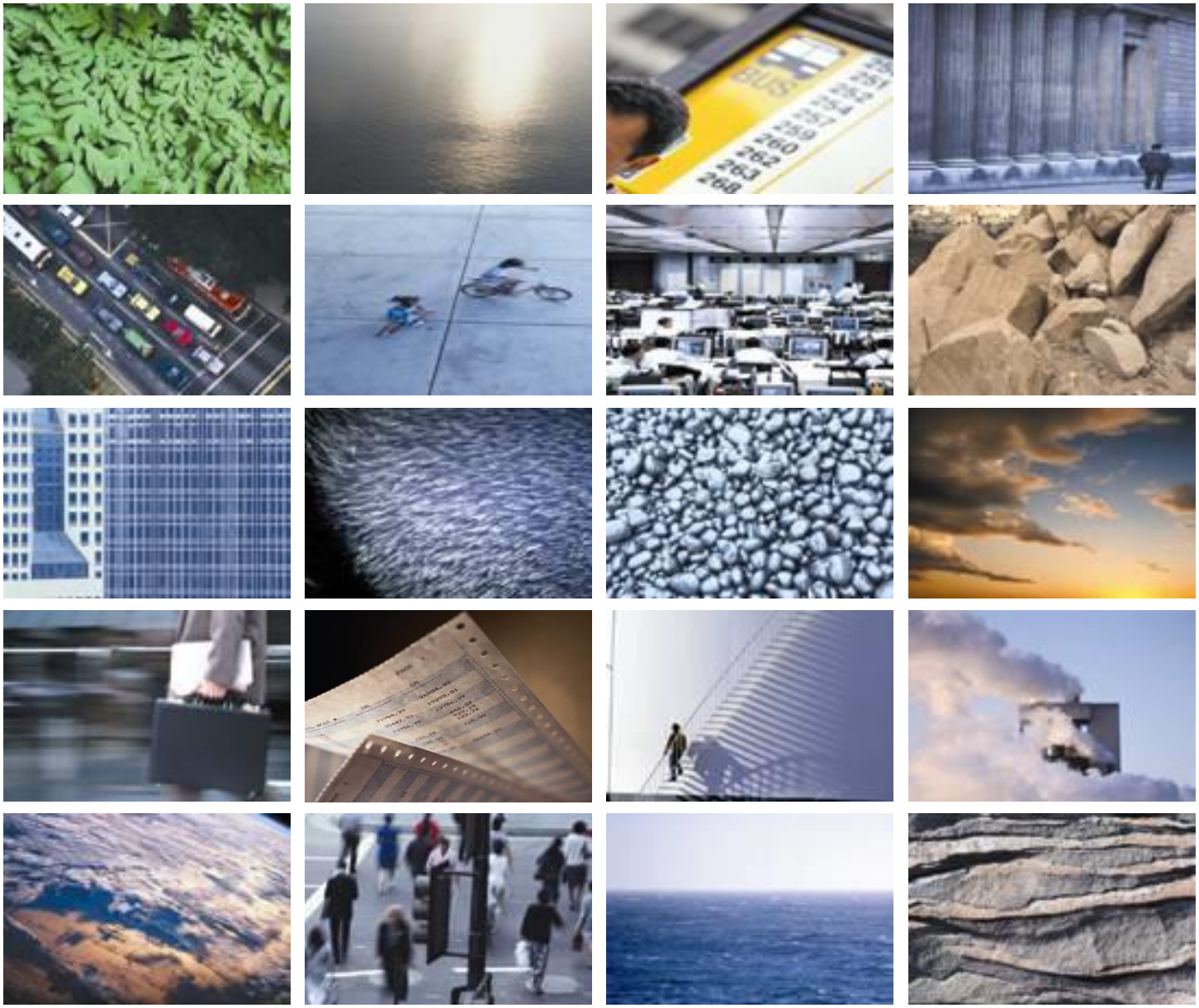

Eerste gefaseerd bodemsaneringsproject - Zesde tussentijds verslag bodemsaneringswerken

Periode juli 2014 - juli 2015

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

5 april 2016

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RAPPORT

Eerste gefaseerd bodemsaneringsproject - Zesde tussentijds verslag bodemsaneringswerken

