.5%, PFHS ± 12%, and PFOS ± 21%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 12%, PFHS ± 19%, PFOS ± 15%, and PFOSA ± 10%.

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

(4) The data uncertainty has been expanded for P263 for PFHS and PFOS to ± 32% based on SRS 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)]**
ISO16-0010-009	P114bis Sample	3.25	1.34	13.3	0.0439
ISO16-0010-009-DUP	P114bis Sample Duplicate	3.31	1.43	13.4	0.0421
	Average	**3.28 [(2)]**	**1.39 [(2)]**	**13.4 [(2)]**	**0.0430**
	%RPD Sample/Sample Dup	**1.8**	**6.5**	**0.75**	**4.2**
ISO16-0010-010	P115 Sample	3.90	2.08	0.307	<0.0248
ISO16-0010-010-DUP	P115 Sample Duplicate	3.74	2.01	0.301	<0.0248
	Average	**3.82 [(2)]**	**2.05 [(2)]**	**0.304 [(2)]**	**<0.0248**
	%RPD Sample/Sample Dup	**4.2**	**3.4**	**2.0**	**NA**
ISO16-0010-011	P116 Sample	1.52	0.724	9.22	0.0983
ISO16-0010-011-DUP	P116 Sample Duplicate	1.65	0.752	10.1	0.111
	Average	**1.59 [(2)]**	**0.738 [(2)]**	**9.66 [(2)]**	**0.105**
	%RPD Sample/Sample Dup	**8.2**	**3.8**	**9.1**	**12**
ISO16-0010-012	Effluent WWTP Sample	22.1	28.2	20.0	0.309
ISO16-0010-012-DUP	Effluent WWTP Sample Duplicate	22.6	29.0	20.1	0.302
	Average	**22.4 [(2)]**	**28.6 [(2)]**	**20.1 [(2)]**	**0.306**
	%RPD Sample/Sample Dup	**2.2**	**2.8**	**0.50**	**2.3**
ISO16-0010-023	13 Sample	115	31.4	385	0.300
ISO16-0010-023-DUP	13 Sample Duplicate	119	31.3	391	0.302
	Average	**117**	**31.4**	**388**	**0.301**
	%RPD Sample/Sample Dup	**3.4**	**0.32**	**1.5**	**0.66**
ISO16-0010-024	5 Sample	72.1	57.5	109	0.367
ISO16-0010-024-DUP	5 Sample Duplicate	73.6	59.6	110	0.377
	Average	**72.9**	**58.6**	**110**	**0.372**
	%RPD Sample/Sample Dup	**2.1**	**3.6**	**0.91**	**2.7**
ISO16-0010-025	Bemalingsstation Sample	62.8	49.0	95.0	0.242
ISO16-0010-025-DUP	Bemalingsstation Sample Duplicate	60.4	48.3	87.1	0.313
	Average	**61.6**	**48.7**	**91.1**	**0.278**
	%RPD Sample/Sample Dup	**3.9**	**1.4**	**8.7**	**26 [(3)]**
ISO16-0010-026	Collector pit Sample	101	34.4	442	19.0
ISO16-0010-026-DUP	Collector pit Sample Duplicate	102	35.2	458	19.8
	Average	**102**	**34.8**	**450**	**19.4**
	%RPD Sample/Sample Dup	**0.99**	**2.30**	**3.6**	**4.1**
ISO16-0010-028	P263 Sample	1910	562	10300	11.6
ISO16-0010-028-DUP	P263 Sample Duplicate	1910	572	10900	12.5
	Average	**1910**	**567 [(4)]**	**10600 [(4)]**	**12.1**
	%RPD Sample/Sample Dup	**0.0**	**1.8**	**5.7**	**7.5**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 23%, PFBS ± 9.5%, PFHS ± 12%, and PFOS ± 21%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 12%, PFHS ± 19%, PFOS ± 15%, and PFOSA ± 10%.

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

(4) The data uncertainty has been expanded for P263 for PFHS and PFOS to ± 32% based on SRS recovery. See section 4 for details.

Table 1 continued. Sample Results Summary [(1)]

3M LIMS ID	Sample Description	PFOA	PFBS	PFHS	PFOS	PFOSA [(2)]
		Concentration (ng/mL)				
ISO16-0010-013	PP01 Sample	74.5	87.9	5760	2100	29.2
ISO16-0010-013-DUP	PP01 Sample Duplicate	74.7	82.0	4630	2000	25.3
	Average	**74.6**	**85.0**	**5200**	**2050**	**27.3**
	%RPD Sample/Sample Dup	**0.27**	**6.9**	**22 [(3)]**	**4.9**	**14**
ISO16-0010-014	PP02 Sample	609	21.5	2580	21300	41.5
ISO16-0010-014-DUP	PP02 Sample Duplicate	626	23.2	2640	22700	40.2
	Average	**618**	**22.4**	**2610**	**22000**	**40.9**
	%RPD Sample/Sample Dup	**2.8**	**7.6**	**2.3**	**6.4**	**3.2**
ISO16-0010-015	PP04 Sample	713	2240	273	6560	61.0
ISO16-0010-015-DUP	PP04 Sample Duplicate	702	2160	268	6260	60.7
	Average	**708**	**2200**	**271**	**6410**	**60.9**
	%RPD Sample/Sample Dup	**1.6**	**3.6**	**1.8**	**4.7**	**0.49**
ISO16-0010-016	PP05 Sample	1710	18300	4150	400	30.7
ISO16-0010-016-DUP	PP05 Sample Duplicate	1710	18500	4110	421	29.2
	Average	**1710**	**18400**	**4130**	**411**	**30.0**
	%RPD Sample/Sample Dup	**0.0**	**1.1**	**0.97**	**5.1**	**5.0**
ISO16-0010-017	PP06 Sample	197	18.1	51.8	1870	61.1
ISO16-0010-017-DUP	PP06 Sample Duplicate	214	19.1	57.2	1930	55.4
	Average	**206**	**18.6**	**54.5**	**1900**	**58.3**
	%RPD Sample/Sample Dup	**8.3**	**5.4**	**9.9**	**3.2**	**9.8**
ISO16-0010-018	PP07 Sample	439	61.8	155	3820	85.1
ISO16-0010-018-DUP	PP07 Sample Duplicate	443	63.5	163	3980	90.1
	Average	**441**	**62.7**	**159**	**3900**	**87.6**
	%RPD Sample/Sample Dup	**0.91**	**2.7**	**5.0**	**4.1**	**5.7**
ISO16-0010-019	PP08 Sample	646	43.8	289	9070	56.1
ISO16-0010-019-DUP	PP08 Sample Duplicate	665	44.3	293	9190	56.1
	Average	**656**	**44.1**	**291**	**9130**	**56.1**
	%RPD Sample/Sample Dup	**2.9**	**1.1**	**1.4**	**1.3**	**0.0**
ISO16-0010-020	PP09 Sample	752	23.7	408	3010	12.5
ISO16-0010-020-DUP	PP09 Sample Duplicate	761	23.7	409	2980	12.3
	Average	**757**	**23.7**	**409**	**3000**	**12.4**
	%RPD Sample/Sample Dup	**1.2**	**0.0**	**0.24**	**1.0**	**1.6**
ISO16-0010-021	PP10 Sample	636	16.2	202	4520	15.8
ISO16-0010-021-DUP	PP10 Sample Duplicate	610	15.8	190	4150	14.7
	Average	**623**	**16.0**	**196**	**4340**	**15.3**
	%RPD Sample/Sample Dup	**4.2**	**2.5**	**6.1**	**8.5**	**7.2**
ISO16-0010-027	P21B Sample	6950	3860	6410	41900	4.50
ISO16-0010-027-DUP	P21B Sample Duplicate	8650	4810	8080	55600	4.77
	Average	**7800**	**4340**	**7250**	**48800**	**4.64**
	%RPD Sample/Sample Dup	**22 [(3)]**	**22 [(3)]**	**23 [(3)]**	**28 [(3)]**	**5.8**

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 ± 23%, PFBS ± 9.5%, PFHS ± 12%, and PFOS ± 21%.
(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 12%, PFHS ± 19%, PFOS ± 15%, and PFOSA ± 10%.
(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
(4) The data uncertainty has been expanded for P263 for PFHS and PFOS to ± 32% based on SRS 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 (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 October 7, 2016 and October 12, 2016 in Nalgene™ (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. Select sample bottles were fortified with surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS at a nominal concentration of 0.1 ng/mL. Collected sample bottles were returned to the laboratory at ambient temperature on October 27, 2016.

2.3 Sample Preparation

Samples prepared using external standard calibration for PFOA, PFBS, PFHS, and PFOS were analyzed using a direct injection of the methanol diluted samples. 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.

Sample locations P121, 3M vijver, Blokkersdijkvijver standard, L21, L22, L31, L4, P114bis, P115, P116, Effluent WWTP, and the Trip Blank and Trip Blank FMS Low were reanalyzed for PFOA, PFHS, and PFOS by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples.

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 standard or method blank 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	10/28/16, 11/1/16, 11/2/16	11/4/16
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	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	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard (1)	Mass Transition Q1/Q3
PFOA	413/369	$[^{13}C_8]$-PFOA	421/376
	413/219		
	413/169		
PFBS	299/99	NA	NA
	299/80		
PFHS	399/99	$[^{13}C_3]$-PFHS	402/80
	399/80		
PFOS	499/99	$[^{13}C_8]$-PFOS	507/80
	499/80		
	499/130		
PFOSA	498/78	$[^{13}C_8]$-PFOSA	506/78
$[^{13}C_4]$-PFOA 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 20 or 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 not used for samples analyzed on 10/28/16 or 11/2/16.

3 Data Analysis

3.1 Calibration

10/28/16 and 11/2/16 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 10/28/16 calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed, and on 11/2/16, calibration standards ranged from 0.5 ng/mL to 150 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.

11/1/16 Internal Standard Calibration: Samples were analyzed for PFOA, PFHS, and PFOS 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. The calibration standards also contained surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

11/4/16 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 10 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) 10/28/16 Analysis	LOQ, ng/mL (2) 11/1/16 Analysis	LOQ, ng/mL (1) 11/2/16 Analysis	LOQ, ng/mL (1) 11/4/16 Analysis
PFOA	0.0192	0.0240	NA	NA
PFBS	0.0200	NA	NA	NA
PFHS	0.0200	0.0250	0.500	NA
PFOS	0.0185	0.0464	0.464	NA
PFOSA	NA	NA	NA	0.0248

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 10/28/16, the high level LCSs were prepared above the resulting ULOQ (50 ng/mL) for PFHS.
- On 11/2/16, the high level LCSs had an average recovery of 121% for PFOS. The mid-level LCSs had an average recovery of 125% for $^{13}C_4$-PFOS.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report. The following calculations were used to generate data in Table 7.

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

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

Table 7. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 10/28/16	PFOA (Linear + Branched)			[$^{13}C_4$]-PFOA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-161028-1	100	110	110	99.8	112	112
LCS-161028-2	100	105	105	99.8	107	107
LCS-161028-3	100	112	112	99.8	111	111
Average ± %RSD	**109% ± 3.3%**			**110% ± 2.4%**		
LCS-161028-4	5000	5450	109	998	1130	113
LCS-161028-5	5000	5380	108	998	1120	112
LCS-161028-6	5000	5550	111	998	1120	112
Average ± %RSD	**109% ± 1.4%**			**112% ± 0.51%**		
LCS-161028-7	35000	31500	90.1			
LCS-161028-8	35000	31000	88.4			
LCS-161028-9	35000	30800	88.1			
Average ± %RSD	**88.9% ± 1.2%**					

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) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 10/28/16	PFBS			PFHS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-161028-1	100	106	106	100	104	104
LCS-161028-2	100	101	101	100	102	102
LCS-161028-3	100	117	117	100	104	104
Average ± %RSD	**108% ± 7.6%**			**103% ± 1.1%**		
LCS-161028-4	5000	5240	105	5000	5220	104
LCS-161028-5	5000	5130	103	5000	5120	102
LCS-161028-6	5000	5200	104	5000	5260	105
Average ± %RSD	**104% ± 0.96%**			**104% ± 1.5%**		
LCS-161028-7	35000	38200	109	35000	>ULOQ	NA
LCS-161028-8	35000	35400	101	35000	>ULOQ	NA
LCS-161028-9	35000	35900	103	35000	>ULOQ	NA
Average ± %RSD	**104% ± 4.0%**			**NA (1)**		

ETS-8-044.3 External Calibration Analyzed 10/28/16	PFOS (Linear + Branched)			[$^{13}C_4$]-PFOS (2)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-161028-1	100	103	103	95.6	108	113
LCS-161028-2	100	101	101	95.6	105	110
LCS-161028-3	100	105	105	95.6	110	115
Average ± %RSD	**103% ± 1.9%**			**113% ± 2.2%**		
LCS-161028-4	5000	5510	110	956	1140	119
LCS-161028-5	5000	5450	109	956	1140	119
LCS-161028-6	5000	5490	110	956	1120	117
Average ± %RSD	**110% ± 0.53%**			**118% ± 0.98%**		
LCS-161028-7	35000	42500	121			
LCS-161028-8	35000	41300	118			
LCS-161028-9	35000	40900	117			
Average ± %RSD	**119% ± 1.8%**					

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) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 11/1/16	PFOA (Linear + Branched)			[$^{13}C_4$]-PFOA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-161101-1	0.190	0.211	111	0.198	0.228	115
LCS-161101-2	0.190	0.203	107	0.198	0.206	104
LCS-161101-3	0.190	0.206	109	0.198	0.212	107
Average ± %RSD	**109% ± 1.8%**			**109% ± 5.2%**		
LCS-161101-4	1.90	2.06	108	1.98	2.27	115
LCS-161101-5	1.90	2.03	107	1.98	2.27	115
LCS-161101-6	1.90	1.98	104	1.98	2.24	113
Average ± %RSD	**106% ± 2.0%**			**114% ± 1.0%**		
LCS-161101-7	33.2	36.9	111			
LCS-161101-8	33.2	37.1	112			
LCS-161101-9	33.2	36.7	111			
Average ± %RSD	**111% ± 0.52%**					

ETS-8-044.3 Internal Calibration Analyzed 11/1/16	PFHS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-161101-1	0.198	0.219	110
LCS-161101-2	0.198	0.221	111
LCS-161101-3	0.198	0.225	114
Average ± %RSD	**112% ± 1.9%**		
LCS-161101-4	1.98	2.15	109
LCS-161101-5	1.98	2.20	111
LCS-161101-6	1.98	2.18	110
Average ± %RSD	**110% ± 0.91%**		
LCS-161101-7	34.7	36.7	106
LCS-161101-8	34.7	36.1	104
LCS-161101-9	34.7	36.4	105
Average ± %RSD	**105% ± 0.95%**		

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) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 11/1/16	PFOS (Linear + Branched)			[$^{13}C_4$]-PFOS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-161101-1	0.184	0.208	113	0.189	0.200	106
LCS-161101-2	0.184	0.203	110	0.189	0.198	105
LCS-161101-3	0.184	0.219	119	0.189	0.204	108
Average ± %RSD	114% ± 4.0%			106% ± 1.4%		
LCS-161101-4	1.84	2.00	109	1.89	2.15	114
LCS-161101-5	1.84	2.08	113	1.89	2.16	114
LCS-161101-6	1.84	1.96	106	1.89	2.10	111
Average ± %RSD	109% ± 3.2%			113% ± 1.5%		
LCS-161101-7	32.2	34.6	107			
LCS-161101-8	32.2	34.1	106			
LCS-161101-9	32.2	33.8	105			
Average ± %RSD	106% ± 0.94%					

ETS-8-044.3 External Calibration Analyzed 11/2/16	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-161102-1	1000	1050	105	1000	1040	104
LCS-161102-2	1000	1000	100	1000	1030	103
LCS-161102-3	1000	1010	101	1000	1020	102
Average ± %RSD	102% ± 2.6%			103% ± 0.97%		
LCS-161102-4	10000	10500	105	10000	11200	112
LCS-161102-5	10000	10600	106	10000	10900	109
LCS-161102-6	10000	10300	103	10000	10200	102
Average ± %RSD	105% ± 1.5%			108% ± 4.8%		
LCS-161102-7	50000	55000	110	50000	59700	119
LCS-161102-8	50000	54500	109	50000	61400	123
LCS-161102-9	50000	55300	111	50000	59900	120
Average ± %RSD	110% ± 0.91%			121% ± 1.7% (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) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 11/2/16	[$^{13}C_4$]-PFOS (2)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-161102-1	382	435	114
LCS-161102-2	382	432	113
LCS-161102-3	382	435	114
Average ± %RSD	**114% ± 0.51%**		
LCS-161102-4	3820	4740	124
LCS-161102-5	3820	4850	127
LCS-161102-6	3820	4760	125
Average ± %RSD	**125% ± 1.2% (3)**		

ETS-8-044.3 Internal Calibration Analyzed 11/4/16	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-161104-1	0.199	0.204	103
LCS-161104-2	0.199	0.206	104
LCS-161104-3	0.199	0.201	101
Average ± %RSD	**103% ± 1.5%**		
LCS-161104-4	19.9	20.0	100
LCS-161104-5	19.9	20.1	101
LCS-161104-6	19.9	20.2	102
Average ± %RSD	**101% ± 0.99%**		
LCS-161104-7	139	139	100
LCS-161104-8	139	140	101
LCS-161104-9	139	140	101
Average ± %RSD	**101% ± 0.57%**		

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) LCSs did not meet acceptance criteria of 100 ± 20%.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.4. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in Table 8 below.

- The data uncertainty for PFOS using external calibration was calculated at ±15% following ETS-12-012.4; however, the method uncertainty was expanded to ±21% based on the recovery of the high-level LCSs analyzed on 11/2/16.

Table 8. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Data Uncertainty
PFOA	External	11.4	±23%
PFBS	External	4.75	±9.5%
PFHS	External	5.95	±12%
PFOS	External	NA	±21%
PFOA	Internal	5.91	±12%
PFHS	Internal	9.33	±19%
PFOS	Internal	7.46	±15%
PFOSA	Internal	5.06	±10%

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 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

Sampling Location	Spike Level	Final Concentration (ng/mL) PFOA	PFBS	PFHS	PFOS	PFOSA
Blokkersdijkvijver standard	FMS	4.79	5.00	5.00	4.64	5.00
P114bis (1)	FMS	4.24	4.42	4.42	4.10	4.42
Effluent WWTP (1)	FMS	8.48	8.85	8.85	8.20	8.85
D09	FMS	100	100	100	100	50.0
13	FMS	155	5.00	155	155	5.00
PP08	FMS	500	100	500	500	100
PP05	FMS	1000	100	1000	1000	100
Trip Blank	Low	4.79	5.00	5.00	4.64	5.00
	High	500	100	500	500	100

(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 18**. All field matrix spikes and surrogate recovery standards met method acceptance criteria except where noted below.

Sample location PP05: The PP05 sample duplicate had a [$^{13}C_4$]-PFOS surrogate recovery of 134%. Since the average [$^{13}C_4$]-PFOS surrogate recovery for all samples in the PP05 sample set was within acceptance criteria, the data uncertainty was not expanded further.

Sample location Bemalingsstation: The Bemalingsstation sample had a [$^{13}C_4$]-PFOS surrogate recovery of 131%. Since the average [$^{13}C_4$]-PFOS surrogate recovery for all samples in the Bemalingsstation sample set was within acceptance criteria, the data uncertainty was not expanded further.

Sample location P263: The P263 sample and sample duplicate had a [$^{13}C_4$]-PFOS surrogate recovery of 133% and 131%. Since the average [$^{13}C_4$]-PFOS surrogate recovery for all samples in the P263 sample set was outside the acceptance criteria, the data uncertainty was expanded to ±32% for PFHS and PFOS.

Table 10. Location ID: D09

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO16-0010-031	D09 Sample	122	NA	2900	NA
ISO16-0010-031-DUP	D09 Sample Duplicate	117	NA	2840	NA
ISO16-0010-031-FMS	D09 FMS	215	95.5	2960	NC
Average Concentration (ng/mL) ± %RPD		120 ng/mL ± 4.2%		2870 ng/mL ± 2.1%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO16-0010-031	D09 Sample	1710	NA	25.7	NA
ISO16-0010-031-DUP	D09 Sample Duplicate	1630	NA	25.8	NA
ISO16-0010-031-FMS	D09 FMS	1770	NC	71.7	91.9
Average Concentration (ng/mL) ± %RPD		1670 ng/mL ± 4.8%		25.8 ng/mL ± 0.39%	

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

Table 11. Location ID: Blokkersdijkvijver standard

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO16-0010-004	Blokkersdijkvijver standard Sample	1.14	NA	0.603	NA
ISO16-0010-004-DUP	Blokkersdijkvijver standard Sample Duplicate	1.11	NA	0.625	NA
ISO16-0010-004-FMS	Blokkersdijkvijver standard FMS	5.43	89.9	5.38	95.3
Average Concentration (ng/mL) ± %RPD		1.13 ng/mL ± 2.7%		0.614 ng/mL ± 3.6%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO16-0010-004	Blokkersdijkvijver standard Sample	6.79	NA	0.144	NA
ISO16-0010-004-DUP	Blokkersdijkvijver standard Sample Duplicate	6.88	NA	0.144	NA
ISO16-0010-004-FMS	Blokkersdijkvijver standard FMS	11.5	101	4.74	91.9 (1)
Average Concentration (ng/mL) ± %RPD		6.84 ng/mL ± 1.3%		0.144 ng/mL ± 0.0%	

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

Table 12. Location ID: P114bis

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO16-0010-009	P114bis Sample	3.25	NA	1.34	NA
ISO16-0010-009-DUP	P114bis Sample Duplicate	3.31	NA	1.43	NA
ISO16-0010-009-FMS	P114bis FMS	7.23	93.2	5.79	99.6
Average Concentration (ng/mL) ± %RPD		3.28 ng/mL ± 1.8%		1.39 ng/mL ± 6.5%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO16-0010-009	P114bis Sample	13.3	NA	0.0439	NA
ISO16-0010-009-DUP	P114bis Sample Duplicate	13.4	NA	0.0421	NA
ISO16-0010-009-FMS	P114bis FMS	17.9	NC	4.16	93.0 (1)
Average Concentration (ng/mL) ± %RPD		13.4 ng/mL ± 0.75%		0.0430 ng/mL ± 4.2%	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) FMS concentration greater than 10 times the sample concentration.

Table 13. Location ID: Effluent WWTP

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO16-0010-012	Effluent WWTP Sample	22.1	NA	28.2	NA
ISO16-0010-012-DUP	Effluent WWTP Sample Duplicate	22.6	NA	29.0	NA
ISO16-0010-012-FMS	Effluent WWTP FMS	29.5	NC	37.5	NC
Average Concentration (ng/mL) ± %RPD		22.4 ng/mL ± 2.2%		28.6 ng/mL ± 2.8%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO16-0010-012	Effluent WWTP Sample	20.0	NA	0.309	NA
ISO16-0010-012-DUP	Effluent WWTP Sample Duplicate	20.1	NA	0.302	NA
ISO16-0010-012-FMS	Effluent WWTP FMS	27.7	NC	8.18	89.0 (1)
Average Concentration (ng/mL) ± %RPD		20.1 ng/mL ± 0.50%		0.306 ng/mL ± 2.3%	

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 14. Location ID: PP05

3M LIMS ID	Sample Description	PFOA Concentration (ng/mL)	PFOA %Recovery	PFBS Concentration (ng/mL)	PFBS %Recovery	PFHS Concentration (ng/mL)	PFHS %Recovery
ISO16-0010-016	PP05 Sample	1710	NA	18300	NA	4150	NA
ISO16-0010-016-DUP	PP05 Sample Duplicate	1710	NA	18500	NA	4110	NA
ISO16-0010-016-FMS	PP05 FMS	2680	97.0	18900	NC	5080	NC
Average Concentration (ng/mL) ± %RPD		**1710 ng/mL ± 0.0%**		**18400 ng/mL ± 1.1%**		**4130 ng/mL ± 0.97%**	

3M LIMS ID	Sample Description	PFOS Concentration (ng/mL)	PFOS %Recovery	PFOSA Concentration (ng/mL)	PFOSA %Recovery
ISO16-0010-016	PP05 Sample	400	NA	30.7	NA
ISO16-0010-016-DUP	PP05 Sample Duplicate	421	NA	29.2	NA
ISO16-0010-016-FMS	PP05 FMS	1650	124	117	87.1
Average Concentration (ng/mL) ± %RPD		**411 ng/mL ± 5.1%**		**30.0 ng/mL ± 5.0%**	

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

Table 15. Location ID: PP08

3M LIMS ID	Sample Description	PFOA Concentration (ng/mL)	PFOA %Recovery	PFBS Concentration (ng/mL)	PFBS %Recovery	PFHS Concentration (ng/mL)	PFHS %Recovery
ISO16-0010-019	PP08 Sample	646	NA	43.8	NA	289	NA
ISO16-0010-019-DUP	PP08 Sample Duplicate	665	NA	44.3	NA	293	NA
ISO16-0010-019-FMS	PP08 FMS	1140	96.9	131	87.0	753	92.4
Average Concentration (ng/mL) ± %RPD		**656 ng/mL ± 2.9%**		**44.1 ng/mL ± 1.1%**		**291 ng/mL ± 1.4%**	

3M LIMS ID	Sample Description	PFOS Concentration (ng/mL)	PFOS %Recovery	PFOSA Concentration (ng/mL)	PFOSA %Recovery
ISO16-0010-019	PP08 Sample	9070	NA	56.1	NA
ISO16-0010-019-DUP	PP08 Sample Duplicate	9190	NA	56.1	NA
ISO16-0010-019-FMS	PP08 FMS	9790	NC	146	89.9
Average Concentration (ng/mL) ± %RPD		**9130 ng/mL ± 1.3%**		**56.1 ng/mL ± 0.0%**	

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

Table 16. Location ID: 13

3M LIMS ID	Sample Description	PFOA Concentration (ng/mL)	PFOA %Recovery	PFHS Concentration (ng/mL)	PFHS %Recovery
ISO16-0010-023	13 Sample	115	NA	31.4	NA
ISO16-0010-023-DUP	13 Sample Duplicate	119	NA	31.3	NA
ISO16-0010-023-FMS	13 FMS	267	96.8	189	102
Average Concentration (ng/mL) ± %RPD		**117 ng/mL ± 3.4%**		**31.4 ng/mL ± 0.32%**	

3M LIMS ID	Sample Description	PFOS Concentration (ng/mL)	PFOS %Recovery	PFOSA Concentration (ng/mL)	PFOSA %Recovery
ISO16-0010-023	13 Sample	385	NA	0.300	NA
ISO16-0010-023-DUP	13 Sample Duplicate	391	NA	0.302	NA
ISO16-0010-023-FMS	13 FMS	548	NC	5.19	97.8 (1)
Average Concentration (ng/mL) ± %RPD		**388 ng/mL ± 1.5%**		**0.301 ng/mL ± 0.66%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) FMS concentration greater than 10 times the sample concentration.

Table 17. Trip Blank

3M LIMS ID	Sample Description	PFOA Concentration (ng/mL)	PFOA %Recovery	PFBS Concentration (ng/mL)	PFBS %Recovery	PFHS Concentration (ng/mL)	PFHS %Recovery
ISO16-0010-029	Trip Blank	<0.0240	NA	<0.200	NA	<0.0250	NA
ISO16-0010-029-FMS-LOW	Trip Blank FMS Low	4.75	99.2	5.08	102	5.38	108
ISO16-0010-029-FMS-HIGH	Trip Blank FMS High	526	105	97.6	97.6	510	102

3M LIMS ID	Sample Description	PFOS Concentration (ng/mL)	PFOS %Recovery	PFOSA Concentration (ng/mL)	PFOSA %Recovery
ISO16-0010-029	Trip Blank	<0.0464	NA	<0.0248	NA
ISO16-0010-029-FMS-LOW	Trip Blank FMS Low	4.57	98.6	5.00	100
ISO16-0010-029-FMS-HIGH	Trip Blank FMS High	542	108	101	101

NA = Not Applicable

Table 18. 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**
ISO16-0010-001	P121 Sample	90.5 (2)	89.2 (2)	NA
ISO16-0010-001-DUP	P121 Sample Duplicate	95.8 (2)	89.1 (2)	NA
ISO16-0010-002	P321 Sample	109	120	NA
ISO16-0010-002-DUP	P321 Sample Duplicate	113	126	NA
ISO16-0010-003	3M vijver Sample	89.1 (2)	83.9 (2)	NA
ISO16-0010-003-DUP	3M vijver Sample Duplicate	92.1 (2)	86.9 (2)	NA
ISO16-0010-004	Blokkersdijkvijver standard Sample	88.2 (2)	87.7 (2)	NA
ISO16-0010-004-DUP	Blokkersdijkvijver standard Sample Duplicate	92.2 (2)	90.4 (2)	NA
ISO16-0010-004-FMS	Blokkersdijkvijver standard FMS	93.4 (2)	82.1 (2)	NA
ISO16-0010-005	L21 Sample	92.5 (2)	89.6 (2)	NA
ISO16-0010-005-DUP	L21 Sample Duplicate	97.6 (2)	86.6 (2)	NA
ISO16-0010-006	L22 Sample	93.7 (2)	85.3 (2)	NA
ISO16-0010-006-DUP	L22 Sample Duplicate	93.2 (2)	85.9 (2)	NA
ISO16-0010-007	L31 Sample	92.2 (2)	89.8 (2)	NA
ISO16-0010-007-DUP	L31 Sample Duplicate	89.0 (2)	89.0 (2)	NA
ISO16-0010-008	L4 Sample	95.7 (2)	85.4 (2)	NA
ISO16-0010-008-DUP	L4 Sample Duplicate	87.3 (2)	85.4 (2)	NA
ISO16-0010-009	P114bis Sample	88.5 (2)	88.1 (2)	NA
ISO16-0010-009-DUP	P114bis Sample Duplicate	88.9 (2)	86.7 (2)	NA
ISO16-0010-009-FMS	P114bis FMS	85.5 (2)	88.0 (2)	NA
ISO16-0010-010	P115 Sample	86.2 (2)	87.6 (2)	NA
ISO16-0010-010-DUP	P115 Sample Duplicate	91.2 (2)	91.6 (2)	NA
ISO16-0010-011	P116 Sample	84.8 (2)	85.7 (2)	NA
ISO16-0010-011-DUP	P116 Sample Duplicate	91.8 (2)	86.4 (2)	NA
ISO16-0010-012	Effleunt WWTP Sample	93.6 (2)	82.4 (2)	NA
ISO16-0010-012-DUP	Effleunt WWTP Sample Duplicate	92.8 (2)	84.8 (2)	NA
ISO16-0010-012-FMS	Effleunt WWTP FMS	89.7 (2)	85.9 (2)	NA
ISO16-0010-013	PP01 Sample	113	119	128
ISO16-0010-013-DUP	PP01 Sample Duplicate	109	120	129
ISO16-0010-014	PP02 Sample	116	117	NA
ISO16-0010-014-DUP	PP02 Sample Duplicate	119	116	NA
ISO16-0010-015	PP04 Sample	119	120	NA
ISO16-0010-015-DUP	PP04 Sample Duplicate	115	117	NA
ISO16-0010-016	PP05 Sample	119	126	NA
ISO16-0010-016-DUP	PP05 Sample Duplicate	122	134 (3)	NA
ISO16-0010-016-FMS	PP05 FMS	112	119	NA
ISO16-0010-017	PP06 Sample	115	123	NA
ISO16-0010-017-DUP	PP06 Sample Duplicate	125	130	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 18 continued. 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**
ISO16-0010-020	PP09 Sample	111	120	NA
ISO16-0010-020-DUP	PP09 Sample Duplicate	121	129	NA
ISO16-0010-021	PP10 Sample	107	113	NA
ISO16-0010-021-DUP	PP10 Sample Duplicate	109	114	NA
ISO16-0010-023	13 Sample	114	123	NA
ISO16-0010-023-DUP	13 Sample Duplicate	110	110	NA
ISO16-0010-023-FMS	13 FMS	107	115	NA
ISO16-0010-024	5 Sample	114	121	NA
ISO16-0010-024-DUP	5 Sample Duplicate	121	130	NA
ISO16-0010-025	Bemalingsstation Sample	122	131 (3)	NA
ISO16-0010-025-DUP	Bemalingsstation Sample Duplicate	119	124	NA
ISO16-0010-026	Collector pit Sample	119	121	NA
ISO16-0010-026-DUP	Collector pit Sample Duplicate	124	124	NA
ISO16-0010-027	P21B Sample	116	124	121
ISO16-0010-027-DUP	P21B Sample Duplicate	107	115	121
ISO16-0010-028	P263 Sample	122	133 (3)	NA
ISO16-0010-028-DUP	P263 Sample Duplicate	117	131 (3)	NA
ISO16-0010-029	Trip Blank	92.6 (2)	87.4 (2)	NA
ISO16-0010-029-FMS-LOW	Trip Blank FMS Low	97.5 (2)	88.8 (2)	NA
ISO16-0010-029-FMS-HIGH	Trip Blank FMS High	103	111	NA
ISO16-0010-030	BD24-3 Sample	122	127	NA
ISO16-0010-030-DUP	BD24-3 Sample Duplicate	110	115	NA
ISO16-0010-031	D09 Sample	119	128	NA
ISO16-0010-031-DUP	D09 Sample Duplicate	123	127	NA
ISO16-0010-031-FMS	D09 FMS	122	129	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%.

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 10-18 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

Digitally signed by Chelsie J. Grochow
DN: c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory - authenticated by LRA,
[redacted] o=3M, cn=Chelsie J. Grochow
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Chelsie Grochow, 3M Report Author

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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.

Digitally signed by Daryl K. Peterson
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SGS

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ANALYTICAL REPORT

IAC16-07321

3M Lab Request Number: E17-0081

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

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Requester

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1. Introduction - Summary.

At the request (Lab Request Number: E17-0081) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected between January 9th and 13th, 2017 from 31 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
D09		2552	81.8	1093	<38.7
D16		58.5	133	942	50.5
D17		455	6682	174	<20.3
D18		191	516	<8.79	<20.3
P119A		113	318	1239	20.5
P119B		479	1423	99.8	<20.3
P121		<0.217	<0.226	1.98	<0.506
L21		<0.217	<0.226	4.72	<0.506
L22		<0.217	<0.226	3.78	<0.506
L31		0.939	<0.226	4.40	<0.506
P115		2.01	2.64	0.259	<0.506
Effluent WWTP		<0.217	<0.226	0.366	<0.506
Bemalingsstation		18.0	21.8	49.2	<0.506
Collector Put		11.3	23.8	87.8	3.69
P27	864	43.6	95.6	1555	31.3
P304	284	123	433	1301	20.8
P305	119	197	150	472	11.8
P42	41.9	456	241	3571	<20.3
M4		161	313	4251	68.9
P118C		53.9	191	1271	36.0
P119C		556	974	2217	41.1
P264		138	194	1197	33.7
P371		160	214	2415	37.7
P374		673	525	2418	12.4
P381		132	512	1156	<11.1
P382		109	311	794	44.4
B3-bis		6.48	42.6	103.2	<1.01

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
B7		1.26	1.31	57.5	<1.00
P378		312	324	1430	<11.1
PA112		31.3	125	390	43.5

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
D09	100
D16	100
D17	100
D18	100
P119A	100
P119B	100
P121	100
L21	100
L22	100
L31	100
P115	100
Effluent WWTP	100
Bemalingsstation	100
Collector Put	100
P27	100
P304	100
P305	100
P42	100
M4	100
P118C	100
P119C	100
P264	100
P265C	100
P371	100
P374	100
P381	100
P382	100
B3-bis	100
B7	100
P378	100
PA112	100

Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
D09	1.0	0.1	Solution A	10.0	0.5mL extract + 10.0mL MeOH (*)
D16	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
D17	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
D18	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P119A	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P119B	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P121	1.0	0.05	Solution A	2.0	-
L21	1.0	0.05	Solution A	2.0	-
L22	1.0	0.05	Solution A	2.0	-
L31	1.0	0.05	Solution A	2.0	-
P115	1.0	0.05	Solution A	2.0	-
Effluent WWTP	1.0	0.05	Solution A	2.0	-
Bemalingsstation	1.0	0.05	Solution A	2.0	-
Collector Put	1.0	0.1	Solution A	10.0	-
P27	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P304	1.0	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P305	1.0	0.1	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
P42	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
M4	1.0	0.2	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P118C	1.0	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P119C	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P264	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P371	1.0	0.1	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
P374	1.0	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P381	1.0	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P382	1.0	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
B3-bis	1.0	0.1	Solution A	5.0	-
B7	1.0	0.05	Solution A	5.0	-
P378	1.0	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
PA112	1.0	0.1	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
Field Trip Blank	1.0	0.1	Solution A	10.0	-

(*): MeOH:LCMS-water (60:40)
Solution A = 1000 ng/mL (nominal) ^{13}C-PFC Surrogate Standards

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ^{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 0.0710 to 0.166 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.217 to 39.0 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within ± 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
20 January 2017	LCS1	80	83.7	88.3	88.1	86.9	98.1	85.2	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, except for the QC Lab High inj2 (^{13}C-PFOS > ± 25%)
Table 8 summarizes the QCs recovery results.

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C-PFOA	PFOS	FOSA	^{13}C-PFOS
10 August 2016	QC Lab Low inj1	10	104	93.5	110	124	117	91.7	110
	QC Lab High inj1	100	87.4	82.0	91.8	84.2	102	85.6	79.8
	QC Lab Low inj2	10	105	95.9	95.8	75.6	116	90.9	96.7
	QC Lab High inj2	100	85.4	79.7	79.8	77.0	102	82.4	71.9(*)

(*) recovery above the acceptance criteria of ± 25 %

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
D09		2552	81.8	1093	<38.7
D16		58.5	133	942	50.5
D17		455	6682	174	<20.3
D18		191	516	<8.79	<20.3
P119A		113	318	1239	20.5
P119B		479	1423	99.8	<20.3
P121		<0.217	<0.226	1.98	<0.506
L21		<0.217	<0.226	4.72	<0.506
L22		<0.217	<0.226	3.78	<0.506
L31		0.939	<0.226	4.40	<0.506
P115		2.01	2.64	0.259	<0.506
Effluent WWTP		<0.217	<0.226	0.366	<0.506
Bemalingsstation		18.0	21.8	49.2	<0.506
Collector Put		11.3	23.8	87.8	3.69
P27	864	43.6	95.6	1555	31.3
P304	284	123	433	1301	20.8
P305	119	197	150	472	11.8
P42	41.9	456	241	3571	<20.3
M4		161	313	4251	68.9
P118C		53.9	191	1271	36.0
P119C		556	974	2217	41.1
P264		138	194	1197	33.7
P371		160	214	2415	37.7
P374		673	525	2418	12.4
P381		132	512	1156	<11.1
P382		109	311	794	44.4
B3-bis		6.48	42.6	103.2	<1.01
B7		1.26	1.31	57.5	<1.00
P378		312	324	1430	<11.1
PA112		31.3	125	390	43.5
Field Trip Blank	<0.799	<0.788	<0.821	<0.799	<1.84

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

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
D09	84.6	73.8	93.5	75.9
D16	107	93.1	106	86.3
D17	83.0	72.4	100	81.5
D18	103	89.7	102	83.1
P119A	89.7	78.2	104	84.1
P119B	93.2	81.1	88.8	72.0
P121	66.3	116	61.9	101
L21	59.1	103	63.5	103
L22	64.8	113	61.7	100
L31	60.4	105	59.5	96.6
P115	50.8	88.4	53.2	86.5
Effluent WWTP	52.8	92.0	57.5	93.4
Bemalingsstation	50.7	88.4	53.3	86.6
Collector Put	94.1	82.0	115	93.7
P27	99.6	86.8	90.1	73.2
P304	94.7	82.5	90.5	73.5
P305	98.8	86.1	109	88.6
P42	86.8	75.6	90.1	73.2
M4	167	72.8	195	79.1
P118C	86.7	75.5	104	84.2
P119C	87.1	75.9	87.1	70.7
P264	89.3	77.7	90.6	73.6
P371	117	102	89.3	72.5
P374	81.7	71.1	93.1	75.6
P381	84.7	73.8	96.6	78.5
P382	89.8	78.3	107	87.1
B3-bis	104	90.6	119	96.5
B7	49.6	86.5	60.8	98.8
P378	91.1	79.3	103	83.7
PA112	103	89.9	112	90.9
Field Trip Blank	106	92.1	126	102

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, except for the QC Lab High inj2 (recovery ^{13}C-PFOS > 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 February 2nd, 2017

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date February 2nd, 2017

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

SGS

3M Corporate Environmental Programs.
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ANALYTICAL REPORT

IAC17-01257-002

3M Lab Request Number: E17-0242

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

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Date issued: May 31st, 2017

Requester

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1. Introduction - Summary.

At the request (Lab Request Number: E17-0242) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, one water sample was collected on April 18th, 2017 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 Noord	0.402	0.802	0.899	<0.025 (a)
Blokkersdijkvijver std (filter)	0.315	0.808	0.566	<0.025 (b)
Blokkersdijkvijver std (niet filter)	0.401	0.829	1.67	<0.025 (c)

(a): The recovery of FOSA for BDV Noord is ±40%
(b): The recovery of FOSA for BDV std (filter) is ±80%.
(c): The recovery of FOSA for BDV std (niet filter) is ±55%

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 Noord	400	0.1	Solution A	0.25	-
		0.25	Solution B	-	1.0
Blokkersdijkvijver std filter)	400	0.1	Solution A	0.25	-
		0.25	Solution B	-	1.0
Blokkersdijkvijver std (niet filter)	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 Noord	40	8
Blokkersdijkvijver std (filter)	40	8
Blokkersdijkvijver std (niet filter)	40	8
Field Trip Blank	40	8

2.3. Determination of suspended solids in water.

To avoid the influence of algae on the analytical result, filtration is proposed prior to extraction at the sampling locations Blokkersdijkvijver - standard.
The filtration was done by using Whatman GF/C glass-fiber filters, with a pore size of 1.2 µm.
The total suspended solids of the samples is determined by pouring a measured volume of sample, typically 200 mL, through a pre-weighed filter then weighing the filter again after drying the filter overnight to remove all water. The gain in weight is a dry weight measure of the particulates present in the water sample. This is expressed in units calculated from the volume of water filtered, milligrams per liter. Table 4 summarizes the results.

Table 4. Suspended solids.

Sample Identification	Suspended solids/ volume (mg/L)
Blokkersdijkvijver - standard (filtered)	0.0005

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
09 May 2017	LCSL01	0.125	90.9	93.0	86.9	91.5	80.0	104	95.8	97.0
	LCSL02	0.125	92.9	81.8	96.6	94.8	82.0	102	93.8	98.2
	Average		**91.9**	**87.4**	**91.8**	**93.2**	**81.0**	**103**	**94.8**	**97.6**
	%RPD		**2.2**	**13**	**11**	**3.5**	**2.5**	**2.0**	**2.1**	**1.2**
	LCSH 01	2.5	96.5	101	89.8	109	81.3	101	102	100
	LCSH 02	2.5	102	107	97.5	112	94.8	115	118	113
	Average		**99.2**	**104**	**93.7**	**111**	**88.1**	**109**	**110**	**107**
	%RPD		**5.3**	**6.0**	**8.2**	**2.4**	**15**	**14**	**14**	**12**

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	87.3	<0.025	86.2
Blokkersdijkvijver Noord	0.402	91.4	0.802	101
Blokkersdijkvijver std (filter)	0.315	89.3	0.808	90.4
Blokkersdijkvijver std (niet filter)	0.401	87.4	0.829	86.4

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	94.7	<0.025	82.1
Blokkersdijkvijver Noord	0.899	100	<0.025	62.0 (a)
Blokkersdijkvijver std (filter)	0.566	104	<0.025	19.6 (a)
Blokkersdijkvijver std (niet filter)	1.67	85.9	<0.025	46.8 (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 Noord is ± 40%.
- The accuracy for FOSA for BDV std (filter) is ± 80%.
- The accuracy for FOSA for BDV std (niet filter) is ± 55%.

5. Conclusion.

- The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130% for three 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 Tine Mandonx (ERM Belgium).

8. Signatures.

Geert De Smet,
Lab Operations Manager

Date May 31st, 2017

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date May 31st, 2017

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

SGS

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

ANALYTICAL REPORT

IAC17-01257-001

3M Lab Request Number: E17-0242

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
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Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: May 31st, 2017

Requester

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1. Introduction - Summary.

At the request (Lab Request Number: E17-0242) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected between April 14th and 18th, 2017 from 11 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C-labeled analogue of PFOA ($C_6F_{13}{}^{13}CF_2{}^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C-labeled analogue of PFOS ($C_7F_{15}{}^{13}CF_2SO3^-$)
- 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		477	2274	4177	<17.0
P114bis		0.910	1.61	9.28	<0.425
Effluent WWTP		22.8	20.1	72.7	<0.425
PP02	<17.3	2192	378	21656	<32.8
PP05	5543	1602	474	92.5	<32.8
PP07	86.0	88.9	339	2595	37.2
PP09	<9.00	233	431	2139	<17.0
12		18.5	73.1	287	<1.55
5		23.8	30.5	49.5	<0.425
Bemalingsstation		19.2	26.3	48.4	<0.425
Collector Put		32.1	99.6	316	9.44

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
P321	100
P114bis	100
Effluent WWTP	100
PP02	100
PP05	100
PP07	100
PP09	100
12	100
5	100
Bemalingsstation	100
Collector Put	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
P321	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P114bis	1.0	0.1	Solution A	2.0	-
Effluent WWTP	1.0	0.1	Solution A	2.0	-
PP02	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP05	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP07	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP09	1.0	0.05	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
12	1.0	0.05	Solution A	10.0	-
5	1.0	0.05	Solution A	2.0	-
Bemalingsstation	1.0	0.05	Solution A	2.0	-
Collector Put	1.0	0.05	Solution A	5.0	-
Field Trip Blank	1.0	0.1	Solution A	10.0	-

(*): MeOH:LCMS-water (60:40)
Solution A = 1000 ng/mL (nominal) ^{13}C-PFC Surrogate Standards

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ^{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 0.0737 to 0.195 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.225 to 45.8 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
9 May 2017	LCS1	80	87.2	91.3	87.6	83.7	105	90.4	108

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing PFBS, PFHS, PFOA, ^{13}C-PFOA, PFOS, FOSA and ^{13}C-PFOS (QC Lab Low and QC Lab High).
The QCs produced recoveries within the method acceptance criteria of ± 25% for accuracy.
Table 8 summarizes the QCs recovery results.

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C-PFOA	PFOS	FOSA	^{13}C-PFOS
10 August 2016	QC Lab Low inj1	10	119	109	118	124	125	117	113
	QC Lab High inj1	100	89.2	80.8	103	86.0	110	94.3	87.2
	QC Lab Low inj2	10	120	102	105	108	118	108	112
	QC Lab High inj2	100	85.8	77.9	89.5	75.3	110	90.1	83.8

3.7. Equations.

Recovery.

$$\text{Recovery (\%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

The following tables summarize the results for the sample locations.

Table 9. Sample Results PFBS, PFHS, PFOA, PFOS and FOSA

	PFBS	PFHS	PFOA	PFOS	FOSA
	ng/ml	ng/ml	ng/ml	ng/ml	ng/ml
P321		477	2274	4177	<17.0
P114bis		0.910	1.61	9.28	<0.425
Effluent WWTP		22.8	20.1	72.7	<0.425
PP02	<17.3	2192	378	21656	<32.8
PP05	5543	1602	474	92.5	<32.8
PP07	86.0	88.9	339	2595	37.2
PP09	<9.00	233	431	2139	<17.0
12		18.5	73.1	287	<1.55
5		23.8	30.5	49.5	<0.425
Bemalingsstation		19.2	26.3	48.4	<0.425
Collector Put		32.1	99.6	316	9.44
Field Trip Blank	<0.818	<0.880	<1.87	<2.16	<1.55

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

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
P321	93.2	81.2	112	90.9
P114bis	60.6	106	59.5	96.7
Effluent WWTP	56.9	99.1	58.7	95.4
PP02	185	80.5	187	75.8
PP05	170	73.9	240	97.5
PP07	90.0	78.4	107	86.7
PP09	86.9	75.7	104	84.4
12	116	101	121	98.1
5	56.3	98.1	55.1	89.4
Bemalingsstation	55.7	97.0	52.2	84.8
Collector Put	111	96.9	114	92.7
Field Trip Blank	126	110	120	97.4

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 Tine Mandonx (ERM Belgium).

8. Signatures.

Geert De Smet,
Lab Operations Manager — Date May 31st, 2017

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories — Date May 31st, 2017

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

IAC17-03038_MO

3M Lab Request Number: E17-0595

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: July 27th, 2017

Requester

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

1. Introduction.

At the request (Lab Request Number: E17-0595) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected between June 28th and July 3th, 2017 from 1 location and analyzed for:

- Total Petroleum Hydrocarbons (TPH)

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

The sample was collected in a 1L glass container containing HCl as a preservative. The bottle was sent by SGS Belgium NV, Division IAC Laboratory.

2.2. Analysis of mineral oil.

The samples was analyzed according to CMA/3/R.1 and WAC/IV/B/025, the official Dutch method.

3. Results.

Table 1 summarizes the sample results.

Table 1. Mineral oil results.

Sample identification	Concentration (in µg/L)				
	Fraction C-10 -C-12	Fraction C-12 -C-20	Fraction C-20 -C-30	Fraction C-30 -C-40	Mineral oil
P28	<150	<150	<150	<150	<500

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 July 27th, 2017

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date July 27th, 2017

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

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The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.

SGS

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

ANALYTICAL REPORT

IAC17-03038

3M Lab Request Number: E17-0595

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: July 20th, 2017

Requester

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

1. Introduction - Summary.

At the request (Lab Request Number: E17-0595) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected between June 28th and July 3th, 2017 from 31 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
Effluent WWTP		2.24	2.05	0.793	<0.269
Bemalingsstation		48.5	72.2	68.2	<0.269
Collector put		27.8	106	311	13.5
BD24-3		37.8	37.8	1068	<5.88
BD24-4		5.40	14.2	4.64	<0.181
D10		85.5	289	19.0	<1.96
D11		29.3	72.0	734	54.6
D14		1.72	2.83	1.22	<0.181
D2		<7.67	<5.24	9.10	<5.88
D5		181	608	487	8.69
ND7		10.2	169	204	140
P118A		1481	1662	125	<10.8
P118B		1953	1619	9337	<20.6
K3	955	45202	<175	4509	<196
P27	516	20.5	117	1324	31.2
P21B	6348	7247	10037	41199	<77.8
P56	5.60	11.4	38.8	312	18.5
L19		62.2	209	3165	21.6
P262		426	1321	3731	28.5
P263		373	2221	7100	<39.3
P340		261	686	2980	51.7
P341		208	558	4006	42.3
P343		340	734	4945	<20.6
P379		19.8	81.5	670	35.0
P380		44.1	105	551	60.8
P372		40.5	56.9	951	23.0
PA109A		36.1	59.8	137	1.22
PA111A		641	820	1514	<10.8

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
Effluent WWTP	100
Bemalingsstation	100
Collector put	100
BD24-3	100
BD24-4	100
D10	100
D11	100
D14	100
D2	100
D5	100
ND7	100
P118A	100
P118B	100
K3	100
P27	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
P18	100
P28	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
Effluent WWTP	1.0	0.05	Solution A	10.0	-
Bemalingsstation	1.0	0.05	Solution A	2.0	-
Collector put	1.0	0.1	Solution A	2.0	1.0mL extract + 1.0mL MeOH (*)
BD24-3	1.0	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
BD24-4	1.0	0.05	Solution A	10.0	-
D10	1.0	0.1	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
D11	1.0	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
D14	1.0	0.05	Solution A	10.0	-
D2	1.0	0.1	Solution A	2.0	1.0mL extract + 5.0mL MeOH (*)
D5	1.0	0.1	Solution A	2.0	1.0mL extract + 2.0mL MeOH (*)
ND7	1.0	0.1	Solution A	5.0	1.0mL extract + 2.0mL MeOH (*)
P118A	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P118B	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
K3	0.5	0.5	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P27	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P21B	0.5	0.25	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P56	1.0	0.1	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
L19	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P262	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P263	0.5	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P340	1.0	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P341	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P343	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P379	1.0	0.1	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P380	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P372	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PA109A	1.0	0.1	Solution A	10.0	-
PA111A	1.0	0.1	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
Field Trip Blank	1.0	0.1	Solution A	10.0	-

(*): MeOH:LCMS-water (60:40)
Solution A = 1000 ng/mL (nominal) ^{13}C-PFC Surrogate Standards

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ^{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 0.0732 to 0.115 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.150 to 101 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 July 2017	LCS1	80	110	79.5	86.1	101	92.6	82.6	87.5

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
10 August 2016	QC Lab Low inj1	10	110	93.9	125	124	114	94.9	110
	QC Lab High inj1	100	84.3	79.1	100	105	98.2	83.3	91.7
	QC Lab Low inj2	10	107	87.6	117	117	118	100	106
	QC Lab High inj2	100	85.7	77.8	109	116	104	85.6	83.2

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
Effluent WWTP		2.24	2.05	0.793	<0.269
Bemalingsstation		48.5	72.2	68.2	<0.269
Collector put		27.8	106	311	13.5
BD24-3		37.8	37.8	1068	<5.88
BD24-4		5.40	14.2	4.64	<0.181
D10		85.5	289	19.0	<1.96
D11		29.3	72.0	734	54.6
D14		1.72	2.83	1.22	<0.181
D2		<7.67	<5.24	9.10	<5.88
D5		181	608	487	8.69
ND7		10.2	169	204	140
P118A		1481	1662	125	<10.8
P118B		1953	1619	9337	<20.6
K3	955	45202	<175	4509	<196
P27	516	20.5	117	1324	31.2
P21B	6348	7247	10037	41199	<77.8
P56	5.60	11.4	38.8	312	18.5
L19		62.2	209	3165	21.6
P262		426	1321	3731	28.5
P263		373	2221	7100	<39.3
P340		261	686	2980	51.7
P341		208	558	4006	42.3
P343		340	734	4945	<20.6
P379		19.8	81.5	670	35.0
P380		44.1	105	551	60.8
P372		40.5	56.9	951	23.0
PA109A		36.1	59.8	137	1.22
PA111A		641	820	1514	<10.8
Field Trip Blank	<0.813	<1.28	<0.874	<1.14	<0.980

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

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
Effluent WWTP	71.6	125	55.8	90.6
Bemalingsstation	71.1	124	51.8	84.1
Collector put	140	122	109	88.8
BD24-3	147	128	101	82.2
BD24-4	67.7	118	44.9	72.9
D10	136	118	116	94.6
D11	143	125	106	85.9
D14	71.9	125	44.3	72.0
D2	111	96.5	127	103
D5	137	119	121	98.5
ND7	141	123	106	86.2
P118A	144	125	92.1	74.7
P118B	86.9	75.8	103	83.9
K3	1078	93.9	935	76.0
P27	139	121	106	86.0
P21B	571	100	495	80.4
P56	144	126	117	94.8
L19	139	121	108	87.3
P262	147	128	89.4	72.6
P263	240	104	182	74.1
P340	139	121	99.6	80.9
P341	140	122	90.6	73.6
P343	146	128	89.1	72.4
P379	147	128	110	89.2
P380	127	110	103	84.0
P372	142	124	103	83.9
PA109A	140	122	107	87.1
PA111A	135	118	105	85.5
Field Trip Blank	132	115	125	102

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 Tine Mandonx (ERM Belgium).

8. Signatures.

Geert De Smet,
Lab Operations Manager

Date July 20th, 2017

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories

Date July 20th, 2017

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

January 2017 Sampling

Laboratory Request Number: ISO17-14-01

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 ISO17-14-01

Water Sample Analysis at 3M Antwerp, Belgium
January 2017 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 collected between January 9, 2017 and January 13, 2017 and returned to the 3M Environmental Laboratory on January 19, 2017 at ambient temperature. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS) (select locations only), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO17-14-01.

The 3M Environmental Laboratory prepared sample containers for sixty-four sampling locations. Each sample set consisted of a field sample and field sample duplicate. Nineteen 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 P265C was not collected. According to the chain of custody, in lieu of sample location Blokkersdijkvijver – oost, sample location Blokkersdijkvijver – noord "North" was 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.

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]**
Zone: 2nd Aquifer					
ISO17-14-01-001	BD24-3; Sample	51.0	44.3	1290	3.59
ISO17-14-01-001-DUP	BD24-3; Sample Dup	49.4	44.1	1270	3.55
	Average	**50.2**	**44.2**	**1280**	**3.57**
	%RPD Sample/Sample Dup	**3.2**	**0.45**	**1.6**	**1.1**
ISO17-14-01-002	D09; Sample	173	3830	1560	25.0
ISO17-14-01-002-DUP	D09; Sample Dup	170	3770	1500	25.7
	Average	**172**	**3800**	**1530**	**25.4**
	%RPD Sample/Sample Dup	**1.7**	**1.6**	**3.9**	**2.8**
ISO17-14-01-003	D10; Sample	234	86.1	23.9	0.322
ISO17-14-01-003-DUP	D10; Sample Dup	223	82.2	22.9	0.299
	Average	**229**	**84.2**	**23.4**	**0.311**
	%RPD Sample/Sample Dup	**4.8**	**4.6**	**4.3**	**7.4**
ISO17-14-01-004	D11; Sample	47.2	33.8	758	100
ISO17-14-01-004-DUP	D11; Sample Dup	47.2	33.9	788	99.0
	Average	**47.2**	**33.9**	**773**	**99.5**
	%RPD Sample/Sample Dup	**0.0**	**0.30**	**3.9**	**1.0**
ISO17-14-01-005	D14; Sample	1.98	1.22	0.573	0.0527
ISO17-14-01-005-DUP	D14; Sample Dup	2.06	1.26	0.931	0.0660
	Average	**2.02**	**1.24**	**0.752**	**0.0594**
	%RPD Sample/Sample Dup	**4.0**	**3.2**	**48 [3]**	**22 [3]**
ISO17-14-01-006	D16; Sample	171	34.0	1170	108
ISO17-14-01-006-DUP	D16; Sample Dup	176	33.5	1170	100
	Average	**174**	**33.8**	**1170**	**104**
	%RPD Sample/Sample Dup	**2.9**	**1.5**	**0.0**	**7.7**
ISO17-14-01-007	D17; Sample	6510	529	214	0.0832
ISO17-14-01-007-DUP	D17; Sample Dup	6130	533	214	0.0818
	Average	**6320**	**531**	**214**	**0.0825**
	%RPD Sample/Sample Dup	**6.0**	**0.75**	**0.0**	**1.7**
ISO17-14-01-008	D18; Sample	394	229	14.8	1.22
ISO17-14-01-008-DUP	D18; Sample Dup	420	232	15.5	1.12
	Average	**407**	**231**	**15.2**	**1.17**
	%RPD Sample/Sample Dup	**6.4**	**1.3**	**4.6**	**8.5**
ISO17-14-01-009	D5; Sample	508	336	476	*6.99*
ISO17-14-01-009-DUP	D5; Sample Dup	498	331	480	*6.96*
	Average	**503**	**334**	**478**	***6.98***
	%RPD Sample/Sample Dup	**2.0**	**1.5**	**0.84**	***0.43***

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 ± 11%, PFHS ± 13%, and PFOS ± 20%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 23%, PFHS ± 18%, PFOS ± 24%, and PFOSA ± 9.8%.

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

(4) Data uncertainty was expanded for PFOA to ±37% based on LCS recovery. See section 4 for more information.

Table 1 continued. Sample Results Summary [1]

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA [2]**
Zone: 2nd Aquifer					
ISO17-14-01-010	ND7; Sample	297	49.7	41.5	59.8
ISO17-14-01-010-DUP	ND7; Sample Dup	289	49.1	40.3	58.9
	Average	**293 [4]**	**49.4**	**40.9**	**59.4**
	%RPD Sample/Sample Dup	**2.7**	**1.2**	**2.9**	**1.5**
ISO17-14-01-011	P118A; Sample	1120	821	5080	25.1
ISO17-14-01-011-DUP	P118A; Sample Dup	1150	825	5020	24.6
	Average	**1140**	**823**	**5050**	**24.9**
	%RPD Sample/Sample Dup	**2.6**	**0.49**	**1.2**	**2.0**
ISO17-14-01-012	P118B; Sample	2180	3160	15300	3.40
ISO17-14-01-012-DUP	P118B; Sample Dup	2150	3220	15700	3.51
	Average	**2170**	**3190**	**15500**	**3.46**
	%RPD Sample/Sample Dup	**1.4**	**1.9**	**2.6**	**3.2**
ISO17-14-01-013	P119A; Sample	275	156	1320	52.8
ISO17-14-01-013-DUP	P119A; Sample Dup	273	155	1370	56.3
	Average	**274 [4]**	**156**	**1350**	**54.6**
	%RPD Sample/Sample Dup	**0.73**	**0.64**	**3.7**	**6.4**
ISO17-14-01-014	P119B; Sample	1000	464	148	0.957
ISO17-14-01-014-DUP	P119B; Sample Dup	1030	483	146	0.836
	Average	**1020**	**474**	**147**	**0.897**
	%RPD Sample/Sample Dup	**3.0**	**4.0**	**1.4**	**13**
ISO17-14-01-015	P121; Sample	0.273	0.107	2.02	0.144
ISO17-14-01-015-DUP	P121; Sample Dup	0.249	0.103	2.07	0.135
	Average	**0.261 [2]**	**0.105 [2]**	**2.05 [2]**	**0.140**
	%RPD Sample/Sample Dup	**9.2**	**3.8**	**2.4**	**6.5**
ISO17-14-01-016	P321; Sample	2000	588	6430	1.46
ISO17-14-01-016-DUP	P321; Sample Dup	2220	615	5920	1.37
	Average	**2110**	**602**	**6180**	**1.42**
	%RPD Sample/Sample Dup	**10**	**4.5**	**8.3**	**6.4**
Zone: Blokkersdijk nature reserve					
ISO17-14-01-017	3M vijver; Sample	0.949	0.455	2.91	<0.0248
ISO17-14-01-017-DUP	3M vijver; Sample Dup	1.03	0.541	3.25	<0.0248
	Average	**0.990**	**0.498**	**3.08**	**<0.0248**
	%RPD Sample/Sample Dup	**8.2**	**17**	**11**	**NA**
ISO17-14-01-018	Blokkersdijkvijver North; Sample	0.827	0.509	1.35	<0.0248
ISO17-14-01-018-DUP	Blokkersdijkvijver North; Sample Dup	0.811	0.487	1.37	<0.0248
	Average	**0.819**	**0.498**	**1.36**	**<0.0248**
	%RPD Sample/Sample Dup	**2.0**	**4.4**	**1.5**	**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 ± 21%, PFBS ± 11%, PFHS ± 13%, and PFOS ± 20%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 23%, PFHS ± 18%, PFOS ± 24%, and PFOSA ± 9.8%.

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

(4) Data uncertainty was expanded for PFOA to ±37% based on LCS recovery. See section 4 for more information.

Table 1 continued. Sample Results Summary [1]

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA [2]
Zone: Blokkersdijk nature reserve					
ISO17-14-01-019	Blokkersdijkvijver standard; Sample	0.803	0.473	0.775	<0.0248
ISO17-14-01-019-DUP	Blokkersdijkvijver standard; Sample Dup	0.784	0.462	0.705	<0.0248
	Average	**0.794**	**0.468**	**0.740**	**<0.0248**
	%RPD Sample/Sample Dup	**2.4**	**2.4**	**9.5**	**NA**
ISO17-14-01-020	L21; Sample	0.295	0.156	5.40	0.0897
ISO17-14-01-020-DUP	L21; Sample Dup	0.282	0.145	5.41	0.0850
	Average	**0.289** [2]	**0.151** [2]	**5.41** [2]	**0.0874**
	%RPD Sample/Sample Dup	**4.5**	**7.3**	**0.19**	**5.4**
ISO17-14-01-021	L22; Sample	0.340	0.0859	4.21	0.0746
ISO17-14-01-021-DUP	L22; Sample Dup	0.369	0.0899	4.36	0.0967
	Average	**0.355** [2]	**0.0879** [2]	**4.29** [2]	**0.0857**
	%RPD Sample/Sample Dup	**8.2**	**4.6**	**3.5**	**26** [3]
ISO17-14-01-022	L31; Sample	0.721	1.02	5.26	0.0404
ISO17-14-01-022-DUP	L31; Sample Dup	0.698	1.01	5.17	0.0541
	Average	**0.710**	**1.02**	**5.22**	**0.0473**
	%RPD Sample/Sample Dup	**3.2**	**0.99**	**1.7**	**29** [3]
ISO17-14-01-023	L4; Sample	2.78	2.25	28.5	1.46
ISO17-14-01-023-DUP	L4; Sample Dup	2.82	2.36	29.8	1.51
	Average	**2.80**	**2.31**	**29.2**	**1.49**
	%RPD Sample/Sample Dup	**1.4**	**4.8**	**4.5**	**3.4**
ISO17-14-01-024	P114bis; Sample	3.62	1.37	9.20	0.0253
ISO17-14-01-024-DUP	P114bis; Sample Dup	3.63	1.43	9.22	0.0330
	Average	**3.63**	**1.40**	**9.21**	**0.0292**
	%RPD Sample/Sample Dup	**0.28**	**4.3**	**0.22**	**26** [3]
ISO17-14-01-025	P115; Sample	3.82	2.13	0.547	<0.0248
ISO17-14-01-025-DUP	P115; Sample Dup	4.05	2.29	0.639	<0.0248
	Average	**3.94**	**2.21**	**0.593**	**<0.0248**
	%RPD Sample/Sample Dup	**5.8**	**7.2**	**16**	**NA**
ISO17-14-01-026	P116; Sample	0.916	0.580	10.7	0.0841
ISO17-14-01-026-DUP	P116; Sample Dup	0.931	0.594	11.1	0.110
	Average	**0.924**	**0.587**	**10.9**	**0.0971**
	%RPD Sample/Sample Dup	**1.6**	**2.4**	**3.7**	**27** [3]
Zone: Effluent WWTP					
ISO17-14-01-027	Effluent WWTP; Sample	0.438	0.430	2.60	0.120
ISO17-14-01-027-DUP	Effluent WWTP; Sample Dup	0.382	0.387	2.30	0.0932
	Average	**0.410** [2]	**0.409** [2]	**2.45** [2]	**0.107**
	%RPD Sample/Sample Dup	**14**	**11**	**12**	**25** [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 ± 11%, PFHS ± 13%, and PFOS ± 20%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 23%, PFHS ± 18%, PFOS ± 24%, and PFOSA ± 9.8%.

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

(4) Data uncertainty was expanded for PFOA to ±37% based on LCS recovery. See section 4 for more information.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA** (2)
Zone: Palingbeek & Tophatgracht					
ISO17-14-01-037	12; Sample	66.8	20.1	276	0.207
ISO17-14-01-037-DUP	12; Sample Dup	68.9	20.4	270	0.204
	Average	**67.9**	**20.3**	**273**	**0.206**
	%RPD Sample/Sample Dup	**3.1**	**1.5**	**2.2**	**1.5**
ISO17-14-01-038	13; Sample	61.4	20.1	230	0.194
ISO17-14-01-038-DUP	13; Sample Dup	60.0	19.4	230	0.201
	Average	**60.7**	**19.8**	**230**	**0.198**
	%RPD Sample/Sample Dup	**2.3**	**3.5**	**0.0**	**3.5**
ISO17-14-01-039	5; Sample	27.0	23.2	57.6	0.213
ISO17-14-01-039-DUP	5; Sample Dup	26.8	23.0	55.4	0.238
	Average	**26.9**	**23.1**	**56.5**	**0.226**
	%RPD Sample/Sample Dup	**0.74**	**0.87**	**3.9**	**11**
ISO17-14-01-040	Bemalingsstation; Sample	25.8	22.4	53.5	0.190
ISO17-14-01-040-DUP	Bemalingsstation; Sample Dup	25.5	22.0	52.1	0.195
	Average	**25.7**	**22.2**	**52.8**	**0.193**
	%RPD Sample/Sample Dup	**1.2**	**1.8**	**2.7**	**2.6**
Zone: Sewer					
ISO17-14-01-041	Collector put; Sample	27.3	13.3	104	5.76
ISO17-14-01-041-DUP	Collector put; Sample Dup	27.7	13.5	104	5.56
	Average	**27.5**	**13.4**	**104**	**5.66**
	%RPD Sample/Sample Dup	**1.5**	**1.5**	**0.0**	**3.5**
Zone: Source area – WWTP					
ISO17-14-01-047	L19; Sample	300	169	3880	37.7
ISO17-14-01-047-DUP	L19; Sample Dup	317	174	4070	37.4
	Average	**309**	**172**	**3980**	**37.6**
	%RPD Sample/Sample Dup	**5.5**	**2.9**	**4.8**	**0.80**
ISO17-14-01-048	M4; Sample	429	189	4930	129
ISO17-14-01-048-DUP	M4; Sample Dup	428	196	4990	129
	Average	**429**	**193**	**4960**	**129**
	%RPD Sample/Sample Dup	**0.23**	**3.6**	**1.2**	**0.0**
ISO17-14-01-049	P118C; Sample	253	80.1	1710	66.7
ISO17-14-01-049-DUP	P118C; Sample Dup	243	77.8	1660	67.9
	Average	**248**	**79.0**	**1690**	**67.3**
	%RPD Sample/Sample Dup	**4.0**	**2.9**	**3.0**	**1.8**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 21%, PFBS ± 11%, PFHS ± 13%, and PFOS ± 20%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 23%, PFHS ± 18%, PFOS ± 24%, and PFOSA ± 9.8%.

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

(4) Data uncertainty was expanded for PFOA to ±37% based on LCS recovery. See section 4 for more information.

Table 1 continued. Sample Results Summary [(1)]

		Concentration (ng/mL)			
3M LIMS ID	Sample Description	PFOA	PFHS	PFOS	PFOSA [(2)]
Zone: Source area - WWTP					
ISO17-14-01-050	P119C; Sample	923	688	2770	61.4
ISO17-14-01-050-DUP	P119C; Sample Dup	990	740	2840	58.5
	Average	**957**	**714**	**2810**	**60.0**
	%RPD Sample/Sample Dup	**7.0**	**7.3**	**2.5**	**4.8**
ISO17-14-01-051	P263; Sample	1210	422	7990	15.3
ISO17-14-01-051-DUP	P263; Sample Dup	1340	451	8720	16.1
	Average	**1280**	**437**	**8360**	**15.7**
	%RPD Sample/Sample Dup	**10**	**6.6**	**8.7**	**5.1**
ISO17-14-01-052	P264; Sample	249	162	1480	78.6
ISO17-14-01-052-DUP	P264; Sample Dup	241	159	1450	73.3
	Average	**245**	**161**	**1470**	**76.0**
	%RPD Sample/Sample Dup	**3.3**	**1.9**	**2.0**	**7.0**
ISO17-14-01-054	P371; Sample	163	142	2770	47.3
ISO17-14-01-054-DUP	P371; Sample Dup	159	139	2830	57.8
	Average	**161**	**141**	**2800**	**52.6**
	%RPD Sample/Sample Dup	**2.5**	**2.1**	**2.1**	**20**
ISO17-14-01-055	P374; Sample	509	760	2640	36.3
ISO17-14-01-055-DUP	P374; Sample Dup	524	784	2870	28.2
	Average	**517**	**772**	**2760**	**32.3**
	%RPD Sample/Sample Dup	**2.9**	**3.1**	**8.3**	**25** [(3)]
ISO17-14-01-056	P381; Sample	477	140	1390	12.9
ISO17-14-01-056-DUP	P381; Sample Dup	462	136	1460	14.4
	Average	**470**	**138**	**1430**	**13.7**
	%RPD Sample/Sample Dup	**3.2**	**2.9**	**4.9**	**11**
ISO17-14-01-057	P382; Sample	379	123	886	77.1
ISO17-14-01-057-DUP	P382; Sample Dup	381	121	833	81.3
	Average	**380**	**122**	**860**	**79.2**
	%RPD Sample/Sample Dup	**0.53**	**1.6**	**6.2**	**5.3**
Zone: Southern site boundary					
ISO17-14-01-058	B3-bis; Sample	36.7	6.00	110	0.217
ISO17-14-01-058-DUP	B3-bis; Sample Dup	37.5	6.35	113	0.203
	Average	**37.1**	**6.18**	**112**	**0.210**
	%RPD Sample/Sample Dup	**2.2**	**5.7**	**2.7**	**6.7**
ISO17-14-01-059	B7; Sample	4.54	1.65	69.2	0.660
ISO17-14-01-059-DUP	B7; Sample Dup	4.13	1.53	65.0	0.578
	Average	**4.34**	**1.59**	**67.1**	**0.619**
	%RPD Sample/Sample Dup	**9.5**	**7.5**	**6.3**	**13**

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 ± 11%, PFHS ± 13%, and PFOS ± 20%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 23%, PFHS ± 18%, PFOS ± 24%, and PFOSA ± 9.8%.

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

(4) Data uncertainty was expanded for PFOA to ±37% based on LCS recovery. See section 4 for more information.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA (2)**
Zone: Source area - WWTP					
ISO17-14-01-060	P372; Sample	65.9	43.6	1180	66.6
ISO17-14-01-060-DUP	P372; Sample Dup	67.6	44.1	1200	70.5
	Average	**66.8**	**43.9**	**1190**	**68.6**
	%RPD Sample/Sample Dup	**2.5**	**1.1**	**1.7**	**5.7**
ISO17-14-01-061	P378; Sample	295	327	1570	2.26
ISO17-14-01-061-DUP	P378; Sample Dup	309	347	1730	2.46
	Average	**302**	**337**	**1650**	**2.36**
	%RPD Sample/Sample Dup	**4.6**	**5.9**	**9.7**	**8.5**
ISO17-14-01-062	PA109A; Sample	43.4	48.4	175	1.62
ISO17-14-01-062-DUP	PA109A; Sample Dup	45.2	50.4	166	1.66
	Average	**44.3**	**49.4**	**171**	**1.64**
	%RPD Sample/Sample Dup	**4.1**	**4.0**	**5.3**	**2.4**
ISO17-14-01-063	PA111A; Sample	642	925	2180	10.7
ISO17-14-01-063-DUP	PA111A; Sample Dup	653	925	2190	10.9
	Average	**648**	**925**	**2190**	**10.8**
	%RPD Sample/Sample Dup	**1.7**	**0.0**	**0.46**	**1.9**
ISO17-14-01-064	PA112; Sample	97.8	34.7	376	75.1
ISO17-14-01-064-DUP	PA112; Sample Dup	97.5	34.5	377	81.2
	Average	**97.7**	**34.6**	**377**	**78.2**
	%RPD Sample/Sample Dup	**0.31**	**0.58**	**0.27**	**7.8**

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 ± 11%, PFHS ± 13%, and PFOS ± 20%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 23%, PFHS ± 18%, PFOS ± 24%, and PFOSA ± 9.8%.

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

(4) Data uncertainty was expanded for PFOA to ±37% based on LCS recovery. See section 4 for more information.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFOA	PFBS	PFHS	PFOS	PFOSA (2)
Zone: Extractions wells P&T						
ISO17-14-01-028	PP01; Sample	45.2	47.0	5510	1900	20.0
ISO17-14-01-028-DUP	PP01; Sample Dup	47.5	49.5	5240	1870	20.8
	Average	**46.4**	**48.3**	**5380**	**1890**	**20.4**
	%RPD Sample/Sample Dup	**5.0**	**5.2**	**5.0**	**1.6**	**3.9**
ISO17-14-01-029	PP02; Sample	403	26.5	2960	24800	48.4
ISO17-14-01-029-DUP	PP02; Sample Dup	381	23.8	2780	22500	45.9
	Average	**392**	**25.2**	**2870**	**23700**	**47.2**
	%RPD Sample/Sample Dup	**5.6**	**11**	**6.3**	**9.7**	**5.3**
ISO17-14-01-030	PP04; Sample	1090	2260	401	4990	45.8
ISO17-14-01-030-DUP	PP04; Sample Dup	1120	2000	391	4450	33.2
	Average	**1110**	**2130**	**396**	**4720**	**39.5**
	%RPD Sample/Sample Dup	**2.7**	**12**	**2.5**	**11**	**32 (3)**
ISO17-14-01-031	PP05; Sample	342	3380	1540	149	23.6
ISO17-14-01-031-DUP	PP05; Sample Dup	319	2850	1440	121	19.7
	Average	**331**	**3120**	**1490**	**135**	**21.7**
	%RPD Sample/Sample Dup	**7.0**	**17**	**6.7**	**21 (3)**	**18**
ISO17-14-01-032	PP06; Sample	119	10.7	40.0	1250	118
ISO17-14-01-032-DUP	PP06; Sample Dup	131	11.7	50.0	1360	100
	Average	**125**	**11.2**	**45.0**	**1310**	**109**
	%RPD Sample/Sample Dup	**9.6**	**8.9**	**22 (3)**	**8.4**	**17**
ISO17-14-01-033	PP07; Sample	477	48.7	214	5640	59.6
ISO17-14-01-033-DUP	PP07; Sample Dup	575	34.1	301	3940	33.9
	Average	**526**	**41.4**	**258**	**4790**	**46.8**
	%RPD Sample/Sample Dup	**19**	**35 (3)**	**34 (3)**	**35 (3)**	**55 (3)**
ISO17-14-01-034	PP08; Sample	487	32.4	261	6820	52.3
ISO17-14-01-034-DUP	PP08; Sample Dup	499	31.9	264	6980	51.6
	Average	**493**	**32.2**	**263**	**6900**	**52.0**
	%RPD Sample/Sample Dup	**2.4**	**1.6**	**1.1**	**2.3**	**1.3**
ISO17-14-01-035	PP09; Sample	631	25.9	391	2600	16.0
ISO17-14-01-035-DUP	PP09; Sample Dup	613	24.1	378	2590	15.8
	Average	**622**	**25.0**	**385**	**2600**	**15.9**
	%RPD Sample/Sample Dup	**2.9**	**7.2**	**3.4**	**0.39**	**1.3**
ISO17-14-01-036	PP10; Sample	600	24.6	374	2580	14.9
ISO17-14-01-036-DUP	PP10; Sample Dup	598	24.4	377	2600	15.1
	Average	**599**	**24.5**	**376**	**2590**	**15.0**
	%RPD Sample/Sample Dup	**0.33**	**0.82**	**0.80**	**0.77**	**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 ± 21%, PFBS ± 11%, PFHS ± 13%, and PFOS ± 20%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 23%, PFHS ± 18%, PFOS ± 24%, and PFOSA ± 9.8%.

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

(4) Data uncertainty was expanded for PFOA to ±37% based on LCS recovery. See section 4 for more information.

Table 1 continued. Sample Results Summary (1)

3M LIMS ID	Sample Description	Concentration (ng/mL) PFOA	PFBS	PFHS	PFOS	PFOSA (2)
Zone: Source area - Building 16						
ISO17-14-01-042	P27; Sample	135	1020	46.8	1870	54.5
ISO17-14-01-042-DUP	P27; Sample Dup	127	962	43.4	1770	56.4
	Average	**131**	**991**	**45.1**	**1820**	**55.5**
	%RPD Sample/Sample Dup	**6.1**	**5.9**	**7.5**	**5.5**	**3.4**
ISO17-14-01-043	P21B; Sample	7280	4580	7450	59300	27.1
ISO17-14-01-043-DUP	P21B; Sample Dup	6970	4780	7310	45000	11.3
	Average	**7130**	**4680**	**7380**	**52300**	**19.2**
	%RPD Sample/Sample Dup	**4.4**	**4.3**	**1.9**	**27 (3)**	**82 (3)**
ISO17-14-01-044	P304; Sample	403	272	126	1340	28.1
ISO17-14-01-044-DUP	P304; Sample Dup	400	270	124	1350	28.5
	Average	**402**	**271**	**125**	**1350**	**28.3**
	%RPD Sample/Sample Dup	**0.75**	**0.74**	**1.6**	**0.74**	**1.4**
ISO17-14-01-045	P305; Sample	137	116	221	506	22.9
ISO17-14-01-045-DUP	P305; Sample Dup	142	119	218	498	21.6
	Average	**140**	**118**	**220**	**502**	**22.3**
	%RPD Sample/Sample Dup	**3.6**	**2.6**	**1.4**	**1.6**	**5.8**
ISO17-14-01-046	P42; Sample	231	42.2	551	4700	14.8
ISO17-14-01-046-DUP	P42; Sample Dup	234	42.6	535	4540	14.9
	Average	**233**	**42.4**	**543**	**4620**	**14.9**
	%RPD Sample/Sample Dup	**1.3**	**0.94**	**2.9**	**3.5**	**0.67**

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 ± 11%, PFHS ± 13%, and PFOS ± 20%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 23%, PFHS ± 18%, PFOS ± 24%, and PFOSA ± 9.8%.

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

(4) Data uncertainty was expanded for PFOA to ±37% based on LCS recovery. See section 4 for more information.

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 9, 2017 and January 13, 2017 in Nalgene™ (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 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, PFHS, and PFOS and PFBS for select locations using a direct injection by methanol dilution method. Samples that required a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples that required a 1:100 dilution were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol. Samples that required a 1:500 dilution were prepared by diluting 0.02 mL of a well-mixed sample with 10.0 mL of methanol. Samples that required a 1:1000 dilution were prepared by first diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol, followed by a second dilution prepared by diluting 1 mL of the diluted sample into 9 mL of methanol. Diluted samples and LCSs were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

Sample locations P121, L21, L22, Effluent WWTP, and Travel Blank sample and FMS Low 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). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples along with the calibration standards and method blanks.

All samples were prepared for PFOSA by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The laboratory control samples were then diluted with methanol in the same manner. 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 the target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). SRSs were added and included in the analysis of these samples. Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.1. 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:

1/27/17 (ETS Kirk) External Standard Calibration Analysis:

- Sample locations ISO17-14-01-001 to ISO17-14-01-033 were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported **except** for the following: D09 (PFHS), D17 (PFOA), P119A (PFOS), P121 (all analytes), L21 (all analytes), L22 (all analytes), Effluent WWTP (all analytes), PP01 (PFHS), PP05 (all analytes), and PP07 (PFHS).

***1/31/17 (ETS Kirk) External Standard Calibration Analysis*:**

- Sample locations ISO17-14-01-034 to ISO17-14-01-065 were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported **except** for the following: P21B (PFOS) and Travel Blank sample and FMS Low.
- In addition, the following samples were re-analyzed and reported: D09 (PFHS), D17 (PFOA), P119A (PFOS), PP01 (PFHS), PP05 (all analytes), and PP07 (PFHS).

2/7/17 (ETS Kirk) External Standard Calibration Analysis:

- Sample location P21B was re-analyzed and reported for PFOS.

***2/8/17 (ETS Kirk) Internal Standard Calibration Analysis*:**

- Sample locations P121, L21, L22, Effluent WWTP, and Travel Blank sample and FMS Low were re-analyzed and reported for PFOA, PFHS, and PFOS.

2/3/17 (ETS Rita) Internal Standard Calibration Analysis:

- Sample locations ISO17-14-01-001 to ISO17-14-01-027 were analyzed for PFOSA. All sample results were reported **except** for the following: D17 and Blokkersdijkvijver – North.

2/6/17 (ETS Rita) Internal Standard Calibration Analysis:

- Sample locations ISO17-14-01-028 to ISO17-14-01-065 were analyzed for PFOSA. All sample results were reported.

2/9/17 (ETS Rita) Internal Standard Calibration Analysis:

- Sample locations D17 and Blokkersdijkvijver – North were re-analyzed and reported for PFOSA.

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	1/27/17, 1/31/17, 2/7/17, 2/8/17	2/3/17, 2/6/17, 2/9/17
Guard column	Prism RP (2.1 mm X 50 mm), 5 μ	Prism RP (2.1 mm X 50 mm), 5 μ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ
Injection Volume	1, 2, or 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	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard	Mass Transition Q1/Q3
PFOA	413/369	[$^{13}C_8$]-PFOA	421/376
	413/219		
	413/169		
PFBS	299/99	NA	NA
	299/80		
PFHS	399/99	[$^{13}C_3$]-PFHS	402/80
	399/80		
PFOS	499/99	[$^{13}C_8$]-PFOS	507/80
	499/80		
	499/130		
PFOSA	498/78	[$^{13}C_8$]-PFOSA	506/78
[$^{13}C_4$]-PFOA	417/372	[$^{13}C_8$]-PFOA	421/376
[$^{13}C_4$]-PFOS	503/80	[$^{13}C_8$]-PFOS	507/80

NA = Not Applicable
The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

3 Data Analysis

3.1 Calibration

1/27/17, 1/31/17, 2/7/17 Analysis (External Standard Calibration): Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed on 1/27/17 and 1/31/17. On 2/7/17, calibration standards ranging from 0.25 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) or 0.25 ng/mL to 25 ng/mL (nominal) on 2/7/17. A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

2/8/17 Analysis (Internal Standard Calibration): Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of twelve calibration standards ranging from 0.0125 ng/mL to 25 ng/mL (nominal) were analyzed. The calibration standards also contained surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

2/3/17, 2/6/17, 2/9/17 Analysis of PFOSA (Internal Standard Calibration): Samples were analyzed for PFOSA against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into laboratory reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 1.0 ng/mL. Calibration standards ranging from

0.025 ng/mL to 200 ng/mL (nominal) were analyzed on 2/3/17 and 2/6/17. Standards ranged from 0.025 ng/mL to 100 ng/mL (nominal) on 2/9/17. 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) 1/27/17, 1/31/17 Analysis	LOQ, ng/mL (2) 2/3/17, 2/6/17, 2/9/17 Analysis	LOQ, ng/mL (1) 2/7/17 Analysis	LOQ, ng/mL (2) 2/8/17 Analysis
PFOA	0.0192	NA	NA	0.0240
PFBS	0.0200	NA	NA	NA
PFHS	0.0200	NA	NA	0.0250
PFOS	0.0185	NA	0.232	0.0232
PFOSA	NA	0.0248	NA	NA

NA = Not Applicable
(1) A dilution factor was not applied to the LOQ.
(2) A dilution factor of 2 was applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of 100%±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:

- 1/27/17: High-level LCSs were spiked above the resulting upper limit of quantitation (ULOQ) for PFBS (25,100 ng/mL), and PFHS (12,600 ng/mL) with a dilution factor of 502 applied. For PFOA, the high-level LCSs had an average recovery of 63.5%. The high-level LCSs showed obvious peak splitting and suppressed response due to oversaturation of the detector. While the LCSs are reported, they were excluded from the overall data uncertainty. However, since sample locations ND7 and P119A were near the upper limit of quantitation, the data uncertainty was expanded to ±37% for those two locations. A method deviation is included with the raw data.

- 1/31/17: High-level LCSs were spiked above the resulting ULOQ for PFHS (25,100 ng/mL) with dilution factor of 502 applied.

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 1/27/17	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-170126-1	100	90.4	90.4	100	94.9	94.9
LCS-170126-2	100	90.3	90.3	100	93.3	93.3
LCS-170126-3	100	88.2	88.2	100	91.6	91.6
Average ± %RSD	**89.6% ± 1.4%**			**93.3% ± 1.8%**		
LCS-170126-4	5000	4850	96.9	5000	4930	98.5
LCS-170126-5	5000	4770	95.5	5000	4980	99.5
LCS-170126-6	5000	4730	94.6	5000	4980	99.6
Average ± %RSD	**95.7% ± 1.2%**			**99.2% ± 0.61%**		
LCS-170126-7	35000	22300	63.6	35000	>ULOQ	NA
LCS-170126-8	35000	22000	63.0	35000	>ULOQ	NA
LCS-170126-9	35000	22300	63.8	35000	>ULOQ	NA
Average ± %RSD	**63.5% ± 0.66% (1)**			**NA (2)**		

NA = Not Applicable
ULOQ = Upper Limit of Quantitation
(1) LCSs did not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 1/27/17	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-170126-1	100	93.1	93.1	100	90.4	90.4
LCS-170126-2	100	93.0	93.0	100	92.7	92.7
LCS-170126-3	100	91.3	91.3	100	87.1	87.1
Average ± %RSD	**92.5% ± 1.1%**			**90.1% ± 3.1%**		
LCS-170126-4	5000	4830	96.5	5000	4990	99.9
LCS-170126-5	5000	4830	96.6	5000	5060	101
LCS-170126-6	5000	4830	96.5	5000	5150	103
Average ± %RSD	**96.5% ± 0.060%**			**101% ± 1.6%**		
LCS-170126-7	35000	>ULOQ	NA	35000	38600	110
LCS-170126-8	35000	>ULOQ	NA	35000	37600	107
LCS-170126-9	35000	>ULOQ	NA	35000	39800	114
Average ± %RSD	**NA (2)**			**110% ± 3.2%**		

ETS-8-044.3 External Calibration Analyzed 1/27/17	[$^{13}C_4$]-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-170126-1	99.8	101	101	95.6	99.1	104
LCS-170126-2	99.8	107	107	95.6	105	110
LCS-170126-3	99.8	96.2	96.4	95.6	96.2	101
Average ± %RSD	**101% ± 5.2%**			**105% ± 4.4%**		
LCS-170126-4	998	1040	105	956	1020	107
LCS-170126-5	998	1050	105	956	1050	110
LCS-170126-6	998	1050	105	956	1070	112
Average ± %RSD	**105% ± 0.0%**			**110% ± 2.3%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantitation
(1) LCSs did not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 1/31/17	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-170131-1	100	96.6	96.6	100	103	103
LCS-170131-2	100	84.1	84.1	100	95.8	95.8
LCS-170131-3	100	91.8	91.8	100	99.8	99.8
Average ± %RSD	**90.8% ± 6.9%**			**99.5% ± 3.6%**		
LCS-170131-4	5000	4710	94.2	5000	5110	102
LCS-170131-5	5000	4830	96.6	5000	5200	104
LCS-170131-6	5000	4900	98.0	5000	5140	103
Average ± %RSD	**96.3% ± 2.0%**			**103% ± 0.97%**		
LCS-170131-7	35000	30600	87.4	35000	34600	98.8
LCS-170131-8	35000	30900	88.4	35000	34700	99.0
LCS-170131-9	35000	30200	86.2	35000	33900	97.0
Average ± %RSD	**87.3% ± 1.3%**			**98.3% ± 1.1%**		

ETS-8-044.3 External Calibration Analyzed 1/31/17	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-170131-1	100	100	100	100	99.6	99.6
LCS-170131-2	100	88.7	88.7	100	85.2	85.2
LCS-170131-3	100	97.8	97.8	100	99.9	99.9
Average ± %RSD	**95.5% ± 6.3%**			**94.9% ± 8.9%**		
LCS-170131-4	5000	5010	100	5000	5370	107
LCS-170131-5	5000	5120	102	5000	5410	108
LCS-170131-6	5000	5070	101	5000	5560	111
Average ± %RSD	**101% ± 0.99%**			**109% ± 1.9%**		
LCS-170131-7	35000	>ULOQ	NA	35000	39000	112
LCS-170131-8	35000	>ULOQ	NA	35000	38300	109
LCS-170131-9	35000	>ULOQ	NA	35000	38700	111
Average ± %RSD	**NA (2)**			**111% ± 1.4%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantitation
(1) LCSs did not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 1/31/17	$[^{13}C_4]$-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-170131-1	99.8	104	104	95.6	108	113
LCS-170131-2	99.8	113	113	95.6	109	115
LCS-170131-3	99.8	105	106	95.6	107	112
Average ± %RSD	**108% ± 4.4%**			**113% ± 1.3%**		
LCS-170131-4	998	1080	108	956	1100	116
LCS-170131-5	998	1130	113	956	1120	118
LCS-170131-6	998	1130	113	956	1140	119
Average ± %RSD	**111% ± 2.6%**			**118% ± 1.3%**		

ETS-8-044.3 Internal Calibration Analyzed 2/3/17	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-170203-1	0.199	0.199	99.8
LCS-170203-2	0.199	0.205	103
LCS-170203-3	0.199	0.200	100
Average ± %RSD	**101% ± 1.8%**		
LCS-170203-4	19.9	20.1	101
LCS-170203-5	19.9	19.5	98.0
LCS-170203-6	19.9	18.9	95.0
Average ± %RSD	**98.0% ± 3.1%**		
LCS-170203-7	139	140	101
LCS-170203-8	139	141	102
LCS-170203-9	139	141	101
Average ± %RSD	**101% ± 0.57%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantitation
(1) LCSs did not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 2/6/17	PFOSA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-170203-1	0.199	0.198	99.3
LCS-170203-2	0.199	0.200	100
LCS-170203-3	0.199	0.207	104
Average ± %RSD	**101% ± 2.5%**		
LCS-170203-4	19.9	19.9	99.9
LCS-170203-5	19.9	19.5	98.0
LCS-170203-6	19.9	18.9	94.8
Average ± %RSD	**97.6% ± 2.6%**		
LCS-170203-7	139	143	103
LCS-170203-8	139	142	102
LCS-170203-9	139	139	100
Average ± %RSD	**102% ± 1.5%**		

ETS-8-044.3 External Calibration Analyzed 2/7/17	PFOS (Linear + Branched)			[$^{13}C_4$]-PFOS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-170207-1	1000	950	95.0	480	494	103
LCS-170207-2	1000	927	92.7	480	466	97.0
LCS-170207-3	1000	946	94.6	480	496	103
Average ± %RSD	**94.1% ± 1.3%**			**101% ± 3.4%**		
LCS-170207-4	25000	23100	92.3	4800	5250	109
LCS-170207-5	25000	24000	95.9	4800	5500	114
LCS-170207-6	25000	23300	93.0	4800	5150	107
Average ± %RSD	**93.7% ± 2.0%**			**110% ± 3.3%**		
LCS-170207-7	100000	99800	99.8			
LCS-170207-8	100000	95000	95.0			
LCS-170207-9	100000	97800	97.8			
Average ± %RSD	**97.5% ± 2.5%**					

NA = Not Applicable
ULOQ = Upper Limit of Quantitation
(1) LCSs did not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 2/8/17	PFOA (Linear + Branched)			PFHS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-170208-1	0.190	0.163	85.7	0.198	0.175	88.4
LCS-170208-2	0.190	0.174	91.3	0.198	0.187	94.5
LCS-170208-3	0.190	0.176	92.8	0.198	0.190	95.9
Average ± %RSD	**89.9% ± 4.2%**			**92.9% ± 4.3%**		
LCS-170208-4	1.90	1.77	93.0	1.98	1.92	96.9
LCS-170208-5	1.90	1.82	96.0	1.98	1.88	95.0
LCS-170208-6	1.90	1.71	90.1	1.98	1.88	94.7
Average ± %RSD	**93.0% ± 3.2%**			**95.5% ± 1.2%**		
LCS-170208-7	33.2	29.3	88.1	34.7	33.8	97.4
LCS-170208-8	33.2	29.5	88.9	34.7	32.9	94.8
LCS-170208-9	33.2	28.2	85.0	34.7	33.3	96.0
Average ± %RSD	**87.3% ± 2.4%**			**96.1% ± 1.4%**		

ETS-8-044.3 Internal Calibration Analyzed 2/8/17	PFOS (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-170208-1	0.184	0.165	89.5
LCS-170208-2	0.184	0.172	93.4
LCS-170208-3	0.184	0.167	90.9
Average ± %RSD	**91.3% ± 2.2%**		
LCS-170208-4	1.84	1.75	95.3
LCS-170208-5	1.84	1.67	90.9
LCS-170208-6	1.84	1.72	93.5
Average ± %RSD	**93.2% ± 2.4%**		
LCS-170208-7	32.2	31.3	97.2
LCS-170208-8	32.2	30.3	94.2
LCS-170208-9	32.2	30.1	93.5
Average ± %RSD	**95.0% ± 2.1%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantitation
(1) LCSs did not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 2/8/17	$[^{13}C_4]$-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-170208-1	0.198	0.201	102	0.189	0.194	103
LCS-170208-2	0.198	0.196	98.9	0.189	0.200	106
LCS-170208-3	0.198	0.187	94.4	0.189	0.198	105
Average ± %RSD	**98.4% ± 3.9%**			**105% ± 1.5%**		
LCS-170208-4	1.98	2.12	107	1.89	2.02	107
LCS-170208-5	1.98	2.18	110	1.89	2.06	109
LCS-170208-6	1.98	2.12	107	1.89	2.01	106
Average ± %RSD	**108% ± 1.6%**			**107% ± 1.4%**		

ETS-8-044.3 Internal Calibration Analyzed 2/9/17	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-170209-1	0.199	0.205	103
LCS-170209-2	0.199	0.193	96.9
LCS-170209-3	0.199	0.214	108
Average ± %RSD	**103% ± 5.4%**		
LCS-170209-4	19.9	20.6	103
LCS-170209-5	19.9	20.3	102
LCS-170209-6	19.9	20.2	101
Average ± %RSD	**102% ± 0.98%**		
LCS-170209-7	69.9	69.0	98.8
LCS-170209-8	69.9	67.9	97.1
LCS-170209-9	69.9	68.5	98.0
Average ± %RSD	**98.0% ± 0.87%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantitation
(1) LCSs did not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.4. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The high-level LCSs analyzed on 1/27/17 were excluded from the analytical data uncertainty for PFOA since they are not representative of normal method performance. The analytical data uncertainty is listed in Table 8 below.

Table 8. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	10.7	±21%
PFBS	External	5.68	±11%
PFHS	External	6.31	±13%
PFOS	External	10.0	±20%
PFOA	Internal	11.5	±23%
PFHS	Internal	8.88	±18%
PFOS	Internal	11.9	±24%
PFOSA	Internal	4.90	±9.8%

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. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

The following calculation was used to generate data in section 4 of the report.

$$FMS\ Recovery = \frac{Sample\ Concentration\ of\ FMS - Average\ Concentration\ (Field\ Sample\ \&\ Field\ Sample\ Dup)}{Spike\ Concentration} \times 100\%$$

Table 9. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
P121, L21, P115	FMS	2.40	2.50	2.50	2.32	2.50
Blokkersdijkvijver – North, B3-bis	FMS	4.79	5.00	5.00	4.64	5.00
PP01	FMS	100	100	100	100	100
Effluent WWTP	FMS	21.0	21.0	21.0	20.9	1.00
D10, Bemalingsstation	FMS	101	101	101	101	1.00
P304	FMS	100	100	100	100	25.0
P378	FMS	511	10.0	510	510	10.0
D11, Collector put	FMS	501	50.0	500	500	50.0
P371	FMS	200	100	200	200	100
P118A, PP09, PA111A	FMS	1000	50.0	1000	1000	50.0
P119C, P264	FMS	1000	100	1000	1000	100
Trip Blank	Low	2.40	2.50	2.50	2.32	2.50
	High	1000	50.0	1000	1000	50.0

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of ±30%, demonstrate that the method is appropriate for the given matrix.

The method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. At times the spike level exceeded the recommended upper limit of 10 times the analyte concentration. In these instances the FMS recovery was reported and flagged as above 10 times the sample concentration.

For PFOA, PFBS, PFHS, and PFOS, when the field matrix spike level was not appropriate as compared to the endogenous sample concentration, the surrogate recovery standards were used to assess method accuracy. All field matrix spike and surrogate recovery standard recoveries met acceptance criteria except where noted below.

Effluent WWTP: Although the surrogate recovery standard [$^{13}C_4$]-PFOS met the method acceptance criteria with recoveries within 100 ± 30%, the analyte area counts were below the LOQ area counts. Upon further evaluation, the samples do not meet the method blank or internal standard response acceptance criteria, therefore, [$^{13}C_4$]-PFOS is not reportable for the sample set. However, all analytes had an appropriate FMS sample that met acceptance criteria. Therefore, the results are reported for PFHS and PFOS.

ND7 and P119A: Since the high level LCSs analyzed on 1/17/17 were appropriate for the sample concentrations for ND7 and P119A for PFOA, the data uncertainty for each location will be expanded based the %bias of the high level LCSs (±37%).

Sample locations with RPDs >30%: Samples reported with an RPD greater than 30% were re-analyzed in attempt to improve the RPD values. For sample locations D14 (PFOS), PP04 (PFOSA), PP07 (PFBS, PFOS, and PFOSA), and P21B (PFOSA) the re-analysis confirmed the initial results, in which the initial results were reported. The re-analysis of PFHS for sample location PP07 did improve with the re-analysis, therefore, the results from the re-analysis were reported even though the RPD was still above 30%.

Table 10. Location ID: D10

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-003	D10; Sample	234	NA	86.1	NA
ISO17-14-01-003-DUP	D10; Sample Duplicate	223	NA	82.2	NA
ISO17-14-01-003-FMS	D10; Sample FMS	311	NC	176	90.9
Average Concentration (ng/mL) ± %RPD		229 ng/mL ± 4.8%		84.2 ng/mL ± 4.6%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-003	D10; Sample	23.9	NA	0.322	NA
ISO17-14-01-003-DUP	D10; Sample Duplicate	22.9	NA	0.299	NA
ISO17-14-01-003-FMS	D10; Sample FMS	115	90.8	1.40	109
Average Concentration (ng/mL) ± %RPD		23.4 ng/mL ± 4.3%		0.311 ng/mL ± 7.4%	

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

Table 11. Location ID: D11

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-004	D11; Sample	47.2	NA	33.8	NA
ISO17-14-01-004-DUP	D11; Sample Duplicate	47.2	NA	33.9	NA
ISO17-14-01-004-FMS	D11; Sample FMS	474	85.2 (1)	472	87.6 (1)
Average Concentration (ng/mL) ± %RPD		47.2 ng/mL ± 0.0%		33.9 ng/mL ± 0.30%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-004	D11; Sample	758	NA	100	NA
ISO17-14-01-004-DUP	D11; Sample Duplicate	788	NA	99.0	NA
ISO17-14-01-004-FMS	D11; Sample FMS	1230	91.4	155	111
Average Concentration (ng/mL) ± %RPD		773 ng/mL ± 3.9%		99.5 ng/mL ± 1.0%	

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

Table 12. Location ID: P118A

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-011	P118A; Sample	1120	NA	821	NA
ISO17-14-01-011-DUP	P118A; Sample Duplicate	1150	NA	825	NA
ISO17-14-01-011-FMS	P118A; Sample FMS	2010	87.3	1790	96.7
Average Concentration (ng/mL) ± %RPD		**1140 ng/mL ± 2.6%**		**823 ng/mL ± 0.49%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-011	P118A; Sample	5080	NA	25.1	NA
ISO17-14-01-011-DUP	P118A; Sample Duplicate	5020	NA	24.6	NA
ISO17-14-01-011-FMS	P118A; Sample FMS	5720	NC	72.1	94.5
Average Concentration (ng/mL) ± %RPD		**5050 ng/mL ± 1.2%**		**24.9 ng/mL ± 2.0%**	

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

Table 13. Location ID: P121

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-015	P121; Sample	0.273	NA	0.107	NA
ISO17-14-01-015-DUP	P121; Sample Duplicate	0.249	NA	0.103	NA
ISO17-14-01-015-FMS	P121; Sample FMS	2.51	93.9	2.42	92.6 (1)
Average Concentration (ng/mL) ± %RPD		**0.261 ng/mL ± 9.2%**		**0.105 ng/mL ± 3.8%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-015	P121; Sample	2.02	NA	0.144	NA
ISO17-14-01-015-DUP	P121; Sample Duplicate	2.07	NA	0.135	NA
ISO17-14-01-015-FMS	P121; Sample FMS	4.18	92.1	2.64	100 (1)
Average Concentration (ng/mL) ± %RPD		**2.05 ng/mL ± 2.4%**		**0.140 ng/mL ± 6.5%**	

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

Table 14. Location ID: Blokkersdijkvijver – North

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-018	Blokkersdijkvijver North	0.827	NA	0.509	NA
ISO17-14-01-018-DUP	Blokkersdijkvijver North Dup	0.811	NA	0.487	NA
ISO17-14-01-018-FMS	Blokkersdijkvijver North FMS	5.26	92.7	5.14	92.8 (1)
Average Concentration (ng/mL) ± %RPD		0.819 ng/mL ± 2.0%		0.498 ng/mL ± 4.4%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-018	Blokkersdijkvijver North	1.35	NA	<0.0248	NA
ISO17-14-01-018-DUP	Blokkersdijkvijver North Dup	1.37	NA	<0.0248	NA
ISO17-14-01-018-FMS	Blokkersdijkvijver North FMS	5.87	97.3	5.35	107 (1)
Average Concentration (ng/mL) ± %RPD		1.36 ng/mL ± 1.5%		<0.0248 ng/mL	

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

Table 15. Location ID: L21

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-020	L21; Sample	0.295	NA	0.156	NA
ISO17-14-01-020-DUP	L21; Sample Duplicate	0.282	NA	0.145	NA
ISO17-14-01-020-FMS	L21; Sample FMS	2.47	91.1	2.41	90.4 (1)
Average Concentration (ng/mL) ± %RPD		0.289 ng/mL ± 4.5%		0.151 ng/mL ± 7.3%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-020	L21; Sample	5.40	NA	0.0897	NA
ISO17-14-01-020-DUP	L21; Sample Duplicate	5.41	NA	0.0850	NA
ISO17-14-01-020-FMS	L21; Sample FMS	6.98	NC	2.54	98.1 (1)
Average Concentration (ng/mL) ± %RPD		5.41 ng/mL ± 0.19%		0.0874 ng/mL ± 5.4%	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) FMS concentration greater than 10 times the sample concentration.

Table 16. Location ID: P115

3M LIMS ID	Sample Description	PFOA Concentration (ng/mL)	PFOA %Recovery	PFHS Concentration (ng/mL)	PFHS %Recovery
ISO17-14-01-025	P115; Sample	3.82	NA	2.13	NA
ISO17-14-01-025-DUP	P115; Sample Duplicate	4.05	NA	2.29	NA
ISO17-14-01-025-FMS	P115; Sample FMS	6.24	96.2	4.59	95.2
Average Concentration (ng/mL) ± %RPD		3.94 ng/mL ± 5.8%		2.21 ng/mL ± 7.2%	

3M LIMS ID	Sample Description	PFOS Concentration (ng/mL)	PFOS %Recovery	PFOSA Concentration (ng/mL)	PFOSA %Recovery
ISO17-14-01-025	P115; Sample	0.547	NA	<0.0248	NA
ISO17-14-01-025-DUP	P115; Sample Duplicate	0.639	NA	<0.0248	NA
ISO17-14-01-025-FMS	P115; Sample FMS	2.88	98.7	2.50	100 (1)
Average Concentration (ng/mL) ± %RPD		0.593 ng/mL ± 16%		<0.0248 ng/mL	

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

Table 17. Location ID: Effluent WWTP

3M LIMS ID	Sample Description	PFOA Concentration (ng/mL)	PFOA %Recovery	PFHS Concentration (ng/mL)	PFHS %Recovery
ISO17-14-01-027	Effluent WWTP; Sample	0.438	NA	0.430	NA
ISO17-14-01-027-DUP	Effluent WWTP; Sample Duplicate	0.382	NA	0.387	NA
ISO17-14-01-027-FMS	Effluent WWTP; Sample FMS	20.3	94.9 (1)	21.8	102 (1)
Average Concentration (ng/mL) ± %RPD		0.410 ng/mL ± 14%		0.409 ng/mL ± 11%	

3M LIMS ID	Sample Description	PFOS Concentration (ng/mL)	PFOS %Recovery	PFOSA Concentration (ng/mL)	PFOSA %Recovery
ISO17-14-01-027	Effluent WWTP; Sample	2.60	NA	0.120	NA
ISO17-14-01-027-DUP	Effluent WWTP; Sample Duplicate	2.30	NA	0.0932	NA
ISO17-14-01-027-FMS	Effluent WWTP; Sample FMS	25.8	112	1.09	98.3
Average Concentration (ng/mL) ± %RPD		2.45 ng/mL ± 12%		0.107 ng/mL ± 25% (2)	

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

Table 18. Location ID: PP01

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-028	PP01; Sample	45.2	NA	47.0	NA	5510	NA
ISO17-14-01-028-DUP	PP01; Sample Duplicate	47.5	NA	49.5	NA	5240	NA
ISO17-14-01-028-FMS	PP01; Sample FMS	138	91.7	151	103	NA (1)	NA
Average Concentration (ng/mL) ± %RPD		46.4 ng/mL ± 5.0%		48.3 ng/mL ± 5.2%		5380 ng/mL ± 5.0%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-028	PP01; Sample	1900	NA	20.0	NA
ISO17-14-01-028-DUP	PP01; Sample Duplicate	1870	NA	20.8	NA
ISO17-14-01-028-FMS	PP01; Sample FMS	2140	NC	122	102
Average Concentration (ng/mL) ± %RPD		1890 ng/mL ± 1.6%		20.4 ng/mL ± 3.9%	

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 19. Location ID: PP09

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-035	PP09; Sample	631	NA	25.9	NA	391	NA
ISO17-14-01-035-DUP	PP09; Sample Duplicate	613	NA	24.1	NA	378	NA
ISO17-14-01-035-FMS	PP09; Sample FMS	1470	84.8	69.9	89.8	1310	92.6
Average Concentration (ng/mL) ± %RPD		622 ng/mL ± 2.9%		25.0 ng/mL ± 7.2%		385 ng/mL ± 3.4%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-035	PP09; Sample	2600	NA	16.0	NA
ISO17-14-01-035-DUP	PP09; Sample Duplicate	2590	NA	15.8	NA
ISO17-14-01-035-FMS	PP09; Sample FMS	3660	NC	64.6	97.4
Average Concentration (ng/mL) ± %RPD		2600 ng/mL ± 0.39%		15.9 ng/mL ± 1.3%	

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

Table 20. Location ID: Bemalingsstation

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-01-040	Bemalingsstation; Sample	25.8	NA	22.4	NA
ISO17-14-01-040-DUP	Bemalingsstation; Sample Duplicate	25.5	NA	22.0	NA
ISO17-14-01-040-FMS	Bemalingsstation; Sample FMS	112	85.5	112	88.9
Average Concentration (ng/mL) ± %RPD		**25.7 ng/mL ± 1.2%**		**22.2 ng/mL ± 1.8%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-01-040	Bemalingsstation; Sample	53.5	NA	0.190	NA
ISO17-14-01-040-DUP	Bemalingsstation; Sample Duplicate	52.1	NA	0.195	NA
ISO17-14-01-040-FMS	Bemalingsstation; Sample FMS	150	96.3	1.26	107
Average Concentration (ng/mL) ± %RPD		**52.8 ng/mL ± 2.7%**		**0.193 ng/mL ± 2.6%**	

NA = Not Applicable

Table 21. Location ID: Collector put

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-01-041	Collector put; Sample	27.3	NA	13.3	NA
ISO17-14-01-041-DUP	Collector put; Sample Duplicate	27.7	NA	13.5	NA
ISO17-14-01-041-FMS	Collector put; Sample FMS	438	82.0 (1)	496	96.5 (1)
Average Concentration (ng/mL) ± %RPD		**27.5 ng/mL ± 1.5%**		**13.4 ng/mL ± 1.5%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-01-041	Collector put; Sample	104	NA	5.76	NA
ISO17-14-01-041-DUP	Collector put; Sample Duplicate	104	NA	5.56	NA
ISO17-14-01-041-FMS	Collector put; Sample FMS	635	106	56.0	101
Average Concentration (ng/mL) ± %RPD		**104 ng/mL ± 0.0%**		**5.66 ng/mL ± 3.5%**	

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

Table 22. Location ID: P304

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-044	P304; Sample	403	NA	272	NA	126	NA
ISO17-14-01-044-DUP	P304; Sample Duplicate	400	NA	270	NA	124	NA
ISO17-14-01-044-FMS	P304; Sample FMS	484	NC	366	NC	215	90.0
Average Concentration (ng/mL) ± %RPD		402 ng/mL ± 0.75%		271 ng/mL ± 0.74%		125 ng/mL ± 1.6%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-044	P304; Sample	1340	NA	28.1	NA
ISO17-14-01-044-DUP	P304; Sample Duplicate	1350	NA	28.5	NA
ISO17-14-01-044-FMS	P304; Sample FMS	1450	NC	57.8	118
Average Concentration (ng/mL) ± %RPD		1350 ng/mL ± 0.74%		28.3 ng/mL ± 1.4%	

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

Table 23. Location ID: P119C

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-050	P119C; Sample	923	NA	688	NA
ISO17-14-01-050-DUP	P119C; Sample Duplicate	990	NA	740	NA
ISO17-14-01-050-FMS	P119C; Sample FMS	1900	94.4	1720	101
Average Concentration (ng/mL) ± %RPD		957 ng/mL ± 7.0%		714 ng/mL ± 7.3%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-050	P119C; Sample	2770	NA	61.4	NA
ISO17-14-01-050-DUP	P119C; Sample Duplicate	2840	NA	58.5	NA
ISO17-14-01-050-FMS	P119C; Sample FMS	3930	NC	162	102
Average Concentration (ng/mL) ± %RPD		2810 ng/mL ± 2.5%		60.0 ng/mL ± 4.8%	

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

Table 24. Location ID: P264

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-052	P264; Sample	249	NA	162	NA
ISO17-14-01-052-DUP	P264; Sample Duplicate	241	NA	159	NA
ISO17-14-01-052-FMS	P264; Sample FMS	1090	84.5	1060	90.0
Average Concentration (ng/mL) ± %RPD		**245 ng/mL ± 3.3%**		**161 ng/mL ± 1.9%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-052	P264; Sample	1480	NA	78.6	NA
ISO17-14-01-052-DUP	P264; Sample Duplicate	1450	NA	73.3	NA
ISO17-14-01-052-FMS	P264; Sample FMS	2430	96.5	181	105
Average Concentration (ng/mL) ± %RPD		**1470 ng/mL ± 2.0%**		**76.0 ng/mL ± 7.0%**	

NA = Not Applicable

Table 25. Location ID: P371

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-054	P371; Sample	163	NA	142	NA
ISO17-14-01-054-DUP	P371; Sample Duplicate	159	NA	139	NA
ISO17-14-01-054-FMS	P371; Sample FMS	314	76.4	296	77.8
Average Concentration (ng/mL) ± %RPD		**161 ng/mL ± 2.5%**		**141 ng/mL ± 2.1%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-054	P371; Sample	2770	NA	47.3	NA
ISO17-14-01-054-DUP	P371; Sample Duplicate	2830	NA	57.8	NA
ISO17-14-01-054-FMS	P371; Sample FMS	2910	NC	158	105
Average Concentration (ng/mL) ± %RPD		**2800 ng/mL ± 2.1%**		**52.6 ng/mL ± 20%**	

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

Table 26. Location ID: B3-bis

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-058	B3-bis; Sample	36.7	NA	6.00	NA
ISO17-14-01-058-DUP	B3-bis; Sample Duplicate	37.5	NA	6.35	NA
ISO17-14-01-058-FMS	B3-bis; Sample FMS	41.6	NC	10.6	88.5
Average Concentration (ng/mL) ± %RPD		37.1 ng/mL ± 2.2%		6.18 ng/mL ± 5.7%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-058	B3-bis; Sample	110	NA	0.217	NA
ISO17-14-01-058-DUP	B3-bis; Sample Duplicate	113	NA	0.203	NA
ISO17-14-01-058-FMS	B3-bis; Sample FMS	117	NC	5.29	102 (1)
Average Concentration (ng/mL) ± %RPD		112 ng/mL ± 2.7%		0.210 ng/mL ± 6.7%	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) FMS concentration greater than 10 times the sample concentration.

Table 27. Location ID: P378

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-061	P378; Sample	295	NA	327	NA
ISO17-14-01-061-DUP	P378; Sample Duplicate	309	NA	347	NA
ISO17-14-01-061-FMS	P378; Sample FMS	689	75.7	763	83.5
Average Concentration (ng/mL) ± %RPD		302 ng/mL ± 4.6%		337 ng/mL ± 5.9%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-061	P378; Sample	1570	NA	2.26	NA
ISO17-14-01-061-DUP	P378; Sample Duplicate	1730	NA	2.46	NA
ISO17-14-01-061-FMS	P378; Sample FMS	1980	NC	12.5	101
Average Concentration (ng/mL) ± %RPD		1650 ng/mL ± 9.7%		2.36 ng/mL ± 8.5%	

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

Table 28. Location ID: PA111A

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-063	PA111A; Sample	642	NA	925	NA
ISO17-14-01-063-DUP	PA111A; Sample Duplicate	653	NA	925	NA
ISO17-14-01-063-FMS	PA111A; Sample FMS	1440	79.3	1790	86.5
Average Concentration (ng/mL) ± %RPD		**648 ng/mL ± 1.7%**		**925 ng/mL ± 0.0%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-063	PA111A; Sample	2180	NA	10.7	NA
ISO17-14-01-063-DUP	PA111A; Sample Duplicate	2190	NA	10.9	NA
ISO17-14-01-063-FMS	PA111A; Sample FMS	3140	NC	62.0	102
Average Concentration (ng/mL) ± %RPD		**2190 ng/mL ± 0.46%**		**10.8 ng/mL ± 1.9%**	

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

Table 29. Location ID: Travel Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-065	Travel Blank	<0.0240	NA	<0.200	NA	<0.0250	NA
ISO17-14-01-065-FMS-LOW	Travel Blank FMS Low	2.22	92.7	2.30	92.0	2.31	92.4
ISO17-14-01-065-FMS-HIGH	Travel Blank FMS High	818	81.8	46.8	93.6	890	89.0

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-01-065	Travel Blank	<0.0232	NA	<0.0248	NA
ISO17-14-01-065-FMS-LOW	Travel Blank FMS Low	2.13	91.9	2.55	102
ISO17-14-01-065-FMS-HIGH	Travel Blank FMS High	981	98.1	51.0	102

NA = Not Applicable

Table 30. 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
ISO17-14-01-001	BD24-3; Sample	108	114	NA
ISO17-14-01-001-DUP	BD24-3; Sample Duplicate	110	108	NA
ISO17-14-01-002	D09; Sample	96.5	100	124
ISO17-14-01-002-DUP	D09; Sample Duplicate	105	111	119
ISO17-14-01-003	D10; Sample	103	112	NA
ISO17-14-01-003-DUP	D10; Sample Duplicate	110	119	NA
ISO17-14-01-003-FMS	D10; Sample FMS	101	109	NA
ISO17-14-01-004	D11; Sample	111	116	NA
ISO17-14-01-004-DUP	D11; Sample Duplicate	107	117	NA
ISO17-14-01-004-FMS	D11; Sample FMS	102	111	NA
ISO17-14-01-005	D14; Sample	86.7 (2)	93.6 (2)	NA
ISO17-14-01-005-DUP	D14; Sample Duplicate	87.8 (2)	84.2 (2)	NA
ISO17-14-01-006	D16; Sample	91.9	98.3	NA
ISO17-14-01-006-DUP	D16; Sample Duplicate	105	113	NA
ISO17-14-01-007	D17; Sample	106	114	NA
ISO17-14-01-007-DUP	D17; Sample Duplicate	101	112	NA
ISO17-14-01-008	D18; Sample	102	117	NA
ISO17-14-01-008-DUP	D18; Sample Duplicate	99.9	112	NA
ISO17-14-01-009	D5; Sample	108	119	NA
ISO17-14-01-009-DUP	D5; Sample Duplicate	107	118	NA
ISO17-14-01-010	ND7; Sample	88.0	105	NA
ISO17-14-01-010-DUP	ND7; Sample Duplicate	92.3	112	NA
ISO17-14-01-011	P118A; Sample	103	120	NA
ISO17-14-01-011-DUP	P118A; Sample Duplicate	107	120	NA
ISO17-14-01-011-FMS	P118A; Sample FMS	104	117	NA
ISO17-14-01-012	P118B; Sample	105	114	NA
ISO17-14-01-012-DUP	P118B; Sample Duplicate	104	114	NA
ISO17-14-01-013	P119A; Sample	103	100	115
ISO17-14-01-013-DUP	P119A; Sample Duplicate	92.5	92.9	117
ISO17-14-01-014	P119B; Sample	98.0	114	NA
ISO17-14-01-014-DUP	P119B; Sample Duplicate	96.6	111	NA
ISO17-14-01-015	P121; Sample	91.2 (2)	83.1 (2)	NA
ISO17-14-01-015-DUP	P121; Sample Duplicate	83.3 (2)	88.2 (2)	NA
ISO17-14-01-015-FMS	P121; Sample FMS	90.5 (2)	82.8 (2)	NA
ISO17-14-01-016	P321; Sample	102	118	NA
ISO17-14-01-016-DUP	P321; Sample Duplicate	101	117	NA
ISO17-14-01-017	3M vijver; Sample	106	117	NA
ISO17-14-01-017-DUP	3M vijver; Sample Duplicate	102	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) NR = Not Reportable; surrogate response below the LOQ and did not meet internal standard or method blank criteria.

Table 30 continued. Surrogate Recovery Standard Results [1]

3M LIMS ID	Sample Description	Percent Recovery (%) $[^{13}C_4]$-PFOA	$[^{13}C_4]$-PFOS	$[^{13}C_4]$-PFOS Re-analysis
ISO17-14-01-018	Blokkersdijkvijver North	108	124	NA
ISO17-14-01-018-DUP	Blokkersdijkvijver North Dup	104	119	NA
ISO17-14-01-018-FMS	Blokkersdijkvijver North FMS	106	123	NA
ISO17-14-01-019	Blokkersdijkvijver std	104	119	NA
ISO17-14-01-019-DUP	Blokkersdijkvijver std Dup	105	121	NA
ISO17-14-01-020	L21; Sample	86.4 [2]	81.5 [2]	NA
ISO17-14-01-020-DUP	L21; Sample Duplicate	87.9 [2]	81.5 [2]	NA
ISO17-14-01-020-FMS	L21; Sample FMS	89.9 [2]	78.5 [2]	NA
ISO17-14-01-021	L22; Sample	85.5 [2]	89.4 [2]	NA
ISO17-14-01-021-DUP	L22; Sample Duplicate	87.3 [2]	88.7 [2]	NA
ISO17-14-01-022	L31; Sample	104	118	NA
ISO17-14-01-022-DUP	L31; Sample Duplicate	104	123	NA
ISO17-14-01-023	L4; Sample	102	116	NA
ISO17-14-01-023-DUP	L4; Sample Duplicate	105	123	NA
ISO17-14-01-024	P114bis; Sample	94.8	111	NA
ISO17-14-01-024-DUP	P114bis; Sample Duplicate	96.0	116	NA
ISO17-14-01-025	P115; Sample	106	119	NA
ISO17-14-01-025-DUP	P115; Sample Duplicate	106	122	NA
ISO17-14-01-025-FMS	P115; Sample FMS	103	120	NA
ISO17-14-01-026	P116; Sample	101	118	NA
ISO17-14-01-026-DUP	P116; Sample Duplicate	101	116	NA
ISO17-14-01-027	Effluent WWTP; Sample	85.8 [2]	NR [3]	NA
ISO17-14-01-027-DUP	Effluent WWTP; Sample Duplicate	96.9 [2]	NR [3]	NA
ISO17-14-01-027-FMS	Effluent WWTP; Sample FMS	85.9 [2]	NR [3]	NA
ISO17-14-01-028	PP01; Sample	102	113	119
ISO17-14-01-028-DUP	PP01; Sample Duplicate	99.6	109	130
ISO17-14-01-028-FMS	PP01; Sample FMS	108	120	NA
ISO17-14-01-029	PP02; Sample	102	108	NA
ISO17-14-01-029-DUP	PP02; Sample Duplicate	93.7	99.7	NA
ISO17-14-01-030	PP04; Sample	106	117	NA
ISO17-14-01-030-DUP	PP04; Sample Duplicate	99.1	112	NA
ISO17-14-01-031	PP05; Sample	104	123	NA
ISO17-14-01-031-DUP	PP05; Sample Duplicate	104	121	NA
ISO17-14-01-032	PP06; Sample	95.1	106	NA
ISO17-14-01-032-DUP	PP06; Sample Duplicate	103	118	NA
ISO17-14-01-033	PP07; Sample	96.5	101	113
ISO17-14-01-033-DUP	PP07; Sample Duplicate	97.2	100	117
ISO17-14-01-034	PP08; Sample	103	107	NA
ISO17-14-01-034-DUP	PP08; Sample Duplicate	112	117	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) NR = Not Reportable; surrogate response below the LOQ and did not meet internal standard or method blank criteria.

Table 30 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
ISO17-14-01-035	PP09; Sample	107	114	NA
ISO17-14-01-035-DUP	PP09; Sample Duplicate	100	109	NA
ISO17-14-01-035-FMS	PP09; Sample FMS	104	112	NA
ISO17-14-01-036	PP10; Sample	99.1	104	NA
ISO17-14-01-036-DUP	PP10; Sample Duplicate	110	116	NA
ISO17-14-01-037	12; Sample	107	118	NA
ISO17-14-01-037-DUP	12; Sample Duplicate	110	118	NA
ISO17-14-01-038	13; Sample	98.3	112	NA
ISO17-14-01-038-DUP	13; Sample Duplicate	99.5	110	NA
ISO17-14-01-039	5; Sample	102	116	NA
ISO17-14-01-039-DUP	5; Sample Duplicate	106	114	NA
ISO17-14-01-040	Bemalingsstation; Sample	99.0	112	NA
ISO17-14-01-040-DUP	Bemalingsstation; Sample Duplicate	109	122	NA
ISO17-14-01-040-FMS	Bemalingsstation; Sample FMS	105	117	NA
ISO17-14-01-041	Collector put; Sample	109	119	NA
ISO17-14-01-041-DUP	Collector put; Sample Duplicate	108	120	NA
ISO17-14-01-041-FMS	Collector put; Sample FMS	107	123	NA
ISO17-14-01-042	P27; Sample	99.8	112	NA
ISO17-14-01-042-DUP	P27; Sample Duplicate	95.7	111	NA
ISO17-14-01-043	P21B; Sample	102	109	113
ISO17-14-01-043-DUP	P21B; Sample Duplicate	90.5	101	113
ISO17-14-01-044	P304; Sample	106	118	NA
ISO17-14-01-044-DUP	P304; Sample Duplicate	107	120	NA
ISO17-14-01-044-FMS	P304; Sample FMS	98.6	113	NA
ISO17-14-01-045	P305; Sample	101	116	NA
ISO17-14-01-045-DUP	P305; Sample Duplicate	95.7	112	NA
ISO17-14-01-046	P42; Sample	97.9	114	NA
ISO17-14-01-046-DUP	P42; Sample Duplicate	100	109	NA
ISO17-14-01-047	L19; Sample	104	114	NA
ISO17-14-01-047-DUP	L19; Sample Duplicate	106	119	NA
ISO17-14-01-048	M4; Sample	106	121	NA
ISO17-14-01-048-DUP	M4; Sample Duplicate	98.7	113	NA
ISO17-14-01-049	P118C; Sample	106	118	NA
ISO17-14-01-049-DUP	P118C; Sample Duplicate	106	117	NA
ISO17-14-01-050	P119C; Sample	106	116	NA
ISO17-14-01-050-DUP	P119C; Sample Duplicate	108	118	NA
ISO17-14-01-050-FMS	P119C; Sample FMS	106	116	NA
ISO17-14-01-051	P263; Sample	112	120	NA
ISO17-14-01-051-DUP	P263; Sample Duplicate	105	116	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) NR = Not Reportable; surrogate response below the LOQ and did not meet internal standard or method blank criteria.

Table 30 continued. Surrogate Recovery Standard Results [1]

		Percent Recovery (%)	
3M LIMS ID	**Sample Description**	**[$^{13}C_4$]-PFOA**	**[$^{13}C_4$]-PFOS**
ISO17-14-01-052	P264; Sample	104	117
ISO17-14-01-052-DUP	P264; Sample Duplicate	104	117
ISO17-14-01-052-FMS	P264; Sample FMS	101	111
ISO17-14-01-054-DUP	P371; Sample Duplicate	97.3	109
ISO17-14-01-054-FMS	P371; Sample FMS	108	114
ISO17-14-01-055	P374; Sample	102	116
ISO17-14-01-055-DUP	P374; Sample Duplicate	105	113
ISO17-14-01-056	P381; Sample	105	117
ISO17-14-01-056-DUP	P381; Sample Duplicate	95.3	107
ISO17-14-01-057	P382; Sample	99.8	111
ISO17-14-01-057-DUP	P382; Sample Duplicate	106	116
ISO17-14-01-058	B3-bis; Sample	104	116
ISO17-14-01-058-DUP	B3-bis; Sample Duplicate	106	117
ISO17-14-01-058-FMS	B3-bis; Sample FMS	109	121
ISO17-14-01-059	B7; Sample	109	122
ISO17-14-01-059-DUP	B7; Sample Duplicate	105	121
ISO17-14-01-060	P372; Sample	104	116
ISO17-14-01-060-DUP	P372; Sample Duplicate	107	121
ISO17-14-01-061	P378; Sample	97.2	109
ISO17-14-01-061-DUP	P378; Sample Duplicate	110	119
ISO17-14-01-061-FMS	P378; Sample FMS	99.7	116
ISO17-14-01-062	PA109A; Sample	102	119
ISO17-14-01-062-DUP	PA109A; Sample Duplicate	102	118
ISO17-14-01-063	PA111A; Sample	104	124
ISO17-14-01-063-DUP	PA111A; Sample Duplicate	105	120
ISO17-14-01-063-FMS	PA111A; Sample FMS	100	116
ISO17-14-01-064	PA112; Sample	104	121
ISO17-14-01-064-DUP	PA112; Sample Duplicate	102	117
ISO17-14-01-065	Travel Blank	89.3 [2]	90.4 [2]
ISO17-14-01-065-FMS-LOW	Travel Blank FMS Low	90.6 [2]	88.5 [2]
ISO17-14-01-065-FMS-HIGH	Travel Blank FMS High	104	120

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) NR = Not Reportable; surrogate response below the LOQ and did not meet internal standard or method blank criteria.

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 10-30 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 Attachments

Chain of Custody Form

8 Signatures

Chelsie Grochow

Digitally signed by Chelsie J. Grochow
DN: c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory - authenticated by LRA, [redacted] o=3M, cn=Chelsie J. Grochow
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Chelsie Grochow, 3M Report Author

Susan S. Wolf

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Susan T. Wolf, 3M Principal Analytical Investigator

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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.

Kent R Lindstrom

Digitally signed by Kent R. Lindstrom
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Reason: [redacted]
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Quality Assurance Representative

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3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

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Project: ISO17-14-01

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 12/22/2016
Project Description: 3M Antwerp Water Sampling for PFCs; January 2017 Sampling
Comments:

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [redacted]
Email Address: [redacted]

* SW = surface water
* GW = ground water
* EW = extraction well

	3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
X	ISO17-14-01-001	BD24-3; Sample	13/1/17	GW	
X	ISO17-14-01-001-DUP	BD24-3; Sample Duplicate	13/1/17	GW	
X	ISO17-14-01-002	D09; Sample	10/01/2017	GW	
X	ISO17-14-01-002-DUP	D09; Sample Duplicate	10/01/2017	GW	
X	ISO17-14-01-003	D10; Sample	11/1/2017	GW	
X	ISO17-14-01-003-DUP	D10; Sample Duplicate	11/1/2017	GW	
X	ISO17-14-01-003-FMS	D10; Sample FMS	11/1/2017	GW	
X	ISO17-14-01-004	D11; Sample	11/1/17	GW	
X	ISO17-14-01-004-DUP	D11; Sample Duplicate	11/1/17	GW	
X	ISO17-14-01-004-FMS	D11; Sample FMS	11/1/17	GW	
X	ISO17-14-01-005	D14; Sample	10/01/17	GW	
X	ISO17-14-01-005-DUP	D14; Sample Duplicate	10/01/17	GW	
X	ISO17-14-01-006	D16; Sample	11/01/17	GW	
X	ISO17-14-01-006-DUP	D16; Sample Duplicate	11/1/17	GW	

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for**
Temperature: 5 **Deg C** ☐ **Received on Ice** ☐ **Other:**

Collected by (print): Tim Mandonx **Collector's signature:** T.M

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
	T. Mandonx	16/01/17	9.00	Fedex	BG /PACE	1-19-17	10:55

(17)

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

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Project: ISO17-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 12/22/2016
Project Description: 3M Antwerp Water Sampling for PFCs; January 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [redacted]
Email Address: [redacted]

GW = groundwater

	3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
X	ISO17-14-01-007	D17; Sample	9/01/2017	GW	
X	ISO17-14-01-007-DUP	D17; Sample Duplicate	9/01/2017	GW	
X	ISO17-14-01-008	D18; Sample	9/01/2017	GW	
X	ISO17-14-01-008-DUP	D18; Sample Duplicate	9/01/2017	GW	
X	ISO17-14-01-009	D5; Sample	9/01/2017	GW	
X	ISO17-14-01-009-DUP	D5; Sample Duplicate	9/01/2017	GW	
X	ISO17-14-01-010	ND7; Sample	9/01/2017	GW	
X	ISO17-14-01-010-DUP	ND7; Sample Duplicate	9/01/2017	GW	
X	ISO17-14-01-011	P118A; Sample	10/01/2017	GW	
X	ISO17-14-01-011-DUP	P118A; Sample Duplicate	10/01/2017	GW	
X	ISO17-14-01-011-FMS	P118A; Sample FMS	10/01/2017	GW	
X	ISO17-14-01-012	P118B; Sample	10/01/2017	GW	
X	ISO17-14-01-012-DUP	P118B; Sample Duplicate	10/01/2017	GW	
X	ISO17-14-01-013	P119A; Sample	11/1/'17	GW	
X	ISO17-14-01-013-DUP	P119A; Sample Duplicate	11/1/'17	GW	
X	ISO17-14-01-014	P119B; Sample	11/1/'17	GW	
X	ISO17-14-01-014-DUP	P119B; Sample Duplicate	11/1/'17	GW	

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for**

Temperature: 5 **Deg C** ☐ **Received on Ice** ☐ **Other:**

Collected by (print): Tim Mandonk **Collector's signature:**

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
	T MANDONK	11/1/17	9:00	Fedex	Brig / PACE	1-19-17	10:55AM

(17)

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone:[redacted]
Alt. Ph[redacted]
Fax: ([redacted]

Project: ISO17-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 12/22/2016
Project Description: 3M Antwerp Water Sampling for PFCs; January 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number:[redacted]
Email Address:[redacted]

GW = groundwater
SW = surface water

	3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
X	ISO17-14-01-015	P121; Sample	12/1/17	GW	
X	ISO17-14-01-015-DUP	P121; Sample Duplicate	12/1/17	GW	
X	ISO17-14-01-015-FMS	P121; Sample FMS	12/1/17	GW	
X	ISO17-14-01-016	P321; Sample	10/1/17	GW	
X	ISO17-14-01-016-DUP	P321; Sample Duplicate	10/1/17	GW	
X	ISO17-14-01-017	3M vijver; Sample	12/1/17	SW	
X	ISO17-14-01-017-DUP	3M vijver; Sample Duplicate	12/1/17	SW	
X	ISO17-14-01-018	Blokkersdijkvijver – ~~oost~~ noord; Sample	12/1/17	SW	
X	ISO17-14-01-018-DUP	Blokkersdijkvijver – ~~oost~~ noord; Sample Duplicate	12/1/17	SW	
X	ISO17-14-01-018-FMS	Blokkersdijkvijver – ~~oost~~ noord; Sample FMS	12/1/17	SW	
X	ISO17-14-01-019	Blokkersdijkvijver – standard; Sample	12/1/17	SW	
X	ISO17-14-01-019-DUP	Blokkersdijkvijver – standard; Sample Duplicate	12/1/17	SW	
X	ISO17-14-01-020	L21; Sample	12/1/17	GW	
X	ISO17-14-01-020-DUP	L21; Sample Duplicate	12/1/17	GW	
X	ISO17-14-01-020-FMS	L21; Sample FMS	12/1/17	GW	
X	ISO17-14-01-021	L22; Sample	12/1/17	GW	
X	ISO17-14-01-021-DUP	L22; Sample Duplicate	12/1/17	GW	

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for**

Temperature: 5 **Deg C** ☐ **Received on Ice** ☐ **Other:**

Collected by (print): Tine Mandonx **Collector's signature:** [signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
	T. MANDONX	16/1/17	9⁰⁰	Fedex	[signature] /PACE	1-14-17	10:55AM

17

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

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3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [redacted]
Alt. Pho[redacted]
Fax: (6[redacted]

Project: ISO17-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202   Site Source: 01WJWT10
Project Number:
Date Created: 12/22/2016
Project Description: 3M Antwerp Water Sampling for PFCs; January 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [redacted]
Email Address: [redacted]

ww= waste water
GW= ground water
SW= surface water
EW= extraction well

	3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
X	ISO17-14-01-022	L31; Sample	12/1/'17	GW	
X	ISO17-14-01-022-DUP	L31; Sample Duplicate	12/1/'17	GW	
X	ISO17-14-01-023	L4; Sample	11/1/'17	GW	
X	ISO17-14-01-023-DUP	L4; Sample Duplicate	11/1/'17	GW	
X	ISO17-14-01-024	P114bis; Sample	12/1/'17	GW	
X	ISO17-14-01-024-DUP	P114bis; Sample Duplicate	12/1/'17	GW	
X	ISO17-14-01-025	P115; Sample	12/1/'17	GW	
X	ISO17-14-01-025-DUP	P115; Sample Duplicate	12/1/'17	GW	
X	ISO17-14-01-025-FMS	P115; Sample FMS	12/1/'17	GW	
X	ISO17-14-01-026	P116; Sample	12/1/'17	GW	
X	ISO17-14-01-026-DUP	P116; Sample Duplicate	12/1/'17	GW	
X	ISO17-14-01-027	Effluent WWTP; Sample	13/1/'17	WW	
X	ISO17-14-01-027-DUP	Effluent WWTP, Sample Duplicate	13/1/'17	WW	
X	ISO17-14-01-027-FMS	Effluent WWTP; Sample FMS	13/1/'17	WW	
X	ISO17-14-01-028	PP01; Sample	13/1/'17	EW	
X	ISO17-14-01-028-DUP	PP01; Sample Duplicate	13/1/'17	EW	
X	ISO17-14-01-028-FMS	PP01; Sample FMS	13/1/'17	EW	

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for**

Temperature: 5 **Deg C** ☐ **Received on Ice** ☐ **Other:**

Collected by (print): Tine Mandonx   **Collector's signature:** [signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
	T. MANDONX	16/1/17	9⁰⁰	Fedex	[signature] /PACE	1-14-17	10:55AM

(17)

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [redacted]
Alt. Pho[redacted]
Fax: (6[redacted]

Project: ISO17-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 12/22/2016
Project Description: 3M Antwerp Water Sampling for PFCs; January 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [redacted]
Email Address: [redacted]

EW= extraction well

	3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
X	ISO17-14-01-029	PP02; Sample	13/1'17	EW	
X	ISO17-14-01-029-DUP	PP02; Sample Duplicate	13/1'17	EW	
X	ISO17-14-01-030	PP04; Sample	13/1'17	EW	
X	ISO17-14-01-030-DUP	PP04; Sample Duplicate	13/1'17	EW	
X	ISO17-14-01-031	PP05; Sample	13/1'17	EW	
X	ISO17-14-01-031-DUP	PP05; Sample Duplicate	13/1'17	EW	
X	ISO17-14-01-032	PP06; Sample	13/1'17	EW	
X	ISO17-14-01-032-DUP	PP06; Sample Duplicate	13/1'17	EW	
X	ISO17-14-01-033	PP07; Sample	13/1'17	EW	
X	ISO17-14-01-033-DUP	PP07; Sample Duplicate	13/1'17	EW	
X	ISO17-14-01-034	PP08; Sample	13/1'17	EW	
X	ISO17-14-01-034-DUP	PP08; Sample Duplicate	13/1'17	EW	
X	ISO17-14-01-035	PP09; Sample	13/1'17	EW	
X	ISO17-14-01-035-DUP	PP09; Sample Duplicate	13/1'17	EW	
X	ISO17-14-01-035-FMS	PP09; Sample FMS	13/1'17	EW	
X	ISO17-14-01-036	PP10; Sample	13/1'17	EW	
X	ISO17-14-01-036-DUP	PP10; Sample Duplicate	13/1'17	EW	

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for**
Temperature: 5 **Deg C** ☐ **Received on Ice** ☐ **Other:**

Collected by (print): Tim Mandonx **Collector's signature:** [signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
	T. MANDONX	11/01/17	9:00	Fedex	[signature] /PACE	1-14-17	10:55AM

(17)

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [redacted]
Alt. Pho[redacted]
Fax: (6[redacted]

Project: ISO17-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 12/22/2016
Project Description: 3M Antwerp Water Sampling for PFCs; January 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [redacted]
Email Address: [redacted]

SW = surface water
WW = waste water
GW = groundwater

	3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
X	ISO17-14-01-037	12; Sample	12/1/'17	SW	
X	ISO17-14-01-037-DUP	12; Sample Duplicate	12/1/'17	SW	
X	ISO17-14-01-038	13; Sample	12/1/'17	SW	
X	ISO17-14-01-038-DUP	13; Sample Duplicate	12/1/'17	SW	
X	ISO17-14-01-039	5; Sample	12/1/'17	SW	
X	ISO17-14-01-039-DUP	5; Sample Duplicate	12/1/'17	SW	
X	ISO17-14-01-040	Bemalingsstation; Sample	12/1/'17	SW	
X	ISO17-14-01-040-DUP	Bemalingsstation; Sample Duplicate	12/1/'17	SW	
X	ISO17-14-01-040-FMS	Bemalingsstation; Sample FMS	12/1/'17	SW	
X	ISO17-14-01-041	Collector put; Sample	13/1/'17	WW	
X	ISO17-14-01-041-DUP	Collector put; Sample Duplicate	13/1/'17	WW	
X	ISO17-14-01-041-FMS	Collector put; Sample FMS	13/1/'17	WW	
X	ISO17-14-01-042	P27; Sample	9/01/2017	GW	
X	ISO17-14-01-042-DUP	P27; Sample Duplicate	9/01/2017	GW	
X	ISO17-14-01-043	P21B; Sample	10/01/2017	GW	
X	ISO17-14-01-043-DUP	P21B; Sample Duplicate	10/01/2017	GW	
X	ISO17-14-01-044	P304; Sample	9/01/2017	GW	

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for**

Temperature: 5 **Deg C** ☐ **Received on Ice** ☐ **Other:**

Collected by (print): Tine Mandonx **Collector's signature:** [signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
	T. MANDONX	16/01/17	9:00	Fedex	[signature] / PACE	1-19-17	10:55AM

(17)

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [redacted]
Alt. Pho[redacted]
Fax: (6[redacted]

Project: ISO17-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 12/22/2016
Project Description: 3M Antwerp Water Sampling for PFCs; January 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [redacted]
Email Address: [redacted]

GW= groundwater

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
X ISO17-14-01-044-DUP	P304; Sample Duplicate	9/01/2017	GW	
X ISO17-14-01-044-FMS	P304; Sample FMS	9/01/2017	GW	
X ISO17-14-01-045	P305; Sample	9/01/2017	GW	
X ISO17-14-01-045-DUP	P305; Sample Duplicate	9/01/2017	GW	
X ISO17-14-01-046	P42; Sample	10/01/2017	GW	
X ISO17-14-01-046-DUP	P42; Sample Duplicate	10/01/2017	GW	
X ISO17-14-01-047	L19; Sample	10/01/2017	GW	
X ISO17-14-01-047-DUP	L19; Sample Duplicate	10/01/2017	GW	
X ISO17-14-01-048	M4; Sample	10/01/2017	GW	
X ISO17-14-01-048-DUP	M4; Sample Duplicate	10/01/2017	GW	
X ISO17-14-01-049	P118C; Sample	10/01/2017	GW	
X ISO17-14-01-049-DUP	P118C; Sample Duplicate	10/01/2017	GW	
X ISO17-14-01-050	P119C; Sample	11/1/2017	GW	
X ISO17-14-01-050-DUP	P119C; Sample Duplicate	11/1/2017	GW	
X ISO17-14-01-050-FMS	P119C; Sample FMS	11/1/2017	GW	
X ISO17-14-01-051	P263; Sample	11/1/2017	GW	
X ISO17-14-01-051-DUP	P263; Sample Duplicate	11/1/2017	GW	

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for**

Temperature: 5 **Deg C** ☐ **Received on Ice** ☐ **Other:**

Collected by (print): Tim Mandonx **Collector's signature:**

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
	T. MANDONX	16/01/17	9 00	Fedex	BG / PACE	1-19-17	10:55AM

(17)

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [redacted]
Alt. Pho[redacted]
Fax: (6[redacted]

Project: ISO17-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 12/22/2016
Project Description: 3M Antwerp Water Sampling for PFCs; January 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number[redacted]
Email Address:[redacted]

GW = groundwater

	3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
X	ISO17-14-01-052	P264; Sample	10/01/2017	GW	
X	ISO17-14-01-052-DUP	P264; Sample Duplicate	10/01/2017	GW	
X	ISO17-14-01-052-FMS	P264; Sample FMS	10/01/2017	GW	
X	ISO17-14-01-053	P265C; Sample	/	—	NOT SAMPLED } WELL DAMAGED
X	ISO17-14-01-053-DUP	P265C; Sample Duplicate	/	—	NOT SAMPLED
X	ISO17-14-01-054	P371; Sample	11/1/'17	GW	
X	ISO17-14-01-054-DUP	P371; Sample Duplicate	11/1/'17	GW	
X	ISO17-14-01-054-FMS	P371; Sample FMS	11/1/'17	GW	
X	ISO17-14-01-055	P374; Sample	11/1/'17	GW	
X	ISO17-14-01-055-DUP	P374; Sample Duplicate	11/1/'17	GW	
X	ISO17-14-01-056	P381; Sample	10/1/17	GW	
X	ISO17-14-01-056-DUP	P381; Sample Duplicate	10/1/17	GW	
X	ISO17-14-01-057	P382; Sample	10/1/17	GW	
X	ISO17-14-01-057-DUP	P382; Sample Duplicate	10/1/17	GW	
X	ISO17-14-01-058	B3-bis; Sample	11/1/'17	GW	
X	ISO17-14-01-058-DUP	B3-bis; Sample Duplicate	11/1/'17	GW	
X	ISO17-14-01-058-FMS	B3-bis; Sample FMS	11/1/'17	GW	

Sample Condition Upon Receipt: ☑ **Acceptable** ☐ **All items accounted for**

Temperature: 5 **Deg C** ☐ **Received on Ice** ☐ **Other:**

Collected by (print): Tine Mandonx **Collector's signature:** [signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
	T. MANDONX	16/01/17	9:00	Fedex	[signature] / PACE	1-19-17	10:55AM

(17)

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [redacted]
Alt. Pho[redacted]
Fax: (6[redacted]

Project: ISO17-14-01 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 12/22/2016
Project Description: 3M Antwerp Water Sampling for PFCs; January 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [redacted]
Email Address: [redacted]

GW = groundwater

	3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
X	ISO17-14-01-059	B7; Sample	11/1/'17	GW	
X	ISO17-14-01-059-DUP	B7; Sample Duplicate	11/1/'17	GW	
X	ISO17-14-01-060	P372; Sample	11/1/'17	GW	
X	ISO17-14-01-060-DUP	P372; Sample Duplicate	11/1/'17	GW	
X	ISO17-14-01-061	P378; Sample	11/1/'17	GW	
X	ISO17-14-01-061-DUP	P378; Sample Duplicate	11/1/'17	GW	
X	ISO17-14-01-061-FMS	P378; Sample FMS	11/1/'17	GW	
X	ISO17-14-01-062	PA109A; Sample	11/1/'17	GW	
X	ISO17-14-01-062-DUP	PA109A; Sample Duplicate	11/1/'17	GW	
X	ISO17-14-01-063	PA111A; Sample	11/1/'17	GW	
X	ISO17-14-01-063-DUP	PA111A; Sample Duplicate	11/1/'17	GW	
X	ISO17-14-01-063-FMS	PA111A; Sample FMS	11/1/'17	GW	
X	ISO17-14-01-064	PA112; Sample	11/1/'17	GW	
X	ISO17-14-01-064-DUP	PA112; Sample Duplicate	11/1/'17	GW	
X	ISO17-14-01-065	Travel Blank	*	*	/
X	ISO17-14-01-065-FMS-HIGH	Travel Blank FMS High	*	*	/
X	ISO17-14-01-065-FMS-LOW	Travel Blank FMS Low	*	*	/

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for**

Temperature: 5 **Deg C** ☐ **Received on Ice** ☐ **Other:**

Collected by (print): Time Mandonx **Collector's signature:** [signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
	T. MANDONX	16/01/17	9 00	Fedex	[signature] /PACE	1-19-17	10:55 AM

* Trip Blank samples prepared by the 3M Environmental Laboratory with the bottle order.

Final Report

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

April 2017 Sampling

Laboratory Request Number: ISO17-14-02

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 ISO17-14-02

Water Sample Analysis at 3M Antwerp, Belgium
April 2017 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 collected between April 14, 2017 and April 18, 2017 and returned to the 3M Environmental Laboratory on April 26, 2017 at ambient temperature. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS) (select locations only), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO17-14-02.

The 3M Environmental Laboratory prepared sample containers for sixty-four sampling locations. Each sample set consisted of a field sample and field sample duplicate. Nineteen 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. According to the chain of custody, in lieu of sample location Blokkersdijkvijver – oost, sample location Blokkersdijkvijver – noord was 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.

Table 1. Sample Results Summary [(1)]

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA [(2)]**
Zone: 2nd Aquifer					
ISO17-14-02-001	BD24-3; Sample	49.1	43.0	1120	5.13
ISO17-14-02-001-DUP	BD24-3; Sample Dup	50.3	41.7	1150	5.47
	Average	**49.7**	**42.4**	**1140**	**5.30**
	%RPD Sample/Sample Dup	**2.4**	**3.1**	**2.6**	**6.4**
ISO17-14-02-002	D09; Sample	176	1350	500	9.28
ISO17-14-02-002-DUP	D09; Sample Dup	181	1370	499	10.1
	Average	**179**	**1360**	**500**	**9.69**
	%RPD Sample/Sample Dup	**2.8**	**1.5**	**0.20**	**8.5**
ISO17-14-02-003	P121; Sample	0.246 [(2)]	0.101 [(2)]	1.56 [(2)]	0.186
ISO17-14-02-003-DUP	P121; Sample Dup	0.235 [(2)]	0.0958 [(2)]	1.41 [(2)]	0.171
	Average	**0.241**	**0.0984**	**1.49**	**0.179**
	%RPD Sample/Sample Dup	**4.6**	**5.3**	**10**	**8.4**
ISO17-14-02-004	P321; Sample	2490	642	5440	0.373
ISO17-14-02-004-DUP	P321; Sample Dup	2540	636	5040	0.420
	Average	**2520**	**639**	**5240**	**0.397**
	%RPD Sample/Sample Dup	**2.0**	**0.94**	**7.6**	**12**
Zone: Blokkersdijk nature reserve					
ISO17-14-02-005	3M vijver; Sample	1.12 [(2)]	0.541 [(2)]	4.45 [(2)]	0.0754
ISO17-14-02-005-DUP	3M vijver; Sample Dup	1.18 [(2)]	0.539 [(2)]	4.33 [(2)]	0.0673
	Average	**1.15**	**0.540**	**4.39**	**0.0714**
	%RPD Sample/Sample Dup	**5.2**	**0.37**	**2.7**	**11**
ISO17-14-02-006	Blokkersdijkvijver noord; Sample	0.827 [(2)]	0.492 [(2)]	0.951 [(2)]	0.0290
ISO17-14-02-006-DUP	Blokkersdijkvijver noord; Sample Dup	0.848 [(2)]	0.492 [(2)]	0.950 [(2)]	0.0351
	Average	**0.838**	**0.492**	**0.951**	**0.0321**
	%RPD Sample/Sample Dup	**2.5**	**0.0**	**0.11**	**19**
ISO17-14-02-007	Blokkersdijkvijver - standard; Sample	0.939 [(2)]	0.510 [(2)]	1.88 [(2)]	0.0316
ISO17-14-02-007-DUP	Blokkersdijkvijver - standard; Sample Dup	0.918 [(2)]	0.517 [(2)]	1.83 [(2)]	0.0367
	Average	**0.929**	**0.514**	**1.86**	**0.0342**
	%RPD Sample/Sample Dup	**2.3**	**1.4**	**2.7**	**15**
ISO17-14-02-008	L21; Sample	0.389 [(2)]	0.202 [(2)]	4.92 [(2)]	0.103
ISO17-14-02-008-DUP	L21; Sample Dup	0.451 [(2)]	0.226 [(2)]	5.09 [(2)]	0.122
	Average	**0.420**	**0.214**	**5.01**	**0.113**
	%RPD Sample/Sample Dup	**15**	**11**	**3.4**	**17**
ISO17-14-02-009	L22; Sample	0.459 [(2)]	0.254 [(2)]	4.46 [(2)]	0.109
ISO17-14-02-009-DUP	L22; Sample Dup	0.462 [(2)]	0.252 [(2)]	4.40 [(2)]	0.118
	Average	**0.461**	**0.253**	**4.43**	**0.114**
	%RPD Sample/Sample Dup	**0.65**	**0.79**	**1.4**	**7.9**

NA = Not Applicable
(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 13%, PFBS ± 16%, PFHS ± 9.2%, and PFOS ± 21%.
(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 8.7%, PFHS ± 14%, PFOS ± 14%, and PFOSA ± 16%.
(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
(4) Data uncertainty was expanded for PFOSA to ±44% based on FMS recovery. See section 4 for more information.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA (2)**
Zone: Blokkersdijk nature reserve					
ISO17-14-02-010	L31; Sample	0.516 (2)	0.691 (2)	5.37 (2)	0.121
ISO17-14-02-010-DUP	L31; Sample Dup	0.518 (2)	0.696 (2)	5.40 (2)	0.112
	Average	**0.517**	**0.694**	**5.39**	**0.117**
	%RPD Sample/Sample Dup	**0.39**	**0.72**	**0.56**	**7.7**
ISO17-14-02-011	L4; Sample	2.41 (2)	2.09 (2)	28.8 (2)	2.93
ISO17-14-02-011-DUP	L4; Sample Dup	2.42 (2)	2.13 (2)	28.7 (2)	3.37
	Average	**2.42**	**2.11**	**28.8**	**3.15**
	%RPD Sample/Sample Dup	**0.41**	**1.9**	**0.35**	**14**
ISO17-14-02-012	P114bis; Sample	3.70 (2)	1.31 (2)	13.0 (2)	0.120
ISO17-14-02-012-DUP	P114bis; Sample Dup	3.55 (2)	1.25 (2)	12.0 (2)	0.0927
	Average	**3.63**	**1.28**	**12.5**	**0.106**
	%RPD Sample/Sample Dup	**4.1**	**4.7**	**8.0**	**26 (3)**
ISO17-14-02-013	P115; Sample	5.05 (2)	2.29 (2)	0.486 (2)	<0.0248
ISO17-14-02-013-DUP	P115; Sample Dup	4.91 (2)	2.42 (2)	0.560 (2)	<0.0248
	Average	**4.98**	**2.36**	**0.523**	**<0.0248**
	%RPD Sample/Sample Dup	**2.8**	**5.5**	**14**	**NA**
ISO17-14-02-014	P116; Sample	1.57 (2)	0.619 (2)	9.69 (2)	0.115
ISO17-14-02-014-DUP	P116; Sample Dup	1.27 (2)	0.630 (2)	9.54 (2)	0.111
	Average	**1.42**	**0.625**	**9.62**	**0.113**
	%RPD Sample/Sample Dup	**21 (3)**	**1.8**	**1.6**	**3.5**
Zone: Effluent WWTP					
ISO17-14-02-015	Effluent WWTP; Sample	27.6 (2)	32.8 (2)	80.9	2.54
ISO17-14-02-015-DUP	Effluent WWTP; Sample Dup	27.6 (2)	34.2 (2)	81.0	2.55
	Average	**27.6**	**33.5**	**81.0**	**2.55**
	%RPD Sample/Sample Dup	**0.0**	**4.2**	**0.12**	**0.39**
Zone: Palingbeek & Tophatgracht					
ISO17-14-02-025	12; Sample	88.0	23.9	319	0.478
ISO17-14-02-025-DUP	12; Sample Dup	87.3	23.8	306	0.525
	Average	**87.7**	**23.9**	**313**	**0.502**
	%RPD Sample/Sample Dup	**0.80**	**0.42**	**4.2**	**9.4**
ISO17-14-02-026	13; Sample	80.2	23.4	258	0.439
ISO17-14-02-026-DUP	13; Sample Dup	80.1	23.4	268	0.429
	Average	**80.2**	**23.4**	**263**	**0.434**
	%RPD Sample/Sample Dup	**0.12**	**0.0**	**3.8**	**2.3**
ISO17-14-02-027	5; Sample	36.0	28.8	57.2	0.227
ISO17-14-02-027-DUP	5; Sample Dup	35.7	29.2	58.5	0.241
	Average	**35.9**	**29.0**	**57.9**	**0.234**
	%RPD Sample/Sample Dup	**0.84**	**1.4**	**2.2**	**6.0**

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 ± 13%, PFBS ± 16%, PFHS ± 9.2%, and PFOS ± 21%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 8.7%, PFHS ± 14%, PFOS ± 14%, and PFOSA ± 16%.

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

(4) Data uncertainty was expanded for PFOSA to ±44% based on FMS recovery. See section 4 for more information.

Table 1 continued. Sample Results Summary [1]

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA [2]**
Zone: Palingbeek & Tophatgracht					
ISO17-14-02-028	Bemalingsstation; Sample	32.7	26.5	54.7	0.220
ISO17-14-02-028-DUP	Bemalingsstation; Sample Dup	32.6	26.1	55.3	0.226
	Average	**32.7**	**26.3**	**55.0**	**0.223**
	%RPD Sample/Sample Dup	**0.31**	**1.5**	**1.1**	**2.7**
Zone: Sewer					
ISO17-14-02-029	Collector put; Sample	113	39.0	360	23.1
ISO17-14-02-029-DUP	Collector put; Sample Dup	116	39.4	369	25.2
	Average	**115**	**39.2**	**365**	**24.2**
	%RPD Sample/Sample Dup	**2.6**	**1.0**	**2.5**	**8.7**
Zone: Source area – WWTP					
ISO17-14-02-031	P263; Sample	1410	448	7890	23.9
ISO17-14-02-031-DUP	P263; Sample Dup	1390	445	7870	23.1
	Average	**1400**	**447**	**7880**	**23.5**
	%RPD Sample/Sample Dup	**1.4**	**0.67**	**0.25**	**3.4**
ISO17-14-02-033	PB8101; Sample	1630	1050	2900	3.14
ISO17-14-02-033-DUP	PB8101; Sample Dup	1580	1040	2870	3.29
	Average	**1610**	**1050**	**2890**	**3.22**
	%RPD Sample/Sample Dup	**3.1**	**0.96**	**1.0**	**4.7**
ISO17-14-02-034	PB8102; Sample	99.2	26.7	734	85.8
ISO17-14-02-034-DUP	PB8102; Sample Dup	95.7	25.3	700	81.7
	Average	**97.5**	**26.0**	**717**	**83.8 [4]**
	%RPD Sample/Sample Dup	**3.6**	**5.4**	**4.7**	**4.9**
ISO17-14-02-035	PB9104; Sample	15.3	4.68	142	2.59
ISO17-14-02-035-DUP	PB9104; Sample Dup	13.9	4.39	141	2.77
	Average	**14.6**	**4.54**	**142**	**2.68**
	%RPD Sample/Sample Dup	**9.6**	**6.4**	**0.71**	**6.7**
ISO17-14-02-036	PB9105; Sample	19.3	3.41	174	1.97
ISO17-14-02-036-DUP	PB9105; Sample Dup	18.7	3.18	172	1.56
	Average	**19.0**	**3.30**	**173**	**1.77**
	%RPD Sample/Sample Dup	**3.2**	**7.0**	**1.2**	**23 [3]**
ISO17-14-02-037	PB9106; Sample	18.9	2.74	168	23.5
ISO17-14-02-037-DUP	PB9106; Sample Dup	19.5	2.87	173	24.6
	Average	**19.2**	**2.81**	**171**	**24.1**
	%RPD Sample/Sample Dup	**3.1**	**4.6**	**2.9**	**4.6**

NA = Not Applicable
(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 13%, PFBS ± 16%, PFHS ± 9.2%, and PFOS ± 21%.
(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 8.7%, PFHS ± 14%, PFOS ± 14%, and PFOSA ± 16%.
(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
(4) Data uncertainty was expanded for PFOSA to ±44% based on FMS recovery. See section 4 for more information.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)				
3M LIMS ID	**Sample Description**	**PFOA**	**PFBS**	**PFHS**	**PFOS**	**PFOSA (2)**
Zone: Extractions wells P&T						
ISO17-14-02-016	PP01; Sample	48.9	46.7	4090	1280	28.1
ISO17-14-02-016-DUP	PP01; Sample Dup	50.2	44.8	3810	1220	29.0
	Average	**49.6**	**45.8**	**3950**	**1250**	**28.6**
	%RPD Sample/Sample Dup	**2.6**	**4.2**	**7.1**	**4.8**	**3.2**
ISO17-14-02-017	PP02; Sample	463	28.2	2840	20900	48.8
ISO17-14-02-017-DUP	PP02; Sample Dup	468	27.3	2860	21000	46.4
	Average	**466**	**27.8**	**2850**	**21000**	**47.6**
	%RPD Sample/Sample Dup	**1.1**	**3.2**	**0.70**	**0.48**	**5.0**
ISO17-14-02-018	PP04; Sample	549	2330	246	5270	80.4
ISO17-14-02-018-DUP	PP04; Sample Dup	533	2260	244	5110	79.0
	Average	**541**	**2300**	**245**	**5190**	**79.7**
	%RPD Sample/Sample Dup	**3.0**	**3.1**	**0.82**	**3.1**	**1.8**
ISO17-14-02-019	PP05; Sample	672	6610	2150	519	21.0
ISO17-14-02-019-DUP	PP05; Sample Dup	632	6350	2060	406	17.9
	Average	**652**	**6480**	**2110**	**463**	**19.5**
	%RPD Sample/Sample Dup	**6.1**	**4.0**	**4.3**	**24 (3)**	**16**
ISO17-14-02-020	PP06; Sample	355	100	98.2	2360	108
ISO17-14-02-020-DUP	PP06; Sample Dup	351	106	96.9	2300	101
	Average	**353**	**103**	**97.6**	**2330**	**105**
	%RPD Sample/Sample Dup	**1.1**	**5.8**	**1.3**	**2.6**	**6.7**
ISO17-14-02-021	PP07; Sample	375	110	116	2860	133
ISO17-14-02-021-DUP	PP07; Sample Dup	385	115	118	2900	118
	Average	**380**	**113**	**117**	**2880**	**126**
	%RPD Sample/Sample Dup	**2.6**	**4.4**	**1.7**	**1.4**	**12**
ISO17-14-02-022	PP08; Sample	596	37.1	264	7510	70.1
ISO17-14-02-022-DUP	PP08; Sample Dup	607	37.1	266	7830	65.9
	Average	**602**	**37.1**	**265**	**7670**	**68.0**
	%RPD Sample/Sample Dup	**1.8**	**0.0**	**0.75**	**4.2**	**6.2**
ISO17-14-02-023	PP09; Sample	673	23.6	408	2370	22.3
ISO17-14-02-023-DUP	PP09; Sample Dup	675	24.1	407	2370	22.9
	Average	**674**	**23.9**	**408**	**2370**	**22.6**
	%RPD Sample/Sample Dup	**0.30**	**2.1**	**0.25**	**0.0**	**2.7**
ISO17-14-02-024	PP10; Sample	556	16.2	193	3700	24.8
ISO17-14-02-024-DUP	PP10; Sample Dup	548	15.9	186	3230	21.9
	Average	**552**	**16.1**	**190**	**3470**	**23.4**
	%RPD Sample/Sample Dup	**1.4**	**1.9**	**3.7**	**14**	**12**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 13%, PFBS ± 16%, PFHS ± 9.2%, and PFOS ± 21%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 8.7%, PFHS ± 14%, PFOS ± 14%, and PFOSA ± 16%.

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

(4) Data uncertainty was expanded for PFOSA to ±44% based on FMS recovery. See section 4 for more information.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)				
3M LIMS ID	Sample Description	PFOA	PFBS	PFHS	PFOS	PFOSA (2)
Zone: Source area - Building 16						
ISO17-14-02-030	P21B; Sample	11400	7370	11700	67500	8.87
ISO17-14-02-030-DUP	P21B; Sample Dup	11100	7200	11500	64800	8.25
	Average	**11300**	**7290**	**11600**	**66200**	**8.56**
	%RPD Sample/Sample Dup	**2.7**	**2.3**	**1.7**	**4.1**	**7.2**

NA = Not Applicable
(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 13%, PFBS ± 16%, PFHS ± 9.2%, and PFOS ± 21%.
(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 8.7%, PFHS ± 14%, PFOS ± 14%, and PFOSA ± 16%.
(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
(4) Data uncertainty was expanded for PFOSA to ±44% based on FMS recovery. See section 4 for more information.

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 April 14, 2017 and April 18, 2017 in Nalgene™ (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 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

Select samples were prepared for PFOA, PFHS, and PFOS and PFBS for select locations using a direct injection by methanol dilution method. Samples that required a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples that required a 1:100 dilution were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol. Samples that required a 1:500 dilution were prepared by diluting 0.02 mL of a well-mixed sample with 10.0 mL of methanol. Samples that required a 1:1000 dilution were prepared by first diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol, followed by a second dilution prepared by diluting 1 mL of the diluted sample into 9 mL of methanol. Diluted samples and LCSs were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

Sample locations P121, 3M vijver, Blokkersdijkvijver – Noord, Blokkersdijkvijver – standard, L21, L22, L31, L4, P114bis, P115, P116, Effluent WWTP, and Travel Blank sample and FMS Low were analyzed for PFOA, PFHS, and PFOS by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples along with the calibration standards and method blanks.

All samples were prepared for PFOSA by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The laboratory control samples were then diluted with methanol in the same manner. 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 the target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). SRSs were added and included in the analysis of these samples. Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.2. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

The following analytical runs were used to report the results herein:

5/22/17 (ETS Kirk) External Standard Calibration Analysis:

- Sample locations ISO17-14-02-001, -002, -004, and -016 to -037 were analyzed for PFOA, PFHS, and PFOS with select locations being analyzed for PFBS. All sample results were reported **except** for the following: P321 (PFOA, PFHS, PFOS), P21B (PFOS), and Travel Blank sample and FMS Low (PFOA, PFHS, PFOS).

5/23/17 (ETS Kirk) Internal Standard Calibration Analysis:

- Sample locations ISO17-14-02-003, and -005 to -015 and Travel Blank sample and FMS Low were analyzed for PFOA, PFHS, and PFOS. All sample results were reported **except** for the following: Effluent WWTP (PFOS).

5/24/17 (ETS Kirk) Internal Standard Calibration Analysis:

- Sample location Effluent WWTP (PFOS).

5/24/17 (ETS Kirk) External Standard Calibration Analysis:

- Sample locations P321 (PFOA, PFHS, PFOS) and P21B (PFOS).

5/25/17 (ETS Rita) Internal Standard Calibration Analysis:

- All sample locations were analyzed for PFOSA. All sample results were reported **except** for the following: PB8102.

5/31/17 (ETS Rita) Internal Standard Calibration Analysis:

- Sample location PB8102 (PFOSA).

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	5/22/17, 5/23/17, 5/24/17	5/25/17, 5/31/17
Guard column	Prism RP (2.1 mm X 50 mm), 5 µ	Prism RP (2.1 mm X 50 mm), 5 µ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 µ	Betasil C18 (2.1 mm X 100 mm), 5 µ
Injection Volume	2 or 5 µL	1 or 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	0.70	300	60.0	40.0
3	9.00	300	5.0	95.0
4	11.0	300	5.0	95.0
5	12.0	300	90.0	10.0
6	14.0	300	90.0	10.0

Table 5. Mass Transitions

Analyte	Mass Transition Q1/Q3	Internal Standard	Mass Transition Q1/Q3
PFOA	413/369	[$^{13}C_8$]-PFOA	421/376
	413/219		
	413/169		
PFBS	299/99	NA	NA
	299/80		
PFHS	399/99	[$^{13}C_3$]-PFHS	402/80
	399/80		
PFOS	499/99	[$^{13}C_8$]-PFOS	507/80
	499/80		
	499/130		
PFOSA	498/78	[$^{13}C_8$]-PFOSA	506/78
[$^{13}C_4$]-PFOA	417/372	[$^{13}C_8$]-PFOA	421/376
[$^{13}C_4$]-PFOS	503/80	[$^{13}C_8$]-PFOS	507/80

NA = Not Applicable
The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

3 Data Analysis

3.1 Calibration

5/22/17 and 5/24/17 Analysis (External Standard Calibration): Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed on 5/22/17. On 5/24/17, calibration standards ranging from 0.25 ng/mL to 100 ng/mL (nominal) were analyzed. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL to 25 ng/mL (nominal) or 0.25 ng/mL to 25 ng/mL (nominal) on 5/24/17. A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

5/23/17 and 5/24/17 Analysis (Internal Standard Calibration): Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of fourteen calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed. The calibration standards also contained surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

5/25/17 and 5/31/17 Analysis of PFOSA (Internal Standard Calibration): Samples were analyzed for PFOSA against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into laboratory reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 1.0 ng/mL. Calibration standards ranging from 0.025 ng/mL to 200 ng/mL (nominal) were analyzed on 5/25/17. Standards ranged from 5.0 ng/mL to 200 ng/mL (nominal) on 5/31/17. Prior to analysis, the calibration standards were diluted 2x by removing a 0.4 mL aliquot and diluting it with 0.4 mL of methanol. A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±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) 5/22/17 Analysis	LOQ, ng/mL (2) 5/23/17 Analysis	LOQ, ng/mL (2) 5/24/17 (internal) Analysis	LOQ, ng/mL (1) 5/24/17 (external) Analysis	LOQ, ng/mL (2) 5/25/17 Analysis	LOQ, ng/mL (2) 5/31/17 Analysis
PFOA	0.0192	0.0240	NA	0.240	NA	NA
PFBS	0.0200	NA	NA	NA	NA	NA
PFHS	0.0200	0.0250	NA	0.250	NA	NA
PFOS	0.0464	0.0464	0.0928	0.232	NA	NA
PFOSA	NA	NA	NA	NA	0.0248	5.00

NA = Not Applicable
(1) A dilution factor was not applied to the LOQ.
(2) A dilution factor of 2 was applied to the LOQ.

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of 100%±25%.

3.5 Blanks

Two types of blanks were prepared and analyzed with the samples: method/solvent blanks and field/trip blanks. Each blank result was reviewed and used to evaluate method performance. The method blanks were used to determine the LOQ for each analyte.

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. LCSs were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. Surrogates [$^{13}C_4$]-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:

- 5/22/17: High-level LCSs were spiked above the resulting upper limit of quantitation (ULOQ) for PFBS (25,100 ng/mL), and PFHS (25,100 ng/mL) with a dilution factor of 501 applied.
- 5/23/17: High-level LCSs were spiked above the resulting ULOQ for PFOA (48.0 ng/mL), PFHS (100 ng/mL), and PFOS (92.8 ng/mL) with dilution factor of 2 applied.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report. 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 5/22/17	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-170519-1	100	95.8	95.8	100	94.9	94.9
LCS-170519-2	100	93.5	93.5	100	93.0	93.0
LCS-170519-3	100	93.0	93.0	100	93.6	93.6
Average ± %RSD	**94.1% ± 1.6%**			**93.8% ± 1.0%**		
LCS-170519-4	5000	5050	101	5000	5000	99.9
LCS-170519-5	5000	5080	102	5000	5250	105
LCS-170519-6	5000	5040	101	5000	5070	101
Average ± %RSD	**101% ± 0.57%**			**102% ± 2.6%**		
LCS-170519-7	35000	30800	88.0	35000	>ULOQ	NA
LCS-170519-8	35000	31100	88.9	35000	>ULOQ	NA
LCS-170519-9	35000	30700	87.9	35000	>ULOQ	NA
Average ± %RSD	**88.3% ± 0.62%**			**NA (1)**		

ETS-8-044.3 External Calibration Analyzed 5/22/17	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-170519-1	100	94.0	94.0	100	92.0	92.0
LCS-170519-2	100	97.9	97.9	100	94.1	94.1
LCS-170519-3	100	96.0	96.0	100	91.2	91.2
Average ± %RSD	**96.0 % ± 2.0%**			**92.4% ± 1.6%**		
LCS-170519-4	5000	4980	99.6	5000	5160	103
LCS-170519-5	5000	5290	106	5000	5060	101
LCS-170519-6	5000	4950	98.9	5000	4900	98.0
Average ± %RSD	**102% ± 3.9%**			**101% ± 2.5%**		
LCS-170519-7	35000	>ULOQ	NA	35000	38400	110
LCS-170519-8	35000	>ULOQ	NA	35000	38000	108
LCS-170519-9	35000	>ULOQ	NA	35000	38800	111
Average ± %RSD	**NA (1)**			**110% ± 1.4%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 5/22/17	[$^{13}C_4$]-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-170519-1	99.8	105	106	95.6	101	106
LCS-170519-2	99.8	107	107	95.6	99.1	104
LCS-170519-3	99.8	108	108	95.6	102	107
Average ± %RSD	**107% ± 0.93%**			**106% ± 1.4%**		
LCS-170519-4	998	1130	114	956	1060	111
LCS-170519-5	998	1110	111	956	1060	111
LCS-170519-6	998	1170	117	956	1050	110
Average ± %RSD	**114% ± 2.6%**			**111% ± 0.52%**		

ETS-8-044.3 Internal Calibration Analyzed 5/23/17	PFOA (Linear + Branched)			PFHS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-170523-1	0.190	0.168	88.5	0.198	0.195	98.7
LCS-170523-2	0.190	0.184	96.8	0.198	0.207	105
LCS-170523-3	0.190	0.180	94.9	0.198	0.211	107
Average ± %RSD	**93.4% ± 4.7%**			**104% ± 4.2%**		
LCS-170523-4	19.0	18.4	96.6	19.8	18.4	92.8
LCS-170523-5	19.0	17.0	89.7	19.8	17.8	89.8
LCS-170523-6	19.0	16.6	87.4	19.8	17.2	86.9
Average ± %RSD	**91.2% ± 5.2%**			**89.8% ± 3.3%**		
LCS-170523-7	133	>ULOQ	NA	139	>ULOQ	NA
LCS-170523-8	133	>ULOQ	NA	139	>ULOQ	NA
LCS-170523-9	133	>ULOQ	NA	139	>ULOQ	NA
Average ± %RSD	**NA (1)**			**NA (1)**		

NA = Not Applicable
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 5/23/17	PFOS (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-170523-1	0.184	0.175	95.4
LCS-170523-2	0.184	0.181	98.6
LCS-170523-3	0.184	0.188	102
Average ± %RSD	**98.7% ± 3.3%**		
LCS-170523-4	18.4	17.5	95.2
LCS-170523-5	18.4	16.3	88.3
LCS-170523-6	18.4	16.1	87.4
Average ± %RSD	**90.3% ± 4.7%**		
LCS-170523-7	129	>ULOQ	NA
LCS-170523-8	129	>ULOQ	NA
LCS-170523-9	129	>ULOQ	NA
Average ± %RSD	**NA (1)**		

ETS-8-044.3 Internal Calibration Analyzed 5/23/17	[$^{13}C_4$]-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-170523-1	0.198	0.171	86.4	0.189	0.169	89.3
LCS-170523-2	0.198	0.182	92.1	0.189	0.165	87.5
LCS-170523-3	0.198	0.172	86.8	0.189	0.165	87.4
Average ± %RSD	**88.4% ± 3.6%**			**88.1% ± 1.2%**		
LCS-170523-4	1.98	1.92	97.1	1.89	1.82	96.3
LCS-170523-5	1.98	1.82	91.9	1.89	1.73	91.5
LCS-170523-6	1.98	1.84	92.9	1.89	1.73	91.7
Average ± %RSD	**94.0% ± 2.9%**			**93.2% ± 2.9%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 5/24/17	PFOS (Linear + Branched)			[$^{13}C_4$]-PFOS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-170523-1	0.184	0.177	96.4	0.189	0.176	93.0
LCS-170523-2	0.184	0.183	99.6	0.189	0.175	92.6
LCS-170523-3	0.184	0.198	107	0.189	0.177	93.8
Average ± %RSD	**101% ± 5.4%**			**93.1% ± 0.66%**		
LCS-170523-4	18.4	17.7	96.4	1.89	1.93	102
LCS-170523-5	18.4	16.9	92.0	1.89	1.81	95.5
LCS-170523-6	18.4	16.3	88.4	1.89	1.81	96.0
Average ± %RSD	**92.3% ± 4.3%**			**97.8% ± 3.7%**		
LCS-170523-7	129	107	83.2			
LCS-170523-8	129	111	86.1			
LCS-170523-9	129	105	81.5			
Average ± %RSD	**83.6% ± 2.8%**					

ETS-8-044.3 External Calibration Analyzed 5/24/17	PFOA (Linear + Branched)			PFHS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-170524-1	1000	990	99.0	1000	1030	103
LCS-170524-2	1000	980	98.0	1000	986	98.6
LCS-170524-3	1000	949	94.9	1000	960	96.0
Average ± %RSD	**97.3% ± 2.2%**			**99.2% ± 3.6%**		
LCS-170524-4	25000	24200	96.7	25000	25500	102
LCS-170524-5	25000	24400	97.6	25000	25400	101
LCS-170524-6	25000	24900	99.6	25000	25400	102
Average ± %RSD	**98.0% ± 1.5%**			**102% ± 0.57%**		
LCS-170524-7	70000	63900	91.3	70000	69100	98.7
LCS-170524-8	70000	65100	93.1	70000	70500	101
LCS-170524-9	70000	63800	91.1	70000	69300	99.0
Average ± %RSD	**91.8% ± 1.2%**			**99.6% ± 1.3%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 5/24/17	PFOS (Linear + Branched)		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-170524-1	1000	988	98.8
LCS-170524-2	1000	964	96.4
LCS-170524-3	1000	947	94.7
Average ± %RSD	**96.6% ± 2.1%**		
LCS-170524-4	25000	24800	99.3
LCS-170524-5	25000	24700	98.7
LCS-170524-6	25000	25000	100
Average ± %RSD	**99.3% ± 0.66%**		
LCS-170524-7	70000	73400	105
LCS-170524-8	70000	75700	108
LCS-170524-9	70000	74800	107
Average ± %RSD	**107% ± 1.4%**		

ETS-8-044.3 External Calibration Analyzed 5/24/17	[$^{13}C_4$]-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-170524-1	499	527	106	478	508	106
LCS-170524-2	499	531	106	478	494	103
LCS-170524-3	499	529	106	478	514	107
Average ± %RSD	**106% ± 0.0%**			**105% ± 2.0%**		
LCS-170524-4	4990	5560	111	4780	5460	114
LCS-170524-5	4990	5620	113	4780	5380	113
LCS-170524-6	4990	5540	111	4780	5540	116
Average ± %RSD	**112% ± 1.0%**			**114% ± 1.3%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 5/25/17	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-170523-1	0.198	0.198	100
LCS-170523-2	0.198	0.195	98.6
LCS-170523-3	0.198	0.190	95.7
Average ± %RSD	**98.1% ± 2.2%**		
LCS-170523-4	19.8	18.9	95.5
LCS-170523-5	19.8	17.9	90.3
LCS-170523-6	19.8	17.3	87.2
Average ± %RSD	**91.0% ± 4.6%**		
LCS-170523-7	139	124	89.2
LCS-170523-8	139	126	90.4
LCS-170523-9	139	123	88.5
Average ± %RSD	**89.4% ± 1.1%**		

ETS-8-044.3 Internal Calibration Analyzed 5/31/17	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-170531-1	19.9	20.2	102
LCS-170531-2	19.9	20.0	100
LCS-170531-3	19.9	20.2	102
Average ± %RSD	**101% ± 1.1%**		
LCS-170531-4	79.8	79.1	99.1
LCS-170531-5	79.8	79.6	99.7
LCS-170531-6	79.8	80.5	101
Average ± %RSD	**99.9% ± 0.97%**		
LCS-170531-7	139	143	103
LCS-170531-8	139	145	104
LCS-170531-9	139	141	102
Average ± %RSD	**103% ± 0.97%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the calibration range.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.4. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in Table 8 below.

Table 8. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	6.71	±13%
PFBS	External	7.93	±16%
PFHS	External	4.62	±9.2%
PFOS	External	10.5	±21%
PFOA	Internal	4.33	±8.7%
PFHS	Internal	7.11	±14%
PFOS	Internal	6.94	±14%
PFOSA	Internal	8.25	±16%

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. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

The following calculation was used to generate data in section 4 of the report.

$$FMS\ Recovery = \frac{Sample\ Concentration\ of\ FMS - Average\ Concentration\ (Field\ Sample\ \&\ Field\ Sample\ Dup)}{Spike\ Concentration} \times 100\%$$

Table 9. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
L31, L4	FMS	2.40	2.50	2.50	2.32	2.50
P21B, P263	FMS	20.0	20.0	20.0	20.0	20.0
5	FMS	51.0	51.0	51.0	50.9	1.00
BD24-3, PB9104, PB9105	FMS	54.8	55.0	55.0	54.6	5.00
PB9106	FMS	105	105	105	105	5.00
PP07	FMS	501	50.0	500	500	50.0
PB8101, PB8102	FMS	952	50.0	950	950	50.0
Trip Blank	Low	2.40	2.50	2.50	2.32	2.50
	High	501	50.0	500	500	50.0

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of ±30%, demonstrate that the method is appropriate for the given matrix.

The method indicates that the target analyte FMS samples should be spiked at approximately 0.5-10 times the expected analyte concentration in the sample. At times the spike level exceeded the recommended upper limit of 10 times the analyte concentration. In these instances the FMS recovery was reported and flagged as above 10 times the sample concentration.

For PFOA, PFBS, PFHS, and PFOS, when the field matrix spike level was not appropriate as compared to the endogenous sample concentration, the surrogate recovery standards were used to assess method accuracy. All field matrix spike and surrogate recovery standard recoveries met acceptance criteria except where noted below.

PB8102: The field matrix spike recovery was 56.5% for PFOSA. The data uncertainty for PFOSA was expanded to ±44% for this location.

Table 10. Location ID: BD24-3

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-02-001	BD24-3; Sample	49.1	NA	43.0	NA
ISO17-14-02-001-DUP	BD24-3; Sample Duplicate	50.3	NA	41.7	NA
ISO17-14-02-001-FMS	BD24-3; FMS	103	97.3	94.5	94.8
Average Concentration (ng/mL) ± %RPD		49.7 ng/mL ± 2.4%		42.4 ng/mL ± 3.1%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-02-001	BD24-3; Sample	1120	NA	5.13	NA
ISO17-14-02-001-DUP	BD24-3; Sample Duplicate	1150	NA	5.47	NA
ISO17-14-02-001-FMS	BD24-3; FMS	1270	NC	10.8	110
Average Concentration (ng/mL) ± %RPD		1140 ng/mL ± 2.6%		5.30 ng/mL ± 6.4%	

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

Table 11. Location ID: L31

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-02-010	L31; Sample	0.516	NA	0.691	NA
ISO17-14-02-010-DUP	L31; Sample Duplicate	0.518	NA	0.696	NA
ISO17-14-02-010-FMS	L31; FMS	2.73	92.4	3.31	105
Average Concentration (ng/mL) ± %RPD		0.517 ng/mL ± 0.39%		0.694 ng/mL ± 0.72%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-02-010	L31; Sample	5.37	NA	0.121	NA
ISO17-14-02-010-DUP	L31; Sample Duplicate	5.40	NA	0.112	NA
ISO17-14-02-010-FMS	L31; FMS	7.91	NC	2.62	100 (1)
Average Concentration (ng/mL) ± %RPD		5.39 ng/mL ± 0.56%		0.117 ng/mL ± 7.7%	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) FMS concentration greater than 10 times the sample concentration.

Table 12. Location ID: L4

3M LIMS ID	Sample Description	PFOA Concentration (ng/mL)	PFOA %Recovery	PFHS Concentration (ng/mL)	PFHS %Recovery
ISO17-14-02-011	L4; Sample	2.41	NA	2.09	NA
ISO17-14-02-011-DUP	L4; Sample Duplicate	2.42	NA	2.13	NA
ISO17-14-02-011-FMS	L4; FMS	4.87	103	4.74	105
Average Concentration (ng/mL) ± %RPD		**2.42 ng/mL ± 0.41%**		**2.11 ng/mL ± 1.9%**	

3M LIMS ID	Sample Description	PFOS Concentration (ng/mL)	PFOS %Recovery	PFOSA Concentration (ng/mL)	PFOSA %Recovery
ISO17-14-02-011	L4; Sample	28.8	NA	2.93	NA
ISO17-14-02-011-DUP	L4; Sample Duplicate	28.7	NA	3.37	NA
ISO17-14-02-011-FMS	L4; FMS	30.9	NC	4.95	72.0
Average Concentration (ng/mL) ± %RPD		**28.8 ng/mL ± 0.35%**		**3.15 ng/mL ± 14%**	

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

Table 13. Location ID: PP07

3M LIMS ID	Sample Description	PFOA Concentration (ng/mL)	PFOA %Recovery	PFBS Concentration (ng/mL)	PFBS %Recovery	PFHS Concentration (ng/mL)	PFHS %Recovery
ISO17-14-02-021	PP07; Sample	375	NA	110	NA	116	NA
ISO17-14-02-021-DUP	PP07; Sample Duplicate	385	NA	115	NA	118	NA
ISO17-14-02-021-FMS	PP07; FMS	852	94.2	158	NC	594	95.4
Average Concentration (ng/mL) ± %RPD		**380 ng/mL ± 2.6%**		**113 ng/mL ± 4.4%**		**117 ng/mL ± 1.7%**	

3M LIMS ID	Sample Description	PFOS Concentration (ng/mL)	PFOS %Recovery	PFOSA Concentration (ng/mL)	PFOSA %Recovery
ISO17-14-02-021	PP07; Sample	2860	NA	133	NA
ISO17-14-02-021-DUP	PP07; Sample Duplicate	2900	NA	118	NA
ISO17-14-02-021-FMS	PP07; FMS	3360	NC	135	NC
Average Concentration (ng/mL) ± %RPD		**2880 ng/mL ± 1.4%**		**126 ng/mL ± 12%**	

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

Table 14. Location ID: 5

3M LIMS ID	Sample Description	PFOA Concentration (ng/mL)	PFOA %Recovery	PFHS Concentration (ng/mL)	PFHS %Recovery
ISO17-14-02-027	5; Sample	36.0	NA	28.8	NA
ISO17-14-02-027-DUP	5; Sample Duplicate	35.7	NA	29.2	NA
ISO17-14-02-027-FMS	5; FMS	82.5	91.5	74.9	90.0
Average Concentration (ng/mL) ± %RPD		**35.9 ng/mL ± 0.84%**		**29.0 ng/mL ± 1.4%**	

3M LIMS ID	Sample Description	PFOS Concentration (ng/mL)	PFOS %Recovery	PFOSA Concentration (ng/mL)	PFOSA %Recovery
ISO17-14-02-027	5; Sample	57.2	NA	0.227	NA
ISO17-14-02-027-DUP	5; Sample Duplicate	58.5	NA	0.241	NA
ISO17-14-02-027-FMS	5; FMS	101	84.7	1.27	104
Average Concentration (ng/mL) ± %RPD		**57.9 ng/mL ± 2.2%**		**0.234 ng/mL ± 6.0%**	

NA = Not Applicable

Table 15. Location ID: P21B

3M LIMS ID	Sample Description	PFOA Concentration (ng/mL)	PFOA %Recovery	PFBS Concentration (ng/mL)	PFBS %Recovery	PFHS Concentration (ng/mL)	PFHS %Recovery
ISO17-14-02-030	P21B; Sample	11400	NA	7370	NA	11700	NA
ISO17-14-02-030-DUP	P21B; Sample Duplicate	11100	NA	7200	NA	11500	NA
ISO17-14-02-030-FMS	P21B; FMS	10300	NC	6650	NC	10500	NC
Average Concentration (ng/mL) ± %RPD		**11300 ng/mL ± 2.7%**		**7290 ng/mL ± 2.3%**		**11600 ng/mL ± 1.7%**	

3M LIMS ID	Sample Description	PFOS Concentration (ng/mL)	PFOS %Recovery	PFOSA Concentration (ng/mL)	PFOSA %Recovery
ISO17-14-02-030	P21B; Sample	67500	NA	8.87	NA
ISO17-14-02-030-DUP	P21B; Sample Duplicate	64800	NA	8.25	NA
ISO17-14-02-030-FMS	P21B; FMS	NA (1)	NA (1)	30.5	110
Average Concentration (ng/mL) ± %RPD		**5.41 ng/mL ± 0.19%**		**8.56 ng/mL ± 7.2%**	

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.

Table 16. Location ID: P263

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-02-031	P263; Sample	1410	NA	448	NA
ISO17-14-02-031-DUP	P263; Sample Duplicate	1390	NA	445	NA
ISO17-14-02-031-FMS	P263; FMS	1400	NC	462	NC
Average Concentration (ng/mL) ± %RPD		**1400 ng/mL ± 1.4%**		**447 ng/mL ± 0.67%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-02-031	P263; Sample	7890	NA	23.9	NA
ISO17-14-02-031-DUP	P263; Sample Duplicate	7870	NA	23.1	NA
ISO17-14-02-031-FMS	P263; FMS	7920	NC	41.5	90.0
Average Concentration (ng/mL) ± %RPD		**7880 ng/mL ± 0.25%**		**23.5 ng/mL ± 3.4%**	

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

Table 17. Location ID: PB8101

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-02-033	PB8101; Sample	1630	NA	1050	NA
ISO17-14-02-033-DUP	PB8101; Sample Duplicate	1580	NA	1040	NA
ISO17-14-02-033-FMS	PB8101; FMS	2460	89.8	1950	95.3
Average Concentration (ng/mL) ± %RPD		**1610 ng/mL ± 3.1%**		**1050 ng/mL ± 0.96%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-02-033	PB8101; Sample	2900	NA	3.14	NA
ISO17-14-02-033-DUP	PB8101; Sample Duplicate	2870	NA	3.29	NA
ISO17-14-02-033-FMS	PB8101; FMS	3870	NC	52.1	97.8 (1)
Average Concentration (ng/mL) ± %RPD		**2890 ng/mL ± 1.0%**		**3.22 ng/mL ± 4.7%**	

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

Table 18. Location ID: PB8102

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-02-034	PB8102; Sample	99.2	NA	26.7	NA
ISO17-14-02-034-DUP	PB8102; Sample Duplicate	95.7	NA	25.3	NA
ISO17-14-02-034-FMS	PB8102; FMS	972	91.9	923	94.4 (1)
Average Concentration (ng/mL) ± %RPD		97.5 ng/mL ± 3.6%		26.0 ng/mL ± 5.4%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-02-034	PB8102; Sample	734	NA	85.8	NA
ISO17-14-02-034-DUP	PB8102; Sample Duplicate	700	NA	81.7	NA
ISO17-14-02-034-FMS	PB8102; FMS	1570	89.8	112	56.5 (2)
Average Concentration (ng/mL) ± %RPD		717 ng/mL ± 4.7%		83.8 ng/mL ± 4.9% (3)	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.
(2) FMS recovery did not meet acceptance criteria of 100 ± 30%.
(3) The data uncertainty was expanded to ±44% for PFOSA.

Table 19. Location ID: PB9104

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-02-035	PB9104; Sample	15.3	NA	4.68	NA
ISO17-14-02-035-DUP	PB9104; Sample Duplicate	13.9	NA	4.39	NA
ISO17-14-02-035-FMS	PB9104; FMS	62.1	86.7	51.0	84.5 (1)
Average Concentration (ng/mL) ± %RPD		14.6 ng/mL ± 9.6%		4.54 ng/mL ± 6.4%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-02-035	PB9104; Sample	142	NA	2.59	NA
ISO17-14-02-035-DUP	PB9104; Sample Duplicate	141	NA	2.77	NA
ISO17-14-02-035-FMS	PB9104; FMS	188	NC	7.56	97.6
Average Concentration (ng/mL) ± %RPD		142 ng/mL ± 0.71%		2.68 ng/mL ± 6.7%	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) FMS concentration greater than 10 times the sample concentration.

Table 20. Location ID: PB9105

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-02-036	PB9105; Sample	19.3	NA	3.41	NA
ISO17-14-02-036-DUP	PB9105; Sample Duplicate	18.7	NA	3.18	NA
ISO17-14-02-036-FMS	PB9105; FMS	68.1	89.6	51.1	86.9 (1)
Average Concentration (ng/mL) ± %RPD		**19.0 ng/mL ± 3.2%**		**3.30 ng/mL ± 7.0%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-02-036	PB9105; Sample	174	NA	1.97	NA
ISO17-14-02-036-DUP	PB9105; Sample Duplicate	172	NA	1.56	NA
ISO17-14-02-036-FMS	PB9105; FMS	217	NC	6.80	101
Average Concentration (ng/mL) ± %RPD		**173 ng/mL ± 1.2%**		**1.77 ng/mL ± 23% (2)**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) FMS concentration greater than 10 times the sample concentration.
(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 21. Location ID: PB9106

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-02-037	PB9106; Sample	18.9	NA	2.74	NA
ISO17-14-02-037-DUP	PB9106; Sample Duplicate	19.5	NA	2.87	NA
ISO17-14-02-037-FMS	PB9106; FMS	109	85.7	92.0	84.9 (1)
Average Concentration (ng/mL) ± %RPD		**19.2 ng/mL ± 3.1%**		**2.81 ng/mL ± 4.6%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-02-037	PB9106; Sample	168	NA	23.5	NA
ISO17-14-02-037-DUP	PB9106; Sample Duplicate	173	NA	24.6	NA
ISO17-14-02-037-FMS	PB9106; FMS	260	85.5	26.4	NC
Average Concentration (ng/mL) ± %RPD		**171 ng/mL ± 2.9%**		**24.1 ng/mL ± 4.6%**	

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 22. Location ID: Travel Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-02-032	Travel Blank	<0.0240	NA	<0.200	NA	<0.0250	NA
ISO17-14-02-032-FMS-LOW	Travel Blank FMS Low	2.35	98.1	2.44	97.6	2.53	101
ISO17-14-02-032-FMS-HIGH	Travel Blank FMS High	505	101	52.0	104	511	102

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-02-032	Travel Blank	<0.0464	NA	<0.0248	NA
ISO17-14-02-032-FMS-LOW	Travel Blank FMS Low	2.25	97.1	2.49	99.6
ISO17-14-02-032-FMS-HIGH	Travel Blank FMS High	508	102	49.9	99.8

NA = Not Applicable

Table 23. 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
ISO17-14-02-001	BD24-3; Sample	113	108	NA
ISO17-14-02-001-DUP	BD24-3; Sample Duplicate	119	115	NA
ISO17-14-02-001-FMS	BD24-3; FMS	116	111	NA
ISO17-14-02-002	D09; Sample	116	116	NA
ISO17-14-02-002-DUP	D09; Sample Duplicate	114	112	NA
ISO17-14-02-003	P121; Sample	93.7 (2)	91.2 (2)	NA
ISO17-14-02-003-DUP	P121; Sample Duplicate	87.8 (2)	91.6 (2)	NA
ISO17-14-02-004	P321; Sample	119	119	NA
ISO17-14-02-004-DUP	P321; Sample Duplicate	122	118	NA
ISO17-14-02-005	3M vijver; Sample	88.3 (2)	96.3(2)	NA
ISO17-14-02-005-DUP	3M vijver; Sample Duplicate	100 (2)	93.1(2)	NA
ISO17-14-02-006	Blokkersdijkvijver - noord; Sample	98.8 (2)	92.0 (2)	NA
ISO17-14-02-006-DUP	Blokkersdijkvijver - noord; Sample Dup	101 (2)	90.4 (2)	NA
ISO17-14-02-007	Blokkersdijkvijver - standard; Sample	99.0 (2)	93.3 (2)	NA
ISO17-14-02-007-DUP	Blokkersdijkvijver - standard; Sample Dup	91.3 (2)	88.6 (2)	NA
ISO17-14-02-008	L21; Sample	89.4 (2)	93.5 (2)	NA
ISO17-14-02-008-DUP	L21; Sample Duplicate	95.9 (2)	87.6 (2)	NA
ISO17-14-02-009	L22; Sample	91.2 (2)	94.5 (2)	NA
ISO17-14-02-009-DUP	L22; Sample Duplicate	92.6 (2)	92.0 (2)	NA
ISO17-14-02-010	L31; Sample	91.5 (2)	92.3 (2)	NA
ISO17-14-02-010-DUP	L31; Sample Duplicate	95.1 (2)	95.6 (2)	NA
ISO17-14-02-010-FMS	L31; FMS	91.5 (2)	92.6 (2)	NA
ISO17-14-02-011	L4; Sample	84.9 (2)	82.9 (2)	NA
ISO17-14-02-011-DUP	L4; Sample Duplicate	89.5 (2)	93.1 (2)	NA
ISO17-14-02-011-FMS	L4; FMS	88.5 (2)	91.8 (2)	NA
ISO17-14-02-012	P114bis; Sample	92.3 (2)	89.6 (2)	NA
ISO17-14-02-012-DUP	P114bis; Sample Duplicate	94.4 (2)	91.6 (2)	NA
ISO17-14-02-013	P115; Sample	103 (2)	96.4 (2)	NA
ISO17-14-02-013-DUP	P115; Sample Duplicate	89.8 (2)	95.4 (2)	NA
ISO17-14-02-014	P116; Sample	89.1 (2)	94.9 (2)	NA
ISO17-14-02-014-DUP	P116; Sample Duplicate	95.9 (2)	90.7 (2)	NA
ISO17-14-02-015	Effluent WWTP; Sample	98.1 (2)	85.4 (2)	92.5 (2)
ISO17-14-02-015-DUP	Effluent WWTP; Sample Duplicate	93.2 (2)	83.5 (2)	91.3 (2)
ISO17-14-02-016	PP01; Sample	115	113	NA
ISO17-14-02-016-DUP	PP01; Sample Duplicate	111	106	NA
ISO17-14-02-017	PP02; Sample	114	108	NA
ISO17-14-02-017-DUP	PP02; Sample Duplicate	114	108	NA
ISO17-14-02-018	PP04; Sample	114	116	NA
ISO17-14-02-018-DUP	PP04; Sample Duplicate	110	105	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.

Table 23 continued. Surrogate Recovery Standard Results (1)

3M LIMS ID	Sample Description	Percent Recovery (%) $[^{13}C_4]$-PFOA	$[^{13}C_4]$-PFOS	$[^{13}C_4]$-PFOS Re-analysis
ISO17-14-02-019	PP05; Sample	114	113	NA
ISO17-14-02-019-DUP	PP05; Sample Duplicate	112	114	NA
ISO17-14-02-020	PP06; Sample	116	115	NA
ISO17-14-02-020-DUP	PP06; Sample Duplicate	116	114	NA
ISO17-14-02-021	PP07; Sample	112	111	NA
ISO17-14-02-021-DUP	PP07; Sample Duplicate	111	113	NA
ISO17-14-02-021-FMS	PP07; FMS	114	114	NA
ISO17-14-02-022	PP08; Sample	109	108	NA
ISO17-14-02-022-DUP	PP08; Sample Duplicate	111	110	NA
ISO17-14-02-023	PP09; Sample	111	113	NA
ISO17-14-02-023-DUP	PP09; Sample Duplicate	105	107	NA
ISO17-14-02-024	PP10; Sample	112	110	NA
ISO17-14-02-024-DUP	PP10; Sample Duplicate	114	109	NA
ISO17-14-02-025	12; Sample	109	110	NA
ISO17-14-02-025-DUP	12; Sample Duplicate	109	105	NA
ISO17-14-02-026	13; Sample	107	104	NA
ISO17-14-02-026-DUP	13; Sample Duplicate	105	107	NA
ISO17-14-02-027	5; Sample	102	103	NA
ISO17-14-02-027-DUP	5; Sample Duplicate	109	109	NA
ISO17-14-02-027-FMS	5; FMS	111	106	NA
ISO17-14-02-028	Bemalingsstation; Sample	110	107	NA
ISO17-14-02-028-DUP	Bemalingsstation; Sample Duplicate	109	108	NA
ISO17-14-02-029	Collector put; Sample	106	107	NA
ISO17-14-02-029-DUP	Collector put; Sample Duplicate	107	103	NA
ISO17-14-02-030	P21B; Sample	106	105	120
ISO17-14-02-030-DUP	P21B; Sample Duplicate	106	103	121
ISO17-14-02-030-FMS	P21B; FMS	108	103	NA
ISO17-14-02-031	P263; Sample	110	111	NA
ISO17-14-02-031-DUP	P263; Sample Duplicate	112	110	NA
ISO17-14-02-031-FMS	P263; FMS	112	111	NA
ISO17-14-02-032	Travel Blank	94.0 (2)	91.3 (2)	NA
ISO17-14-02-032-FMS-LOW	Travel Blank FMS Low	96.6 (2)	92.7 (2)	NA
ISO17-14-02-032-FMS-HIGH	Travel Blank FMS High	104	101	NA
ISO17-14-02-033	PB8101; Sample	103	105	NA
ISO17-14-02-033-DUP	PB8101; Sample Duplicate	105	107	NA
ISO17-14-02-033-FMS	PB8101; FMS	104	105	NA
ISO17-14-02-034	PB8102; Sample	109	107	NA
ISO17-14-02-034-DUP	PB8102; Sample Duplicate	95.0	94.6	NA
ISO17-14-02-034-FMS	PB8102; FMS	109	107	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.

Table 23 continued. Surrogate Recovery Standard Results [1]

		Percent Recovery (%)	
3M LIMS ID	Sample Description	[$^{13}C_4$]-PFOA	[$^{13}C_4$]-PFOS
ISO17-14-02-035	PB9104; Sample	112	110
ISO17-14-02-035-DUP	PB9104; Sample Duplicate	110	111
ISO17-14-02-035-FMS	PB9104; FMS	111	110
ISO17-14-02-036	PB9105; Sample	117	111
ISO17-14-02-036-DUP	PB9105; Sample Duplicate	111	109
ISO17-14-02-036-FMS	PB9105; FMS	111	109
ISO17-14-02-037	PB9106; Sample	114	109
ISO17-14-02-037-DUP	PB9106; Sample Duplicate	114	109
ISO17-14-02-037-FMS	PB9106; FMS	113	109

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.

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 Attachments

Chain of Custody Form

8 Signatures

Chelsie Grochow

Digitally signed by Chelsie J. Grochow
DN: c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory - authenticated by LRA, [redacted] o=3M, cn=Chelsie J. Grochow
Reason: I am the author of this document
Date: 2017.06.30 10:22:50 -05'00'

Chelsie Grochow, 3M Report Author

Susan T. Wolf
c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory - authenticated by LRA, [redacted] o=3M, cn=Susan T. Wolf
I have reviewed this document
2017.06.30 10:03:03 -05'00'

Susan T. Wolf, 3M Principal Analytical Investigator

Digitally signed by William K. Reagen
DN: c=US, st=MN, l=St. Paul, ou=Laboratory Director, ou=3M Environmental Laboratory - authenticated by LRA, [redacted] o=3M, cn=William K. Reagen
Reason: I am approving this document
Date: 2017.07.05 07:38:43 -05'00'

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.

Digitally signed by Matthew D. Naruko-Stewart
DN: c=US, st=MN, l=St. Paul, ou=Quality Assurance Unit, ou=3M Environmental Laboratory - authenticated by LRA [redacted] o=3M, cn=Matthew D. Naruko-Stewart
Reason: I agree to the terms defined by the placement of my signature on this document
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Quality Assurance Representative

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Final Report

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

July 2017 Sampling

Laboratory Request Number: ISO17-14-03

Report Date – Date of Last Signature

Testing Laboratory

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

Requester

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

The testing reported herein meet the requirements of ANSI/ISO/IEC 17025:2005 "General Requirements for the Competence of Testing and Calibration Laboratories", in accordance with A2LA Certificate # 2052.01. Additionally, the laboratory's quality system has been audited and was determined to be in conformance with the EPA GLPs (40 CFR 792) by an independent A2LA assessment.

3M EHS Laboratory

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

Analytical Report ISO17-14-03

Water Sample Analysis at 3M Antwerp, Belgium
July 2017 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M EHS Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. All samples were collected between June 28, 2017 and July 3, 2017 and returned to the 3M EHS Laboratory on July 10, 2017 at ambient temperature. The samples were analyzed for Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS) (select locations), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO17-14-03.

The 3M EHS Laboratory prepared sample containers for seventy-four sampling locations. Each sample set consisted of a field sample and field sample duplicate. Eighteen locations also included a target analyte field matrix spike. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with a mass-labeled internal standard mix and select sample bottles were fortified with surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS prior to being sent to the field for sample collection. Sample location P265C 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.

Table 1. Sample Results Summary [(1)]

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
Zone: 2nd Aquifer					
ISO17-14-03-001	BD24-3; Sample	44.1	37.2	1210	5.51
ISO17-14-03-001-DUP	BD24-3; Sample Dup	43.4	38.5	1180	5.44
	Average	**43.8**	**37.9**	**1200**	**5.48 (2)**
	%RPD Sample/Sample Dup	**1.6**	**3.4**	**2.5**	**1.3**
ISO17-14-03-002	BD24-4; Sample	12.8	5.85	6.33	0.470
ISO17-14-03-002-DUP	BD24-4; Sample Dup	12.5	5.38	5.46	0.458
	Average	**12.7**	**5.62**	**5.90**	**0.464 (2)**
	%RPD Sample/Sample Dup	**2.4**	**8.4**	**15**	**2.6**
ISO17-14-03-003	D09; Sample	126	4880	1750	28.2
ISO17-14-03-003-DUP	D09; Sample Dup	123	4760	1700	27.6
	Average	**125**	**4820**	**1730**	**27.9 (2)**
	%RPD Sample/Sample Dup	**2.4**	**2.5**	**2.9**	**2.2**
ISO17-14-03-004	D10; Sample	309	108	23.3	0.443
ISO17-14-03-004-DUP	D10; Sample Dup	310	109	24.2	0.458
	Average	**310**	**109**	**23.8**	**0.451 (2)**
	%RPD Sample/Sample Dup	**0.32**	**0.92**	**3.8**	**3.3**
ISO17-14-03-005	D11; Sample	83.4	45.6	834	93.9
ISO17-14-03-005-DUP	D11; Sample Dup	79.0	45.0	836	97.7
	Average	**81.2**	**45.3**	**835**	**95.8 (2)**
	%RPD Sample/Sample Dup	**5.4**	**1.3**	**0.24**	**4.0**
ISO17-14-03-006	D14; Sample	2.45	1.34	0.423	<0.0248
ISO17-14-03-006-DUP	D14; Sample Dup	2.35	1.33	0.352	<0.0248
	Average	**2.40**	**1.34**	**0.388**	**<0.0248 (2)**
	%RPD Sample/Sample Dup	**4.2**	**0.75**	**18**	**NA**
ISO17-14-03-007	D16; Sample	198	53.9	1240	103
ISO17-14-03-007-DUP	D16; Sample Dup	195	54.3	1230	102
	Average	**197**	**54.1**	**1240**	**103 (2)**
	%RPD Sample/Sample Dup	**1.5**	**0.74**	**0.81**	**0.98**
ISO17-14-03-008	D17; Sample	779	76.4	389	40.8
ISO17-14-03-008-DUP	D17; Sample Dup	760	75.5	375	39.4
	Average	**770 (4)**	**76.0**	**382**	**40.1 (2)**
	%RPD Sample/Sample Dup	**2.5**	**1.2**	**3.7**	**3.5**
ISO17-14-03-009	D18; Sample	208	109	53.5	5.85
ISO17-14-03-009-DUP	D18; Sample Dup	207	108	50.9	5.81
	Average	**208**	**109**	**52.2**	**5.83 (2)**
	%RPD Sample/Sample Dup	**0.48**	**0.92**	**5.0**	**0.69**

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 ± 25%, PFHS ± 20%, PFOS ± 28%, and PFOSA ± 31%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 14%, PFBS ± 13%, PFHS ± 20%, PFOS ± 17%, and PFOSA ± 17%.

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

(4) The data uncertainty for PFOA has been expanded for location D1 to ± 31% based on surrogate recovery. See section 4 for details.

(5) The data uncertainty for PFOSA has been expanded for location Collector put to ± 36% based on 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**
Zone: 2nd Aquifer					
ISO17-14-03-010	D2; Sample	0.295	0.146	8.14	0.794
ISO17-14-03-010-DUP	D2; Sample Dup	0.319	0.154	8.52	0.845
	Average	**0.307 (2)**	**0.150 (2)**	**8.33 (2)**	**0.820 (2)**
	%RPD Sample/Sample Dup	**7.8**	**5.3**	**4.6**	**6.2**
ISO17-14-03-011	D5; Sample	646	276	641	16.6
ISO17-14-03-011-DUP	D5; Sample Dup	626	275	609	15.3
	Average	**636**	**276**	**625**	**16.0 (2)**
	%RPD Sample/Sample Dup	**3.1**	**0.36**	**5.1**	**8.2**
ISO17-14-03-012	ND7; Sample	158	12.6	221	164
ISO17-14-03-012-DUP	ND7; Sample Dup	157	12.0	224	165
	Average	**158**	**12.3**	**223**	**165 (2)**
	%RPD Sample/Sample Dup	**0.63**	**4.9**	**1.3**	**0.61**
ISO17-14-03-013	P118A; Sample	1570	1910	153	1.61
ISO17-14-03-013-DUP	P118A; Sample Dup	1610	1900	161	1.60
	Average	**1590**	**1910**	**157**	**1.61 (2)**
	%RPD Sample/Sample Dup	**2.5**	**0.52**	**5.1**	**0.62**
ISO17-14-03-014	P118B; Sample	2030	3320	15900	6.42
ISO17-14-03-014-DUP	P118B; Sample Dup	2000	3270	15300	5.74
	Average	**2020**	**3300**	**15600**	**6.08 (2)**
	%RPD Sample/Sample Dup	**1.5**	**1.5**	**3.8**	**11**
ISO17-14-03-015	P119A; Sample	340	176	2000	84.2
ISO17-14-03-015-DUP	P119A; Sample Dup	349	175	1980	88.8
	Average	**345**	**176**	**1990**	**86.5 (2)**
	%RPD Sample/Sample Dup	**2.6**	**0.57**	**1.0**	**5.3**
ISO17-14-03-016	P119B; Sample	550	222	139	1.14
ISO17-14-03-016-DUP	P119B; Sample Dup	550	225	116	1.14
	Average	**550**	**224**	**128**	**1.14 (2)**
	%RPD Sample/Sample Dup	**0.0**	**1.3**	**18**	**0.0**
ISO17-14-03-017	P121; Sample	0.309	0.293	1.40	0.202
ISO17-14-03-017-DUP	P121; Sample Dup	0.250	0.122	1.44	0.193
	Average	**0.280 (2)**	**0.208 (2)**	**1.42 (2)**	**0.198 (2)**
	%RPD Sample/Sample Dup	**21 (3)**	**82 (3)**	**2.8**	**4.6**
ISO17-14-03-018	P321; Sample	976	322	4200	14.8
ISO17-14-03-018-DUP	P321; Sample Dup	998	330	4360	14.8
	Average	**987**	**326**	**4280**	**14.8 (2)**
	%RPD Sample/Sample Dup	**2.2**	**2.5**	**3.7**	**0.0**

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 ± 25%, PFHS ± 20%, PFOS ± 28%, and PFOSA ± 31%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 14%, PFBS ± 13%, PFHS ± 20%, PFOS ± 17%, and PFOSA ± 17%.

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

(4) The data uncertainty for PFOA has been expanded for location D1 to ± 31% based on surrogate recovery. See section 4 for details.

(5) The data uncertainty for PFOSA has been expanded for location Collector put to ± 36% based on 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**
Zone: Blokkersdijk nature reserve					
ISO17-14-03-019	3M vijver; Sample	2.00	0.928	5.90	0.0793
ISO17-14-03-019-DUP	3M vijver; Sample Dup	2.05	0.949	5.73	0.0681
	Average	**2.03 (2)**	**0.939 (2)**	**5.82 (2)**	**0.0737 (2)**
	%RPD Sample/Sample Dup	**2.5**	**2.2**	**2.9**	**15**
ISO17-14-03-020	Blokkersdijkvijver Noord; Sample	1.26	0.639	2.06	0.0784
ISO17-14-03-020-DUP	Blokkersdijkvijver Noord; Sample Dup	1.15	0.685	2.09	0.0607
	Average	**1.21 (2)**	**0.662 (2)**	**2.08 (2)**	**0.0696 (2)**
	%RPD Sample/Sample Dup	**9.1**	**6.9**	**1.4**	**25 (3)**
ISO17-14-03-021	Blokkersdijkvijver standard; Sample	1.43	0.738	6.38	0.226
ISO17-14-03-021-DUP	Blokkersdijkvijver standard; Sample Dup	1.52	0.769	6.85	0.235
	Average	**1.48 (2)**	**0.754 (2)**	**6.62 (2)**	**0.231 (2)**
	%RPD Sample/Sample Dup	**6.1**	**4.1**	**7.1**	**3.9**
ISO17-14-03-022	L21; Sample	0.416	0.198	6.26	0.296
ISO17-14-03-022-DUP	L21; Sample Dup	0.419	0.178	5.99	0.285
	Average	**0.418 (2)**	**0.188 (2)**	**6.13 (2)**	**0.291 (2)**
	%RPD Sample/Sample Dup	**0.72**	**11**	**4.4**	**3.8**
ISO17-14-03-023	L22; Sample	0.791	0.368	4.99	0.0926
ISO17-14-03-023-DUP	L22; Sample Dup	0.776	0.356	4.95	0.111
	Average	**0.784**	**0.362**	**4.97**	**0.102 (2)**
	%RPD Sample/Sample Dup	**1.9**	**3.3**	**0.80**	**18**
ISO17-14-03-024	L31; Sample	1.25	0.939	7.58	0.188
ISO17-14-03-024-DUP	L31; Sample Dup	1.13	0.950	7.47	0.146
	Average	**1.19**	**0.945**	**7.53**	**0.167 (2)**
	%RPD Sample/Sample Dup	**10**	**1.2**	**1.5**	**25 (3)**
ISO17-14-03-025	L4; Sample	2.81	2.18	33.0	0.568
ISO17-14-03-025-DUP	L4; Sample Dup	2.94	2.28	32.5	0.544
	Average	**2.88**	**2.23**	**32.8**	**0.556 (2)**
	%RPD Sample/Sample Dup	**4.5**	**4.5**	**1.5**	**4.3**
ISO17-14-03-026	P114bis; Sample	2.55	1.11	11.9	0.0472
ISO17-14-03-026-DUP	P114bis; Sample Dup	2.59	1.11	11.4	0.0581
	Average	**2.57**	**1.11**	**11.7**	**0.0527 (2)**
	%RPD Sample/Sample Dup	**1.6**	**0.0**	**4.3**	**21 (3)**
ISO17-14-03-027	P115; Sample	5.32	2.33	0.437	<0.0248
ISO17-14-03-027-DUP	P115; Sample Dup	4.96	2.30	0.448	<0.0248
	Average	**5.14**	**2.32**	**0.443**	**<0.0248 (2)**
	%RPD Sample/Sample Dup	**7.0**	**1.3**	**2.5**	**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 ± 25%, PFHS ± 20%, PFOS ± 28%, and PFOSA ± 31%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 14%, PFBS ± 13%, PFHS ± 20%, PFOS ± 17%, and PFOSA ± 17%.

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

(4) The data uncertainty for PFOA has been expanded for location D1 to ± 31% based on surrogate recovery. See section 4 for details.

(5) The data uncertainty for PFOSA has been expanded for location Collector put to ± 36% based on 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
Zone: Blokkersdijk nature reserve					
ISO17-14-03-028	P116; Sample	1.39	0.654	8.76	0.139
ISO17-14-03-028-DUP	P116; Sample Dup	1.52	0.620	9.39	0.106
	Average	**1.46**	**0.637**	**9.08**	**0.123 (2)**
	%RPD Sample/Sample Dup	**8.9**	**5.3**	**6.9**	**27 (3)**
Zone: Effluent WWTP					
ISO17-14-03-029	Effluent WWTP; Sample	2.27	2.79	0.890	<0.0248
ISO17-14-03-029-DUP	Effluent WWTP; Sample Dup	2.34	3.01	0.930	0.0276
	Average	**2.31 (2)**	**2.90 (2)**	**0.910 (2)**	**0.0276 (2)**
	%RPD Sample/Sample Dup	**3.0**	**7.6**	**4.4**	**NA**
Zone: Palingbeek & Tophatgracht					
ISO17-14-03-039	12; Sample	108	26.3	365	0.355
ISO17-14-03-039-DUP	12; Sample Dup	107	26.7	369	0.335
	Average	**108**	**26.5**	**367**	**0.345 (2)**
	%RPD Sample/Sample Dup	**0.93**	**1.5**	**1.1**	**5.8**
ISO17-14-03-040	13; Sample	94.4	27.1	298	0.274
ISO17-14-03-040-DUP	13; Sample Dup	91.6	27.4	299	0.268
	Average	**93.0**	**27.3**	**299**	**0.271 (2)**
	%RPD Sample/Sample Dup	**3.0**	**1.1**	**0.34**	**2.2**
ISO17-14-03-041	5; Sample	88.0	77.5	103	0.372
ISO17-14-03-041-DUP	5; Sample Dup	87.2	78.9	104	0.377
	Average	**87.6**	**78.2**	**104**	**0.375 (2)**
	%RPD Sample/Sample Dup	**0.91**	**1.8**	**0.97**	**1.3**
ISO17-14-03-042	Bemalingsstation; Sample	70.2	62.5	87.7	0.303
ISO17-14-03-042-DUP	Bemalingsstation; Sample Dup	71.3	63.3	89.9	0.284
	Average	**70.8**	**62.9**	**88.8**	**0.294 (2)**
	%RPD Sample/Sample Dup	**1.6**	**1.3**	**2.5**	**6.5**
Zone: Sewer					
ISO17-14-03-043	Collector put; Sample	107	37.9	370	24.6
ISO17-14-03-043-DUP	Collector put; Sample Dup	107	38.6	370	25.2
	Average	**107**	**38.3**	**370**	**24.9 (5)**
	%RPD Sample/Sample Dup	**0.0**	**1.8**	**0.0**	**2.4**
Zone: Source area – WWTP					
ISO17-14-03-051	L19; Sample	191	90.7	4420	50.7
ISO17-14-03-051-DUP	L19; Sample Dup	187	86.5	4010	41.3
	Average	**189**	**88.6**	**4220**	**46.0 (2)**
	%RPD Sample/Sample Dup	**2.1**	**4.7**	**9.7**	**20**

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 ± 14%, PFBS ± 13%, PFHS ± 20%, PFOS ± 17%, and PFOSA ± 17%.

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

(4) The data uncertainty for PFOA has been expanded for location D1 to ± 31% based on surrogate recovery. See section 4 for details.

(5) The data uncertainty for PFOSA has been expanded for location Collector put to ± 36% based on 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
Zone: Source area - WWTP					
ISO17-14-03-052	M4; Sample	4360	460	11600	212
ISO17-14-03-052-DUP	M4; Sample Dup	4300	452	11300	220
	Average	**4330**	**456**	**11500**	**216 (2)**
	%RPD Sample/Sample Dup	**1.4**	**1.8**	**2.6**	**3.7**
ISO17-14-03-053	P118C; Sample	150	35.7	1130	104
ISO17-14-03-053-DUP	P118C; Sample Dup	134	34.0	1130	120
	Average	**142**	**34.9**	**1130**	**112**
	%RPD Sample/Sample Dup	**11**	**4.9**	**0.0**	**14**
ISO17-14-03-054	P119C; Sample	712	427	3780	93.0
ISO17-14-03-054-DUP	P119C; Sample Dup	708	418	3610	95.5
	Average	**710**	**423**	**3700**	**94.3**
	%RPD Sample/Sample Dup	**0.56**	**2.1**	**4.6**	**2.7**
ISO17-14-03-055	P262; Sample	1160	527	4480	74.5
ISO17-14-03-055-DUP	P262; Sample Dup	1160	530	4170	86.6
	Average	**1160**	**529**	**4330**	**80.6 (2)**
	%RPD Sample/Sample Dup	**0.0**	**0.57**	**7.2**	**15**
ISO17-14-03-056	P263; Sample	1640	506	9800	15.1
ISO17-14-03-056-DUP	P263; Sample Dup	1620	490	9990	14.0
	Average	**1630**	**498**	**9900**	**14.6 (2)**
	%RPD Sample/Sample Dup	**1.2**	**3.2**	**1.9**	**7.6**
ISO17-14-03-057	P264; Sample	322	196	1490	128
ISO17-14-03-057-DUP	P264; Sample Dup	313	199	1520	136
	Average	**318**	**198**	**1510**	**132 (2)**
	%RPD Sample/Sample Dup	**2.8**	**1.5**	**2.0**	**6.1**
ISO17-14-03-059	P340; Sample	631	305	3610	108
ISO17-14-03-059-DUP	P340; Sample Dup	642	307	3690	110
	Average	**637**	**306**	**3650**	**109 (2)**
	%RPD Sample/Sample Dup	**1.7**	**0.65**	**2.2**	**1.8**
ISO17-14-03-060	P341; Sample	543	265	5450	94.8
ISO17-14-03-060-DUP	P341; Sample Dup	551	275	5640	87.7
	Average	**547**	**270**	**5550**	**91.3 (2)**
	%RPD Sample/Sample Dup	**1.5**	**3.7**	**3.4**	**7.8**
ISO17-14-03-061	P343; Sample	667	405	6920	23.4
ISO17-14-03-061-DUP	P343; Sample Dup	653	394	6750	22.1
	Average	**660**	**400**	**6840**	**22.8 (2)**
	%RPD Sample/Sample Dup	**2.1**	**2.8**	**2.5**	**5.7**

NA = Not Applicable
(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 14%, PFBS ± 25%, PFHS ± 20%, PFOS ± 28%, and PFOSA ± 31%.
(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 14%, PFBS ± 13%, PFHS ± 20%, PFOS ± 17%, and PFOSA ± 17%.
(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
(4) The data uncertainty for PFOA has been expanded for location D1 to ± 31% based on surrogate recovery. See section 4 for details.
(5) The data uncertainty for PFOSA has been expanded for location Collector put to ± 36% based on 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**
Zone: Source area - WWTP					
ISO17-14-03-062	P371; Sample	466	187	4450	31.9
ISO17-14-03-062-DUP	P371; Sample Dup	472	189	4450	30.5
	Average	**469**	**188**	**4450**	**31.2 (2)**
	%RPD Sample/Sample Dup	**1.3**	**1.1**	**0.0**	**4.5**
ISO17-14-03-063	P374; Sample	728	1190	2190	38.4
ISO17-14-03-063-DUP	P374; Sample Dup	744	1170	2260	37.8
	Average	**736**	**1180**	**2230**	**38.1 (2)**
	%RPD Sample/Sample Dup	**2.2**	**1.7**	**3.1**	**1.6**
ISO17-14-03-064	P379; Sample	74.3	22.0	760	91.9
ISO17-14-03-064-DUP	P379; Sample Dup	75.0	21.4	768	89.2
	Average	**74.7**	**21.7**	**764**	**90.6 (2)**
	%RPD Sample/Sample Dup	**0.94**	**2.8**	**1.0**	**3.0**
ISO17-14-03-065	P380; Sample	119	42.7	622	115
ISO17-14-03-065-DUP	P380; Sample Dup	122	44.7	644	133
	Average	**121**	**43.7**	**633**	**124 (2)**
	%RPD Sample/Sample Dup	**2.5**	**4.6**	**3.5**	**15**
ISO17-14-03-066	P381; Sample	604	167	1270	19.4
ISO17-14-03-066-DUP	P381; Sample Dup	592	164	1290	17.7
	Average	**598**	**166**	**1280**	**18.6 (2)**
	%RPD Sample/Sample Dup	**2.0**	**1.8**	**1.6**	**9.2**
ISO17-14-03-067	P382; Sample	485	138	891	125
ISO17-14-03-067-DUP	P382; Sample Dup	507	137	887	120
	Average	**496**	**138**	**889**	**123 (2)**
	%RPD Sample/Sample Dup	**4.4**	**0.73**	**0.45**	**4.1**
Zone: Southern site boundary					
ISO17-14-03-068	B3-bis; Sample	21.7	5.03	176	0.819
ISO17-14-03-068-DUP	B3-bis; Sample Dup	22.9	6.05	183	0.698
	Average	**22.3**	**5.54**	**180**	**0.759 (2)**
	%RPD Sample/Sample Dup	**5.4**	**18**	**3.9**	**16**
ISO17-14-03-069	B7; Sample	81.7	11.4	285	4.45
ISO17-14-03-069-DUP	B7; Sample Dup	58.3	8.37	254	3.93
	Average	**70.0**	**9.89**	**270**	**4.19 (2)**
	%RPD Sample/Sample Dup	**33 (3)**	**31 (3)**	**12**	**12**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 14%, PFBS ± 25%, PFHS ± 20%, PFOS ± 28%, and PFOSA ± 31%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 14%, PFBS ± 13%, PFHS ± 20%, PFOS ± 17%, and PFOSA ± 17%.

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

(4) The data uncertainty for PFOA has been expanded for location D1 to ± 31% based on surrogate recovery. See section 4 for details.

(5) The data uncertainty for PFOSA has been expanded for location Collector put to ± 36% based on 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**
Zone: Southern site boundary					
ISO17-14-03-070	P372; Sample	68.1	42.9	1190	58.2
ISO17-14-03-070-DUP	P372; Sample Dup	65.9	44.3	1210	61.2
	Average	**67.0**	**43.6**	**1200**	**59.7** [(2)]
	%RPD Sample/Sample Dup	**3.3**	**3.2**	**1.7**	**5.0**
ISO17-14-03-071	P378; Sample	265	268	1110	3.09
ISO17-14-03-071-DUP	P378; Sample Dup	260	272	1100	2.92
	Average	**263**	**270**	**1110**	**3.01** [(2)]
	%RPD Sample/Sample Dup	**1.9**	**1.5**	**0.90**	**5.7**
ISO17-14-03-072	PA109A; Sample	52.2	54.3	173	3.39
ISO17-14-03-072-DUP	PA109A; Sample Dup	54.4	57.3	169	3.20
	Average	**53.3**	**55.8**	**171**	**3.30** [(2)]
	%RPD Sample/Sample Dup	**4.1**	**5.4**	**2.3**	**5.8**
ISO17-14-03-073	PA111A; Sample	836	1080	2300	17.1
ISO17-14-03-073-DUP	PA111A; Sample Dup	860	1090	2320	19.2
	Average	**848**	**1090**	**2310**	**18.2** [(2)]
	%RPD Sample/Sample Dup	**2.8**	**0.92**	**0.87**	**12**
ISO17-14-03-074	PA112; Sample	149	33.8	357	108
ISO17-14-03-074-DUP	PA112; Sample Dup	151	32.1	358	106
	Average	**150**	**33.0**	**358**	**107** [(2)]
	%RPD Sample/Sample Dup	**1.3**	**5.2**	**0.28**	**1.9**

		Concentration (ng/mL)				
3M LIMS ID	**Sample Description**	**PFOA**	**PFBS**	**PFHS**	**PFOS**	**PFOSA**
Zone: Extractions wells P&T						
ISO17-14-03-030	PP01; Sample	57.3	53.7	4540	1250	26.5
ISO17-14-03-030-DUP	PP01; Sample Dup	54.1	54.2	4530	1230	26.5
	Average	**55.7**	**54.0**	**4540**	**1240**	**26.5** [(2)]
	%RPD Sample/Sample Dup	**5.7**	**0.93**	**0.22**	**1.6**	**0.0**
ISO17-14-03-031	PP02; Sample	504	32.3	3270	25000	31.9
ISO17-14-03-031-DUP	PP02; Sample Dup	510	32.6	3290	25600	56.7
	Average	**507**	**32.5**	**3280**	**25300**	**44.3** [(2)]
	%RPD Sample/Sample Dup	**1.2**	**0.92**	**0.61**	**2.4**	**56** [(3)]
ISO17-14-03-032	PP04; Sample	1530	1560	463	2750	19.0
ISO17-14-03-032-DUP	PP04; Sample Dup	1550	1600	464	2740	19.0
	Average	**1540**	**1580**	**464**	**2750**	**19.0** [(2)]
	%RPD Sample/Sample Dup	**1.3**	**2.5**	**0.22**	**0.36**	**0.0**

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 ± 25%, PFHS ± 20%, PFOS ± 28%, and PFOSA ± 31%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 14%, PFBS ± 13%, PFHS ± 20%, PFOS ± 17%, and PFOSA ± 17%.

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

(4) The data uncertainty for PFOA has been expanded for location D1 to ± 31% based on surrogate recovery. See section 4 for details.

(5) The data uncertainty for PFOSA has been expanded for location Collector put to ± 36% based on 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**
Zone: Extractions wells P&T						
ISO17-14-03-033	PP05; Sample	866	7940	3400	524	15.8
ISO17-14-03-033-DUP	PP05; Sample Dup	910	8350	3680	661	15.9
	Average	**888**	**8145**	**3540**	**593**	**15.9 (2)**
	%RPD Sample/Sample Dup	**5.0**	**5.0**	**7.9**	**23 (3)**	**0.63**
ISO17-14-03-034	PP06; Sample	163	21.9	55.4	1630	77.9
ISO17-14-03-034-DUP	PP06; Sample Dup	166	21.5	54.5	1590	75.3
	Average	**165**	**21.7**	**55.0**	**1610**	**76.6 (2)**
	%RPD Sample/Sample Dup	**1.8**	**1.8**	**1.6**	**2.5**	**3.4**
ISO17-14-03-035	PP07; Sample	332	161	105	3120	131
ISO17-14-03-035-DUP	PP07; Sample Dup	334	162	104	2590	86.4
	Average	**333**	**162**	**105**	**2860**	**109 (2)**
	%RPD Sample/Sample Dup	**0.60**	**0.62**	**0.96**	**19**	**41 (3)**
ISO17-14-03-036	PP08; Sample	604	32.6	271	7480	54.9
ISO17-14-03-036-DUP	PP08; Sample Dup	632	32.9	267	7480	58.7
	Average	**618**	**32.8**	**269**	**7480**	**56.8 (2)**
	%RPD Sample/Sample Dup	**4.5**	**0.92**	**1.5**	**0.0**	**6.7**
ISO17-14-03-037	PP09; Sample	751	23.9	395	2630	13.4
ISO17-14-03-037-DUP	PP09; Sample Dup	746	23.6	389	2510	13.3
	Average	**749**	**23.8**	**392**	**2570**	**13.4 (2)**
	%RPD Sample/Sample Dup	**0.67**	**1.3**	**1.5**	**4.7**	**0.75**
ISO17-14-03-038	PP10; Sample	603	17.2	204	3710	17.9
ISO17-14-03-038-DUP	PP10; Sample Dup	596	17.8	207	3840	19.4
	Average	**600**	**17.5**	**206**	**3780**	**18.7 (2)**
	%RPD Sample/Sample Dup	**1.2**	**3.4**	**1.5**	**3.4**	**8.0**
Zone: Source area - Building 16						
ISO17-14-03-044	K3; Sample	89.0	850	83900	4610	14.8
ISO17-14-03-044-DUP	K3; Sample Dup	87.1	862	84800	4760	15.1
	Average	**88.1**	**856**	**84400**	**4690**	**15.0 (2)**
	%RPD Sample/Sample Dup	**2.2**	**1.4**	**1.1**	**3.2**	**2.0**
ISO17-14-03-045	P27; Sample	150	571	19.4	1690	64.7
ISO17-14-03-045-DUP	P27; Sample Dup	130	549	18.9	1670	62.8
	Average	**140**	**560**	**19.2**	**1680**	**63.8 (2)**
	%RPD Sample/Sample Dup	**14**	**3.9**	**2.6**	**1.2**	**3.0**
ISO17-14-03-046	P21B; Sample	10300	6440	11000	72600	4.38
ISO17-14-03-046-DUP	P21B; Sample Dup	10800	7040	11500	69200	4.53
	Average	**10600**	**6740**	**11300**	**70900**	**4.46 (2)**
	%RPD Sample/Sample Dup	**4.7**	**8.9**	**4.4**	**4.8**	**3.4**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical data uncertainties associated with the reported results are as follows: PFOA ± 14%, PFBS ± 25%, PFHS ± 20%, PFOS ± 28%, and PFOSA ± 31%.

(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 14%, PFBS ± 13%, PFHS ± 20%, PFOS ± 17%, and PFOSA ± 17%.

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

(4) The data uncertainty for PFOA has been expanded for location D1 to ± 31% based on surrogate recovery. See section 4 for details.

(5) The data uncertainty for PFOSA has been expanded for location Collector put to ± 36% based on 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**
Zone: Source area - Building 16						
ISO17-14-03-047	P304; Sample	471	277	135	1170	28.1
ISO17-14-03-047-DUP	P304; Sample Dup	481	280	138	1190	26.5
	Average	**476**	**279**	**137**	**1180**	**27.3 (2)**
	%RPD Sample/Sample Dup	**2.1**	**1.1**	**2.2**	**1.7**	**5.9**
ISO17-14-03-048	P305; Sample	239	155	320	678	9.82
ISO17-14-03-048-DUP	P305; Sample Dup	238	157	322	583	10.9
	Average	**239**	**156**	**321**	**631**	**10.4 (2)**
	%RPD Sample/Sample Dup	**0.42**	**1.3**	**0.62**	**15**	**10**
ISO17-14-03-049	P42; Sample	284	51.3	501	3790	17.1
ISO17-14-03-049-DUP	P42; Sample Dup	274	47.4	477	3560	15.5
	Average	**279**	**49.4**	**489**	**3680**	**16.3 (2)**
	%RPD Sample/Sample Dup	**3.6**	**7.9**	**4.9**	**6.3**	**9.8**
ISO17-14-03-050	P56; Sample	38.6	6.16	15.9	351	30.4
ISO17-14-03-050-DUP	P56; Sample Dup	38.3	7.98	16.5	356	30.1
	Average	**38.5**	**7.07**	**16.2**	**354**	**30.3 (2)**
	%RPD Sample/Sample Dup	**0.78**	**26 (3)**	**3.7**	**1.4**	**0.99**

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 ± 25%, PFHS ± 20%, PFOS ± 28%, and PFOSA ± 31%.
(2) Sample set reported using internal standard calibration. The analytical data uncertainty associated with the reported results are as follows: PFOA ± 14%, PFBS ± 13%, PFHS ± 20%, PFOS ± 17%, and PFOSA ± 17%.
(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
(4) The data uncertainty for PFOA has been expanded for location D1 to ± 31% based on surrogate recovery. See section 4 for details.
(5) The data uncertainty for PFOSA has been expanded for location Collector put to ± 36% based on field matrix spike recovery. See section 4 for details.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis".

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected between June 28, 2017 and July 3, 2017 in Nalgene™ (high-density polyethylene) bottles prepared at the 3M EHS Laboratory. Prior to sample collection, bottles designated for field matrix spikes were spiked in the laboratory with a known volume of an appropriate matrix spiking solution containing the analytes of interest. All sample locations were spiked with a mass-labeled internal standard mix at a nominal concentration of 1 ng/mL and select samples were fortified with surrogate recovery standards (SRSs) at a nominal concentration of 0.1 ng/mL prior to sample collection.

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 10 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 D2, P121, 3M vijver, Blokkersdijkvijver Noord, Blokkersdijkvijver standard, L21, Effluent WWTP, and the Trip Blank and Trip Blank FMS Low were reanalyzed for PFOA, PFHS, and PFOS by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples along with the calibration standards and method blanks.

All samples were prepared for PFOSA by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). 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. 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-12-010.2. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

The following analytical runs were used to report the results herein:

***7/12/17 (ETS Kirk) External Standard Calibration Analysis*:**

- BD24-3, BD24-4, D09, D10, D11, D14, D16, D17, D18, D5, ND7, P118A, P118B, P119A, P119B, P321, L22, L31, L4, P114bis, P115, P116, PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09, and PP10 (PFOA, [$^{13}C_4$]-PFOA)

***7/27/17 (ETS Kirk) External Standard Calibration Analysis*:**
- BD24-3, BD24-4, D09, D10, D11, D14, D16, D17, D18, D5, ND7, P118A, P118B, P119A, P119B, P321, L22, L31, L4, P114bis, P115, P116, and PP02 (PFHS, PFOS, [$^{13}C_4$]-PFOS)
- PP01, PP04, PP05, PP06, PP07, PP08, PP09, and PP10 (PFBS, PFHS, PFOS, [$^{13}C_4$]-PFOS)

8/2/17 (ETS Kirk) External Standard Calibration Analysis:
- 12, 13, 5, Bemalingsstation, Collector put, L19, P118C, P119C, P264, P340, P341, P343, P371, P374, P379, P380, P381, P382, B3-bis, B7, P372, P378, PA109A, PA111A, and PA112 (PFOA, PFHS, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- P304, P305, P42, P42, P56, and Trip Blank FMS High (PFOA, PFBS, PFHS, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- M4 and P263 (PFOA, PFHS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- P262 (PFHS, [$^{13}C_4$]-PFOS)
- K3, P27 (PFOA, PFBS, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- P21B (PFOA, PFBS, PFHS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)

***8/3/17 (ETS Rita) Internal Standard Calibration Analysis*:**
- BD24-3, BD24-4, D09, D10, D11, D16, D17, D18, D2, D5, ND7, P118A, P118B, P119A, P119B, P121, P321, Blokkersdijkvijver standard, L21, L4, PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09, and PP10 (PFOSA)

***8/7/17 (ETS Rita) Internal Standard Calibration Analysis*:**
- D14, 3M vijver, Blokkersdijkvijver Noord, L22, L31, P114bis, P115, P116, and Effluent WWTP (PFOSA)

***8/8/17 (ETS Rita) External Standard Calibration Analysis*:**
- Collector put, P118C, and P119C (PFOSA)

8/9/17 (ETS Kirk) Internal Standard Calibration Analysis:
- D2, 3M vijver, Blokkersdijkvijver Noord, Blokkersdijkvijver standard, L21, and Effluent WWTP (PFOA, PFHS, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- P121 (PFOA, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)
- Trip Blank and Trip Blank FMS Low (PFOA, PFBS, PFHS, PFOS, [$^{13}C_4$]-PFOA, [$^{13}C_4$]-PFOS)

8/10/17 (ETS Kirk) External Standard Calibration Analysis:
- M4 and P263 (PFOS, [$^{13}C_4$]-PFOS)
- P262 (PFOA, [$^{13}C_4$]-PFOA)

***8/14/17 (ETS Rita) Internal Standard Calibration Analysis*:**
- M4 and P380 (PFOSA)

8/15/17 (ETS Kirk) External Standard Calibration Analysis:
- K3 (PFHS, [$^{13}C_4$]-PFOS)
- P21B and P262 (PFOS, [$^{13}C_4$]-PFOS)

8/16/17 (ETS Kirk) Internal Standard Calibration Analysis:
- P121 (PFHS, [$^{13}C_4$]-PFOS)

8/25/17 (ETS DaVinci) External Standard Calibration Analysis:
- PP02 (PFBS, [$^{13}C_4$]-PFOS)
- P27 (PFHS, [$^{13}C_4$]-PFOS)

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk	ETS Rita	ETS DaVinci
Liquid Chromatograph	Agilent 1260	Agilent 1260	Agilent 1260
Analysis Method	ETS-8-044.3	ETS-8-044.3	ETS-8-044.3
Analysis Date	7/12/17, 7/27/17, 8/2/17, 8/9/17, 8/10/17, 8/15/17, 8/16/17	8/3/17, 8/7/17, 8/8/17, 8/14/17	8/25/17
Guard column	Prism RP (2.1 mm X 50 mm), 5 μ	Prism RP (2.1 mm X 50 mm), 5 μ	Prism RP (2.1 mm X 50 mm), 5 μ
Analytical column	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ	Betasil C18 (2.1 mm X 100 mm), 5 μ
Injection Volume	1, 2 or 5 μL	2 or 5 μL	5 μL
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 6500+	AB Sciex Triple Quad 6500
Ion Source	Turbo Spray	Turbo Spray	Turbo Spray
Polarity	Negative	Negative	Negative
Software	Analyst 1.6.3	Analyst 1.6.3	Analyst 1.6.3

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.3 Analysis				
Step Number	Total Time (min)	Flow Rate (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 [(1)]	Mass Transition Q1/Q3
PFOA	413/369	[$^{13}C_8$]-PFOA	421/376
	413/219		
	413/169		
PFBS	299/99	[$^{18}O_2$]-PFBS	303/84
	299/80		
PFHS	399/99	[$^{13}C_3$]-PFHS	402/80
	399/80		
PFOS	499/99	[$^{13}C_8$]-PFOS	507/80
	499/80		
	499/130		
PFOSA	498/78	[$^{13}C_8$]-PFOSA	506/78
[$^{13}C_4$]-PFOA	417/372	[$^{13}C_8$]-PFOA	421/376
[$^{13}C_4$]-PFOS	503/80	[$^{13}C_8$]-PFOS	507/80

The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

(1) Internal standards were not used for the analysis on 7/12/17, 7/27/17, 8/2/17, 8/10/17, 8/15/17, and 8/25/17.

3 Data Analysis

3.1 Calibration

7/12/17, 7/27/17, 8/2/17, 8/10/17, 8/15/17, 8/25/17 Analysis (External Standard Calibration): Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed on 7/12/17, 7/27/17, and 8/2/17, from 0.25 ng/mL to 100 ng/mL (nominal) on 8/10/17, from 0.25 ng/mL to 150 ng/mL (nominal) on 8/15/17, and from 0.02 ng/mL to 5 ng/mL (nominal) on 8/25/17. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL (or 0.25 ng/mL on 8/10/17 and 8/15/17) to 25 ng/mL (or 5.0 ng/mL on 8/25/17) (nominal). A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

8/9/17 and 8/16/17 Analysis (Internal Standard Calibration): Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of 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 and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

8/3/17, 8/7/17, 8/8/17, 8/14/17 Analysis of PFOSA (Internal Standard Calibration): Samples were analyzed for PFOSA against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into laboratory reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 1.0 ng/mL. Prior to analysis, the calibration standards were diluted by removing a 0.4 mL aliquot and diluting it with 0.4 mL of methanol. Calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed on 8/3/17 and 8/8/17. Standards ranged from 0.0125 ng/mL to 12.5 ng/mL (nominal) on 8/7/17 and from 0.5 ng/mL to 100 ng/mL (nominal) on 8/14/17. 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.

8/8/17 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. Prior to analysis, the calibration standards were diluted by removing a 0.4 mL aliquot and diluting it with 0.4 mL of methanol. Calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) (nominal) were analyzed. A quadratic, 1/x weighted, calibration curve of the standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point.

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±25% (100±30% for the lowest curve point) were met for all analytes. The correlation coefficient (r) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes.

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) 7/12/17 External Analysis	LOQ, ng/mL (1) 7/27/17 External Analysis	LOQ, ng/mL (1) 8/2/17 External Analysis	LOQ, ng/mL (2) 8/3/17 Internal Analysis	LOQ, ng/mL (2) 8/7/17 Internal Analysis
PFOA	0.0479	NA	0.0479	NA	NA
PFBS	NA	0.0200	0.100	NA	NA
PFHS	NA	0.0200	0.0500	NA	NA
PFOS	NA	0.0185	0.0927	NA	NA
PFOSA	NA	NA	NA	0.0990	0.0248
Analyte	**LOQ, ng/mL (2) 8/8/17 Internal Analysis**	**LOQ, ng/mL (2) 8/8/17 External Analysis**	**LOQ, ng/mL (2) 8/9/17 Internal Analysis**	**LOQ, ng/mL (1) 8/10/17 External Analysis**	**LOQ, ng/ mL (3) 8/14/17 Internal Analysis**
PFOA	NA	NA	0.0240	0.240	NA
PFBS	NA	NA	0.0250	0.250	NA
PFHS	NA	NA	0.0250	0.250	NA
PFOS	NA	NA	0.0232	0.232	NA
PFOSA	0.0500	0.500	NA	NA	2.48
Analyte	**LOQ, ng/ mL (1) 8/15/17 External Analysis**	**LOQ, ng/mL (2) 8/16/17 Internal Analysis**	**LOQ, ng/mL (1) 8/25/17 External Analysis**		
PFOA	NA	NA	NA		
PFBS	NA	NA	0.0200		
PFHS	0.250	0.0500	0.0200		
PFOS	0.232	NA	NA		
PFOSA	NA	NA	NA		

NA = Not Applicable
(1) A dilution factor was not applied to the LOQ.
(2) A dilution factor of 2 was applied to the LOQ.
(3) 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:

- 8/2/17: Low-level LCSs had an average recovery of 125% for PFBS.
- 8/8/17 External: Low-level LCSs were spiked below the resulting LLOQ for PFOSA (0.5 ng/mL).
- 8/10/17: High-level LCSs were spiked above the resulting ULOQ for PFOA, PFBS, PFHS, and PFOS (50,000 ng/mL with dilution factor applied). Sample results reported from this run were bracketed by the low and mid-level LCSs which met method acceptance criteria, and the data are reported.
- 8/15/17: Mid-level LCSs had an average recovery of 72.4% for PFOS. High-level LCSs had an average recovery of 76.8% for PFOS.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report. The following calculations were used to generate data in Table 7.

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

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

Table 7. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 7/12/17	PFOA (Linear + Branched)			[$^{13}C_4$]-PFOA		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-170712-1	100	100	100	99.8	113	114
LCS-170712-2	100	99.8	99.8	99.8	120	121
LCS-170712-3	100	107	107	99.8	117	117
Average ± %RSD	**102% ± 4.0%**			**117% ± 3.0%**		
LCS-170712-4	5000	5040	101	998	1170	117
LCS-170712-5	5000	4960	99.1	998	1170	118
LCS-170712-6	5000	5060	101	998	1150	116
Average ± %RSD	**100% ± 1.1%**			**117% ± 0.85%**		
LCS-170712-7	35000	33000	94.2			
LCS-170712-8	35000	32800	93.8			
LCS-170712-9	35000	33000	94.3			
Average ± %RSD	**94.1% ± 0.28%**					

ETS-8-044.3 External Calibration Analyzed 7/27/17	PFBS			PFHS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-170712-1	100	107	107	100	101	101
LCS-170712-2	100	98.9	98.9	100	96.9	96.9
LCS-170712-3	100	108	108	100	106	106
Average ± %RSD	**105% ± 4.8%**			**101% ± 4.5%**		
LCS-170712-4	5000	5150	103	5000	5170	103
LCS-170712-5	5000	5170	103	5000	5200	104
LCS-170712-6	5000	5230	105	5000	5200	104
Average ± %RSD	**104% ± 1.1%**			**104% ± 0.56%**		
LCS-170712-7	35000	36300	104	35000	36400	104
LCS-170712-8	35000	36200	103	35000	36100	103
LCS-170712-9	35000	35500	101	35000	36300	104
Average ± %RSD	**103% ± 1.5%**			**104% ± 0.56%**		

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 7/27/17	PFOS (Linear + Branched)			[$^{13}C_4$]-PFOS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-170712-1	100	90.9	90.9	95.6	111	116
LCS-170712-2	100	88.9	88.9	95.6	109	114
LCS-170712-3	100	114	114	95.6	110	115
Average ± %RSD	**97.9% ± 14%**			**115% ± 0.87%**		
LCS-170712-4	5000	5050	101	956	1120	117
LCS-170712-5	5000	5130	103	956	1120	117
LCS-170712-6	5000	5120	102	956	1100	115
Average ± %RSD	**102% ± 0.98%**			**116% ± 0.99%**		
LCS-170712-7	35000	38900	111			
LCS-170712-8	35000	39900	114			
LCS-170712-9	35000	39400	112			
Average ± %RSD	**112% ± 1.4%**					

ETS-8-044.3 External Calibration Analyzed 8/2/17	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-170801-1	100	110	110	100	101	101
LCS-170801-2	100	106	106	100	144	144
LCS-170801-3	100	113	113	100	131	131
Average ± %RSD	**110% ± 3.2%**			**125% ± 18% (1)**		
LCS-170801-4	5000	5390	108	5000	5280	106
LCS-170801-5	5000	5220	104	5000	5160	103
LCS-170801-6	NA	NA	NA	NA	NA	NA
Average ± %RPD	**106% ± 3.8%**			**105% ± 2.9%**		
LCS-170801-7	35000	33900	97.0	35000	35900	103
LCS-170801-8	35000	34300	98.1	35000	36300	104
LCS-170801-9	35000	33400	95.3	35000	34900	99.7
Average ± %RSD	**96.8% ± 1.5%**			**102% ± 2.2%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs do not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 8/2/17	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-170801-1	100	106	106	100	102	102
LCS-170801-2	100	115	115	100	91.9	91.9
LCS-170801-3	100	114	114	100	95.9	95.9
Average ± %RSD	**112% ± 4.4%**			**96.6% ± 5.3%**		
LCS-170801-4	5000	5370	107	5000	5340	107
LCS-170801-5	5000	5280	106	5000	5120	102
LCS-170801-6	NA	NA	NA	NA	NA	NA
Average ± %RPD	**107% ± 0.94%**			**105% ± 4.8%**		
LCS-170801-7	35000	35600	102	35000	38600	110
LCS-170801-8	35000	36500	104	35000	38800	111
LCS-170801-9	35000	35200	101	35000	37700	108
Average ± %RSD	**102% ± 1.5%**			**110% ± 1.4%**		

ETS-8-044.3 External Calibration Analyzed 8/2/17	[$^{13}C_4$]-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-170801-1	99.8	118	118	95.6	112	117
LCS-170801-2	99.8	118	119	95.6	109	114
LCS-170801-3	99.8	116	116	95.6	115	120
Average ± %RSD	**118% ± 1.3%**			**117% ± 2.6%**		
LCS-170801-4	998	1180	118	956	1160	122
LCS-170801-5	998	1090	109	956	1060	111
LCS-170801-6	NA	NA	NA	NA	NA	NA
Average ± %RPD	**114% ± 7.9%**			**117% ± 9.4%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs do not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 8/3/17	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-170803-1	NA	NA	NA
LCS-170803-2	0.199	0.237	119
LCS-170803-3	0.199	0.233	117
Average ± %RPD	**118% ± 1.7%**		
LCS-170803-4	19.9	19.0	95.7
LCS-170803-5	19.9	18.8	94.2
LCS-170803-6	19.9	19.1	96.1
Average ± %RSD	**95.3% ± 1.1%**		
LCS-170803-7	139	140	100
LCS-170803-8	139	132	95.0
LCS-170803-9	139	138	99.3
Average ± %RSD	**98.1% ± 2.8%**		

ETS-8-044.3 Internal Calibration Analyzed 8/8/17	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-170807-1	0.199	0.208	105
LCS-170807-2	0.199	0.215	108
LCS-170807-3	0.199	0.211	106
Average ± %RSD	**106% ± 1.4%**		
LCS-170807-4	19.9	20.3	102
LCS-170807-5	19.9	18.8	94.5
LCS-170807-6	19.9	18.7	93.8
Average ± %RSD	**96.8% ± 4.7%**		
LCS-170807-7	139	138	98.9
LCS-170807-8	139	133	96.0
LCS-170807-9	139	134	96.3
Average ± %RSD	**97.1% ± 1.6%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs do not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 8/8/17	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-170807-1	0.199	BLOQ	NA
LCS-170807-2	0.199	BLOQ	NA
LCS-170807-3	0.199	BLOQ	NA
Average ± %RSD	**NA (2)**		
LCS-170807-4	19.9	20.2	102
LCS-170807-5	19.9	20.6	104
LCS-170807-6	19.9	21.4	108
Average ± %RSD	**105% ± 2.9%**		
LCS-170807-7	139	165	119
LCS-170807-8	139	132	95.1
LCS-170807-9	139	139	99.9
Average ± %RSD	**105% ± 12%**		

ETS-8-044.3 Internal Calibration Analyzed 8/9/17	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-170809-1	0.190	0.169	88.9	0.198	0.200	101
LCS-170809-2	0.190	0.185	97.4	0.198	0.191	96.3
LCS-170809-3	0.190	0.189	99.7	0.198	0.199	101
Average ± %RSD	**95.3% ± 6.0%**			**99.4% ± 2.7%**		
LCS-170809-4	1.90	1.81	95.4	1.98	1.86	94.1
LCS-170809-5	1.90	1.79	94.0	1.98	1.91	96.5
LCS-170809-6	1.90	1.79	94.0	1.98	1.83	92.2
Average ± %RSD	**94.5% ± 0.86%**			**94.3% ± 2.3%**		
LCS-170809-7	6.69	6.80	102	6.99	6.75	96.5
LCS-170809-8	6.69	6.41	95.8	6.99	6.70	95.9
LCS-170809-9	6.69	6.57	98.2	6.99	6.68	95.5
Average ± %RSD	**98.7% ± 3.2%**			**96.0% ± 0.52%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs do not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 8/9/17	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-170809-1	0.198	0.205	103	0.184	0.181	98.2
LCS-170809-2	0.198	0.189	95.4	0.184	0.186	101
LCS-170809-3	0.198	0.196	99.2	0.184	0.170	92.3
Average ± %RSD	**99.2% ± 3.8%**			**97.2% ± 4.6%**		
LCS-170809-4	1.98	2.01	102	1.84	1.81	98.2
LCS-170809-5	1.98	1.88	95.2	1.84	1.62	87.9
LCS-170809-6	1.98	1.96	98.9	1.84	1.74	94.7
Average ± %RSD	**98.7% ± 3.4%**			**93.6% ± 5.6%**		
LCS-170809-7	6.99	7.13	102	6.48	6.31	97.4
LCS-170809-8	6.99	6.66	95.3	6.48	6.51	100
LCS-170809-9	6.99	6.83	97.7	6.48	6.40	98.8
Average ± %RSD	**98.3% ± 3.5%**			**98.7% ± 1.3%**		

ETS-8-044.3 Internal Calibration Analyzed 8/9/17	[$^{13}C_4$]-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-170809-1	0.198	0.193	97.7	0.189	0.193	102
LCS-170809-2	0.198	0.207	104	0.189	0.198	105
LCS-170809-3	0.198	0.211	107	0.189	0.189	99.8
Average ± %RSD	**103% ± 4.6%**			**102% ± 2.6%**		
LCS-170809-4	1.98	1.98	99.8	1.89	2.06	109
LCS-170809-5	1.98	2.05	104	1.89	1.88	99.7
LCS-170809-6	1.98	2.03	103	1.89	1.98	105
Average ± %RSD	**102% ± 2.1%**			**105% ± 4.5%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs do not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 8/10/17	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-170810-1	1000	1120	112	1000	1050	105
LCS-170810-2	1000	1090	109	1000	1050	105
LCS-170810-3	1000	1070	107	1000	1010	101
Average ± %RSD	**109% ± 2.3%**			**104% ± 2.2%**		
LCS-170810-4	25000	26800	107	25000	27600	110
LCS-170810-5	25000	25700	103	25000	27300	109
LCS-170810-6	25000	25600	102	25000	28200	113
Average ± %RSD	**104% ± 2.5%**			**111% ± 1.9%**		
LCS-170810-7	70000	>ULOQ	NA	70000	>ULOQ	NA
LCS-170810-8	70000	>ULOQ	NA	70000	>ULOQ	NA
LCS-170810-9	70000	>ULOQ	NA	70000	>ULOQ	NA
Average ± %RSD	**NA (2)**			**NA (2)**		

ETS-8-044.3 External Calibration Analyzed 8/10/17	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-170810-1	1000	1090	109	1000	1080	108
LCS-170810-2	1000	1080	108	1000	1040	104
LCS-170810-3	1000	1080	108	1000	1030	103
Average ± %RSD	**108% ± 0.53%**			**105% ± 2.5%**		
LCS-170810-4	25000	27300	109	25000	29600	118
LCS-170810-5	25000	26900	108	25000	28300	113
LCS-170810-6	25000	27300	109	25000	29500	118
Average ± %RSD	**109% ± 0.53%**			**116% ± 2.5%**		
LCS-170810-7	70000	>ULOQ	NA	70000	>ULOQ	NA
LCS-170810-8	70000	>ULOQ	NA	70000	>ULOQ	NA
LCS-170810-9	70000	>ULOQ	NA	70000	>ULOQ	NA
Average ± %RSD	**NA (2)**			**NA (2)**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs do not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 8/10/17	[$^{13}C_4$]-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-170810-1	499	572	115	478	542	113
LCS-170810-2	499	549	110	478	527	110
LCS-170810-3	499	544	109	478	538	113
Average ± %RSD	**111% ± 2.9%**			**112% ± 1.5%**		
LCS-170810-4	4990	5470	110	4780	5400	113
LCS-170810-5	4990	5460	110	4780	5240	110
LCS-170810-6	4990	5580	112	4780	5560	116
Average ± %RSD	**111% ± 1.0%**			**113% ± 2.7%**		

ETS-8-044.3 Internal Calibration Analyzed 8/14/17	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-170814-1	19.9	20.5	103
LCS-170814-2	19.9	18.4	92.7
LCS-170814-3	19.9	19.8	99.3
Average ± %RPD	**98.3% ± 5.3%**		
LCS-170814-4	99.8	102	102
LCS-170814-5	99.8	102	102
LCS-170814-6	99.8	105	105
Average ± %RSD	**103% ± 1.7%**		
LCS-170814-7	347	353	102
LCS-170814-8	347	345	99.5
LCS-170814-9	347	344	99.2
Average ± %RSD	**100% ± 1.5%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs do not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 8/15/17	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-170815-1	1000	1100	110	1000	1110	111
LCS-170815-2	1000	1090	109	1000	1080	108
LCS-170815-3	1000	1120	112	1000	1160	116
Average ± %RSD	**110% ± 1.4%**			**112% ± 3.6%**		
LCS-170815-4	25000	27700	111	25000	18200	72.9
LCS-170815-5	25000	27400	110	25000	17800	71.3
LCS-170815-6	25000	27500	110	25000	18300	73.1
Average ± %RSD	**110% ± 0.52%**			**72.4% ± 1.4% (1)**		
LCS-170815-7	100000	115000	115	100000	78800	78.8
LCS-170815-8	100000	112000	112	100000	75900	75.9
LCS-170815-9	100000	112000	112	100000	75700	75.7
Average ± %RSD	**113% ± 1.5%**			**76.8% ± 2.3% (1)**		

ETS-8-044.3 External Calibration Analyzed 8/15/17	[$^{13}C_4$]-PFOS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-170815-1	478	565	118
LCS-170815-2	478	534	112
LCS-170815-3	478	552	116
Average ± %RSD	**115% ± 2.6%**		
LCS-170815-4	4780	5670	119
LCS-170815-5	4780	5530	116
LCS-170815-6	4780	5480	115
Average ± %RSD	**117% ± 1.8%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs do not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 8/16/17	PFHS			$[^{13}C_4]$-PFOS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-170816-1	0.198	0.244	123	0.189	0.191	101
LCS-170816-2	0.198	0.234	118	0.189	0.191	101
LCS-170816-3	0.198	0.209	106	0.189	0.196	103
Average ± %RSD	**116% ± 7.6%**			**102% ± 1.1%**		
LCS-170816-4	1.98	2.08	105	1.89	2.10	111
LCS-170816-5	1.98	1.97	99.4	1.89	1.95	103
LCS-170816-6	1.98	2.15	109	1.89	1.95	103
Average ± %RSD	**104% ± 4.6%**			**106% ± 4.4%**		
LCS-170816-7	6.99	7.03	101			
LCS-170816-8	6.99	6.80	97.3			
LCS-170816-9	6.99	6.83	97.7			
Average ± %RSD	**98.7% ± 2.1%**					

ETS-8-044.3 External Calibration Analyzed 8/25/17	PFBS			PFHS		
Lab ID	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery	Spiked Concentration (ng/mL)	Calculated Concentration (ng/mL)	%Recovery
LCS-170825-1	20.0	21.0	105	20.0	19.0	94.9
LCS-170825-2	20.0	19.6	98.0	20.0	19.2	96.0
LCS-170825-3	20.0	20.2	101	20.0	19.2	95.8
Average ± %RSD	**101% ± 3.5%**			**95.6% ± 0.61%**		
LCS-170825-4	200	199	99.3	200	200	100
LCS-170825-5	200	210	105	200	211	105
LCS-170825-6	200	209	104	200	213	106
Average ± %RSD	**103% ± 3.0%**			**104% ± 3.1%**		
LCS-170825-7	349	343	98.3	349	355	102
LCS-170825-8	349	342	97.9	349	336	96.2
LCS-170825-9	349	349	100	349	357	102
Average ± %RSD	**98.7% ± 1.1%**			**100% ± 3.3%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs do not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 8/25/17	[$^{13}C_4$]-PFOS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-170825-1	19.1	22.4	117
LCS-170825-2	19.1	23.1	121
LCS-170825-3	19.1	21.5	112
Average ± %RSD	**117% ± 3.9%**		
LCS-170825-4	191	229	120
LCS-170825-5	191	232	122
LCS-170825-6	191	211	111
Average ± %RSD	**118% ± 5.0%**		

NA = Not Applicable
BLOQ = Below Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs do not meet acceptance criteria of 100 ± 20%.
(2) LCSs spiked outside the calibration range.

3.7 Analytical Data Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.4. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

When determining the analytical data uncertainty assigned to the sample results in Table 1, in addition to the analytical method uncertainty, the batch LCS samples prepared with the projects samples and field QC data are also reviewed. The analytical data uncertainty is listed in Table 8 below.

- The data uncertainty for PFBS using external calibration was calculated at ±16% following ETS-12-012.4; however, the method uncertainty was expanded to ±25% based on the recovery of the low-level LCSs analyzed on 8/2/17.
- The data uncertainty for PFOS using external calibration was calculated at ±24% following ETS-12-012.4; however, the method uncertainty was expanded to ±28% based on the recovery of the high-level LCSs analyzed on 8/15/17.

Table 8. Analytical Data Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Analytical Data Uncertainty
PFOA	External	6.95	±14%
PFBS	External	NA	±25%
PFHS	External	9.78	±20%
PFOS	External	NA	±28%
PFOSA	External	15.3	±31%
PFOA	Internal	6.90	±14%
PFBS	Internal	6.36	±13%
PFHS	Internal	10.2	±20%
PFOS	Internal	8.59	±17%
PFOSA	Internal	8.44	±17%

NA = Not Applicable

3.8 Field Matrix Spikes (FMS)

Target analyte field matrix spike samples were collected for select sampling points to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions 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. 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

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
BD24-4, 3M viver, P116, Effluent WWTP, and B7	FMS	5.00	5.00	5.00	5.00	5.00
D18	FMS	10.0	10.0	10.0	10.0	10.0
Collector Put	FMS	20.0	20.0	20.0	20.0	20.0
P27, P305, L19, M4, P341, P380 and PA112	FMS	100	100	100	100	100
12	FMS	102	102	102	102	2.00
P119B (1)	FMS	457	8.97	457	457	8.97
PP02 and PP10	FMS	500	50.0	500	500	50.0
Trip Blank	Low	2.50	2.50	2.50	2.50	2.50
	High	500	50.0	500	500	50.0

(1) Sample container for the FMS sample was overfilled by more than 10%. The FMS true values were adjusted accordingly.

4 Data Summary and Discussion

The tables below summarize the sample results and field matrix spike recoveries for sampling locations as well as the Trip Blank. Each table provides the average concentration and the relative percent difference (%RPD) of the sample and sample duplicate. Results and average values are rounded to three significant figures. Percent relative difference (%RPD) values are rounded to two significant figures. Because of rounding, values vary slightly from those listed in the raw data. Field matrix spikes meeting the method acceptance criteria of ±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.

D17, D18, P321, PP05: Surrogate recovery standard [$^{13}C_4$]-PFOA analyzed on 7/12/17 had one sample within each sample set with recoveries greater than 130%. However, the average surrogate recovery for the D18, P321, and PP05 sample sets was within the acceptance criteria of 100 ± 30%. Therefore, the results are reported and the analytical uncertainty was not expanded. For D17, the average surrogate recovery was 131%; therefore, the analytical uncertainty was expanded to ±31% for PFOA.

BD24-4, P119B, P321: Surrogate recovery standard [$^{13}C_4$]-PFOS analyzed on 7/27/17 had one sample within each sample set with recoveries greater than 130%. However, the average surrogate recovery for all sample sets was within the acceptance criteria of 100 ± 30%. Therefore, the results are reported and the analytical uncertainty was not expanded.

PP02: The FMS sample analyzed on 8/25/17 had a [$^{13}C_4$]-PFOS surrogate recovery of 69.1%. However, the average surrogate recovery for the sample set was within the acceptance criteria of 100 ± 30%. Therefore, the results are reported and the analytical uncertainty was not expanded.

Collector Put: The field matrix spike had a recovery of 136% for PFOSA. The analytical uncertainty was expanded to ±36% for PFOSA.

PP02 and PP07: The initial analysis of sample sets PP02 and PP07 on 8/3/17 for PFOSA produced sample/sample duplicate RPD value of 56% for PP02 and 41% for location PP07. The sample sets were re-analyzed on 8/8/17, confirming the high RPD values. The results from the initial analysis of the samples is reported.

Table 10. Location ID: BD24-4

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-002	BD24-4; Sample	12.8	NA	5.85	NA
ISO17-14-03-002-DUP	BD24-4; Sample Duplicate	12.5	NA	5.38	NA
ISO17-14-03-002-FMS	BD24-4; FMS	17.0	87.0	10.3	93.7
Average Concentration (ng/mL) ± %RPD		**12.7 ng/mL ± 2.4%**		**5.62 ng/mL ± 8.4%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-002	BD24-4; Sample	6.33	NA	0.470	NA
ISO17-14-03-002-DUP	BD24-4; Sample Duplicate	5.46	NA	0.458	NA
ISO17-14-03-002-FMS	BD24-4; FMS	10.2	86.1	5.81	107 (1)
Average Concentration (ng/mL) ± %RPD		**5.90 ng/mL ± 15%**		**0.464 ng/mL ± 2.6%**	

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

Table 11. Location ID: D18

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-009	D18; Sample	208	NA	109	NA
ISO17-14-03-009-DUP	D18; Sample Duplicate	207	NA	108	NA
ISO17-14-03-009-FMS	D18; FMS	215	NC	120	NC
Average Concentration (ng/mL) ± %RPD		**208 ng/mL ± 0.48%**		**109 ng/mL ± 0.92%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-009	D18; Sample	53.5	NA	5.85	NA
ISO17-14-03-009-DUP	D18; Sample Duplicate	50.9	NA	5.81	NA
ISO17-14-03-009-FMS	D18; FMS	58.2	NC	17.1	113
Average Concentration (ng/mL) ± %RPD		**52.2 ng/mL ± 5.0%**		**5.83 ng/mL ± 0.69%**	

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

Table 12. Location ID: P119B

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-016	P119B; Sample	550	NA	222	NA
ISO17-14-03-016-DUP	P119B; Sample Duplicate	550	NA	225	NA
ISO17-14-03-016-FMS	P119B; FMS	1050	109	715	108
Average Concentration (ng/mL) ± %RPD		**550 ng/mL ± 0.0%**		**224 ng/mL ± 1.3%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-016	P119B; Sample	139	NA	1.14	NA
ISO17-14-03-016-DUP	P119B; Sample Duplicate	116	NA	1.14	NA
ISO17-14-03-016-FMS	P119B; FMS	591	101	11.0	110
Average Concentration (ng/mL) ± %RPD		**128 ng/mL ± 18%**		**1.14 ng/mL ± 0.0%**	

NA = Not Applicable

Table 13. Location ID: 3M vijver

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-019	3M vijver; Sample	2.00	NA	0.928	NA
ISO17-14-03-019-DUP	3M vijver; Sample Duplicate	2.05	NA	0.949	NA
ISO17-14-03-019-FMS	3M vijver; FMS	7.51	110	6.06	102
Average Concentration (ng/mL) ± %RPD		**2.03 ng/mL ± 2.5%**		**0.939 ng/mL ± 2.2%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-019	3M vijver; Sample	5.90	NA	0.0793	NA
ISO17-14-03-019-DUP	3M vijver; Sample Duplicate	5.73	NA	0.0681	NA
ISO17-14-03-019-FMS	3M vijver; FMS	11.8	120	5.48	108 (1)
Average Concentration (ng/mL) ± %RPD		**5.82 ng/mL ± 2.9%**		**0.0737 ng/mL ± 15%**	

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

Table 14. Location ID: P116

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-03-028	P116; Sample	1.39	NA	0.654	NA
ISO17-14-03-028-DUP	P116; Sample Duplicate	1.52	NA	0.620	NA
ISO17-14-03-028-FMS	P116; FMS	6.43	99.5	5.64	100
Average Concentration (ng/mL) ± %RPD		**1.46 ng/mL ± 8.9%**		**0.637 ng/mL ± 5.3%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-03-028	P116; Sample	8.76	NA	0.139	NA
ISO17-14-03-028-DUP	P116; Sample Duplicate	9.39	NA	0.106	NA
ISO17-14-03-028-FMS	P116; FMS	14.0	98.5	5.45	107 (1)
Average Concentration (ng/mL) ± %RPD		**9.08 ng/mL ± 6.9%**		**0.123 ng/mL ± 27% (2)**	

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

Table 15. Location ID: Effluent WWTP

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-03-029	Effluent WWTP; Sample	2.27	NA	2.79	NA
ISO17-14-03-029-DUP	Effluent WWTP; Sample Duplicate	2.34	NA	3.01	NA
ISO17-14-03-029-FMS	Effluent WWTP; FMS	7.53	105	8.39	110
Average Concentration (ng/mL) ± %RPD		**2.31 ng/mL ± 3.0%**		**2.90 ng/mL ± 7.6%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-03-029	Effluent WWTP; Sample	0.890	NA	<0.0248	NA
ISO17-14-03-029-DUP	Effluent WWTP; Sample Duplicate	0.930	NA	0.0276	NA
ISO17-14-03-029-FMS	Effluent WWTP; FMS	5.93	100	5.54	110 (1)
Average Concentration (ng/mL) ± %RPD		**0.910 ng/mL ± 4.4%**		**0.0276 ng/mL**	

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

Table 16. Location ID: PP02

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-031	PP02; Sample	504	NA	32.3	NA	3270	NA
ISO17-14-03-031-DUP	PP02; Sample Dup	510	NA	32.6	NA	3290	NA
ISO17-14-03-031-FMS	PP02; FMS	1040	107	84.5	104	3720	NC
Average Concentration (ng/mL) ± %RPD		**507 ng/mL ± 1.2%**		**32.5 ng/mL ± 0.92%**		**3280 ng/mL ± 0.61%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-031	PP02; Sample	25000	NA	31.9	NA
ISO17-14-03-031-DUP	PP02; Sample Dup	25600	NA	56.7	NA
ISO17-14-03-031-FMS	PP02; FMS	25300	NC	85.8	83.0
Average Concentration (ng/mL) ± %RPD		**25300 ng/mL ± 2.4%**		**44.3 ng/mL ± 56% (1)**	

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

Table 17. Location ID: PP10

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-038	PP10; Sample	603	NA	17.2	NA	204	NA
ISO17-14-03-038-DUP	PP10; Sample Dup	596	NA	17.8	NA	207	NA
ISO17-14-03-038-FMS	PP10; FMS	1110	102	70.2	105	735	106
Average Concentration (ng/mL) ± %RPD		**600 ng/mL ± 1.2%**		**17.5 ng/mL ± 3.4%**		**206 ng/mL ± 1.5%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-038	PP10; Sample	3710	NA	17.9	NA
ISO17-14-03-038-DUP	PP10; Sample Dup	3840	NA	19.4	NA
ISO17-14-03-038-FMS	PP10; FMS	4320	NC	69.9	103
Average Concentration (ng/mL) ± %RPD		**3780 ng/mL ± 3.4%**		**18.7 ng/mL ± 8.0%**	

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

Table 18. Location ID: 12

3M LIMS ID	Sample Description	PFOA Concentration (ng/mL)	PFOA %Recovery	PFHS Concentration (ng/mL)	PFHS %Recovery
ISO17-14-03-039	12; Sample	108	NA	26.3	NA
ISO17-14-03-039-DUP	12; Sample Duplicate	107	NA	26.7	NA
ISO17-14-03-039-FMS	12; FMS	204	94.6	124	95.6
Average Concentration (ng/mL) ± %RPD		108 ng/mL ± 0.93%		26.5 ng/mL ± 1.5%	

3M LIMS ID	Sample Description	PFOS Concentration (ng/mL)	PFOS %Recovery	PFOSA Concentration (ng/mL)	PFOSA %Recovery
ISO17-14-03-039	12; Sample	365	NA	0.355	NA
ISO17-14-03-039-DUP	12; Sample Duplicate	369	NA	0.335	NA
ISO17-14-03-039-FMS	12; FMS	460	91.2	2.60	113
Average Concentration (ng/mL) ± %RPD		367 ng/mL ± 1.1%		0.345 ng/mL ± 5.8%	

NA = Not Applicable

Table 19. Location ID: Collector put

3M LIMS ID	Sample Description	PFOA Concentration (ng/mL)	PFOA %Recovery	PFHS Concentration (ng/mL)	PFHS %Recovery
ISO17-14-03-043	Collector put; Sample	107	NA	37.9	NA
ISO17-14-03-043-DUP	Collector put; Sample Duplicate	107	NA	38.6	NA
ISO17-14-03-043-FMS	Collector put; FMS	127	NC	56.9	93.3
Average Concentration (ng/mL) ± %RPD		107 ng/mL ± 0.0%		38.3 ng/mL ± 1.8%	

3M LIMS ID	Sample Description	PFOS Concentration (ng/mL)	PFOS %Recovery	PFOSA Concentration (ng/mL)	PFOSA %Recovery
ISO17-14-03-043	Collector put; Sample	370	NA	24.6	NA
ISO17-14-03-043-DUP	Collector put; Sample Duplicate	370	NA	25.2	NA
ISO17-14-03-043-FMS	Collector put; FMS	393	NC	52.0	136 (1)
Average Concentration (ng/mL) ± %RPD		370 ng/mL ± 0.0%		24.9 ng/mL ± 2.4% (2)	

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) The data uncertainty was expanded for PFOSA to ±36%.

Table 20. Location ID: P27

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-045	P27; Sample	150	NA	571	NA	19.4	NA
ISO17-14-03-045-DUP	P27; Sample Dup	130	NA	549	NA	18.9	NA
ISO17-14-03-045-FMS	P27; FMS	233	93.0	628	NC	113	93.9
Average Concentration (ng/mL) ± %RPD		**140 ng/mL ± 14%**		**560 ng/mL ± 3.9%**		**19.2 ng/mL ± 2.6%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-045	P27; Sample	1690	NA	64.7	NA
ISO17-14-03-045-DUP	P27; Sample Dup	1670	NA	62.8	NA
ISO17-14-03-045-FMS	P27; FMS	1660	NC	169	105
Average Concentration (ng/mL) ± %RPD		**1680 ng/mL ± 1.2%**		**63.8 ng/mL ± 3.0%**	

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

Table 21. Location ID: P305

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-048	P305; Sample	239	NA	155	NA	320	NA
ISO17-14-03-048-DUP	P305; Sample Dup	238	NA	157	NA	322	NA
ISO17-14-03-048-FMS	P305; FMS	335	NC	241	85.0	414	NC
Average Concentration (ng/mL) ± %RPD		**239 ng/mL ± 0.42%**		**156 ng/mL ± 1.3%**		**321 ng/mL ± 0.62%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-048	P305; Sample	678	NA	9.82	NA
ISO17-14-03-048-DUP	P305; Sample Dup	583	NA	10.9	NA
ISO17-14-03-048-FMS	P305; FMS	685	NC	118	108
Average Concentration (ng/mL) ± %RPD		**631 ng/mL ± 15%**		**10.4 ng/mL ± 10%**	

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

Table 22. Location ID: L19

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-03-051	L19; Sample	191	NA	90.7	NA
ISO17-14-03-051-DUP	L19; Sample Duplicate	187	NA	86.5	NA
ISO17-14-03-051-FMS	L19; FMS	278	89.0	181	92.4
Average Concentration (ng/mL) ± %RPD		**189 ng/mL ± 2.1%**		**88.6 ng/mL ± 4.7%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-03-051	L19; Sample	4420	NA	50.7	NA
ISO17-14-03-051-DUP	L19; Sample Duplicate	4010	NA	41.3	NA
ISO17-14-03-051-FMS	L19; FMS	4170	NC	143	97.0
Average Concentration (ng/mL) ± %RPD		**4220 ng/mL ± 9.7%**		**46.0 ng/mL ± 20%**	

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

Table 23. Location ID: M4

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-03-052	M4; Sample	4360	NA	460	NA
ISO17-14-03-052-DUP	M4; Sample Duplicate	4300	NA	452	NA
ISO17-14-03-052-FMS	M4; FMS	4680	NC	573	NC
Average Concentration (ng/mL) ± %RPD		**4330 ng/mL ± 1.4%**		**456 ng/mL ± 1.8%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO17-14-03-052	M4; Sample	11600	NA	212	NA
ISO17-14-03-052-DUP	M4; Sample Duplicate	11300	NA	220	NA
ISO17-14-03-052-FMS	M4; FMS	NA (1)	NA (1)	268	NC
Average Concentration (ng/mL) ± %RPD		**11500 ng/mL ± 2.6%**		**216 ng/mL ± 3.7%**	

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 24. Location ID: P341

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-060	P341; Sample	543	NA	265	NA
ISO17-14-03-060-DUP	P341; Sample Duplicate	551	NA	275	NA
ISO17-14-03-060-FMS	P341; FMS	646	NC	361	NC
Average Concentration (ng/mL) ± %RPD		**547 ng/mL ± 1.5%**		**270 ng/mL ± 3.7%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-060	P341; Sample	5450	NA	94.8	NA
ISO17-14-03-060-DUP	P341; Sample Duplicate	5640	NA	87.7	NA
ISO17-14-03-060-FMS	P341; FMS	5680	NC	166	74.8
Average Concentration (ng/mL) ± %RPD		**5550 ng/mL ± 3.4%**		**91.3 ng/mL ± 7.8%**	

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

Table 25. Location ID: P380

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-065	P380; Sample	119	NA	42.7	NA
ISO17-14-03-065-DUP	P380; Sample Duplicate	122	NA	44.7	NA
ISO17-14-03-065-FMS	P380; FMS	225	105	147	103
Average Concentration (ng/mL) ± %RPD		**121 ng/mL ± 2.5%**		**43.7 ng/mL ± 4.6%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-065	P380; Sample	622	NA	115	NA
ISO17-14-03-065-DUP	P380; Sample Duplicate	644	NA	133	NA
ISO17-14-03-065-FMS	P380; FMS	692	NC	217	93.0
Average Concentration (ng/mL) ± %RPD		**633 ng/mL ± 3.5%**		**124 ng/mL ± 15%**	

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

Table 26. Location ID: B7

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-069	B7; Sample	81.7	NA	11.4	NA
ISO17-14-03-069-DUP	B7; Sample Duplicate	58.3	NA	8.37	NA
ISO17-14-03-069-FMS	B7; FMS	96.0	NC	16.0	122
Average Concentration (ng/mL) ± %RPD		70.0 ng/mL ± 33% (1)		9.89 ng/mL ± 31% (1)	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-069	B7; Sample	285	NA	4.45	NA
ISO17-14-03-069-DUP	B7; Sample Duplicate	254	NA	3.93	NA
ISO17-14-03-069-FMS	B7; FMS	316	NC	10.1	118
Average Concentration (ng/mL) ± %RPD		270 ng/mL ± 12%		4.19 ng/mL ± 12%	

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

Table 27. Location ID: PA112

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-074	PA112; Sample	149	NA	33.8	NA
ISO17-14-03-074-DUP	PA112; Sample Duplicate	151	NA	32.1	NA
ISO17-14-03-074-FMS	PA112; FMS	239	89.0	128	95.1
Average Concentration (ng/mL) ± %RPD		150 ng/mL ± 1.3%		33.0 ng/mL ± 5.2%	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-074	PA112; Sample	357	NA	108	NA
ISO17-14-03-074-DUP	PA112; Sample Duplicate	358	NA	106	NA
ISO17-14-03-074-FMS	PA112; FMS	423	NC	192	85.0
Average Concentration (ng/mL) ± %RPD		358 ng/mL ± 0.28%		107 ng/mL ± 1.9%	

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

Table 28. Trip Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-075	Trip Blank	<0.0240	NA	<0.0250	NA	<0.0250	NA
ISO17-14-03-075-FMS-LOW	Trip Blank FMS Low	2.61	104	2.42	96.8	2.41	96.4
ISO17-14-03-075-FMS-HIGH	Trip Blank FMS High	501	100	55.2	110	499	100

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO17-14-03-075	Trip Blank	<0.0464	NA	<0.0500	NA
ISO17-14-03-075-FMS-LOW	Trip Blank FMS Low	2.32	92.8	2.76	110
ISO17-14-03-075-FMS-HIGH	Trip Blank FMS High	508	102	54.7	109

NA = Not Applicable

Table 29. 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
ISO17-14-03-001	BD24-3; Sample	124	119	NA
ISO17-14-03-001-DUP	BD24-3; Sample Duplicate	128	123	NA
ISO17-14-03-002	BD24-4; Sample	129	131 (2)	NA
ISO17-14-03-002-DUP	BD24-4; Sample Duplicate	126	125	NA
ISO17-14-03-002-FMS	BD24-4; FMS	123	123	NA
ISO17-14-03-003	D09; Sample	123	121	NA
ISO17-14-03-003-DUP	D09; Sample Duplicate	126	123	NA
ISO17-14-03-004	D10; Sample	127	123	NA
ISO17-14-03-004-DUP	D10; Sample Duplicate	124	127	NA
ISO17-14-03-005	D11; Sample	123	126	NA
ISO17-14-03-005-DUP	D11; Sample Duplicate	125	119	NA
ISO17-14-03-006	D14; Sample	122	120	NA
ISO17-14-03-006-DUP	D14; Sample Duplicate	119	121	NA
ISO17-14-03-007	D16; Sample	124	125	NA
ISO17-14-03-007-DUP	D16; Sample Duplicate	123	124	NA
ISO17-14-03-008	D17; Sample	138 (2)	130	NA
ISO17-14-03-008-DUP	D17; Sample Duplicate	123	124	NA
ISO17-14-03-009	D18; Sample	121	121	NA
ISO17-14-03-009-DUP	D18; Sample Duplicate	132 (2)	128	NA
ISO17-14-03-009-FMS	D18; FMS	127	121	NA
ISO17-14-03-010	D2; Sample	95.8 (3)	98.2 (3)	NA
ISO17-14-03-010-DUP	D2; Sample Duplicate	97.1 (3)	104 (3)	NA
ISO17-14-03-011	D5; Sample	128	128	NA
ISO17-14-03-011-DUP	D5; Sample Duplicate	121	120	NA
ISO17-14-03-012	ND7; Sample	120	119	NA
ISO17-14-03-012-DUP	ND7; Sample Duplicate	127	123	NA
ISO17-14-03-013	P118A; Sample	126	121	NA
ISO17-14-03-013-DUP	P118A; Sample Duplicate	123	119	NA
ISO17-14-03-014	P118B; Sample	125	122	NA
ISO17-14-03-014-DUP	P118B; Sample Duplicate	123	120	NA
ISO17-14-03-015	P119A; Sample	124	121	NA
ISO17-14-03-015-DUP	P119A; Sample Duplicate	121	116	NA
ISO17-14-03-016	P119B; Sample	132	132 (2)	NA
ISO17-14-03-016-DUP	P119B; Sample Duplicate	128	123	NA
ISO17-14-03-016-FMS	P119B; FMS	130	124	NA
ISO17-14-03-017	P121; Sample	97.3 (3)	100 (3)	104 (2)
ISO17-14-03-017-DUP	P121; Sample Duplicate	91.8 (3)	97.9 (3)	101 (2)
ISO17-14-03-018	P321; Sample	119	113	NA
ISO17-14-03-018-DUP	P321; Sample Duplicate	139 (2)	132 (2)	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) Surrogate recovery standard did not meet acceptance criteria of 100 ± 30%.
(3) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 29 continued. Surrogate Recovery Standard Results (1)

3M LIMS ID	Sample Description	Percent Recovery (%) [$^{13}C_4$]-PFOA	[$^{13}C_4$]-PFOS	[$^{13}C_4$]-PFOS Re-analysis
ISO17-14-03-019	3M vijver; Sample	93.5 (3)	96.5 (3)	NA
ISO17-14-03-019-DUP	3M vijver; Sample Duplicate	98.0 (3)	93.0 (3)	NA
ISO17-14-03-019-FMS	3M vijver; FMS	108 (3)	99.2 (3)	NA
ISO17-14-03-020	Blokkersdijkvijver-Noord; Sample	104 (3)	97.9 (3)	NA
ISO17-14-03-020-DUP	Blokkersdijkvijver-Noord; Sample Duplicate	91.9 (3)	91.9 (3)	NA
ISO17-14-03-021	Blokkersdijkvijver-standard; Sample	102 (3)	101 (3)	NA
ISO17-14-03-021-DUP	Blokkersdijkvijver-standard; Sample Duplicate	96.4 (3)	102 (3)	NA
ISO17-14-03-022	L21; Sample	89.6 (3)	107 (3)	NA
ISO17-14-03-022-DUP	L21; Sample Duplicate	95.5 (3)	99.9 (3)	NA
ISO17-14-03-023	L22; Sample	125	117	NA
ISO17-14-03-023-DUP	L22; Sample Duplicate	119	118	NA
ISO17-14-03-024	L31; Sample	124	117	NA
ISO17-14-03-024-DUP	L31; Sample Duplicate	124	114	NA
ISO17-14-03-025	L4; Sample	124	118	NA
ISO17-14-03-025-DUP	L4; Sample Duplicate	122	120	NA
ISO17-14-03-026	P114bis; Sample	125	120	NA
ISO17-14-03-026-DUP	P114bis; Sample Duplicate	124	119	NA
ISO17-14-03-027	P115; Sample	129	119	NA
ISO17-14-03-027-DUP	P115; Sample Duplicate	122	121	NA
ISO17-14-03-028	P116; Sample	126	120	NA
ISO17-14-03-028-DUP	P116; Sample Duplicate	118	114	NA
ISO17-14-03-028-FMS	P116; FMS	125	124	NA
ISO17-14-03-029	Effluent WWTP; Sample	96.9 (3)	96.1 (3)	NA
ISO17-14-03-029-DUP	Effluent WWTP; Sample Duplicate	110 (3)	99.4 (3)	NA
ISO17-14-03-029-FMS	Effluent WWTP; FMS	96.4 (3)	94.5 (3)	NA
ISO17-14-03-030	PP01; Sample	127	124	NA
ISO17-14-03-030-DUP	PP01; Sample Duplicate	123	120	NA
ISO17-14-03-031	PP02; Sample	119	117	70.3
ISO17-14-03-031-DUP	PP02; Sample Duplicate	130	124	71.6
ISO17-14-03-031-FMS	PP02; FMS	126	120	69.1 (2)
ISO17-14-03-032	PP04; Sample	122	124	NA
ISO17-14-03-032-DUP	PP04; Sample Duplicate	129	127	NA
ISO17-14-03-033	PP05; Sample	132 (2)	125	NA
ISO17-14-03-033-DUP	PP05; Sample Duplicate	126	124	NA
ISO17-14-03-034	PP06; Sample	127	121	NA
ISO17-14-03-034-DUP	PP06; Sample Duplicate	126	121	NA
ISO17-14-03-035	PP07; Sample	125	120	NA
ISO17-14-03-035-DUP	PP07; Sample Duplicate	127	120	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) Surrogate recovery standard did not meet acceptance criteria of 100 ± 30%.
(3) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 29 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
ISO17-14-03-036	PP08; Sample	126	121	NA
ISO17-14-03-036-DUP	PP08; Sample Duplicate	127	120	NA
ISO17-14-03-037	PP09; Sample	130	123	NA
ISO17-14-03-037-DUP	PP09; Sample Duplicate	126	119	NA
ISO17-14-03-038	PP10; Sample	124	119	NA
ISO17-14-03-038-DUP	PP10; Sample Duplicate	126	121	NA
ISO17-14-03-038-FMS	PP10; FMS	123	121	NA
ISO17-14-03-039	12; Sample	123	124	NA
ISO17-14-03-039-DUP	12; Sample Duplicate	122	115	NA
ISO17-14-03-039-FMS	12; FMS	118	117	NA
ISO17-14-03-040	13; Sample	113	120	NA
ISO17-14-03-040-DUP	13; Sample Duplicate	113	113	NA
ISO17-14-03-041	5; Sample	115	114	NA
ISO17-14-03-041-DUP	5; Sample Duplicate	119	119	NA
ISO17-14-03-042	Bemalingsstation; Sample	119	125	NA
ISO17-14-03-042-DUP	Bemalingsstation; Sample Duplicate	120	119	NA
ISO17-14-03-043	Collector put; Sample	115	114	NA
ISO17-14-03-043-DUP	Collector put; Sample Duplicate	118	112	NA
ISO17-14-03-043-FMS	Collector put; FMS	104	107	NA
ISO17-14-03-044	K3; Sample	120	127	120
ISO17-14-03-044-DUP	K3; Sample Duplicate	114	119	115
ISO17-14-03-045	P27; Sample	122	119	117
ISO17-14-03-045-DUP	P27; Sample Duplicate	118	117	114
ISO17-14-03-045-FMS	P27; FMS	118	118	113
ISO17-14-03-046	P21B; Sample	118	119	108
ISO17-14-03-046-DUP	P21B; Sample Duplicate	104	108	114
ISO17-14-03-047	P304; Sample	116	120	NA
ISO17-14-03-047-DUP	P304; Sample Duplicate	118	117	NA
ISO17-14-03-048	P305; Sample	118	115	NA
ISO17-14-03-048-DUP	P305; Sample Duplicate	117	121	NA
ISO17-14-03-048-FMS	P305; FMS	117	119	NA
ISO17-14-03-049	P42; Sample	119	113	NA
ISO17-14-03-049-DUP	P42; Sample Duplicate	119	114	NA
ISO17-14-03-050	P56; Sample	114	116	NA
ISO17-14-03-050-DUP	P56; Sample Duplicate	119	116	NA
ISO17-14-03-051	L19; Sample	116	115	NA
ISO17-14-03-051-DUP	L19; Sample Duplicate	114	112	NA
ISO17-14-03-051-FMS	L19; FMS	120	117	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) Surrogate recovery standard did not meet acceptance criteria of 100 ± 30%.
(3) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 29 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
ISO17-14-03-052	M4; Sample	111	109	120
ISO17-14-03-052-DUP	M4; Sample Duplicate	114	113	117
ISO17-14-03-052-FMS	M4; FMS	115	115	NA
ISO17-14-03-053	P118C; Sample	123	117	NA
ISO17-14-03-053-DUP	P118C; Sample Duplicate	116	116	NA
ISO17-14-03-054	P119C; Sample	120	124	NA
ISO17-14-03-054-DUP	P119C; Sample Duplicate	114	115	NA
ISO17-14-03-055	P262; Sample	113	91.9	119
ISO17-14-03-055-DUP	P262; Sample Duplicate	117	91.8	117
ISO17-14-03-056	P263; Sample	107	108	116
ISO17-14-03-056-DUP	P263; Sample Duplicate	115	116	120
ISO17-14-03-057	P264; Sample	117	120	NA
ISO17-14-03-057-DUP	P264; Sample Duplicate	116	117	NA
ISO17-14-03-059	P340; Sample	120	120	NA
ISO17-14-03-059-DUP	P340; Sample Duplicate	118	123	NA
ISO17-14-03-060	P341; Sample	121	119	NA
ISO17-14-03-060-DUP	P341; Sample Duplicate	123	124	NA
ISO17-14-03-060-FMS	P341; FMS	120	116	NA
ISO17-14-03-061	P343; Sample	120	118	NA
ISO17-14-03-061-DUP	P343; Sample Duplicate	118	117	NA
ISO17-14-03-062	P371; Sample	118	121	NA
ISO17-14-03-062-DUP	P371; Sample Duplicate	113	117	NA
ISO17-14-03-063	P374; Sample	118	121	NA
ISO17-14-03-063-DUP	P374; Sample Duplicate	116	116	NA
ISO17-14-03-064	P379; Sample	117	121	NA
ISO17-14-03-064-DUP	P379; Sample Duplicate	115	124	NA
ISO17-14-03-065	P380; Sample	115	121	NA
ISO17-14-03-065-DUP	P380; Sample Duplicate	121	120	NA
ISO17-14-03-065-FMS	P380; FMS	119	120	NA
ISO17-14-03-066	P381; Sample	119	119	NA
ISO17-14-03-066-DUP	P381; Sample Duplicate	117	125	NA
ISO17-14-03-067	P382; Sample	113	121	NA
ISO17-14-03-067-DUP	P382; Sample Duplicate	116	118	NA
ISO17-14-03-068	B3-bis; Sample	119	111	NA
ISO17-14-03-068-DUP	B3-bis; Sample Duplicate	117	118	NA
ISO17-14-03-069	B7; Sample	113	111	NA
ISO17-14-03-069-DUP	B7; Sample Duplicate	105	108	NA
ISO17-14-03-069-FMS	B7; FMS	120	121	NA
ISO17-14-03-070	P372; Sample	119	119	NA
ISO17-14-03-070-DUP	P372; Sample Duplicate	123	127	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) Standard recovery standard did not meet acceptance criteria of 100 ± 30%.
(3) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

Table 29 continued. Surrogate Recovery Standard Results [1]

		Percent Recovery (%)	
3M LIMS ID	Sample Description	[$^{13}C_4$]-PFOA	[$^{13}C_4$]-PFOS
ISO17-14-03-071	P378; Sample	117	123
ISO17-14-03-071-DUP	P378; Sample Duplicate	117	121
ISO17-14-03-072	PA109A; Sample	114	120
ISO17-14-03-072-DUP	PA109A; Sample Duplicate	121	119
ISO17-14-03-073	PA111A; Sample	116	125
ISO17-14-03-073-DUP	PA111A; Sample Duplicate	119	124
ISO17-14-03-074	PA112; Sample	121	126
ISO17-14-03-074-DUP	PA112; Sample Duplicate	112	117
ISO17-14-03-074-FMS	PA112; FMS	117	125
ISO17-14-03-075	Trip Blank; Sample	95.7 (3)	94.7 (3)
ISO17-14-03-075-FMS-LOW	Trip Blank; FMS Low	105 (3)	100 (3)
ISO17-14-03-075-FMS-HIGH	Trip Blank; FMS High	115	117

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) Standard recovery standard did not meet acceptance criteria of 100 ± 30%.
(3) The surrogate recovery standards were added to the sample bottle prior to the sampling event.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M EHS Laboratory method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 10-29 of this report.

6 Data / Sample Retention

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

7 Attachment

Attachment A: Chain of Custody Form

8 Signatures

Digitally signed by Chelsie J. Grochow
DN: c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory - authenticated by LRA, [redacted] o=3M, cn=Chelsie J. Grochow
Reason: I am the author of this document
Date: 2017.09.18 14:08:18 -05'00'

Chelsie Grochow, 3M Report Author

Susan T. Wolf
c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory - authenticated by LRA [redacted] o=3M, cn=Susan T. Wolf
I have reviewed this document
2017.09.18 12:07:44 -05'00'

Susan T. Wolf, 3M Principal Analytical Investigator

Digitally signed by William K. Reagen
DN: c=US, st=MN, l=St. Paul, ou=Laboratory Director, ou=3M Environmental Laboratory - authenticated by LRA, [redacted] o=3M, cn=William K. Reagen
Reason: I am approving this document
Date: 2017.09.18 16:13:43 -05'00'

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

The 3M EHS Laboratory's Quality Assurance Unit has audited the data and report for this project.

Digitally signed by Tanya K. Rude
DN: c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory - authenticated by LRA, [redacted] o=3M, cn=Tanya K. Rude
Reason: I agree to the terms defined by the placement of my signature on this document
Date: 2017.09.18 12:24:12 -05'00'

Quality Assurance Representative

This test report shall not be reproduced except in full, without written approval of the 3M EHS Laboratory.

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone: [redacted]
Alt. Pho[redacted]
Fax: (6[redacted]

Project: ISO17-14-03

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 03/15/2017
Project Description: 3M Antwerp Water Sampling for PFCs; June-July 2017 Sampling
Comments:

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [redacted]
Email Address: [redacted]

GW = groundwater

DAY/MONTH/YEAR

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
✓ ISO17-14-03-001	BD24-3; Sample V	3/7/'17	GW	/
✓ ISO17-14-03-001-DUP	BD24-3; Sample Duplicate V	3/7/'17	GW	/
✓ ISO17-14-03-002	BD24-4; Sample V	3/7/'17	GW	/
✓ ISO17-14-03-002-DUP	BD24-4; Sample Duplicate V	3/7/'17	GW	/
✓ ISO17-14-03-002-FMS	BD24-4; FMS V	3/7/'17	GW	/
✓ ISO17-14-03-003	D09; Sample V	28/6/'17	GW	/
✓ ISO17-14-03-003-DUP	D09; Sample Duplicate ✓	28/6/'17	GW	/
✓ ISO17-14-03-004	D10; Sample V	3/7/'17	GW	/
✓ ISO17-14-03-004-DUP	D10; Sample Duplicate V	3/7/'17	GW	/
✓ ISO17-14-03-005	D11; Sample V	28/6/'17	GW	/
✓ ISO17-14-03-005-DUP	D11; Sample Duplicate V	28/6/'17	GW	/
✓ ISO17-14-03-006	D14; Sample V	30/6/'17	GW	/
✓ ISO17-14-03-006-DUP	D14; Sample Duplicate V	30/6/'17	GW	/
✓ ISO17-14-03-007	D16; Sample V	28/6/'17	GW	/

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for**

Temperature: 15 **Deg C** ☐ **Received on Ice** ☐ **Other:**

7/10/17 KPH
☒ ACCEPTABLE
☒ 22C

Collected by (print): Tine Mandonx (TMA) **Collector's signature:**

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
14	TMA	3/7/'16		FEDEX	[signature]	7/10/17	10:00 AM

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone[redacted]
Alt. Ph[redacted]
Fax: ([redacted]

Project: ISO17-14-03 (cont.)

GW = groundwater

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 03/15/2017
Project Description: 3M Antwerp Water Sampling for PFCs; June-July 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number[redacted]
Email Address:[redacted]

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
✓ ISO17-14-03-007-DUP	D16; Sample Duplicate ✓	28/6/'17	GW	/
✓ ISO17-14-03-008	D17; Sample ✓	28/6/'17	GW	/
✓ ISO17-14-03-008-DUP	D17; Sample Duplicate ✓	28/6/'17	GW	/
✓ ISO17-14-03-009	D18; Sample ✓	28/6/'17	GW	/
✓ ISO17-14-03-009-DUP	D18; Sample Duplicate ✓	28/6/'17	GW	/
✓ ISO17-14-03-009-FMS	D18; FMS ✓	28/6/'17	GW	/
✓ ISO17-14-03-010	D2; Sample ✓	3/7/'17	GW	/
✓ ISO17-14-03-010-DUP	D2; Sample Duplicate ✓	3/7/'17	GW	/
✓ ISO17-14-03-011	D5; Sample ✓	29/6/'17	GW	/
✓ ISO17-14-03-011-DUP	D5; Sample Duplicate ✓	29/6/'17	GW	/
✓ ISO17-14-03-012	ND7; Sample ✓	29/6/'17	GW	/
✓ ISO17-14-03-012-DUP	ND7; Sample Duplicate ✓	29/6/'17	GW	/
✓ ISO17-14-03-013	P118A; Sample ✓	29/6/'17	GW	/
✓ ISO17-14-03-013-DUP	P118A; Sample Duplicate ✓	29/6/'17	GW	/
✓ ISO17-14-03-014	P118B; Sample ✓	30/6/'17	GW	/
✓ ISO17-14-03-014-DUP	P118B; Sample Duplicate ✓	30/6/'17	GW	/
✓ ISO17-14-03-015	P119A; Sample ✓	29/6/'17	GW	/

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for** 7/10/17 KPH

Temperature: 15 **Deg C** ☐ **Received on Ice** ☐ **Other:** ☑ ACCEPTABLE ☑ 22C

Collected by (print): Tine Mandonx (TMA) **Collector's signature:** [signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	3/7/'17		FEDEX	[signature]	7/10/17	10:05AM

Page 2 of 11

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phone[redacted]
Alt. Ph
Fax: ([redacted]

Project: ISO17-14-03 (cont.)

GW: groundwater
SW: surface water

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 03/15/2017
Project Description: 3M Antwerp Water Sampling for PFCs; June-July 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number[redacted]
Email Address

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
✓ ISO17-14-03-015-DUP	P119A; Sample Duplicate V	29/6/'17	GW	/
✓ ISO17-14-03-016	P119B; Sample V	29/6/'17	GW	/
✓ ISO17-14-03-016-DUP	P119B; Sample Duplicate V	29/6/'17	GW	/
✓ ISO17-14-03-016-FMS	P119B; FMS V	29/6/'17	GW	/
✓ ISO17-14-03-017	P121; Sample V	3/7/'17	GW	/
✓ ISO17-14-03-017-DUP	P121; Sample Duplicate V	3/7/'17	GW	/
✓ ISO17-14-03-018	P321; Sample V	29/6/'17	GW	/
✓ ISO17-14-03-018-DUP	P321; Sample Duplicate V	29/6/'17	GW	/
✓ ISO17-14-03-019	3M vijver; Sample V	29/6/'17	SW	/
✓ ISO17-14-03-019-DUP	3M vijver; Sample Duplicate V	29/6/'17	SW	/
✓ ISO17-14-03-019-FMS	3M vijver; FMS V	29/6/'17	SW	/
✓ ISO17-14-03-020	Blokkersdijkvijver-Noord; Sample V	29/6/'17	SW	/
✓ ISO17-14-03-020-DUP	Blokkersdijkvijver-Noord; Sample Duplicate V	29/6/'17	SW	/
✓ ISO17-14-03-021	Blokkersdijkvijver-standard; Sample V	29/6/'17	SW	/
✓ ISO17-14-03-021-DUP	Blokkersdijkvijver-standard; Sample Duplicate V	29/6/'17	SW	/
✓ ISO17-14-03-022	L21; Sample V	3/7/'17	GW	/
✓ ISO17-14-03-022-DUP	L21; Sample Duplicate V	3/7/'17	GW	/

Sample Condition Upon Receipt: ☒ Acceptable ☐ All items accounted for

Temperature: 15 Deg C ☐ Received on Ice ☐ Other:

7/10/17 KPH
☑ ACCEPTABLE
☑ 22C

Collected by (print): Tine Mondox (TMA) **Collector's signature:**

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	3/7/17		FEDEX	Kathy H.	7/10/17	10:15AM

Attachment A: Chain of Custody Form

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phon[redacted]
Alt. P[redacted]
Fax: [redacted]

Project: ISO17-14-03 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 03/15/2017
Project Description: 3M Antwerp Water Sampling for PFCs; June-July 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number[redacted]
Email Address[redacted]

GW: groundwater
ww: waste water

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
✓ ISO17-14-03-023	L22; Sample ✓	3/7/'17	GW	✓
✓ ISO17-14-03-023-DUP	L22; Sample Duplicate ✓	3/7/'17	GW	✓
✓ ISO17-14-03-024	L31; Sample ✓	3/7/'17	GW	✓
✓ ISO17-14-03-024-DUP	L31; Sample Duplicate ✓	3/7/'17	GW	✓
✓ ISO17-14-03-025	L4; Sample ✓	3/7/'17	GW	✓
✓ ISO17-14-03-025-DUP	L4; Sample Duplicate ✓	3/7/'17	GW	✓
✓ ISO17-14-03-026	P114bis; Sample ✓	3/7/'17	GW	✓
✓ ISO17-14-03-026-DUP	P114bis; Sample Duplicate ✓	3/7/'17	GW	✓
✓ ISO17-14-03-027	P115; Sample ✓	3/7/'17	GW	✓
✓ ISO17-14-03-027-DUP	P115; Sample Duplicate ✓	3/7/'17	GW	✓
✓ ISO17-14-03-028	P116; Sample ✓	3/7/'17	GW	✓
✓ ISO17-14-03-028-DUP	P116; Sample Duplicate ✓	3/7/'17	GW	✓
✓ ISO17-14-03-028-FMS	P116; FMS ✓	3/7/'17	GW	✓
✓ ISO17-14-03-029	Effluent WWTP; Sample ✓	30/6/'17	~~GW~~	✓ WW
✓ ISO17-14-03-029-DUP	Effluent WWTP; Sample Duplicate ✓	30/6/'17	~~GW~~	✓ WW
✓ ISO17-14-03-029-FMS	Effluent WWTP; FMS ✓	30/6/'17	~~GW~~	✓ WW
✓ ISO17-14-03-030	PP01; Sample ✓	30/6/'17	~~GW~~	✓ GW

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for**

Temperature: 15 **Deg C** ☐ **Received on Ice** ☐ **Other:**

7/10/17 KPH
☑ ACCEPTABLE
☑ 22 C

Collected by (print): Tine Mandonx (TMA) **Collector's signature:** [signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	3/7/'17		FEDEX	[signature]	7/10/17	10:25AM

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phon[redacted]
Alt. P[redacted]
Fax: [redacted]

Project: ISO17-14-03 (cont.)

GW : groundwater

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 03/15/2017
Project Description: 3M Antwerp Water Sampling for PFCs; June-July 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number[redacted]
Email Address:[redacted]

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
✓ ISO17-14-03-030-DUP	PP01; Sample Duplicate ✓	30/6/17		GW /
✓ ISO17-14-03-031	PP02; Sample ✓	30/6/17	GW	/
✓ ISO17-14-03-031-DUP	PP02; Sample Duplicate ✓	30/6/17	GW	/
✓ ISO17-14-03-031-FMS	PP02; FMS ✓	30/6/17	GW	/
✓ ISO17-14-03-032	PP04; Sample ✓	30/6/17	GW	/
✓ ISO17-14-03-032-DUP	PP04; Sample Duplicate ✓	30/6/17	GW	/
✓ ISO17-14-03-033	PP05; Sample ✓	30/6/17	GW	the extra bottle will be filled during the cleaning in Sept. 2017
✓ ISO17-14-03-033-DUP	PP05; Sample Duplicate ✓	30/6/17	GW	/
✓ ISO17-14-03-034	PP06; Sample ✓	30/6/17	GW	/
✓ ISO17-14-03-034-DUP	PP06; Sample Duplicate ✓	30/6/17	GW	/
✓ ISO17-14-03-035	PP07; Sample ✓	30/6/17	GW	/
✓ ISO17-14-03-035-DUP	PP07; Sample Duplicate ✓	30/6/17	GW	/
✓ ISO17-14-03-036	PP08; Sample ✓	30/6/17	GW	/
✓ ISO17-14-03-036-DUP	PP08; Sample Duplicate ✓	30/6/17	GW	/
✓ ISO17-14-03-037	PP09; Sample ✓	30/6/17	GW	/
✓ ISO17-14-03-037-DUP	PP09; Sample Duplicate ✓	30/6/17	GW	/
✓ ISO17-14-03-038	PP10; Sample ✓	30/6/17	GW	/

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for**
Temperature: 15 **Deg C** ☐ **Received on Ice** ☐ **Other:**

7/10/17 KPIL
☑ ACCEPTABLE
☑ 3.2 C

Collected by (print): Tine Mandoux (TMA) **Collector's signature:**

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	3/7/17		FEDEX	Katty Hoge	7/10/17	10:30AM

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phon[redacted]
Alt. P[redacted]
Fax: [redacted]

Project: ISO17-14-03 (cont.)

GW: groundwater
SW: surface water
WW: waste water

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 03/15/2017
Project Description: 3M Antwerp Water Sampling for PFCs; June-July 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number[redacted]
Email Address[redacted]

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
✓ ISO17-14-03-038-DUP	PP10; Sample Duplicate ✓	30/6/'17	GW	✓
✓ ISO17-14-03-038-FMS	PP10; FMS ✓	30/6/'17	GW	✓
✓ ISO17-14-03-039	12; Sample ✓	29 ~~30~~/6/'17	SW	✓
✓ ISO17-14-03-039-DUP	12; Sample Duplicate ✓	29 ~~30~~/6/'17	SW	✓
✓ ISO17-14-03-039-FMS	12; FMS ✓	29 ~~30~~/6/'17	SW	✓
✓ ISO17-14-03-040	13; Sample ✓	29/6/'17	SW	✓
✓ ISO17-14-03-040-DUP	13; Sample Duplicate ✓	29/6/'17	SW	✓
✓ ISO17-14-03-041	5; Sample ✓	29/6/'17	SW	✓
✓ ISO17-14-03-041-DUP	5; Sample Duplicate ✓	29/6/'17	SW	✓
✓ ISO17-14-03-042	Bemalingsstation; Sample ✓	29/6/'17	SW	✓
✓ ISO17-14-03-042-DUP	Bemalingsstation; Sample Duplicate ✓	29/6/'17	SW	✓
✓ ISO17-14-03-043	Collector put; Sample ✓	30/6/'17	WW	✓
✓ ISO17-14-03-043-DUP	Collector put; Sample Duplicate ✓	30/6/'17	WW	✓
✓ ISO17-14-03-043-FMS	Collector put; FMS ✓	30/6/'17	WW	✓
✓ ISO17-14-03-044	K3; Sample ✓	28/~~6~~6/'17	GW	✓
✓ ISO17-14-03-044-DUP	K3; Sample Duplicate ✓	28/6/'17	GW	✓
✓ ISO17-14-03-045	P27; Sample ✓	28/6/'17	GW	✓

Sample Condition Upon Receipt: ☒ Acceptable ☐ All items accounted for
Temperature: 15 Deg C ☐ Received on Ice ☐ Other:

7/10/17 KPH
☑ ACCEPTABLE
☑ 22 C

Collected by (print): Tine Mandonx (TMA) **Collector's signature:** [signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	3/7/'17		FEDEX	[signature]	7/10/17	10:49 AM

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phon[redacted]
Alt. P[redacted]
Fax: [redacted]

Project: ISO17-14-03 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 03/15/2017
Project Description: 3M Antwerp Water Sampling for PFCs; June-July 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [redacted]
Email Address: [redacted]

GW: groundwater

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO17-14-03-045-DUP	P27; Sample Duplicate	28/6/17	GW	/
ISO17-14-03-045-FMS	P27; FMS	28/6/17	GW	/
ISO17-14-03-046	P21B; Sample	28/6/17	GW	/
ISO17-14-03-046-DUP	P21B; Sample Duplicate	28/6/17	GW	/
ISO17-14-03-047	P304; Sample	29/6/17	GW	/
ISO17-14-03-047-DUP	P304; Sample Duplicate	29/6/17	GW	/
ISO17-14-03-048	P305; Sample	29/6/17	GW	/
ISO17-14-03-048-DUP	P305; Sample Duplicate	29/6/17	GW	/
ISO17-14-03-048-FMS	P305; FMS	29/6/17	GW	/
ISO17-14-03-049	P42; Sample	28/6/17	GW	/
ISO17-14-03-049-DUP	P42; Sample Duplicate	28/6/17	GW	/
ISO17-14-03-050	P56; Sample	28/6/17	GW	/
ISO17-14-03-050-DUP	P56; Sample Duplicate	28/6/17	GW	/
ISO17-14-03-051	L19; Sample	29/6/17	GW	/
ISO17-14-03-051-DUP	L19; Sample Duplicate	29/6/17	GW	/
ISO17-14-03-051-FMS	L19; FMS	29/6/17	GW	/
ISO17-14-03-052	M4; Sample	30/6/17	GW	/

Sample Condition Upon Receipt: ☒ Acceptable ☐ All items accounted for
Temperature: 15 Deg C ☐ Received on Ice ☐ Other:

☑ 7/10/17 KPH
☑ ACCEPTABLE
☑ 22 C

Collected by (print): Tine Mandonx (TMA) **Collector's signature:** [signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	3/7/17		FEDEX	[signature]	7/10/17	10:55AM

Attachment A: Chain of Custody Form

3M EHS LABORATORY
REPORT NO. ISO17-14-03

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phon[redacted]
Alt. P[redacted]
Fax: [redacted]

Project: ISO17-14-03 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 03/15/2017
Project Description: 3M Antwerp Water Sampling for PFCs; June-July 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [redacted]
Email Address: [redacted]

GW: groundwater

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO17-14-03-052-DUP	M4; Sample Duplicate	30/6/'17	GW	/
ISO17-14-03-052-FMS	M4; FMS	30/6/'17	GW	/
ISO17-14-03-053	P118C; Sample	30/6/'17	GW	/
ISO17-14-03-053-DUP	P118C; Sample Duplicate	30/6/'17	GW	/
ISO17-14-03-054	P119C; Sample	30/6/'17	GW	/
ISO17-14-03-054-DUP	P119C; Sample Duplicate	30/6/'17	GW	/
ISO17-14-03-055	P262; Sample	3/7/'17	GW	/
ISO17-14-03-055-DUP	P262; Sample Duplicate	3/7/'17	GW	/
ISO17-14-03-056	P263; Sample	3/7/'17	GW	/
ISO17-14-03-056-DUP	P263; Sample Duplicate	3/7/'17	GW	/
ISO17-14-03-057	P264; Sample	3/7/'17	GW	/
ISO17-14-03-057-DUP	P264; Sample Duplicate	3/7/'17	GW	/
ISO17-14-03-058	P265C; Sample	/	/	WELL GONE /not available
ISO17-14-03-058-DUP	P265C; Sample Duplicate	/	/	"
ISO17-14-03-059	P340; Sample	29/6/'17	GW	/
ISO17-14-03-059-DUP	P340; Sample Duplicate	29/6/'17	GW	/
ISO17-14-03-060	P341; Sample	29/6/'17	GW	/

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for** 7/10/17 KPH
Temperature: 15 **Deg C** ☐ **Received on Ice** ☐ **Other:** ☒ ACCEPTABLE ☒ 22 C

Collected by (print): Tine Mandonx (TMA) **Collector's signature:** [illegible]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	3/7/'17		FEDEX	[illegible]	7/10/17	11:00AM

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Pho[redacted]
Alt. [redacted]
Fax[redacted]

Project: ISO17-14-03 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 03/15/2017
Project Description: 3M Antwerp Water Sampling for PFCs; June-July 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number[redacted]
Email Address[redacted]

GW. groundwater

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
✓ ISO17-14-03-060-DUP	P341; Sample Duplicate ✓	29/6/'17	GW	/
✓ ISO17-14-03-060-FMS	P341; FMS ✓	29/6/'17	GW	/
✓ ISO17-14-03-061	P343; Sample ✓	29/6/'17	GW	/
✓ ISO17-14-03-061-DUP	P343; Sample Duplicate ✓	29/6/'17	GW	/
✓ ISO17-14-03-062	P371; Sample ✓	28/6/'17	GW	/
✓ ISO17-14-03-062-DUP	P371; Sample Duplicate ✓	28/6/'17	GW	/
✓ ISO17-14-03-063	P374; Sample ✓	28/6/'17	GW	/
✓ ISO17-14-03-063-DUP	P374; Sample Duplicate ✓	28/6/'17	GW	/
✓ ISO17-14-03-064	P379; Sample ✓	28/6/'17	GW	/
✓ ISO17-14-03-064-DUP	P379; Sample Duplicate ✓	28/6/'17	GW	/
✓ ISO17-14-03-065	P380; Sample ✓	28/6/'17	GW	/
✓ ISO17-14-03-065-DUP	P380; Sample Duplicate ✓	28/6/'17	GW	/
✓ ISO17-14-03-065-FMS	P380; FMS ✓	28/6/'17	GW	/
✓ ISO17-14-03-066	P381; Sample ✓	28/6/'17	GW	/
✓ ISO17-14-03-066-DUP	P381; Sample Duplicate ✓	28/6/'17	GW	/
✓ ISO17-14-03-067	P382; Sample ✓	28/6/'17	GW	/
✓ ISO17-14-03-067-DUP	P382; Sample Duplicate ✓	28/6/'17	GW	/

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for**
Temperature: 15 **Deg C** ☐ **Received on Ice** ☐ **Other:**

7/10/17 KAH
☑ ACCEPTABLE
☑ 22C

Collected by (print): Tine Mandonx (TMA) **Collector's signature:** TMA

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	3/7/'17		FEDEX	[signature]	7/10/17	11:05AM

Attachment A: Chain of Custody Form

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phon[redacted]
Alt. [redacted]
Fax: [redacted]

Project: ISO17-14-03 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 03/15/2017
Project Description: 3M Antwerp Water Sampling for PFCs; June-July 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number[redacted]
Email Address[redacted]

GW: groundwater

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
✓ ISO17-14-03-068	B3-bis; Sample ✓	30/6/'17	GW	/
✓ ISO17-14-03-068-DUP	B3-bis; Sample Duplicate ✓	30/6/'17	GW	/
✓ ISO17-14-03-069	B7; Sample ✓	30/6/'17	GW	/
✓ ISO17-14-03-069-DUP	B7; Sample Duplicate ✓	30/6/'17	GW	/
✓ ISO17-14-03-069-FMS	B7; FMS ✓	30/6/'17	GW	/
✓ ISO17-14-03-070	P372; Sample ✓	28/6/'17	GW	/
✓ ISO17-14-03-070-DUP	P372; Sample Duplicate ✓	28/6/'17	GW	/
✓ ISO17-14-03-071	P378; Sample ✓	30/6/'17	GW	/
✓ ISO17-14-03-071-DUP	P378; Sample Duplicate ✓	30/6/'17	GW	/
✓ ISO17-14-03-072	PA109A; Sample ✓	30/6/'17	GW	/
✓ ISO17-14-03-072-DUP	PA109A; Sample Duplicate ✓	30/6/'17	GW	/
✓ ISO17-14-03-073	PA111A; Sample ✓	30/6/'17	GW	/
✓ ISO17-14-03-073-DUP	PA111A; Sample Duplicate ✓	30/6/'17	GW	/
✓ ISO17-14-03-074	PA112; Sample ✓	30/6/'17	GW	/
✓ ISO17-14-03-074-DUP	PA112; Sample Duplicate ✓	30/6/'17	GW	/
✓ ISO17-14-03-074-FMS	PA112; FMS ✓	30/6/'17	GW	/
✓ ISO17-14-03-075	Trip Blank; Sample ✓	/	/	*

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for**
Temperature: 15 **Deg C** ☐ **Received on Ice** ☐ **Other:**

7/10/17 KPH
☒ ACCEPTABLE
☒ 22C

Collected by (print): Tine Mandonx (TMA) **Collector's signature:** [signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
17	TMA	3/7/'17		FEDEX	[signature]	7/10/17	11:10AM

3M ENVIRONMENTAL LABORATORY
Chain-of-Custody

Shipping Address:
3M Environmental Laboratory
3M Center, Bldg 260-5N-17
St. Paul, MN 55144

Phon[redacted]
Alt. P
Fax:

Project: ISO17-14-03 (cont.)

Requester: Cauberghe, Nicole (ZWIJNDRECH-3
Department: 832202 Site Source: 01WJWT10
Project Number:
Date Created: 03/15/2017
Project Description: 3M Antwerp Water Sampling for PFCs; June-July 2017 Sampling

Completion Date:
Project Lead: Susan T. Wolf
Phone Number: [redacted]
Email Address:

3M Sample Number	Sample Description	Date/Time Sampled	Matrix	Comment
ISO17-14-03-075-FMS-HIGH	Trip Blank; FMS High ✓	/	/	*
ISO17-14-03-075-FMS-LOW	Trip Blank; FMS Low ✓	/	/	*

* Trip Blank samples prepared by the 3M EHS Laboratory with the bottle order.

Sample Condition Upon Receipt: ☒ **Acceptable** ☐ **All items accounted for**
Temperature: 15 **Deg C** ☐ **Received on Ice** ☐ **Other:**

7/10/17 KPH
☑ ACCEPTABLE
☑ 22C

Collected by (print): Tine Mandonx (TMA) **Collector's signature:** [signature]

Item #	Relinquished by:	Date	Time	Shipped Via	Received by:	Date	Time
2	TMA	3/7/17		FEDEX	[signature]	7/10/17	11:10AM

SGS

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

ANALYTICAL REPORT

IAC16-05382-002

3M Lab Request Number: E16-0801

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: November 17th, 2016

Requester

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

1. Introduction - Summary.

At the request (Lab Request Number: E16-0801) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, one water sample was collected on Octobre 11th, 2016 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%. 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	1.31	3.94	9.52	0.093

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 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, except for PFHS LCSH.
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	112	114	107	117	103	115	102	108
	LCSL02	0.125	107	106	107	104	103	104	105	108
	Average		**109**	**110**	**107**	**111**	**103**	**109**	**103**	**108**
	%RPD		**5.4**	**7.4**	**0.3**	**12**	**0.4**	**10**	**2.4**	**0.2**
	LCSH 01	2.5	117	116	103	118	87.9	107	96.0	100
	LCSH 02	2.5	112	108	107	115	103	117	105	111
	Average		**115**	**112**	**105**	**117**	**95.5**	**112**	**101**	**106**
	%RPD		**3.9**	**6.4**	**4.3**	**2.7**	**16(a)**	**9.1**	**9.3**	**10**

(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	82.9	<0.025	102
3M vijver	1.31	91.5	3.94	96.2

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	114	<0.025	111
3M vijver	9.52	115	0.093	75.6

All PFHS, PFOA, PFOS and FOSA results have an accuracy of ± 30%.

5. Conclusion.

- The recovery of the surrogate recovery standard fell within the method acceptance criterion 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 Ruben Maes (ERM Belgium).

8. Signatures.

Geert De Smet,
Lab Operations Manager — Date November 17th, 2016

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories — Date November 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

IAC16-05382-001

3M Lab Request Number: E16-0801

Analysis of Perfluorinated Compounds in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: November 17th, 2016

Requester

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

1. Introduction - Summary.

At the request (Lab Request Number: E16-0801) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected between Octobre 7th and 11th, 2016 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%. See Section 4 of the report for additional information on the method QC used to assess method uncertainty.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
L4		2.23	1.72	5.64	0.400
P116		0.515	<0.369	5.93	<0.129
Effluent WWTP		31.4	22.8	14.9	<0.129
PP01	144	6538	24.5	2512	15.5
PP04	2783	296	784	6427	24.2
PP06	24.7	56.6	215	2176	38.7
PP08	52.2	298	512	7905	16.9
PP10	18.9	188	630	4593	8.72
13		29.3	107	356	<0.384
5		52.5	75.8	78.9	<0129
Bemalingsstation		15.8	46.5	203	6.11
Collector Put		91.8	118	158	<0.384

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
L4	100
P116	100
Effluent WWTP	100
PP01	100
PP04	100
PP06	100
PP08	100
PP10	100
13	100
5	100
Bemalingsstation	100
Collector Put	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 " Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)". Briefly, 1.0 mL sample was spiked with surrogate standard, diluted with 10.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. Afterwards an amount of the dilution was transferred to a centrifuge tube and diluted with MeOH:LCMS-water (60:40). The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
L4	1.0	0.05	Solution A	1.0	-
P116	1.0	0.05	Solution A	1.0	-
Effluent WWTP	1.0	0.05	Solution A	1.0	-
PP01	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP04	1.0	0.2	Solution A	10.0	0.5mL extract + 10.0mL MeOH (*)
PP06	1.0	0.100	Solution A	10.0	1.0mL extract + 3.0mL MeOH (*)
PP08	1.0	0.2	Solution A	10.0	0.5mL extract + 10.0mL MeOH (*)
PP10	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
13	1.0	0.100	Solution A	5.0	-
5	1.0	0.05	Solution A	1.0	-
Bemalingsstation	1.0	0.05	Solution A	2.0	-
Collector Put	1.0	0.1	Solution A	5.0	-
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 0.0630 to 0.18 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.129 to 42.3 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within ± 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
16 Octobre 2016	LCS1	80	99.2	92.8	93.2	80.8	101	98.0	84.3

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
10 August 2016	QC Lab Low inj1	10	99.9	95.1	100	124	118	108	98.0
	QC Lab High inj1	100	85.0	75.5	91.9	75.4	98.3	90.8	76.3
	QC Lab Low inj2	10	117	108	120	120	123	116	103
	QC Lab High inj2	100	109	82.0	105	76.2	109	95.1	75.8

3.7. Equations.

Recovery.

$$\text{Recovery (\%)} = \frac{\text{Found concentration}}{\text{Spiked concentration}} * 100$$

4. Data Summary.

The following tables summarize the results for the sample locations.

Table 9. Sample Results PFBS, PFHS, PFOA, PFOS and FOSA

	PFBS	PFHS	PFOA	PFOS	FOSA
	ng/ml	ng/ml	ng/ml	ng/ml	ng/ml
L4		2.23	1.72	5.64	0.400
P116		0.515	<0.369	5.93	<0.129
Effluent WWTP		31.4	22.8	14.9	<0.129
PP01	144	6538	24.5	2512	15.5
PP04	2783	296	784	6427	24.2
PP06	24.7	56.6	215	2176	38.7
PP08	52.2	298	512	7905	16.9
PP10	18.9	188	630	4593	8.72
13		29.3	107	356	<0.384
5		52.5	75.8	78.9	<0129
Bemalingsstation		15.8	46.5	203	6.11
Collector Put		91.8	118	158	<0.384
Field Trip Blank	<0.932	<0.966	<2.00	<0.999	<0.699

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

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
L4	62.0	108	44.0	71.5
P116	55.5	96.7	45.2	73.4
Effluent WWTP	72.2	126	56.6	91.9
PP01	142	124	144	117
PP04	286	125	282	114
PP06	146	127	134	109
PP08	296	129	257	105
PP10	134	116	108	87.7
13	135	118	127	103
5	70.7	123	53.5	87.0
Bemalingsstation	63.6	111	58.6	95.2
Collector Put	139	121	121	98.3
Field Trip Blank	123	107	121	98.2

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 November 17th, 2016

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories — Date November 17th, 2016

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

Bijlage 4

Overzicht veldmetingen en analyseresultaten

Puntlocatie	Top filter (cm)	Bodem filter (cm)	Datum bemonstering	Grondwaterstand (cm)	Redox (mV)	pH	EC (µS/cm)	Temperatuur (°C)
12			14-04-2017		42,3	7,57	764	12
12			29-06-2017		-39,2	8,45	1011	17,73
13			11-10-2016		82	7,65	857	9,27
13			14-04-2017		45,2	7,66	763	
13			29-06-2017		-27	9,33	991	16,71
3M vijver			11-10-2016		94,6	6,99	973	10,47
3M vijver			14-04-2017		118	8,06	654	8,61
3M vijver			29-06-2017		32,5	8,91	1164	19,09
5			12-10-2016		97,8	7,67	1294	10,72
5			14-04-2017		47,9	7,75	1063	11,9
5			29-06-2017		-53	8,62	1780	20,71
B3-bis	250	350	11-01-2017	225	-182,7	7,52	238	9,94
B3-bis	250	350	30-06-2017	279	-176,1	7,33	422	13,99
B7	180	380	11-01-2017	233	-7,8	7,68	287	9,42
B7	180	380	30-06-2017	283	-89	7,84	338	14,03
BD24-3	1700	1800	03-07-2017	340	-123,2	7,02	812	13,91
BD24-3	1700	1800	07-10-2016	337	-124,4	7,37	726	12,11
BD24-3	1700	1800	13-01-2017	304	45,8	7,44	834	9,93
BD24-3	1700	1800	14-04-2017	301		7,43	980	11,1
BD24-4	2200	2400	03-07-2017	338	-89,1	7,26	1892	13
Bemalingsstation			11-10-2016		113,6	7,55	1518	11,2
Bemalingsstation			14-04-2017		44,9	7,73	1145	12
Bemalingsstation			29-06-2017		-43,4	8,55	1896	20,64
Blokkersdijk - std			11-10-2016		104,2	7,86	509	9,19
Blokkersdijk - std			18-04-2017		99,5	8,61	393	8,87
Blokkersdijk - std			29-06-2017		-26,1	9,82	409	20,89
Blokkersdijkvijver N			18-04-2017		101,5	8,48	402	9,18
Blokkersdijkvijver N			29-07-2017		-43,5	9,8	472	19
Collector pit			10-10-2016		-55,8	7,53	644	17,3
Collector pit			30-06-2017		-91,3	7,53	1164	20,03
D10	14	16	03-07-2017	448	-132,1	6,78	1754	14,78
D10	14	16	10-01-2017	422		7,15	2012	11,1
D11	1490	1590	11-01-2017	324		7,49	490	11,5
D11	1490	1590	28-06-2017	362	-102	6,86	445	6,86
D14	1550	1650	10-01-2017	489		6,9	3950	10,6
D14	1550	1650	30-06-2017	508	-109,9	7,15	2980	15,01
D16	1400	1600	10-01-2017	327		7,34	718	12,3
D16	1400	1600	29-06-2017	370		7,3	658	15,9
D17	1400	1600	09-01-2017	355		6,99	1478	14,8
D18	1400	1600	09-01-2017	260		7,19	1001	11,4
D2	2400	2616	03-07-2017	569	-8,8	7,08	347	14,93
D5	1400	1600	12-01-2017	342	0	7,14	931	12,2
D5	1400	1600	29-06-2017	364	0	7,23	922	14,6
D9	1300	1500	09-01-2017	330	0	7,21	1074	15,5
D9	1300	1500	12-10-2016	360	-167,4	7,05	7,42	16,16
D9	1300	1500	14-04-2017	336	-132,2	6,98	1176	14,96
D9	1300	1500	28-06-2017	360		7,21	830	16,9
Effluent WWTP			30-06-2017		-91,2	7,89	5740	22,53
K3	360	560	28-06-2017	250		7,34	581	16
L19	280	480	10-01-2017	340		7,21	862	10,5
L19	280	480	29-06-2017	343		7,29	761	19,8
L21	300	500	03-07-2017	237	-155,7	7,09	400	16,69
L21	300	500	11-10-2016	218	-61,8	7,46	432	16,57
L21	300	500	12-01-2017	192		7,63	505	9,6

Puntlocatie	Top filter (cm)	Bodem filter (cm)	Datum bemonstering	Grondwaterstand (cm)	Redox (mV)	pH	EC (µS/cm)	Temperatuur (°C)
L21	300	500	14-04-2017	198	-169,9	7,34	377	10,37
L22	200	400	03-07-2017	292	-183,5	7,09	601	16,73
L22	200	400	11-10-2016	224	-76,4	7,49	420	16,15
L22	200	400	12-01-2017	184		7,54	617	10
L22	200	400	14-04-2017	259	-165,8	7,3	440	10,21
L31	120	320	03-07-2017	262	-109,7	7,05	677	14,82
L31	120	320	11-10-2016	247	97,7	7,47	462	16,64
L31	120	320	12-01-2017	232		7,31	767	8,2
L31	120	320	14-04-2017	230	79,9	7,24	503	9,49
L4	200	400	03-07-2017	250	-165,4	7,27	910	15,9
L4	200	400	07-10-2016	238	-158,3	7,18	682	14,75
L4	200	400	10-01-2017	280		7,37	980	9,3
L4	200	400	14-04-2017	183		7,17	850	10,6
M4	100	300	10-01-2017	184		7,24	1237	12,9
M4	100	300	30-06-2017	201		7,17	1883	20
ND7	1400	1600	09-01-2017	289		7,19	811	12,2
ND7	1400	1600	28-06-2017	321		7	359	15
P114bis	360	460	03-07-2017	203	-143,2	7,23	1882	16,11
P114bis	360	460	12-01-2017	140		7,07	2780	10,6
P114bis	360	460	12-10-2016	194	-80,7	6,93	1304	13,84
P114bis	360	460	14-04-2017	169		7,17	1786	10,5
P115	330	430	12-01-2017	124		7,01	3300	9,9
P115	330	430	12-10-2016	208	-77,2	6,9	2514	13,89
P115	330	430	14-04-2017	138		7,03	3270	9,8
P116	250	350	03-07-2017	294	-187,7	7,13	612	14,96
P116	250	350	11-10-2016	263	-156,6	7,68	543	15,34
P116	250	350	12-01-2017	235		7,62	659	10
P116	250	350	14-04-2017	266	170,7	7,49	523	10,29
P118A	2250	2350	10-01-2017	297	-138,4	6,77	1280	10,95
P118B	1300	1400	10-01-2017	299	-106,7	7,86	1989	10,73
P118B	1300	1400	30-06-2017	336	-118,1	7,16	2344	13,3
P118C	250	450	11-01-2017	262	-157,7	6,68	934	10,15
P118C	250	450	30-06-2017	308	-171	7,29	1187	13,32
P119A	23	24,2	11-01-2017	378		6,8	810	11,2
P119A	23	24,2	30-06-2017	407		6,89	781	15,4
P119B	1300	1400	11-01-2017	377		7,07	1626	11
P119B	1300	1400	30-06-2017	411		7,05	1619	15,7
P119C	250	450	11-01-2017	165		7,53	737	10,3
P119C	250	450	30-06-2017	231		7,51	713	14
P121	2350	2450	03-07-2017	483	-175,2	6,58	2596	14,4
P121	2350	2450	12-01-2017	447		6,86	3170	11,7
P121	2350	2450	12-10-2016	484	-48,8	6,72	2376	12,07
P18	140	340	28-06-2017	290		7,32	863	14,9
P21B	350	550	10-01-2017	119				
P21B	350	550	12-10-2016	124	-161,4	7,38	1069	13,64
P21B	350	550	14-04-2017	116				
P21B	350	550	29-06-2017	128		7,47	2220	16,4
P262	620	720	03-07-2017	438	-145,5	6,98	639	13,11
P263	740	840	03-07-2017	547		7,49	1705	16,3
P263	740	840	11-01-2017	512		7,51	1714	11,6
P263	740	840	12-10-2016	540	-120,1	7,45	1245	13,01
P263	740	840	14-04-2017	505	-175,6	7,46	1265	11,84
P264	500	550	03-07-2017	317	-178	6,94	707	12,39
P264	500	550	10-01-2017	277	-169,9	6,56	565	10,87

Puntlocatie	Top filter (cm)	Bodem filter (cm)	Datum bemonstering	Grondwaterstand (cm)	Redox (mV)	pH	EC (µS/cm)	Temperatuur (°C)
P27	185	285	09-01-2017	170		7,47	8,07	8,1
P27	185	285	28-06-2017	179		7,36	666	17,8
P28	110	310	29-06-2017	221		13,08	36600	16,2
P304	500	600	09-01-2017	240		7,18	8,94	14,4
P304	500	600	28-06-2017	262		7,13	810	16,8
P305	350	550	09-01-2017	196		7,07	790	12,7
P305	350	550	29-06-2017	202		7,07	878	17,1
P321	1550	1650	10-01-2017	384		7,17	1515	12,6
P321	1550	1650	12-10-2016	412	-93,6	7,12	1240	13,7
P321	1550	1650	14-04-2017	477		7,19	1593	12,8
P321	1550	1650	30-06-2017	418		7,36	1322	15
P340	400	600	30-06-2017	237		7,16	1473	17,3
P341	360	560	29-06-2017	292		7,32	975	15
P343	510	610	30-06-2017	387		7,13	1254	15,5
P371	150	350	10-01-2017	244		7,26	699	9,9
P371	150	350	28-06-2017	371	-25,4	6,59	800	14,42
P372	480	580	10-01-2017	250		7,27	465	11,8
P372	480	580	28-06-2017	644	-122,6	6,56	389	13,42
P374	500	600	10-01-2017	301		6,89	1355	11,7
P374	500	600	28-06-2017	337	-101,5	6,47	1246	13,36
P378	350	550	11-01-2017	230	-140,5	5,98	916	11,19
P378	350	550	30-06-2017	272	-125,9	6,96	733	13,9
P379	350	550	28-06-2017	270	-136,1	6,72	528	14,97
P380	350	550	28-06-2017	272	-140	6,87	443	13,82
P381	500	600	10-01-2017	290	-159,2	6,79	656	10,56
P382	250	350	10-01-2017	285	-171,7	7,19	898	10,13
P42	300	500	10-01-2017	384		7,04	1194	11,6
P42	300	500	29-06-2017	297		6,45	1626	18,4
P56	120	320	28-06-2017	200		7,36	498	16,8
PA109A	500	600	11-01-2017	310	-168,5	5,93	450	11,34
PA109A	500	600	30-06-2017	362	-171,5	7	506	13,9
PA111A	400	600	11-01-2017	261	-77,8	6,63	1194	11,29
PA111A	400	600	30-06-2017	304	-65,6	6,83	1251	14,7
PA112	345	545	11-01-2017	260	-186,6	7,42	907	10,99
PA112	345	545	30-06-2017	296	-176,6	7,67	577	13,28
PP01	260	560	07-10-2016	382	-22,3	7,56	615	17,48
PP01	260	560	30-07-2017	382	-86,4	6,98	767	16,41
PP02	320	620	07-10-2016	370	-33,6	7,14	787	17,3
PP02	320	620	30-07-2017	370	-71,5	7,35	1343	21,06
PP04	200	600	07-10-2016	179	-33,2	7,59	677	17,05
PP04	200	600	30-07-2017	179	-120,1	6,99	928	15,16
PP06	130	530	07-10-2016	292	-62,7	7,32	589	17,3
PP06	130	530	30-07-2017	292	-88,8	7,34	1046	18,1
PP07	200	600	07-10-2016	432	-38,1	7	861	17,3
PP07	200	600	30-07-2017	432	-61,4	7,24	1601	20,67
PP08	320	720	07-10-2016	505	-17,3	6,9	1188	17,3
PP08	320	720	30-07-2017	547	-48,8	7,16	1628	19,95
PP09	300	600	06-10-2017	0	-20,6	7,1	887	17,3
PP09	300	600	30-07-2017	0	-64,4	7,12	1300	19,73
PP10	300	600	07-10-2016	270	-23,9	7,1	1297	17,3
PP10	300	600	30-07-2017	270	-87,7	7,2	1392	20

Puntlocatie	B3-bis	B3-bis	B7	B7	BD24-3	BD24-3	BD24-3	BD24-3	BD24-4	D10
Bemonsteringsdatum	11/01/2017	30/06/2017	11/01/2017	30/06/2017	7/10/2016	13/01/2017	14/04/2017	3/07/2017	3/07/2017	11/01/2017
Top Filter	2,5	2,5	1,8	1,8	17	17	17	17	22	15
Bodem Filter	3,5	3,5	3,8	3,8	18	18	18	18	24	16
PFBS (µg/l)										
PFHS (µg/l)	6,18	5,54	1,59	9,89	59,6	44,2	42,4	37,9	5,62	84,2
PFOA (µg/l)	37,1	22,3	4,34	70	83,1	50,2	49,7	43,8	12,7	229
PFOS (µg/l)	112	180	67,1	270	1470	1280	1140	1200	5,9	23,4
PFOSA (µg/l)	0,21	0,759	0,619	4,19	3,66	3,57	5,3	5,48	0,464	0,311

Puntlocatie	D10	D11	D11	D14	D14	D16	D16	D17	D17
Bemonsteringsdatum	3/07/2017	11/01/2017	28/06/2017	10/01/2017	30/06/2017	11/01/2017	28/06/2017	9/01/2017	28/06/2017
Top Filter	15	14,9	14,9	15,5	15,5				
Bodem Filter	16	15,9	15,9	16,5	16,5	15,68	15,68	15	15
PFBS (µg/l)									
PFHS (µg/l)	109	33,9	45,3	1,24	1,34	33,8	54,1	531	76
PFOA (µg/l)	310	47,2	81,2	2,02	2,4	174	197	6320	770
PFOS (µg/l)	23,8	773	835	0,752	0,388	1170	1240	214	382
PFOSA (µg/l)	0,451	99,5	95,8	0,0594	<0,0248	104	103	0,0825	40,1

Puntlocatie	D18	D18	D2	D5	D5	D9	D9	D9	D9	K3
Bemonsteringsdatum	9/01/2017	28/06/2017	3/07/2017	9/01/2017	29/06/2017	12/10/2016	10/01/2017	14/04/2017	28/06/2017	28/06/2017
Top Filter	13	13				13	13	13	13	3,6
Bodem Filter	15	15				15	15	15	15	5,6
PFBS (µg/l)										856
PFHS (µg/l)	231	109	0,15	334	276	2870	3800	1360	4820	84400
PFOA (µg/l)	407	208	0,307	503	636	120	172	179	125	88,1
PFOS (µg/l)	15,2	52,2	8,33	478	625	1670	1530	500	1730	4690
PFOSA (µg/l)	1,17	5,83	0,82	6,98	16	25,8	25,4	9,69	27,9	15

Puntlocatie	L19	L19	L21	L21	L21	L21	L22	L22	L22	L22
Bemonsteringsdatum	10/01/2017	29/06/2017	11/10/2016	12/01/2017	14/04/2017	3/07/2017	11/10/2016	12/01/2017	14/04/2017	3/07/2017
Top Filter	2,8	2,8	3	3	3	3	2	2	2	2
Bodem Filter	4,8	4,8	5	5	5	5	4	4	4	4
PFBS (µg/l)										
PFHS (µg/l)	172	88,6	0,158	0,151	0,214	0,188	0,12	0,0879	0,253	0,362
PFOA (µg/l)	309	189	0,318	0,289	0,42	0,418	0,488	0,355	0,461	0,784
PFOS (µg/l)	3980	4220	5,22	5,41	5,01	6,13	4,94	4,29	4,43	4,97
PFOSA (µg/l)	37,6	46	0,0506	0,0874	0,113	0,291	0,0791	0,0857	0,114	0,102

Puntlocatie	L31	L31	L31	L31	L4	L4	L4	L4
Bemonsteringsdatum	11/10/2016	12/01/2017	14/04/2017	3/07/2017	11/10/2016	11/01/2017	14/04/2017	3/07/2017
Top Filter	1,2	1,2	1,2	1,2	2	2	2	2
Bodem Filter	3,2	3,2	3,2	3,2	4	4	4	4
PFBS (µg/l)								
PFHS (µg/l)	0,395	1,02	0,694	0,945	2,8	2,31	2,11	2,23
PFOA (µg/l)	0,723	0,71	0,517	1,19	3,15	2,8	2,42	2,88
PFOS (µg/l)	9,2	5,22	5,39	7,53	31,8	29,2	28,8	32,8
PFOSA (µg/l)	0,133	0,0473	0,117	0,167	1,11	1,49	3,15	0,556

Puntlocatie	M4	M4	ND7	ND7	P114bis	P114bis	P114bis	P114bis
Bemonsteringsdatum	10/01/2017	30/06/2017	9/01/2017	29/06/2017	12/10/2016	12/01/2017	14/04/2017	3/07/2017
Top Filter	1	1			3,6	3,6	3,6	3,6
Bodem Filter	3	3			4,6	4,6	4,6	4,6
PFBS (µg/l)								
PFHS (µg/l)	193	456	49,4	12,3	1,39	1,4	1,28	1,11
PFOA (µg/l)	429	4330	293	158	3,28	3,63	3,63	2,57
PFOS (µg/l)	4960	11500	40,9	223	13,4	9,21	12,5	11,7
PFOSA (µg/l)	129	216	59,4	165	0,043	0,0292	0,106	0,0527

Puntlocatie	P115	P115	P115	P115	P116	P116	P116	P116
Bemonsteringsdatum	12/10/2016	12/01/2017	14/04/2017	3/07/2017	11/10/2016	12/01/2017	14/04/2017	3/07/2017
Top Filter	3,3	3,3	3,3	3,3	2,5	2,5	2,5	2,5
Bodem Filter	4,3	4,3	4,3	4,3	3,5	3,5	3,5	3,5
PFBS (µg/l)								
PFHS (µg/l)	2,05	2,21	2,36	2,32	0,738	0,587	0,625	0,637
PFOA (µg/l)	3,82	3,94	4,98	5,14	1,59	0,924	1,42	1,46
PFOS (µg/l)	0,304	0,593	0,523	0,443	9,66	10,9	9,62	9,08
PFOSA (µg/l)	<0,0248	<0,0248	<0,0248	<0,0248	0,105	0,0971	0,113	0,123

Puntlocatie	P118A	P118A	P118B	P118B	P118C	P118C	P119A	P119A	P119B	P119B
Bemonsteringsdatum	10/01/2017	29/06/2017	10/01/2017	30/06/2017	10/01/2017	30/06/2017	11/01/2017	29/06/2017	11/01/2017	29/06/2017
Top Filter	22,5	22,5	13	13	2,5	2,5	23,5	23,5	13	13
Bodem Filter	23,5	23,5	14	14	4,5	4,5	24,5	24,5	14	14
PFBS (µg/l)										
PFHS (µg/l)	823	1910	3190	3300	79	34,9	156	176	479	224
PFOA (µg/l)	1140	1590	2170	2020	248	142	274	345	1423	550
PFOS (µg/l)	5050	157	15500	15600	1690	1130	1350	1990	99,8	128
PFOSA (µg/l)	24,9	1,61	3,46	6,08	67,3	112	54,6	86,5	0,897	1,14

Puntlocatie	P119C	P119C	P121	P121	P121	P121	P21B	P21B	P21B	P21B
Bemonsteringsdatum	11/01/2017	30/06/2017	12/10/2016	12/01/2017	18/04/2017	3/07/2017	12/10/2016	10/01/2017	14/04/2017	28/06/2017
Top Filter	2,5	2,5	23,5	23,5	23,5	23,5	3,5	3,5	3,5	3,5
Bodem Filter	4,5	4,5	24,5	24,5	24,5	24,5	5,5	5,5	5,5	5,5
PFBS (µg/l)							4340	4680	7290	6740
PFHS (µg/l)	714	423	0,113	0,105	0,0984	0,208	7250	7380	11600	11300
PFOA (µg/l)	957	710	0,285	0,261	0,241	0,28	7800	7130	11300	10600
PFOS (µg/l)	2810	3700	3,86	2,05	1,49	1,42	48800	52300	66200	70900
PFOSA (µg/l)	60	94,3	0,234	0,14	0,179	0,198	4,64	19,2	8,56	4,46

Puntlocatie	P262	P263	P263	P263	P263	P264	P264	P27	P27
Bemonsteringsdatum	3/07/2017	12/10/2016	11/01/2017	14/04/2017	3/07/2017	10/01/2017	3/07/2017	9/01/2017	28/06/2017
Top Filter	6,2	7,4	7,4	7,4	7,4	5	5		
Bodem Filter	7,2	8,4	8,4	8,4	8,4	5,5	5,5		
PFBS (µg/l)								991	560
PFHS (µg/l)	529	567	437	447	498	161	198	45,1	19,2
PFOA (µg/l)	1160	1910	1280	1400	1630	245	318	131	140
PFOS (µg/l)	4330	10600	8360	7880	9900	1470	1510	1820	1680
PFOSA (µg/l)	80,6	12,1	15,7	23,5	14,6	76	132	55,5	63,8

Puntlocatie	P304	P304	P305	P305	P321	P321	P321	P321	P28
Bemonsteringsdatum	9/01/2017	29/06/2017	9/01/2017	29/06/2017	12/10/2016	10/01/2017	14/04/2017	29/06/2017	29/06/2017
Top Filter	4	4	3,5	3,5	15,5	15,5	15,5	15,5	1,1
Bodem Filter	6	6	5,5	5,5	16,5	16,5	16,5	16,5	3,1
PFBS (µg/l)	271	279	118	156					
PFHS (µg/l)	125	137	220	321	758	602	639	326	
PFOA (µg/l)	402	476	140	239	2610	2110	2520	987	
PFOS (µg/l)	1350	1180	502	631	4860	6180	5240	4280	
PFOSA (µg/l)	28,3	27,3	22,3	10,4	0,229	1,42	0,397	14,8	
Minerale olie (µg/l)									<500

Puntlocatie	P340	P341	P343	P371	P371	P372	P372	P374	P374	P378
Bemonsteringsdatum	29/06/2017	29/06/2017	29/06/2017	11/01/2017	28/06/2017	11/01/2017	28/06/2017	11/01/2017	28/06/2017	11/01/2017
Top Filter	4	3,5	4,8	1,5	1,5	4,8	4,8	5	5	3,5
Bodem Filter	6	5,5	6,8	3,5	3,5	5,8	5,8	6	6	5,5
PFBS (µg/l)										
PFHS (µg/l)	306	270	400	141	188	43,9	43,6	772	1180	337
PFOA (µg/l)	637	547	660	161	469	66,8	67	517	736	302
PFOS (µg/l)	3650	5550	6840	2800	4450	1190	1200	2760	2230	1650
PFOSA (µg/l)	109	91,3	22,8	52,6	31,2	68,6	59,7	32,3	38,1	2,36

Puntlocatie	P378	P379	P380	P381	P381	P382	P382	P42	P42
Bemonsteringsdatum	30/06/2017	28/06/2017	28/06/2017	10/01/2017	28/06/2017	10/01/2017	28/06/2017	10/01/2017	28/06/2017
Top Filter	3,5	3,5	3,5	5	5	2,5	2,5	3	3
Bodem Filter	5,5	5,5	5,5	6	6	3,5	3,5	5	5
PFBS (µg/l)								42,4	49,4
PFHS (µg/l)	270	21,7	43,7	138	166	122	138	543	489
PFOA (µg/l)	263	74,7	121	470	598	380	496	233	279
PFOS (µg/l)	1110	764	633	1430	1280	860	889	4620	3680
PFOSA (µg/l)	3,01	90,6	124	13,7	18,6	79,2	123	14,9	16,3

Puntlocatie	P56	PA109A	PA109A	PA111A	PA111A	PA112	PA112
Bemonsteringsdatum	28/06/2017	11/01/2017	30/06/2017	11/01/2017	30/06/2017	11/01/2017	30/06/2017
Top Filter	1,2	5	5	4	4	3,45	3,45
Bodem Filter	3,2	6	6	6	6	5,45	5,45
PFBS (µg/l)	7,07						
PFHS (µg/l)	16,2	49,4	55,8	925	1090	34,6	33
PFOA (µg/l)	38,5	44,3	53,3	648	848	97,7	150
PFOS (µg/l)	354	171	171	2190	2310	377	358
PFOSA (µg/l)	30,3	1,64	3,3	10,8	18,2	78,2	107

Puntlocatie	3M Vijver	3M Vijver	3M Vijver	3M Vijver	Blokkersdijk standaard	Blokkersdijk standaard	Blokkersdijk standaard	Blokkersdijk standaard	Blokkersdijk noord	Blokkersdijk noord
Bemonsteringsdatum	11/10/2016	12/01/2017	18/04/2017	29/06/2017	11/10/2016	12/01/2017	18/04/2017	29/06/2017	12/01/2017	18/04/2017
PFBS (µg/l)										
PFHS (µg/l)	0,676	0,498	0,54	0,939	0,614	0,468	0,514	0,754	0,498	0,492
PFOA (µg/l)	1,72	0,99	1,15	2,03	1,13	0,794	0,929	1,48	0,819	0,838
PFOS (µg/l)	4,68	3,08	4,39	5,82	6,84	0,74	1,86	6,62	1,36	0,951
PFOSA (µg/l)	0,0327	0,01	0,0714	0,0737	0,144	0,01	0,0342	0,231	0,01	0,0321

Puntlocatie	Blokkersdijk noord	12	12	12	13	13	13	13
Bemonsteringsdatum	29/06/2017	12/01/2017	14/04/2017	29/06/2017	11/10/2016	12/01/2017	14/04/2017	29/06/2017
PFBS (µg/l)								
PFHS (µg/l)	0,662	20,3	23,9	26,5	31,4	19,8	23,4	27,3
PFOA (µg/l)	1,21	67,9	87,7	108	117	60,7	80,2	93
PFOS (µg/l)	2,08	273	313	367	388	230	263	299
PFOSA (µg/l)	0,0696	0,206	0,502	0,345	0,301	0,198	0,434	0,271

Puntlocatie	5	5	5	5	Bemalingsstatioı	Bemalingsstation	Bemalingsstation	Bemalingsstation	collectorput	collectorput
Bemonsteringsdatum	11/10/2016	12/01/2017	14/04/2017	29/06/2017	11/10/2016	12/01/2017	14/04/2017	29/06/2017	10/10/2016	13/01/2017
PFBS (µg/l)										
PFHS (µg/l)	58,6	23,1	29	78,2	48,7	18,0	26,3	62,9	34,8	13,4
PFOA (µg/l)	72,9	26,9	35,9	87,6	61,6	21,8	32,7	70,8	102	27,5
PFOS (µg/l)	110	56,5	57,9	104	91,1	49,2	55	88,8	450	104
PFOSA (µg/l)	0,372	0,226	0,234	0,375	0,278	0,193	0,223	0,294	19,4	5,66

Puntlocatie	collectorput	collectorput	effluent WW'	effluent WWT	effluent WWT	effluent WWT
Bemonsteringsdatum	14/04/2017	30/06/2017	10/10/2016	13/01/2017	14/04/2017	30/06/2017
PFBS (µg/l)						
PFHS (µg/l)	39,2	38,3	28,6	0,409	33,5	2,9
PFOA (µg/l)	115	107	22,4	0,41	27,6	2,31
PFOS (µg/l)	365	370	20,1	2,45	81	0,91
PFOSA (µg/l)	24,2	24,9	0,306	0,107	2,55	0,0276

Bijlage 5

Statistische evaluatie analyseresultaten Blokkersdijk

Memo

To	3M: Jim Kotsmith; Nicole Cauberghe; Charlotte Tack Weston: Jai Kesari; Dave Cairns
Copied to	ERM: Dirk Nuyens; Mattias Verbeeck
From	ERM: Stephen Geiger; Nanda Hermes
Ref/Project number	0398993
Subject	Evaluation of the statistical trend analysis of the ground- and surface water concentrations collected in the Blokkersdijk nature reserve
Date	29-November-2017

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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 3-January-2017 memo which presented an evaluation of the statistical trend of the PFOS concentrations. Performance of this PFOS trend analysis is part of the remedial action plan(1) that was drafted for the 3M site in Zwijndrecht.

Since 2009, periodic sampling has been conducted to collect water samples in the 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 31-March-2016 memo argued that there were substantial differences between laboratories, and the 3M lab data should be used for the trend analysis. The current trend analysis includes sampling results collected from January 2014 through July 2017, and analyzed by the 3M lab.

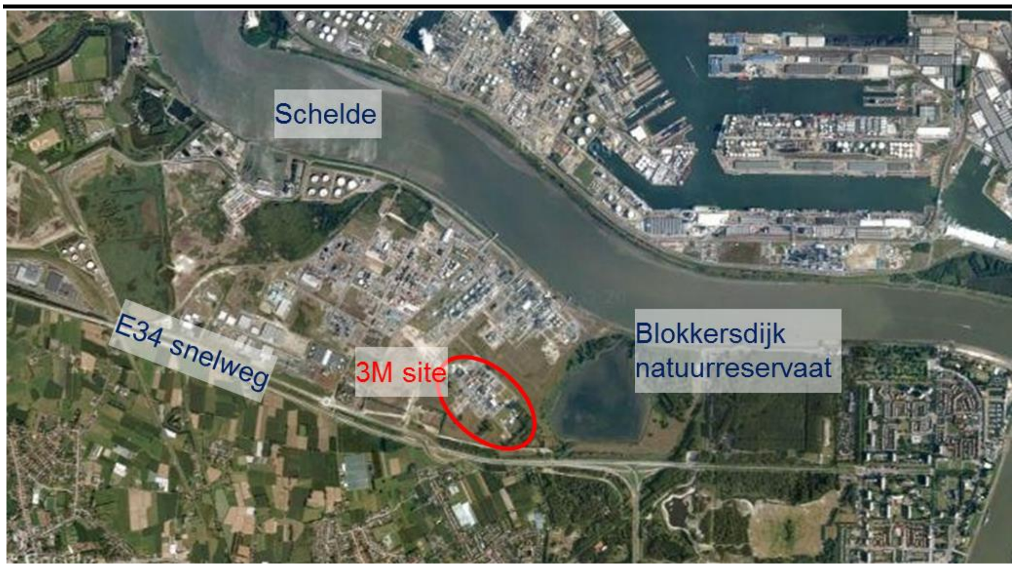
The results of the revised evaluation will be included in the sixth annual interim report ("*Eerste gefaseerd bodemsaneringsproject - Zesde tussentijds verslag bodemsaneringswerken, Periode*

(1)"Eerste gefaseerd bodemsaneringsproject 3M Belgium NV, haven 1005 – Canadastraat 11, 2070 Zwijndrecht", opgesteld door Arcadis op oktober 2008 *juli 2014 - juli 2015*", dd. 31 maart 2016) that was submitted to the Flemish environmental agency OVAM.

Background

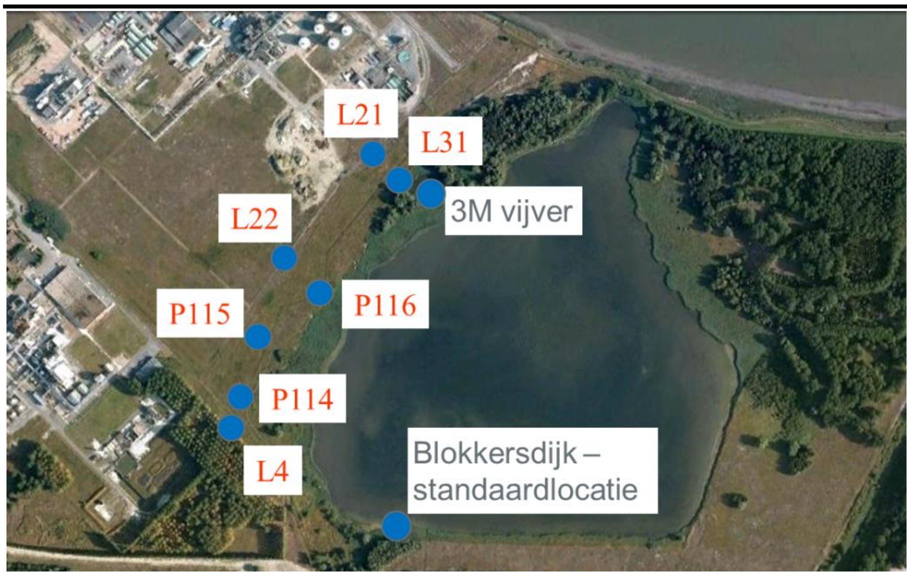
The Blokkerdijk nature reserve lies east and adjacent to the 3M site (see Figure 1). During the evaluation of remedial options in 2008, monitoring of the water quality in the nature reserve was selected as the best remedial technique and 'a statistically increasing trend' was defined as trigger value. 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 July 2017.

The trend analysis was conducted as follows:

1. Data distribution was determined using the Shapiro-Wilks and Lillifors tests for each location;
2. Q-Q plots were developed for each location to evaluate if data outliers were present in the data;
3. Time series figures were created for each location showing PFOS concentrations over time; and
4. Trend analyses were conducted using ordinary least-square (OLS) regression on 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 wells L22 with a significantly decreasing trend and P116 with a significantly increasing trend in PFOS. None of the other wells showed any significant trend.

<u>Well L21</u>

Figure 1 (see Annex 2) shows well L21 with an apparent upward trend in the data set. 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 did not show a significant trend in PFOS (Table 2; see Annex 3.

Well L22

Figure 1 (see Annex 2) shows well L22 with an apparent downward trend in the data set. However, two intermediate values are substantially higher than the norm for this well. The Q-Q plots 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 (Figure 1; see Annex 2). The data set does not follow a defined distribution (i.e., is non-parametric), and the Mann-Kendall regression shows a significant downward trend in PFOS; However, the downward trend seems to consist of a difference between the earliest three samples and the final seven samples. The final seven samples (January 2016 through July 2017) appear to have similar concentrations in PFOS, as shown in Figure 1(see Annex 2). A trend analysis was conducted on just these seven samples, and no significant trend was observed.

Well L31

Figure 1 (see Annex 2) shows well L31 with no apparent trend in the data set, and the Q-Q plots did not indicate any data outliers. The 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 trend in the 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 data set. The Q-Q plots did not indicate any potential outliers. The data were normally distributed, and the OLS regress 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 data set. 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 data set. 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 a significant increasing trend in PFOS (Table 2; see Annex 3). However, the increasing trend seems to consist of a difference between the earliest eight samples and the final seven samples. The final seven samples (January 2016 through July 2017) appear to have similar concentrations in PFOS, as shown in Figure 1(see Annex 2). A trend analysis was conducted on just these seven samples, and no significant trend was observed.

3M vijver

Figure 1 (see Annex 2) shows the 3M vijver PFOS concentrations with no apparent trend in the data set.. The Q-Q plots (log-transformed) do not indicate data outliers. The data is log-normally distributed, and the OLS regression indicates no significant trend in PFOS (Table 2; see Annex 3).

Blokkersdijk vijver standard

Figure 1 (see Annex 2) shows the Blokkersdijk vijver standard_PFOS concentrations with no apparent trend in the data set. The Q-Q plot (log-transformed) did not indicate data outliers (Figure 2; see Annex 4). The data set was log-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 downward trend (L22) and one location showed a statistically significant upward trend (P116). Note that the trends observed in these two wells appear to result from a difference between the earliest and latest samples, and are not evident as a continuing trend in the data. There was no significant trend in PFOS from latest seven observations from these two wells. 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

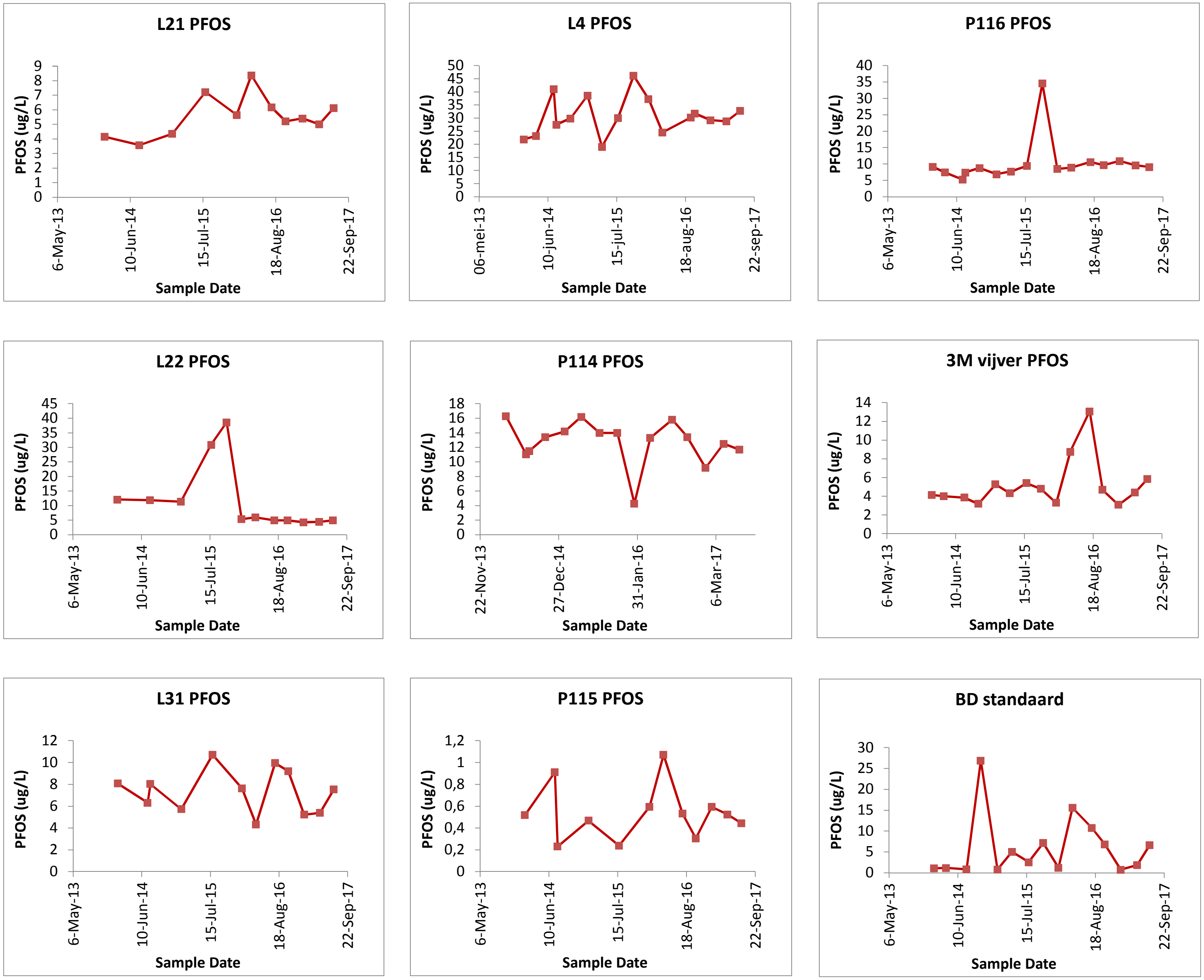
L21		L22		L31		L4		P114		P115		P116		3M vijver		BD standaard	
Date	PFOS (ug/L)	Date	PFOS (ug/L)	Date	PFOS (ug/L)	Date	PFOS (ug/L)	Date	PFOS (ug/L)	Date	PFOS (ug/L)	Date	PFOS (ug/L)	Date	PFOS (ug/L)	Date	PFOS (ug/L)
21-Jan-14	4,16	21-Jan-14	12,1	21-jan-14	8,07	21-jan-14	21,9	02-apr-14	16,3	21-jan-14	0,518	21-jan-14	9,17	23-jan-14	4,13	23-jan-14	1,06
30-Jul-14	3,58	30-Jul-14	11,9	14-jul-14	6,3	02-apr-14	23,2	14-jul-14	11,05	14-jul-14	0,911	02-apr-14	7,46	02-apr-14	4	02-apr-14	1,17
27-Jan-15	4,36	27-Jan-15	11,4	30-jul-14	8,03	14-jul-14	41,04	30-jul-14	11,5	30-jul-14	<0.464	14-jul-14	5,25	31-jul-14	3,85	31-jul-14	0,86
28-Jul-15	7,23	23-Jul-15	30,9	27-jan-15	5,72	30-jul-14	27,5	20-okt-14	13,4	27-jan-15	0,467	30-jul-14	7,42	21-okt-14	3,18	21-okt-14	26,9
16-jan-16	5,65	21-Oct-15	38,6	28-jul-15	10,7	20-okt-14	29,9	27-jan-15	14,2	23-jul-15	0,238	20-okt-14	8,77	27-jan-15	5,27	27-jan-15	0,792
06-apr-16	8,37	16-jan-16	5,39	16-jan-16	7,61	27-jan-15	38,6	21-apr-15	16,2	16-jan-16	0,594	27-jan-15	6,87	21-apr-15	4,3	21-apr-15	5,01
26-jul-16	6,17	06-apr-16	6	06-apr-16	4,32	21-apr-15	19	23-jul-15	14	06-apr-16	1,07	21-apr-15	7,71	27-jul-15	5,39	27-jul-15	2,54
11-okt-16	5,22	26-jul-16	4,935	26-jul-16	9,94	23-jul-15	30	21-okt-15	14	26-jul-16	0,532	23-jul-15	9,43	20-okt-15	4,78	20-okt-15	7,18
12-jan-17	5,41	11-okt-16	4,94	11-okt-16	9,2	21-okt-15	46,2	16-jan-16	4,29	12-okt-16	0,304	21-okt-15	34,6	16-jan-16	3,29	16-jan-16	1,23
14-apr-17	5,01	12-jan-17	4,29	12-jan-17	5,22	16-jan-16	37,3	06-apr-16	13,3	12-jan-17	0,593	16-jan-16	8,51	07-apr-16	8,73	07-apr-16	15,6
03-jul-17	6,13	14-apr-17	4,43	14-apr-17	5,39	06-apr-16	24,5	26-jul-16	15,8	14-apr-17	0,523	06-apr-16	8,88	28-jul-16	13,05	28-jul-16	10,75
		03-jul-17	4,97	03-jul-17	7,53	19-sep-16	30,2	12-okt-16	13,4	03-jul-17	0,443	27-jul-16	10,6	11-okt-16	4,68	11-okt-16	6,84
						11-okt-16	31,8	12-jan-17	9,21			11-okt-16	9,66	12-jan-17	3,08	12-jan-17	0,74
						11-jan-17	29,2	14-apr-17	12,5			12-jan-17	10,9	18-apr-17	4,39	18-apr-17	1,86
						14-apr-17	28,8	03-jul-17	11,7			14-apr-17	9,62	29-jun-17	5,82	29-jun-17	6,62
						03-jul-17	32,8					03-jul-17	9,08				

All data was generated by 3M
Data in green are recently added since the last trend analysis
Data highlighted in yellow are non-detect values

Annex 2

Figure 1: Time series graphs of PFOS

Figure 1. Time series plots of PFOS data.



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	NP	N	N	N	N	NP	LN	LN
OLS trend	0,185	0,21	0,708	0,604	0,389	0,983	0,55	0,29	0,93
Ln-OLS trend	0,109	0,037	0,641	0,458	0,501	0,821	0,246	0,289	0,37
Mann-Kendall Trend	0,175	0,006	0,225	0,196	0,138	NA	0,011	0,138	0,244

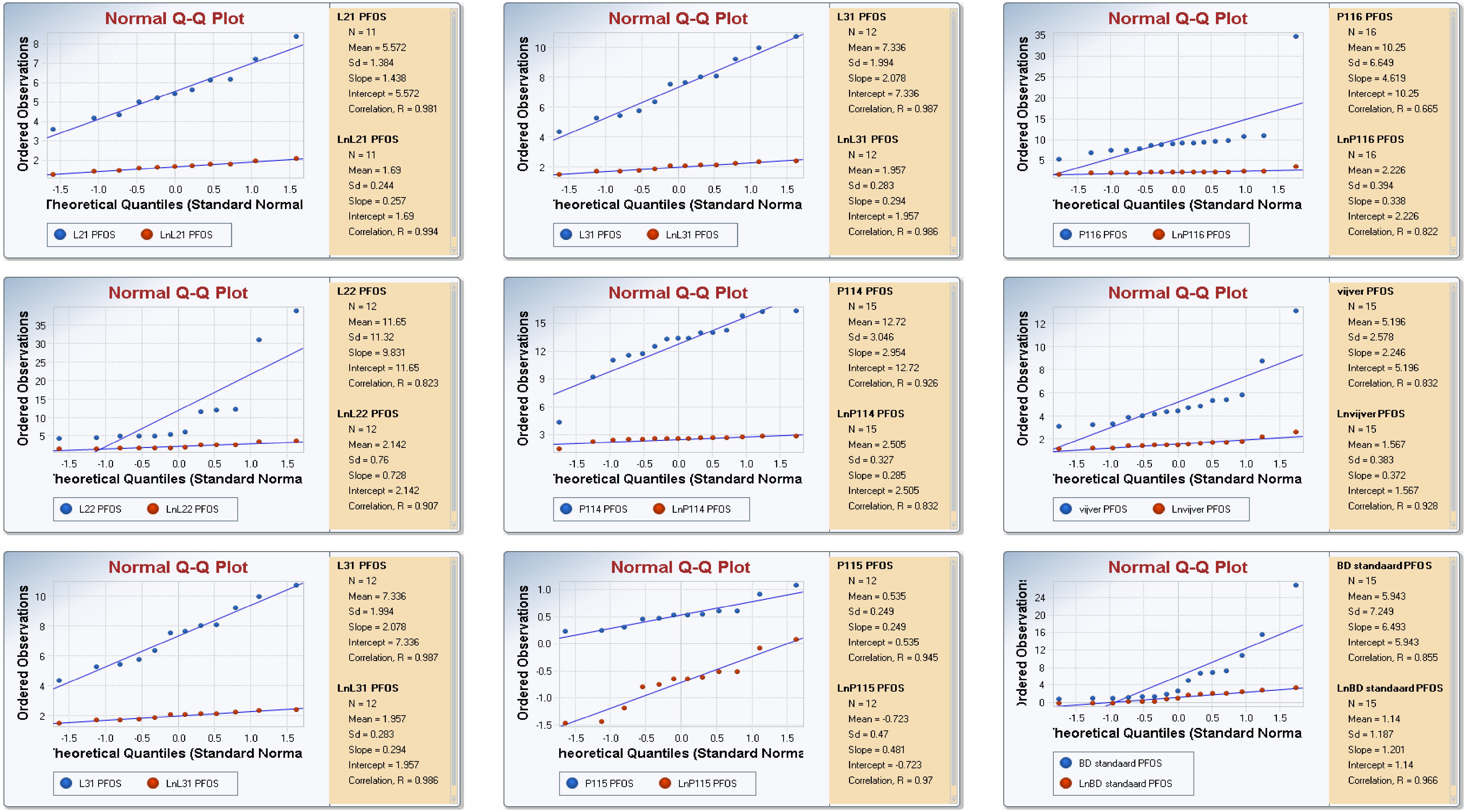
N = normal
LN = lognormal
NP = non-parametric

indicates significant trend

Annex 4

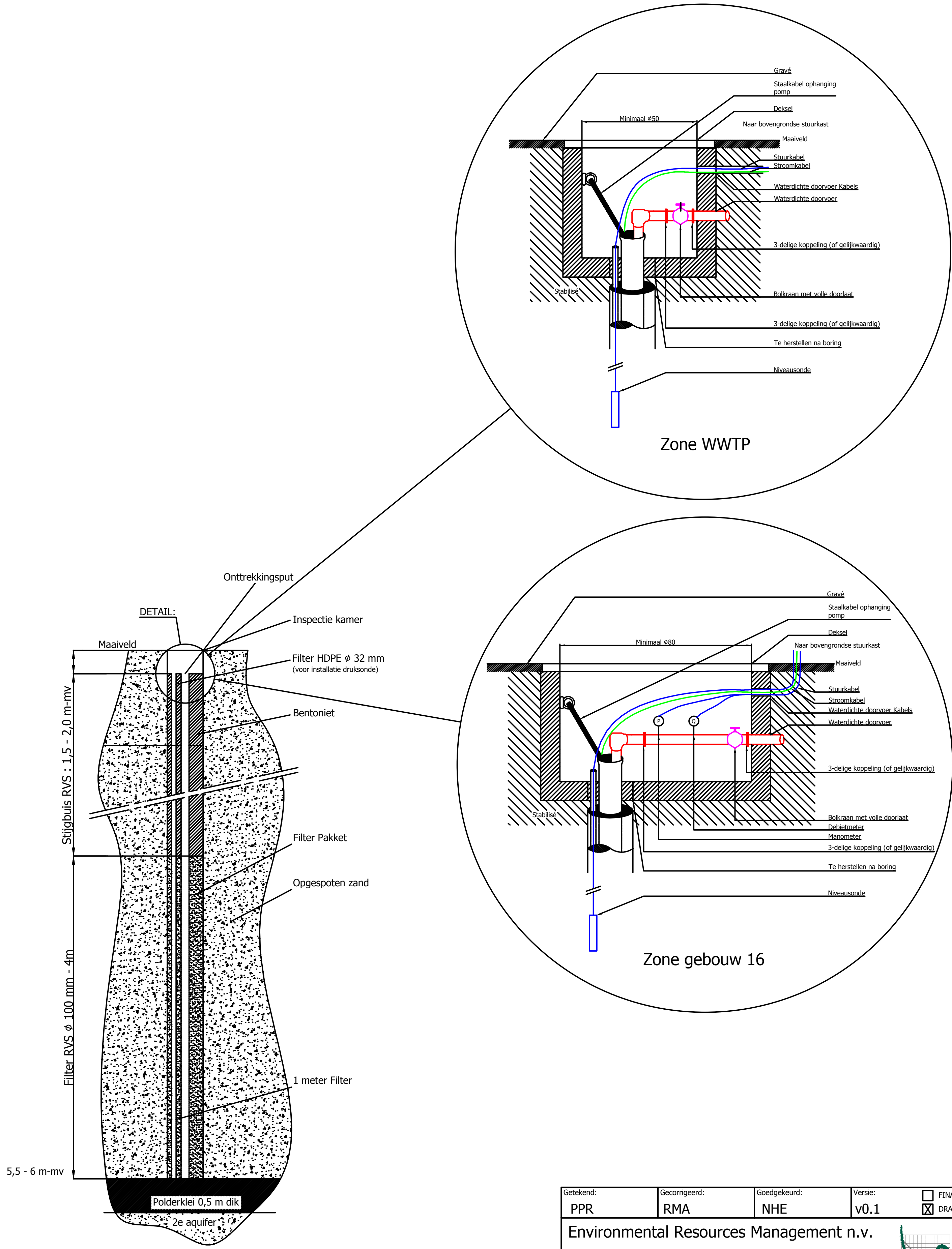
Figure 2: Q-Q plots (all data)

Figure 2. Q-Q plots for the 3M data sets.



Bijlage 6

Schematische weergave pompput en inspectiekamer



Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
PPR	RMA	NHE	v0.1	☐ FINAL ☒ DRAFT

Environmental Resources Management n.v.

Cantersteen 47
1000 Brussel
Tel.: 02/550.02.80
Fax.: 02/550.02.99

Posthoflei 5
2600 Antwerpen
Tel.: 03/287.36.50
Fax.: 03/287.36.79

Place de l'Université 16
1348 Louvain-la-Neuve
Tel.: 010/48.35.39
Fax.: 010/48.35.36

ERM

Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.: 0341743	Titel: 7de Tussentijds verslag Bodemsaneringswerken
Kaart: 10	Beschrijving: Doorsnede Pompput en inspectiekamer
Fase: TTV-7	

Schaal:	Formaat:	Bestandsnaam:	Datum:
-/-	A4	3M-Doorsnedes.dwg	22/12/2016

Bijlage 7

Documenten schadegeval hydraulische olie

Melding van de vaststelling van bodemverontreiniging

SAMEN MAKEN WE MORGEN MOOIER

OVAM

//

Openbare Vlaamse Afvalstoffenmaatschappij
Schadegevallen
Stationsstraat 110, 2800 MECHELEN
T 015 284 488
F 015 284 408
www.ovam.be
schade@ovam.be

In te vullen door de behandelende afdeling

Referentie OVAM

dossiernummer

ontvangstdatum

Waarvoor dient dit formulier?

Met dit formulier kunt u aan de OVAM melden dat er een geval van bodemverontreiniging werd vastgesteld.
Meer informatie vindt u op de website van de OVAM: www.ovam.be/schadegevallen.
Het meldingsformulier wordt bij voorkeur via email (schade@ovam.be) aan de OVAM overgemaakt. Doorfaxen of opsturen met de post blijft uiteraard ook mogelijk (zie onze contactgegevens bovenaan dit formulier).

Identiteit van de melder of vaststeller

1 Vul hieronder uw persoonlijke gegevens in.

(bedrijfs)naam	3M Belgium bvba
voor- en achternaam	Charlotte Tack
straat en nummer	Canadastraat 11
postnummer en gemeente	2070 Zwijndrecht
telefoonnummer	[redacted]
e-mailadres	[redacted]

hoedanigheid

☐ eigenaar ☐ exploitant ☐ gebruiker
☐ schadelijder ☐ politie ☐ gemeente
☐ bodemsaneringsdeskundige ☐ verzekeraar of expert
☒ milieucoördinator ☐ andere:

2 Wanneer is de bodemverontreiniging ontstaan?

datum dag 05 maand 07 jaar 2017

☐ Onbekend

3 Wanneer werd de bodemverontreiniging vastgesteld?

datum dag 05 maand 07 jaar 2017

Vlaamse overheid

4 Is de grond waar de bodemverontreiniging is ontstaan (het bronperceel), bekend.

☒ ja. *Ga naar vraag 5.*

☐ nee. *Ga naar vraag 12.*

Gegevens van het bronperceel

Het bronperceel is bekend

5 Betreft het een particulier terrein?

☐ ja ☒ Nee

6 Betreft het een terrein waarop een inrichting gevestigd is vallende onder klasse 1?

☒ ja ☐ Nee

Vul de adresgegevens van het bronperceel in.

straat en nummer: Canadastraat 11

postnummer en gemeente: 2070 Zwijndrecht

kadastrale gegevens: afdeling 1 sectie A grondnummer 467E

7 Vul de eigenaarsgegevens van het bronperceel in.

naam van de **exploitant**: 3M Belgium bvba

adres van de exploitant: Hermeslaan 7
1831 Diegem

telefoonnummer:

naam van de **gebruiker**:

adres van de gebruiker:

telefoonnummer:

naam van de **eigenaar**: 3M Belgium bvba

adres van de eigenaar: Hermeslaan 7
1831 Diegem

telefoonnummer:

8 Veroorzaakt de bodemverontreiniging van het bronperceel ook verontreiniging op de buurtpercelen?
Aanwijzing.

☐ Ja. *Ga naar vraag 7.*

☒ Nee*. Ga naar vraag 8.*

9 Vul de gegevens van de verontreinigde buurtpercelen in..

naam van de eigenaar	Adres	Kadastrale gegevens		
		afdeling	sectie	grondnr.

10 Kruis de oorzaak van de bodemverontreiniging aan.

- ☐ lek in leiding tank voor verwarming gebouw
- ☐ lek in bovengr. tank dat gebruikt wordt/werd voor verwarming gebouw
- ☐ afspuiten **hoge druk** asbesthoudend dak
- ☐ overvulling
- ☐ lek in leiding andere dan hierboven vermeld
- ☐ lek in ondergr. tank dat gebruikt wordt/werd voor verwarming gebouw
- ☐ brand met asbesthoudend materiaal
- ☐ lek
- ☐ ongeval met voertuig
- ☐ lek in tank andere dan hierboven vermeld
- ☐ breuk in afvoer
- ☐ morsen
- ☐ vandalisme
- ☒ andere oorzaak, namelijk: Slang van graafmachine met hydraulische olie gesprongen

11 Kruis aan welk product er vrijgekomen is.
Ga daarna naar vraag 19.

- ☐ benzine
- ☐ diesel
- ☐ stookolie
- ☐ asbest
- ☐ solventen
- ☐ smeermiddelen
- ☐ zuren
- ☐ pcb-houdende olie
- ☒ ander product, namelijk: Hydraulische olie

Het bronperceel is niet bekend

12 Vul de adresgegevens in van het perceel waar de verontreiniging het eerst werd vastgesteld.

straat en nummer ....

postnummer en gemeente ....

kadastrale gegevens afdeling .... sectie .... grondnummer ....

13 Vul de eigenaarsgegevens in van het perceel waar de verontreiniging het eerst werd vastgesteld.

naam van de **exploitant** ....

adres van de exploitant ....

....

telefoonnummer: ....

klasse 1-bedrijf ☐ ja ☐ nee

naam van de **gebruiker** ....

adres van de gebruiker

telefoonnummer:

naam van de **eigenaar**

adres van de eigenaar

telefoonnummer:

14 Is de bodemverontreiniging ook vastgesteld op buurtpercelen?

- ☐ Ja. *Ga naar vraag 15.*
- ☐ nee. *Ga naar vraag 16.*

15 Vul de gegevens van de buurtpercelen in.

naam van de eigenaar	**adres**	**Kadastrale gegevens**		
		afdeling	**sectie**	**grondnr.**

16 Welke onderzoeken of inspecties werden op de percelen uitgevoerd?

- ☐ een buurtonderzoek
- ☐ een rioolinspectie

17 Zijn er in de buurt potentiële bronnen voor de bodemverontreiniging?

- ☐ Ja. *Ga naar vraag 17.:*
- ☐ Nee. *Ga naar vraag 18.*

18 Vul de gegevens in van de locaties waar zich potentiële bronnen voor de bodemverontreiniging bevinden.
Een voorbeeld van een potentiële bron is een boven- of ondergrondse tank op de locatie.

naam van de eigenaar	**adres**	**Kadastrale gegevens**		
		afdeling	**sectie**	**grondnr.**

19 Kruis aan welk product er vrijgekomen is.

- ☐ benzine
- ☐ Diesel
- ☐ Stookolie
- ☐ asbest
- ☐ solventen
- ☐ smeermiddelen
- ☐ Zuren
- ☐ pcb-houdende olie
- ☐ ander product, namelijk:

Genomen maatregelen tegen de bodemverontreiniging

- ☒ de verontreinigde grond werd afgegraven
- ☒ de bron of het lek werd gestopt
- ☐ er was een interventie door de brandweer of de politie
- ☐ de verzekeringsmaatschappij werd ingelicht
- ☐ er werden andere maatregelen genomen, namelijk:
- ☒ er werd een bodemdeskundige aangesteld, namelijk: Environmental Resources Management nv - ERM nv
- ☐ er werden absorberende korrels gestrooid
- ☐ de drijflaag werd afgezogen
- ☒ er werden stalen genomen en analyses uitgevoerd
- ☐ er werd puur product weggenomen
- ☒ Op 5 juli 2017 is door het springen van een slang met hydraulische olie ca. 10 tot 20 liter product op grind en stelconplaten terecht gekomen.
  Op 6 juli 2017 is ter hoogte van de geimpacteerde zone (ca. 2m bij 3m) de halfverharding (grind) verwijderd en een beperkte hoeveelheid grond tot circa 20 cm diep afgegraven. Hierbij zijn door de erkend bodemsaneringsdeskundige ERM nv grondstalen genomen van de afgegraven grond, alsook van de bodem en wanden van de ontgravingsput.
  De verwijderde kiezels en afgegraven grond zijn op een folie gelegd en afgedekt met een zeil om verspreiding naar de grond en/of het grondwater te voorkomen.
  Twee bodemstalen, twee wandstalen en een mengstaal van de afgegraven grond zijn naar het door OVAM erkend laboratorium SGS gestuurd voor analyse op minerale olie.
  In functie van deze resultaten zullen de eventueel verder te nemen acties met 3M en OVAM worden besproken.
- ☐ ……

Ondertekening

20 Vul de onderstaande verklaring in.

Ik verklaar dat alle gegevens in deze melding naar waarheid zijn ingevuld.

datum dag 07 maand 07 jaar 2017

handtekening ……

SAMEN MAKEN WE MORGEN MOOIER
OVAM
Openbare Vlaamse
Afvalstoffenmaatschappij
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B-2800 Mechelen
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www.ovam.be

DS-LB-SDW-20170403518

3M
Hermeslaan 7
1831 DIEGEM

UW BERICHT VAN •
UW KENMERK •
BIJLAGEN • Besluit
MECHELEN • 12.07.2017

AFDELING • Doelgroepgericht Saneren
TEAM • Lokale en Bovenlokale besturen
CONTACTPERSOON • Sofie De Wit ([redacted]
ONS KENMERK • DS-LB-SDW-20170403518 (Dossiernummer: 732)

Zwijndrecht – Canadastraat 11: melding schadegeval

Geachte heer,
Geachte mevrouw,

Op 7 juli 2017 werd de OVAM via een melding schadegeval in kennis gesteld van een mogelijke bodemverontreiniging ter hoogte van Canadastraat 11 in Zwijndrecht. Dit bronperceel is kadastraal bekend als:

Gemeente-nummer	Afdeling	Sectie	Grondnr.	Bisnr.	Exp.1	Exp.2
11056	ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	467		E	

De verontreiniging is ontstaan naar aanleiding van een schadegeval waarbij door het springen van een slang van een graafmachine een hoeveelheid hydraulische olie is vrijgekomen. Het schadegeval heeft zich voorgedaan op een grond:

— waarop een inrichting gevestigd is die krachtens titel V van het decreet van 5 april 1995 houdende algemene bepalingen inzake milieubeleid ingedeeld wordt in de eerste klasse

Op grond van het decreet van 27 oktober 2006 betreffende de bodemsanering en de bodembescherming (Bodemdecreet) heeft de OVAM de bevoegdheid om maatregelen op te leggen ter behandeling van de bodemverontreiniging die ontstaan is ingevolge dit schadegeval. Als bijlage vindt u het besluit waarbij de OVAM die maatregelen oplegt.

De verplichting om over te gaan tot die maatregelen ligt, conform artikel 80 van het Bodemdecreet, bij:

1° de exploitant, vermeld in titel V van het decreet van 5 april 1995 houdende algemene bepalingen inzake milieubeleid, als op de grond waar de bodemverontreiniging tot stand is gekomen een inrichting of activiteit gevestigd is die vergunnings- of meldingsplichtig is krachtens titel V;

2° bij gebrek aan exploitant, de gebruiker van de grond waar de bodemverontreiniging tot stand kwam;

3° bij gebrek aan exploitant en gebruiker, de eigenaar van de grond waar de bodemverontreiniging tot stand kwam.

In uw hoedanigheid van exploitant van de grond waar het schadegeval zich heeft voorgedaan, bent u krachtens artikel 80 van het Bodemdecreet ertoe gehouden de maatregelen binnen de vastgestelde termijn uit te voeren onder leiding van een erkende bodemsaneringsdeskundige. Een lijst met alle erkende bodemsaneringsdeskundigen kan u terugvinden via www.ovam.be/schadegevallen. Gelieve uw deskundige in te lichten over de inhoud van deze brief en het bijgevoegde besluit.

Als u de maatregelen niet of in onvoldoende mate uitvoert, kan de OVAM de maatregelen zelf uitvoeren en de kosten ervan op u verhalen. Met behoud van de toepassing van het Strafwetboek zijn, in voorkomend geval, de strafbepalingen van artikel 173 van het Bodemdecreet van toepassing.

Als de opgelegde termijn van 180 dagen overschreden wordt, zoals bepaald in het besluit, kan u als saneringsplichtige verplicht worden om een beschrijvend bodemonderzoek en eventuele bodemsanering uit te voeren.

Binnen zestig dagen nadat u het besluit ontvangen hebt, kunt u met een verzoekschrift een vordering tot schorsing of nietigverklaring indienen bij de Raad van State. U verstuurt dat verzoekschrift aangetekend.

Hoogachtend,

20170403518

Ann Cuyckens
Afdelingshoofd

Bijlagen:

— het besluit tot het nemen van maatregelen voor de bodemverontreiniging naar aanleiding van het schadegeval

Kopie:

3M Belgium NV
Charlotte Tack
Milieucoördinator
Canadastraat 11
2070 ZWIJNDRECHT

GEMEENTE ZWIJNDRECHT
Milieudienst
Binnenplein 1
2070 ZWIJNDRECHT

SAMEN MAKEN WE
MORGEN MOOIER
OVAM

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Afvalstoffenmaatschappij
Stationsstraat 110
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www.ovam.be

Besluit van de OVAM Maatregelen tot behandeling schadegeval

Naam:	Sofie De Wit
Dossier:	732
Datum:	12.07.2017

Zwijndrecht – Canadastraat 11

Gelet op het Decreet van 27 oktober 2006 betreffende de bodemsanering en de bodembescherming (Bodemdecreet), artikel 74 tot en met 82;

Overwegende dat op de grond gelegen aan de Canadastraat 11 te Zwijndrecht er zich een schadegeval heeft voorgedaan waarbij door het springen van een slang van een graafmachine een hoeveelheid hydraulische olie is vrijgekomen. De grond is kadastraal gekend als:

Gemeente-nummer	Afdeling	Sectie	Grondnr.	Bisnr.	Exp.1	Exp.2
11056	ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	467		E	

De exploitant op deze grond is:

Perceel	Gegevens exploitant
467 E	3M Belgium bvba, Hermeslaan, 7, 1831, Machelen

Overeenkomstig artikel 75 van het Bodemdecreet is de OVAM de bevoegde overheid voor het betreffende schadegeval, omdat het schadegeval plaatsvond op een grond:

— waarop een inrichting of activiteit gevestigd is die krachtens titel V van het decreet van 5 april 1995 houdende algemene bepalingen inzake milieubeleid ingedeeld wordt in de eerste klasse

De OVAM is van oordeel dat maatregelen noodzakelijk zijn om de (mogelijke) bodemverontreiniging ingevolge het schadegeval te behandelen. De maatregelen tot behandeling van de schadegevallen kunnen worden uitgevoerd binnen 180 dagen na de melding of vaststelling van het schadegeval. Het schadegeval valt dan ook onder het toepassingsgebied van de schadegevallenregeling, vermeld in artikel 74 tot en met artikel 82 van het Bodemdecreet.

Op basis van de beschikbare gegevens zijn volgende maatregelen al genomen:

— de bron of het lek werden gestopt;
— er werd een bodemsaneringsdeskundige aangesteld;
— de verontreinigde grond werd ontgraven;
— stalen en analyses werden uitgevoerd;

Op basis van de verkregen gegevens dienen volgende maatregelen genomen te worden om de bodemverontreiniging te behandelen vóór 6 januari 2018:

— afhankelijk van de bevindingen van de erkende bodemsaneringsdeskundige: overgaan tot bijkomende ontgraving van de verontreinigde grond, onder leiding van de erkende bodemsaneringsdeskundige;
— de vervuilde ontgraven grond laten verwerken in een centrum voor grondreiniging;
— de bewijzen van afvoer van de verontreinigde grond en/of de verwerkingsattesten van de verontreinigde grond verwerven;
— controlestaalnames van bodem en grondwater (putbodem en –wanden) laten uitvoeren door een erkende bodemsaneringsdeskundige, conform de geldende richtlijnen van de OVAM;
— de saneringswerkzaamheden uitvoeren volgens de geldende codes van goede praktijk;
— het evaluatierapport, opgesteld door een bodemsaneringsdeskundige conform de betreffende richtlijn, aan de OVAM via het webloket bezorgen;
— de OVAM informeren binnen de termijn vermeld in dit besluit of tijdig in te lichten bij eventuele vertragingen;
— de bodemsaneringsdeskundige dient er op toe te zien dat de opgelegde maatregelen overeenkomen met de effectief uit te voeren (vergunningsplichtige) maatregelen. Indien nodig vraagt hij een nieuw aangepast besluit op aan de bevoegde overheid.

Als die maatregelen handelingen, inrichtingen of activiteiten omvatten die meldings- of vergunningsplichtig zijn krachtens titel V van het decreet van 5 april 1995 houdende algemene bepalingen inzake milieubeleid of krachtens titel IV, hoofdstuk II, van de Vlaamse Codex Ruimtelijke Ordening, geldt de beslissing als meldingsakte of omgevingsvergunning.

Besluit

Artikel 1. Het schadegeval dat zich heeft voorgedaan op 5 juli 2017 op de grond, kadastraal bekend als:

Gemeente-nummer	Afdeling	Sectie	Grondnr.	Bisnr.	Exp.1	Exp.2
11056	ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	467		E	

en gelegen in de Canadastraat 11 te Zwijndrecht, wordt gekwalificeerd als een schadegeval dat valt onder het toepassingsgebied van de schadegevallenregeling, vermeld in artikel 74 tot en met 82 van het decreet van 27 oktober 2006 betreffende de bodemsanering en de bodembescherming.

Art. 2. De volgende maatregelen ter behandeling van de (mogelijke) bodemverontreiniging ingevolge het schadegeval op de grond, vermeld in artikel 1, moeten **vóór 6 januari 2018** worden uitgevoerd :

- afhankelijk van de bevindingen van de erkende bodemsaneringsdeskundige: overgaan tot bijkomende ontgraving van de verontreinigde grond, onder leiding van de erkende bodemsaneringsdeskundige;
- de vervuilde ontgraven grond laten verwerken in een centrum voor grondreiniging;
- de bewijzen van afvoer van de verontreinigde grond en/of de verwerkingsattesten van de verontreinigde grond verwerven;
- controlestaalnames van bodem en grondwater (putbodem en –wanden) laten uitvoeren door een erkende bodemsaneringsdeskundige, conform de geldende richtlijnen van de OVAM;
- de saneringswerkzaamheden uitvoeren volgens de geldende codes van goede praktijk;
- het evaluatierapport, opgesteld door een bodemsaneringsdeskundige conform de betreffende richtlijn, aan de OVAM via het webloket bezorgen;
- de OVAM informeren binnen de termijn vermeld in dit besluit of tijdig in te lichten bij eventuele vertragingen;
- de bodemsaneringsdeskundige dient er op toe te zien dat de opgelegde maatregelen overeenkomen met de effectief uit te voeren (vergunningsplichtige) maatregelen. Indien nodig vraagt hij een nieuw aangepast besluit op aan de bevoegde overheid.

Deze maatregelen moeten worden uitgevoerd door 3M.

Art. 3. De maatregelen, vermeld in artikel 2, kunnen ten allen tijde door een beslissing van de OVAM aangepast of opgeheven worden.

Hoogachtend,

20170403531

Ann Cuyckens
Afdelingshoofd

	DAGBOEK MILIEUKUNDIG TOEZICHT*
	Volgnummer en terreinbezoek : 1
	Datum : 06/07/2017
	Projectidentificatie : 0398993 *OVAM dossier: 732*

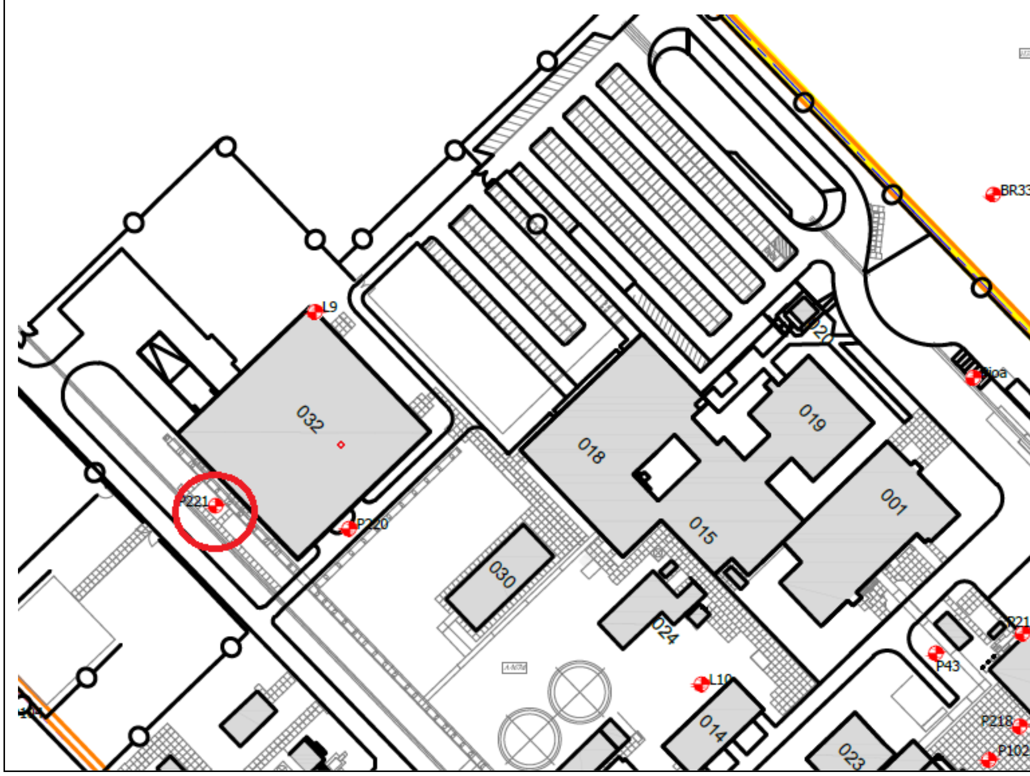
Locatie: 3M	**Weersomstandigheden: Warm, 28°C**
Straat en huisnummer: Canadastraat 11 Gemeente: Zwijndrecht	*Spill Hydraulische olie ten ZW van Gebouw 32*

Aanwezigen en hun taak

Naam	Functie	Firma	Uur in	Uur uit
Erik Boeckx	Milieukundige begeleiding	ERM	11:00	13:00

Beschrijving van de lopende bodemsaneringswerken op het ogenblik van de controle

Op 6 juli 2017 is er een spill geweest op de site van 3M te Zwijndrecht ten gevolge van een breuk in een hydraulische leiding van een kraan. Hierbij kwam een 10-tal liter (geraamd) hydraulische olie vrij. De MSDS sheet is als bijlage toegevoegd. De olie kwam deels terecht op Stelcon-dals en deels op een met kiezel verharde ondergrond. De geïmpacteerde zone situeert zich ten zuidwesten van gebouw 32.



* conform OVAM document "Bodemsaneringswerken en nazorg, standaardprocedure, oktober 2014"

Het vrijgekomen product op de steenslag verharding is afgegraven (2x2m) tot een diepte van 20 cm. Er was een zichtbare donkere verkleuring en afwijkende geur. De vrijgekomen grond met steenslag (+/- 1m³) is tijdelijk op een folie geplaatst. Hiervan is door ERM een mengstaal genomen. Van de putbodem en de putwanden zijn eveneens stalen genomen. Aangezien 2 wanden gevormd worden door de Stelcon dals, zijn deze niet bemonsterd. Van de putbodem is eveneens een dieper monster genomen. Van de afgegraven grond is eveneens een mengstaal genomen. Na ontgraving was er geen verkleuring of afwijkende geur vast te stellen.

Health & Safety
Geen opmerkingen

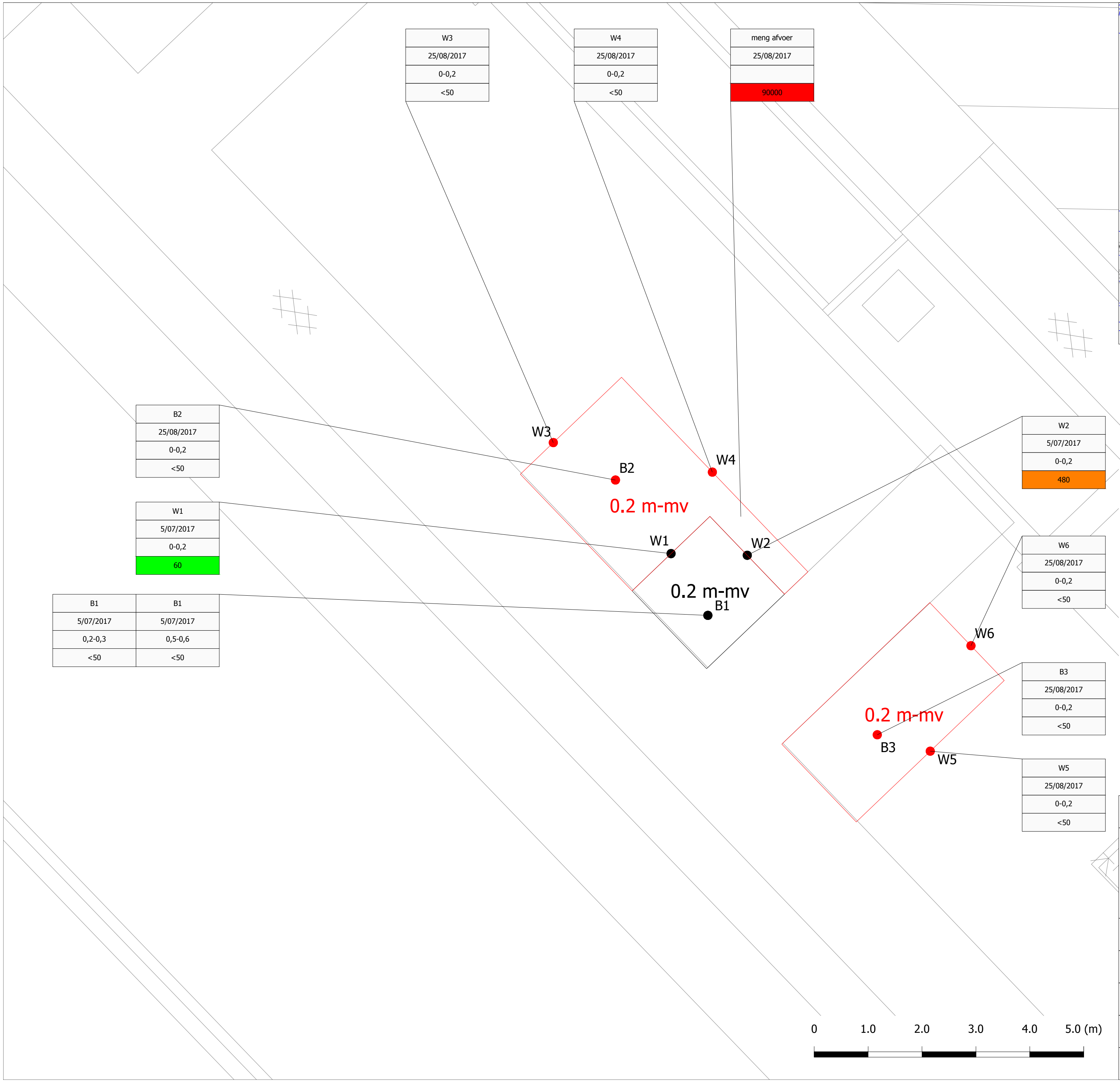
Milieukundige vaststellingen	
Visueel Zie foto-reportage.	*Monstername(s) en Analyse(s)* – zie plan voor locatie monsternamepunten • B1 (20-30) AG17145539 • B1 (50-60) AG1714554A • W1(0-20) AG17145517 • W2(0-20) AG17145528 • Meng Afvoer AG1714558E

Bindend advies en/of opmerkingen

1. De noodzaak voor eventuele verdere acties dient geëvalueerd te worden op basis van de analyseresultaten van de grondstalen.
2. Het incident dient gemeld te worden aan OVAM (lopende)

Voor de bodemsaneringsdeskundige	**Voor de opdrachtgever**
Naam Erik Boeckx	Handtekening en naam

Foto-reportage



Legende:

- Grondstaal (05/07/2017)
- Ontgravingsvak (05/07/2017)
- Grondstaal (25/08/2017)
- Ontgravingsvak (25/08/2017)

W5	Staalnamepunt
25/08/2017	Staalnamedatum
0-0,2	Staalnamediepte (m-mv)
<50	Hydraulische olie (mg/kg ds)

(green)	SW
(orange)	RW
(red)	BSN

Drafted by:	Corrected by:	Approved by:	Version:	
NVE	TMA	NHE	v1.0	X FINAL X DRAFT

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ERM

Client / Project Name: 3M Belgium bvba

Location: Zwijndrecht

Project Number: 0435617

Title: Periodiek Oriënterend Bodemonderzoek

Kaart: 8

Description: Schadegeval Minerale Olie 05/07/2017

Phase: POBO

Scale: 1:100 | Paper Size: A3 | File Name: 3M Zwijndrecht-2017.dwg | Date: 08/03/2018

Foto 1 : Situering achter Gebouw 32

Foto 2: Ontgravingszone

Foto 3: Ontgravingsvak

Foto 4: Ontgravingszone

Foto 5: Product

Foto 6: Tijdelijk depot

		
Foto 1. Eerste fase ontgraving – restanten spil	Foto 2. Eerste fase ontgraving– restanten spil	Foto 3. Restanten spil
		
Foto 4. Tweede fase ontgraving (25/08/17)	Foto 5. Tweede fase ontgraving (25/08/17)	Foto 6. Afwerking - aangevuld

Toetsing analyseresultaten - Grond

												Toetsingswaarden			
Perceelnummer															
Onderzochte zone												3,0% klei	1,0% OM		
Boornummer	B1	B1	W1	W2	meng afvoer	W3	W4	W5	W6	B2	B3				
Datum staalname	5/07/2017	5/07/2017	5/07/2017	5/07/2017	5/07/2017	25/08/2017	25/08/2017	25/08/2017	25/08/2017	25/08/2017	25/08/2017				
Zintuiglijk waarneembare verontr.												**SW**	**RW**	**80% BSN**	**BSN - 5**
Interval van waarneming															
Staalnamediepte (m-mv)	0,2-0,3	0,5-0,6	0-0,2	0-0,2		0-0,2	0-0,2	0-0,2	0-0,2	0-0,2	0-0,2				
Droge stofgehalte (%)	94	94,2	85,8	86,8	88,8	87,5	83,9	91,5	93,5	89,7	90,2	-	-		-
Minerale olie (mg/kg ds)	<50	<50	*60*	*480*	***90000***	<50	<50	<50	<50	<50	<50	50,0	300,0	600,0	750,0

(1) = normen voor EOX: SW = detectielimiet en BSN = interne OVAM-norm

\- = geen normen beschikbaar in Vlarebo

VERWERKINGSCERTIFICAAT

Bremcon nv verklaart dat de hierna genoemde afvalstof gerecycleerd of gereinigd werd op haar verwerkingscentrum te Zwijndrecht (Burcht).

De recyclage of reiniging werd uitgevoerd volgens de OVAM Codes van Goede Praktijk en de geldende regelgevingen VLAREMA en VLAREBO.

Aanbieder:	Van Goethem en co bvba	
Afvalstof:	verontreinigde grond	
Herkomst:	3M	
Aanvoer:	augustus 2017	
Hoeveelheid:	8,60	ton

Opgemaakt te Zwijndrecht (Burcht), op 18 september 2017

ing. Chris Heymans
Manager grondreinigingscentrum

Bremcon nv
Maatschappelijke zetel:
Kruibeeksesteenweg 162
2070 Zwijndrecht (Burcht)

Telefoon: +32 (03) 253.25.50
Telefax: +32 (03) 253.25.59
E-mail: info@bremcon.be
website: www.bremcon.be

Exploitatiezetel:
Kruibeeksesteenweg 154
2070 Zwijndrecht (Burcht)

Ond. Nr. 0459.343.302
OVAM-nummer: 10452
IBAN BE58 0014 5451 3279
BIC GEBABEBB

Beste mevrouw Tack,

Wij kunnen hiermee akkoord gaan.

Zowel de rapportage omtrent dit schadegeval (hydraulische olie 5/7/17)- dat aangepakt werd werd binnen de schadegevallenprocedure- alsook het incident met ricinusolie van 30/8/17 (waarvoor de schadegevallenprocedure niet werd opgestart) dient dan opgenomen te worden in het periodiek oriënterend bodemonderzoek van 2017. Eventuele verdere contactname omtrent deze incidenten dient dan rechtstreeks te gebeuren met mijn collega Filip Collet.

Met vriendelijke groeten,

Sofie De Wit | Afdeling Doelgroepgericht Saneren| Programma Lokale en Bovenlokale Besturen
OVAM | Stationsstraat 110 - 2800 MECHELEN
T 015 284 471 | F 015 284 408
E [redacted] | www.ovam.be

Van: "Charlotte Tack" <[redacted]>
Aan: [redacted]
Cc: "Filip Collet" <[redacted]>, "anne diels" <[redacted]>, "Nynke De Schutter" <[redacted]>
Verzonden: Vrijdag 13 oktober 2017 13:28:41
Onderwerp: Schadegeval hydraulische olie

Beste mevrouw De Wit,

Op 7 juli 2017 werd door 3M Belgium bvba aan OVAM een officiële melding gedaan van een schadegeval met hydraulische olie op het terrein van 3M te Zwijndrecht.
Het schadegeval vond plaats op 5 juli 2017, de melding werd conform de vereisten binnen 14 dagen aan OVAM gemeld (uw referentie DS-LB-SDW-20170403518 – dossiernummer 732) .

Op het moment van de melding was reeds –als voorzorgsmaatregel– de bulk van de grondverontreiniging afgegraven en waren er controle stalen van de bodem en de wand genomen voor analyse op minerale olie onder begeleiding van de erkende bodemdeskundige ERM nv. Uit deze controlestalen bleek dat op 1 locatie nog een overschrijding van de richtwaarde van minerale olie werd vastgesteld. Visueel werd er ook op een andere locatie in deze zone nog verontreiniging vastgesteld. In overleg met de bodemdeskundige (ERM) is vervolgens een bijkomende ontgraving uitgevoerd. De grondconcentraties gemeten in de controlestalen na de bijkomende ontgraving zijn alle lager dan de richtwaarde. Op basis van de grondanalyses is daarna besloten dat het niet nodig is verder grond af te graven. Als bijlage een plan met aanduiding van de ontgraven zone, alsook de analysecertificaten.

In uw schrijven van 12 juli 2017 vermeldt u dat een evaluatierapport ingediend moet worden voor 6 januari 2018. Omwille van de periodieke onderzoeksplicht die op het terrein van 3M rust, dient in 2017 ook een periodiek oriënterend bodemonderzoek uitgevoerd te worden. Kunt u akkoord gaan dat de rapportage van dit schadegeval opgenomen wordt in het periodiek oriënterend bodemonderzoek, zodat er geen afzonderlijk evaluatierapport opgemaakt dient te worden?

Alvast bedankt voor uw reactie.

Vriendelijke groeten,

3M Science. Applied to Life.™

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

3M.com

Vlaanderen is materiaalbewust

Deel 4: KAARTMATERIAAL

Eerste gefaseerd bodemsaneringsproject -
Achtste tussentijds verslag bodemsaneringswerken
Periode juli 2016 – juli 2017

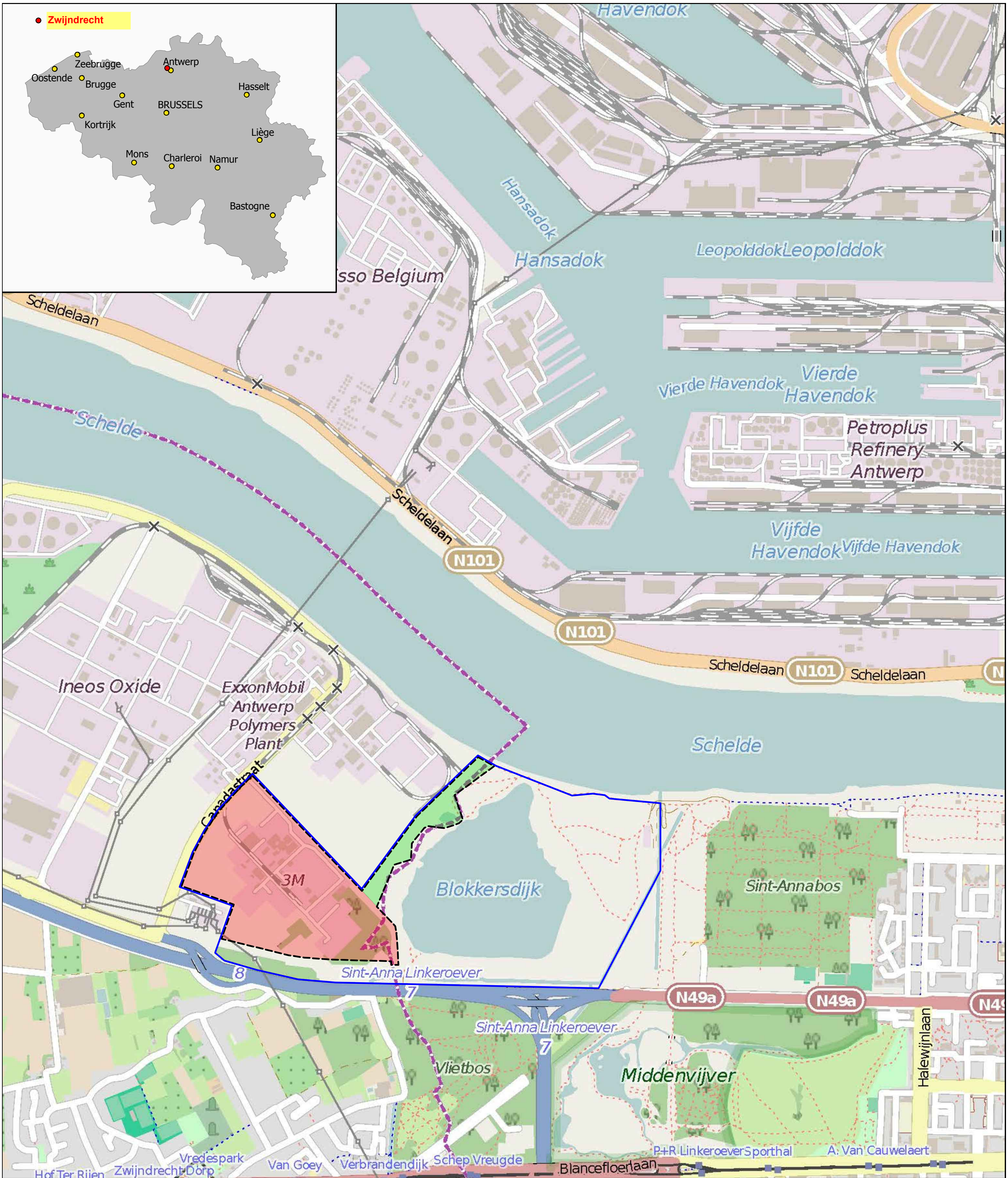
3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

14 augustus 2018

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)

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ERM

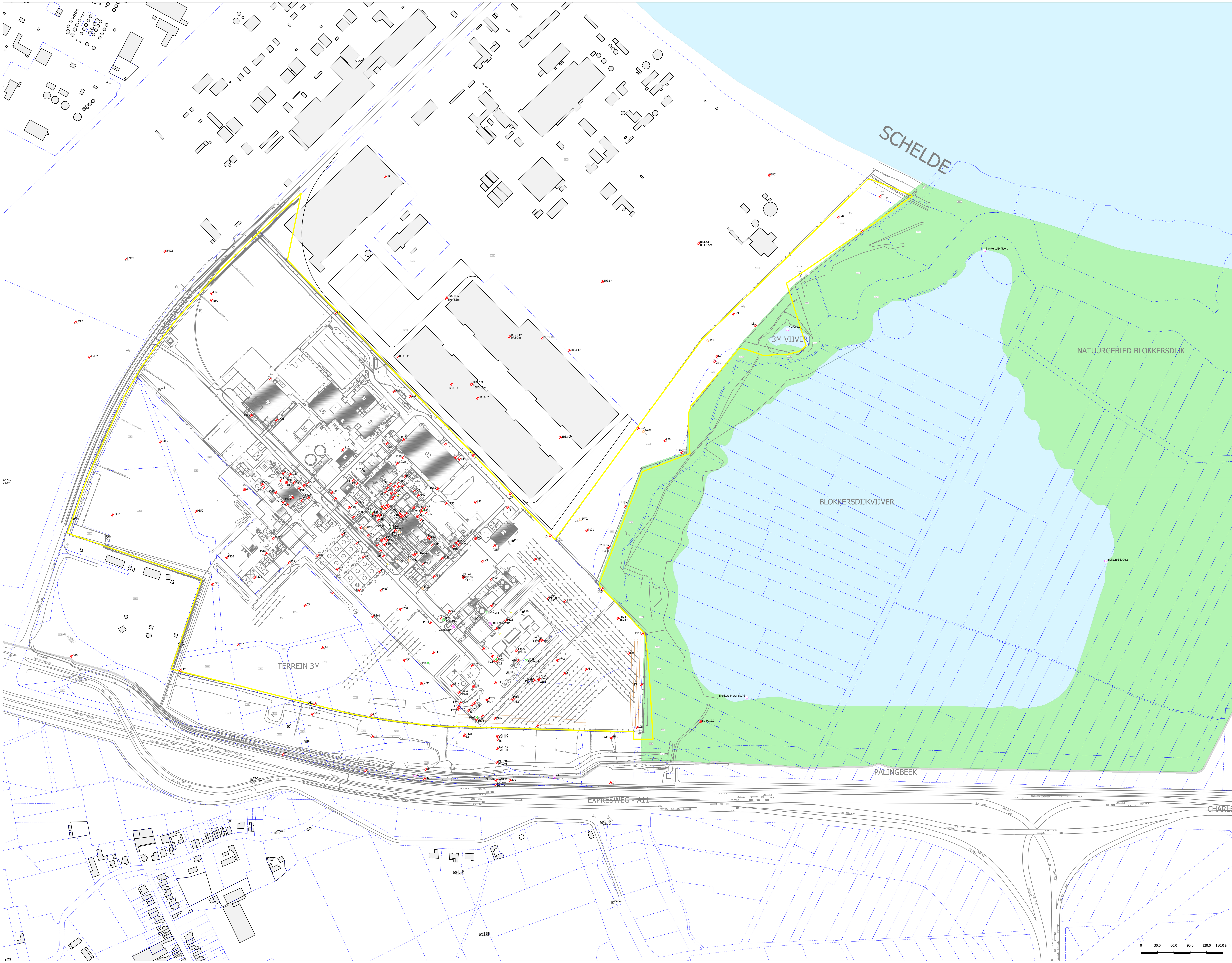
Client / Project Name:	Location:
3M Belgium bvba	Zwijndrecht

Project Number:	Title:
0398993	TTR8
Kaart:	**Description:**
1	Topografische situering © OpenStreetMap-contributors
Phase:	
TTR8	

Scale:	Paper Size:	File Name:	Date:
1:25 000	A4	3M Zwijndrecht_Topo.dwg	10/04/2018

Kaart 2

Overzichtsplan onderzoekslocatie



Legende:

- 3M terreingrens
- Kadastrale grens
- 267v2 Kadastrale nummer
- Peilbuis
- Peilbuis verdwenen
- Onttrekkingsput
- Oppervlaktewaterstaalnamepunt
- Regenwaterrioleringsput

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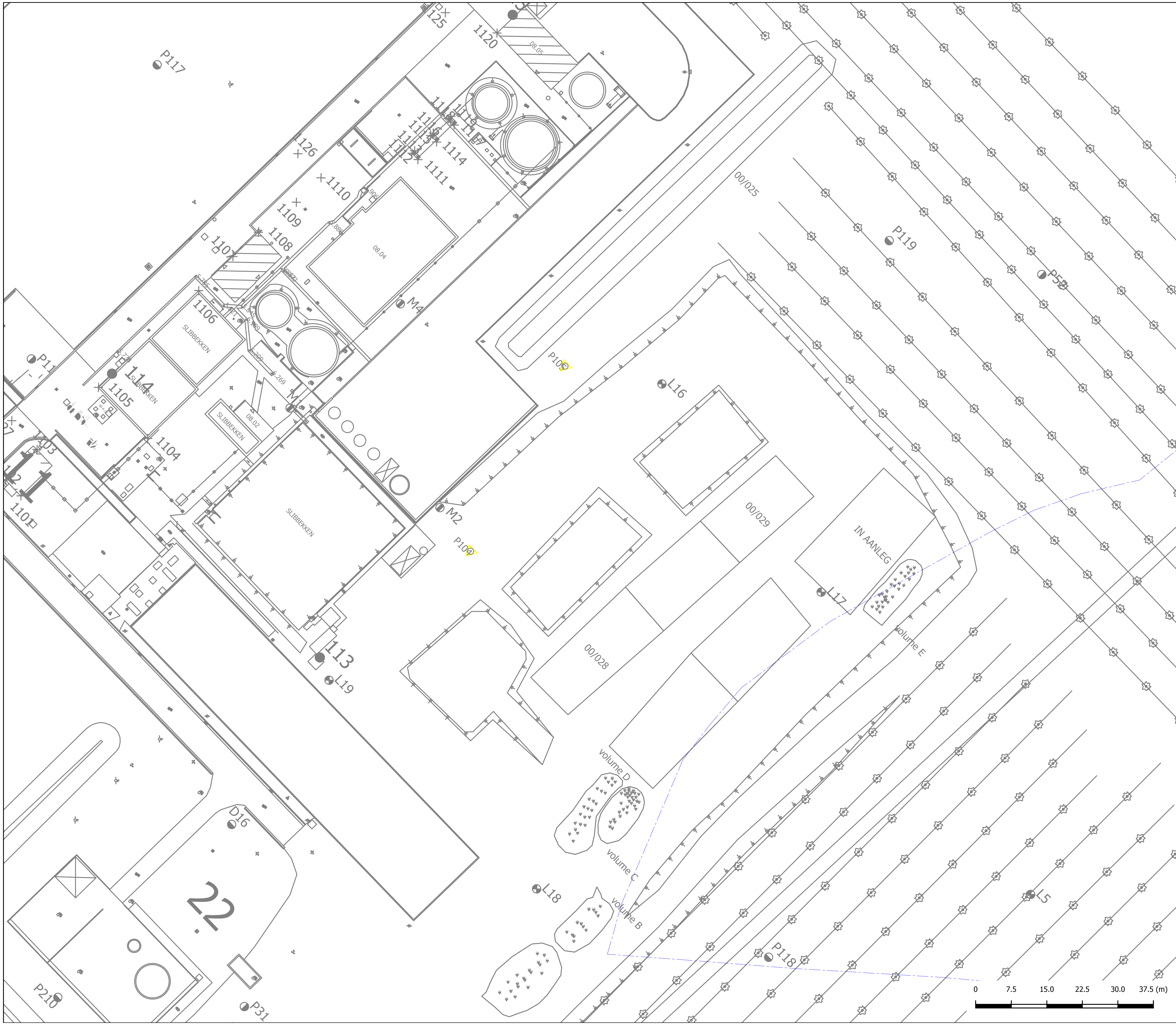
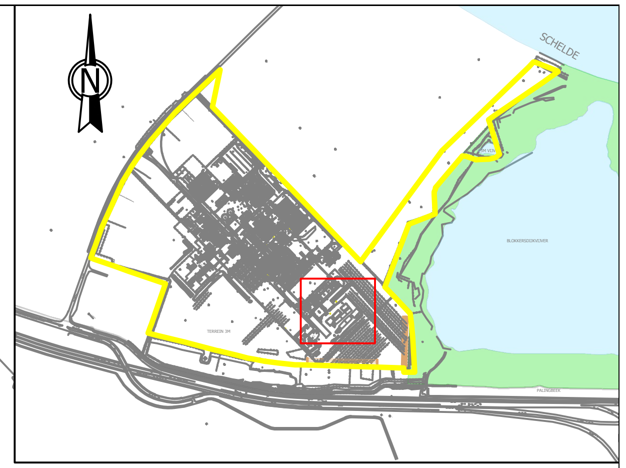
Klant / Projectnaam: 3M Belgium bvba		Locatie: Zwijndrecht	
Projectnr.: 0398993	Titel: TTR8		
Kaart: 2	Beschrijving: Overzicht van de onderzoekslocatie met aanduiding van Peilbuizen		
Fase: TTR8			
Schaal: 1:3000	Formaat: A1	Bestandsnaam: 3M Zwijndrecht-2017.dwg	Datum: 12/04/2018

Kaart 3

Detailplan ligging grondhopen





Legende:

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ERM

Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

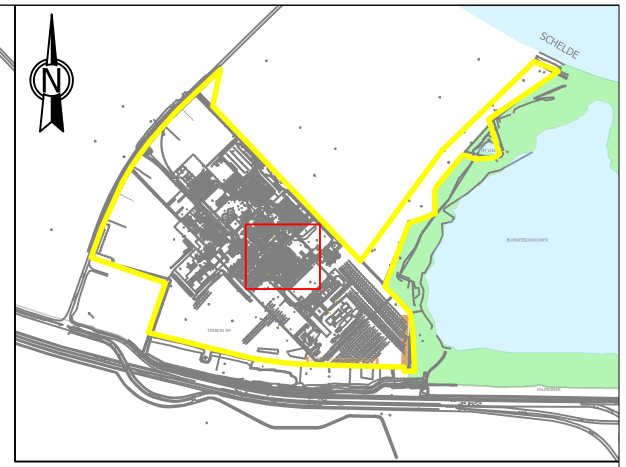
Projectnr.:	Titel:
0398993	TTR8
Kaart: 3	Beschrijving: Overzicht Grondhopen
Fase: TTR8	

Schaal:	Formaat:	Bestandsnaam:	Datum:
1:750	A3	3M Zwijndrecht-2017.dwg	12/04/2018

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:

- 3M terreingrens
- Kadastrale grens
- 267v2 Kadastrale nummer

- Peilbuis
- Peilbuis verdwenen
- Onttrekkingsput
- Oppervlaktewaterstaalnamepunt
- Regenwaterrioleringsput

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ERM

Klant / Projectnaam: 3M Belgium bvba	Locatie: Zwijndrecht
Projectnr.: 0398993	Titel: TTR8
Kaart: 4A	Beschrijving: Overzicht monsternamelocaties Bronzone Gebouw 16 en zone ondergrondse tanken
Fase: TTR8	

Schaal:	Formaat:	Bestandsnaam:	Datum:
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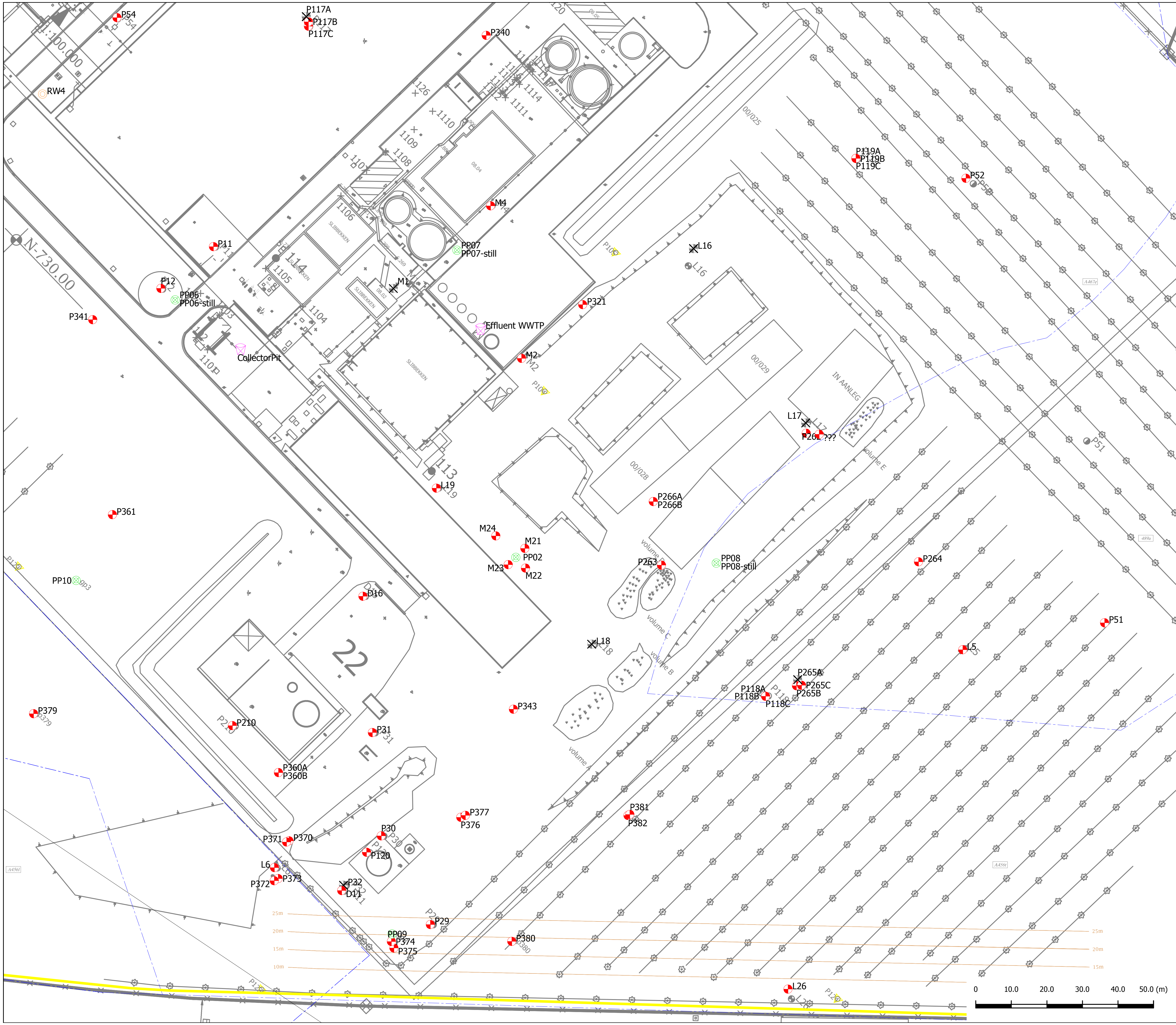

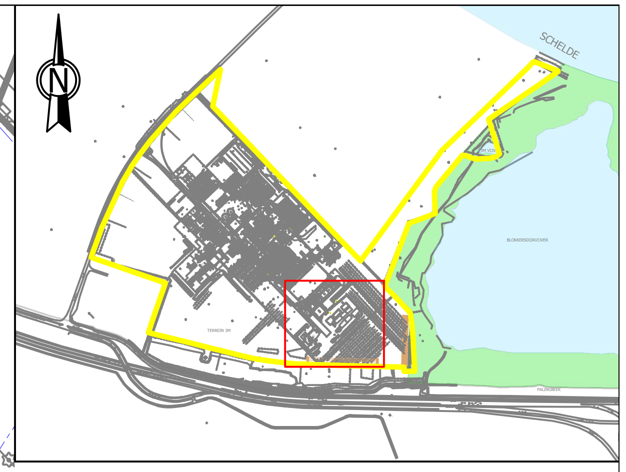

Legende:

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- Peilbuis verdwenen
- Onttrekkingsput
- Oppervlaktewaterstaalnamepunt
- Regenwaterrioleringsput

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ERM

Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.:	Titel:
0398993	TTR8
Kaart: 4B	Beschrijving: Overzicht monsternamelocaties Gebouw WWTP
Fase: TTR8	

Schaal:	Formaat:	Bestandsnaam:	Datum:
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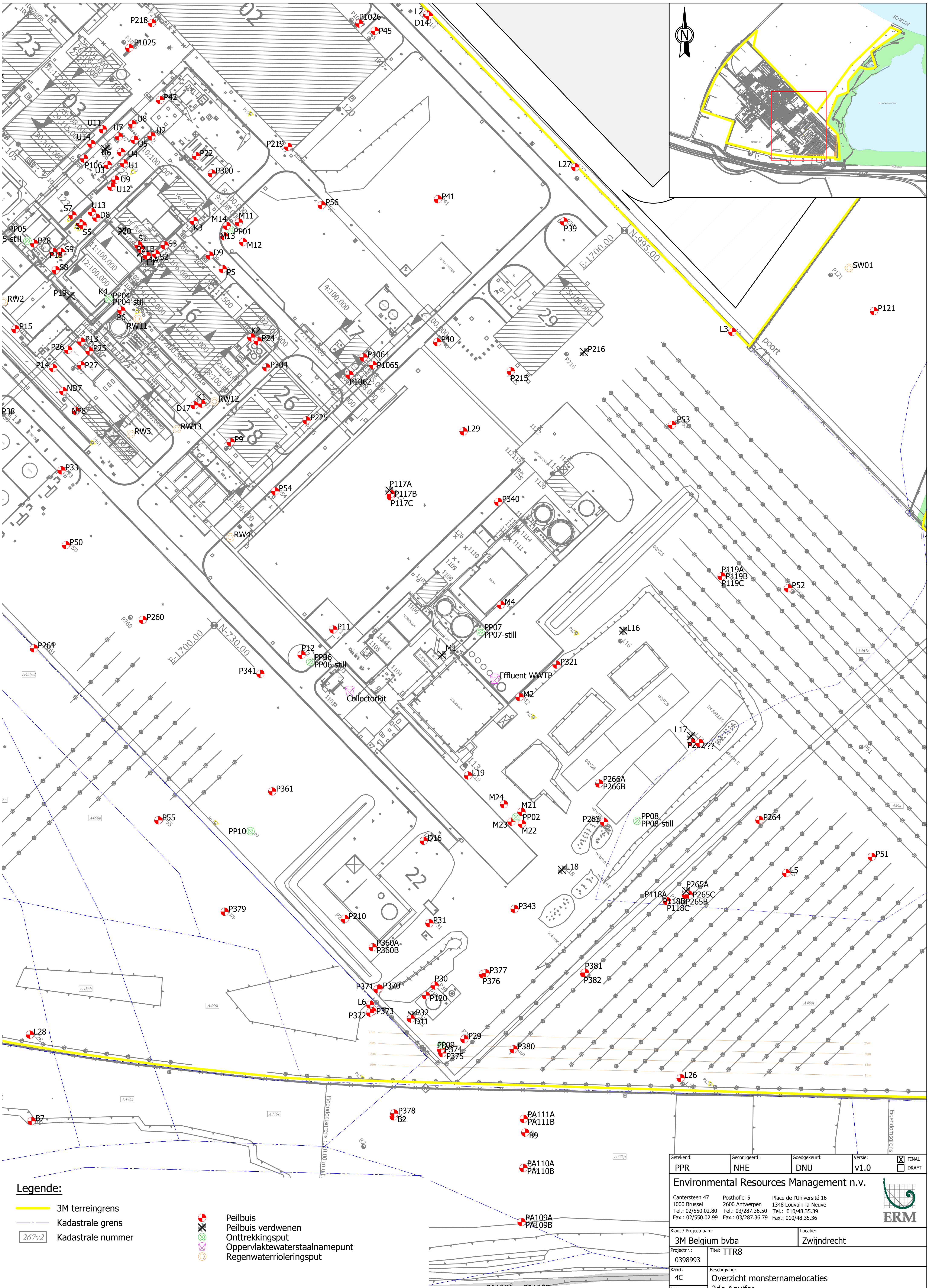

Legende:

- 3M terreingrens
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- Oppervlaktewaterstaalnamepunt
- Regenwaterrioleringsput

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ERM

Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.: 0398993	Titel: TTR8
Kaart: 4C	Beschrijving: Overzicht monsternamelocaties 2de Aquifer
Fase: TTR8	

Schaal:	Formaat:	Bestandsnaam:	Datum:
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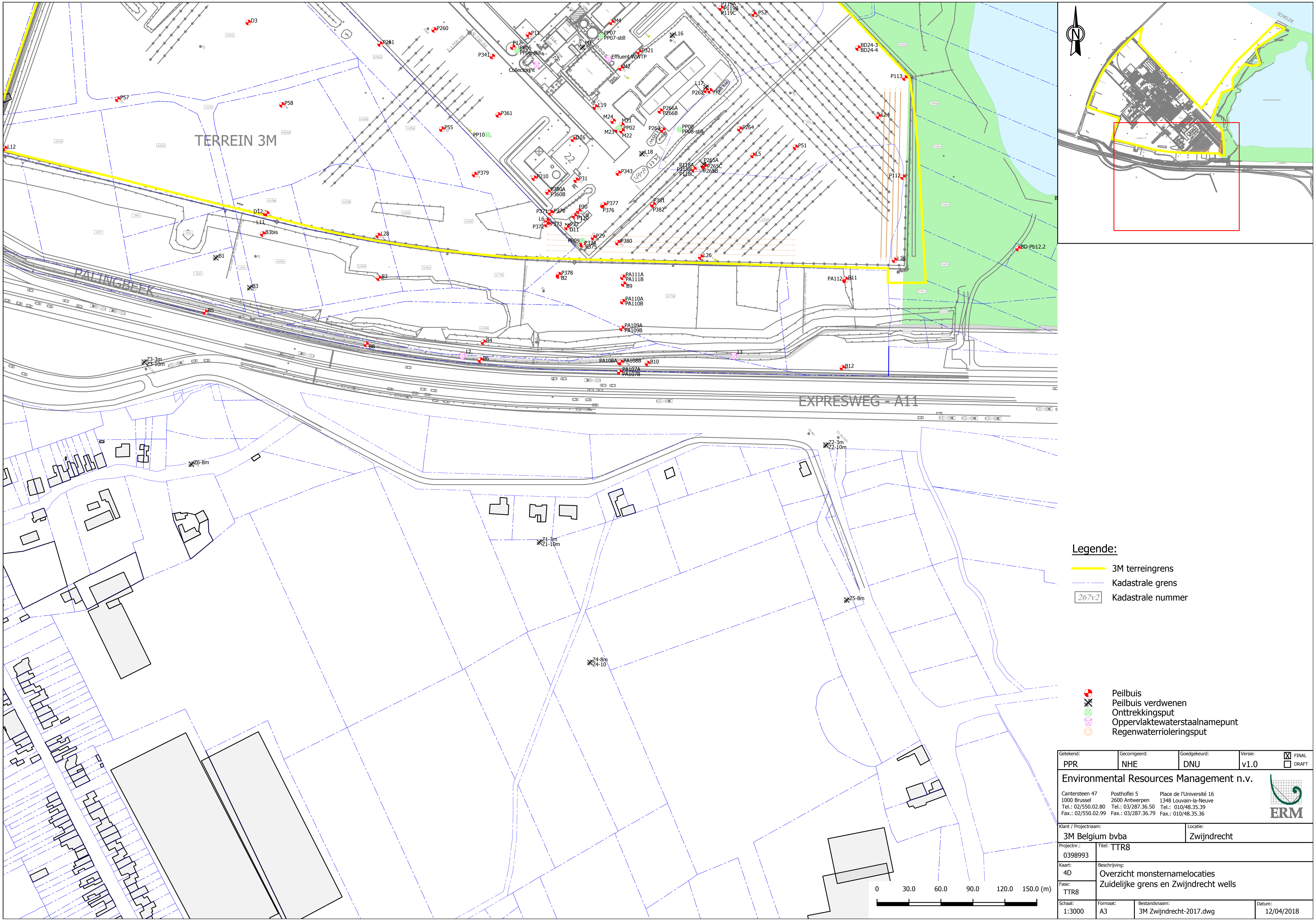

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- 267v2 Kadastrale nummer

- Peilbuis
- Peilbuis verdwenen
- Onttrekkingsput
- Oppervlaktewaterstaalnamepunt
- Regenwaterrioleringsput

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Fax.: 010/48.35.36

ERM

Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.:	Titel:
0398993	TTR8

Kaart:	Beschrijving:
4D	Overzicht monsternamelocaties Zuidelijke grens en Zwijndrecht wells
Fase:	
TTR8	

Schaal:	Formaat:	Bestandsnaam:	Datum:
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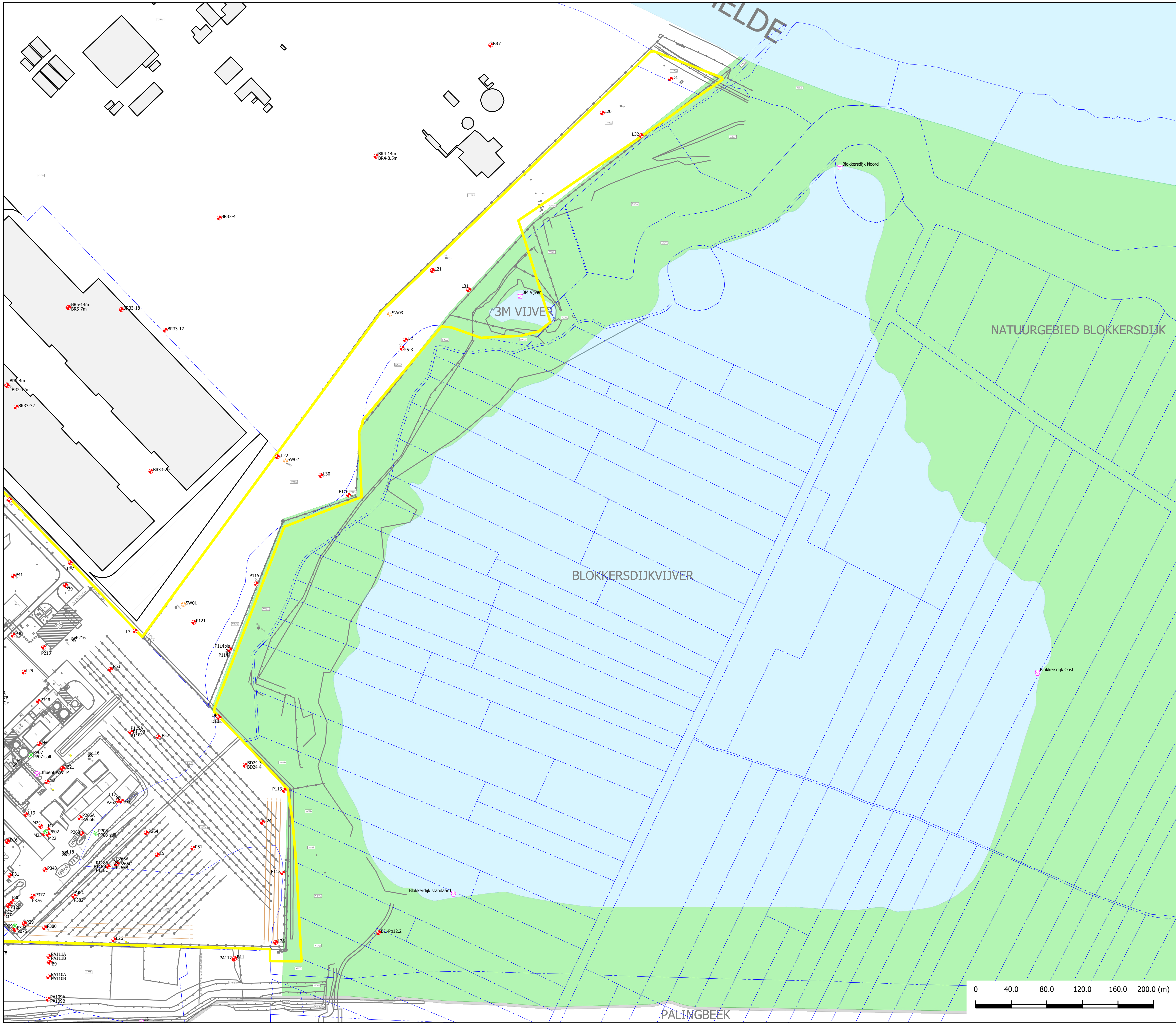

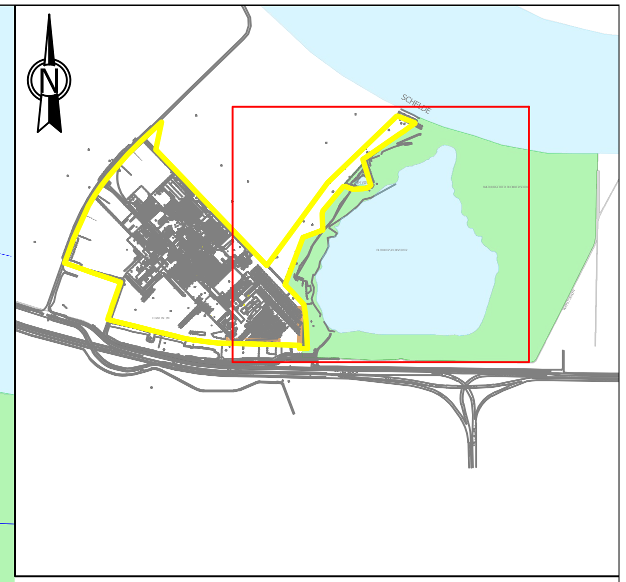

Legende:

- 3M terreingrens
- Kadastrale grens
- 267v2 Kadastrale nummer

- Peilbuis
- Peilbuis verdwenen
- Onttrekkingsput
- Oppervlaktewaterstaalnamepunt
- Regenwaterrioleringsput

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ERM

Klant / Projectnaam: 3M Belgium bvba	Locatie: Zwijndrecht
Projectnr.: 0398993	Titel: TTR8
Kaart: 4E	Beschrijving: Overzicht monsternamelocaties Blokkersdijk Noord
Fase: TTR8	

Schaal:	Formaat:	Bestandsnaam:	Datum:
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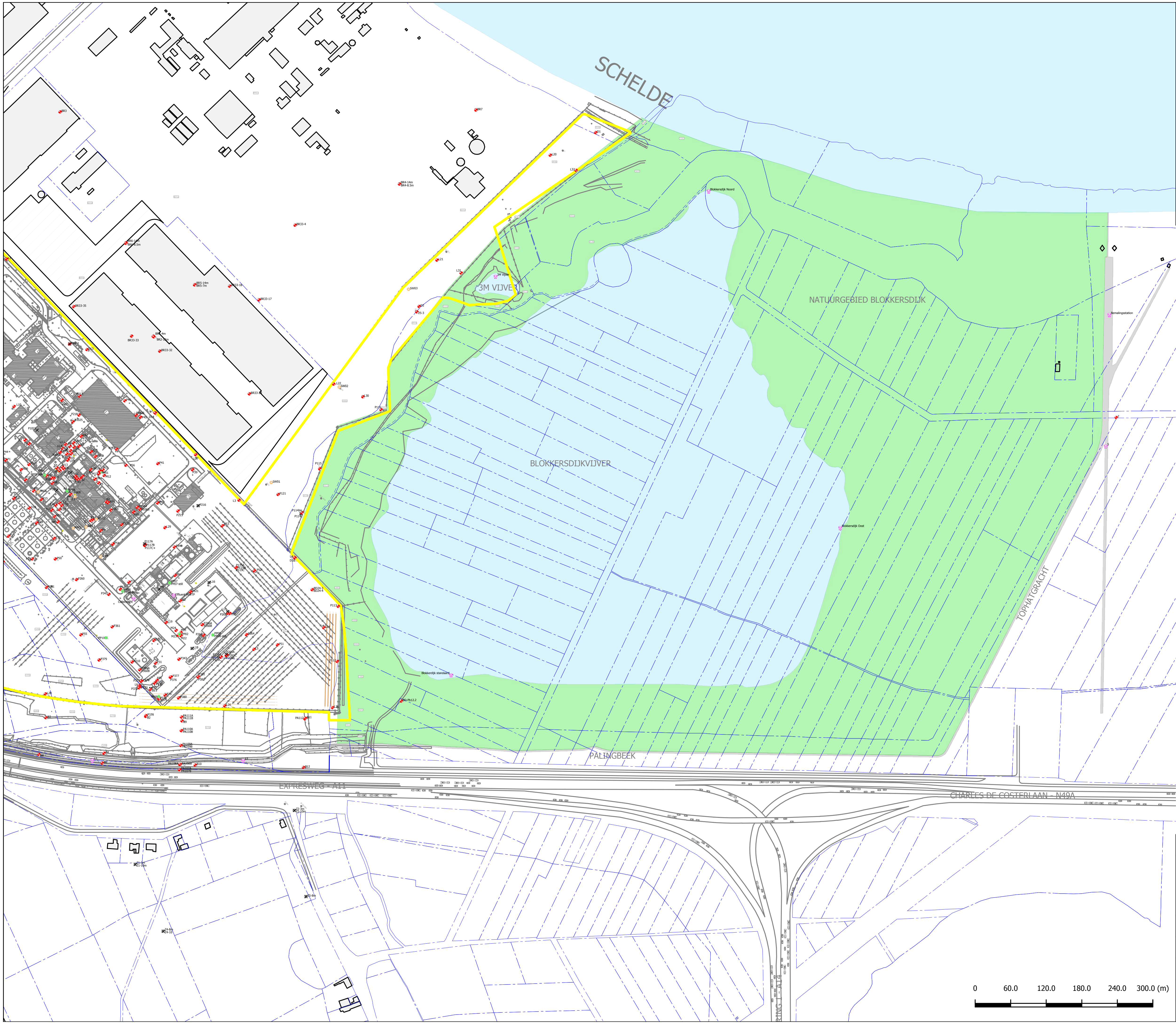

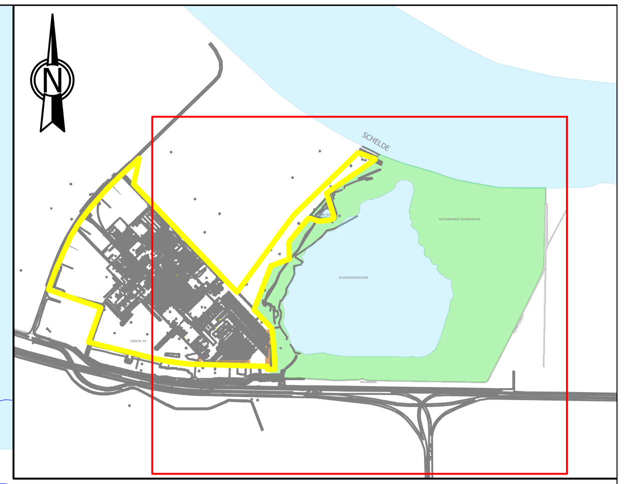

Legende:

- 3M terreingrens
- Kadastrale grens
- 267v2 Kadastrale nummer

- Peilbuis
- Peilbuis verdwenen
- Onttrekkingsput
- Oppervlaktewaterstaalnamepunt
- Regenwaterrioleringsput

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
PPR	NHE	DNU	v1.0	☒ FINAL ☐ DRAFT

Environmental Resources Management n.v.

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1000 Brussel
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Fax.: 02/550.02.99

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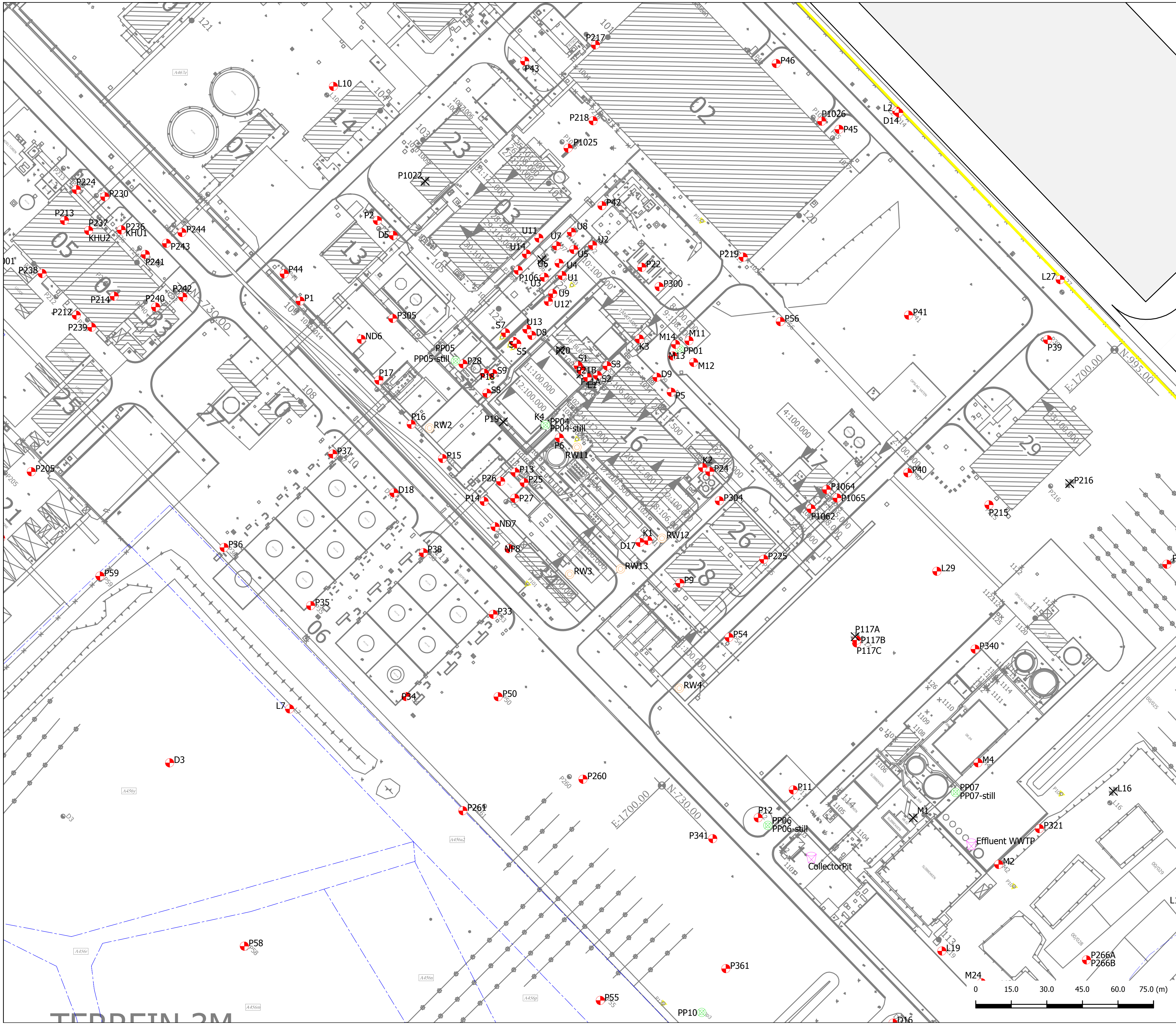
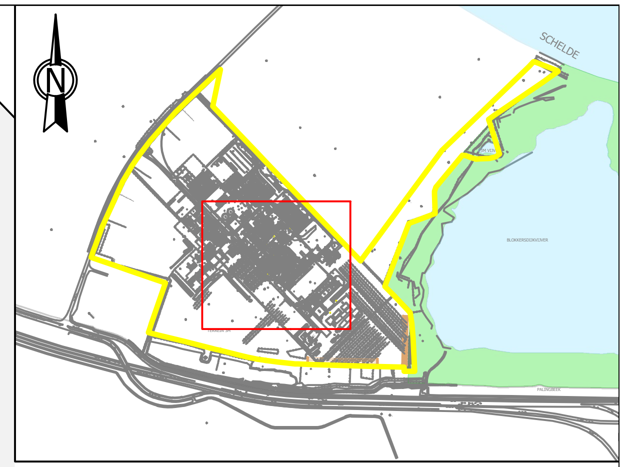
Fax.: 010/48.35.36

ERM

Klant / Projectnaam: 3M Belgium bvba	Locatie: Zwijndrecht
Projectnr.: 0398993	Titel: TTR8
Kaart: 4F	Beschrijving: Overzicht monsternamelocaties Palingbeek
Fase: TTR8	
Schaal: 0.166667	Formaat: A3
Bestandsnaam: 3M Zwijndrecht-2017.dwg	Datum: 12/04/2018





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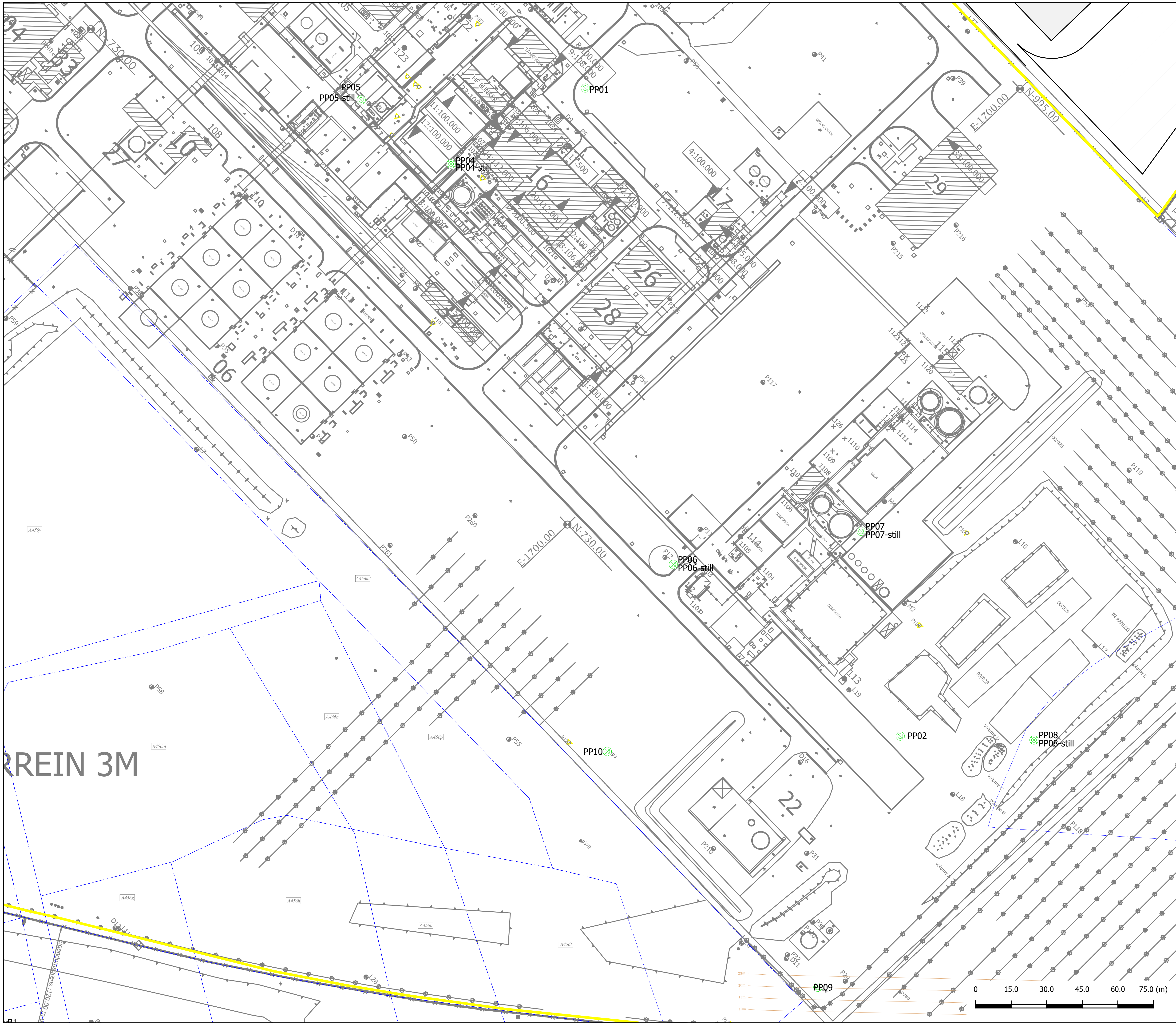
ERM

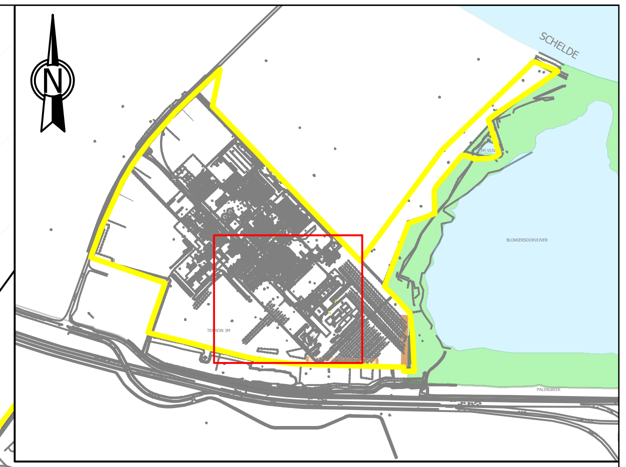
Klant / Projectnaam: 3M Belgium bvba	Locatie: Zwijndrecht
Projectnr.: 0398993	Titel: TTR8
Kaart: 4G	Beschrijving: Overzicht monsternamelocaties Regenwaterriolering
Fase: TTR8	

Schaal:	Formaat:	Bestandsnaam:	Datum:
1:1500	A3	3M Zwijndrecht-2017.dwg	12/04/2018

Kaart 5

Onttrekkingsfilters grondwateronttrekking





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- 3M terreingrens
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Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
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ERM

Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.: 0398993	Titel: TTR8
Kaart: 5	Beschrijving: Onttrekkingsfilters grondwateronttrekking
Fase: TTR8	

Schaal:	Formaat:	Bestandsnaam:	Datum:
1:1500	A3	3M Zwijndrecht-2017.dwg	12/04/2018