Periode juli 2014 – juli 2015

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

Projectnr. : 0287933

Ref. : R01 0287933TTV6 v1.docx

Voor en namens ERM nv

Opgemaakt door: Ruben Maes en
Nanda Hermes

Goedgekeurd door: Dirk Nuyens
Functie: Principal Partner
Handtekening:



Datum: 5 april 2016

Dit document is opgemaakt door Environmental Resources Management - ERM n.v., met de grootst mogelijke zorg en onder de voorwaarden en binnen het budget zoals overeengekomen met de opdrachtgever. We wijzen elke aansprakelijkheid af voor aangelegenheden die vallen buiten de overeenkomst die met de opdrachtgever zijn afgesloten.

Dit document is vertrouwelijk en we aanvaarden geen enkele aansprakelijkheid ten over staan van partijen, andere dan de opdrachtgever, die op enige wijze kennis hebben gekregen van de inhoud van dit document

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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 -
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In Tabel 0.1 zijn de administratieve gegevens van het rapport samengevat. De administratieve gegevens van het perceel waarop het schadegeval is voorgevallen, zijn opgenomen in Tabel 0.2.

Tabel 0.1 Administratieve gegevens van het rapport

Titel:	Eerste gefaseerd bodemsaneringsproject - Zesde tussentijds verslag bodemsaneringswerken: Periode juli 2014 – juli 2015 3M Belgium bvba Canadastraat 11 te 2070 Zwijndrecht
Referentie EBSD:	R01 0287933TTV6 v1.docx
Rapportdatum:	5 april 2016
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:	-

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

Titel:	Eerste gefaseerd bodemsaneringsproject - Zesde tussentijds verslag bodemsaneringswerken: Periode juli 2014 – juli 2015 3M Belgium bvba Canadastraat 11 te 2070 Zwijndrecht
Referentie EBSD:	R01 0287933TTV6 v1.docx
Rapportdatum:	5 april 2016

Hoedanigheid:	☒ Eigenaar ☐ Gebruiker ☒ Exploitant ☐ Optredend in opdracht van de eigenaar/gebruiker/exploitant ☐ Andere: ……

Naam contactpersoon:	Charlotte Tack
- Telefoon:	[redacted]
- Fax:	[redacted]
- E-mail:	[redacted]

Naam contactpersoon ter plaatse:	Kristof Verstraeten
- Telefoon:	[redacted]
- Fax:	-
- 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)	Periode Van	Periode Tot	Persoon (Eigenaar / gebruiker / exploitant) Type (3)	Naam	Adres	Letter (4)	Huidig bestemmingstype (1)	Toekomstig bestemmingstype (1)	Grondwaterkwetsbaarheid (2)
11056	A	467D	Canadastraat 11	Zwijndrecht	321 598			Ex	3M Belgium bvba	Canadastraat 11 2070 Zwijndrecht	A	V	V	Ca1/ v

Overige percelen: *zie conformverklaring BSP in Bijlage 1*

(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.

D EEL 2: RAPPORT

Eerste gefaseerd bodemsaneringsproject -
Zesde tussentijds verslag bodemsaneringswerken
Periode juli 2014 - juli 2015

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

5 april 2016

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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 (FCs) and mercury, and groundwater impacted with FCs and aromatic compounds (mainly Xylenes), were identified. In 2008, the first phase of the remedial action plan (RAP) was submitted by Arcadis Gedas nv and in early 2009 the Flemish Soil Agency (OVAM) approved the RAP.

Currently, the first phase of the RAP is being implemented to remediate groundwater containing FCs in the production area (Building 16) and the waste water treatment plant (WWTP) area. In addition, the Blokkersdijk nature reserve and the second aquifer are being monitored regularly to assess the potential for further migration of FCs in these areas. The second phase of the RAP plan will address the FCs present near the southern site boundary and in the "Palingbeek" (a ditch), but can only be implemented when the work plan for the civil works related to the Oosterweel connection (a road infrastructure project) has been finalized. At present, the work plan and schedule are still under development.

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 2014 and July 2015.

In the period between July 2014 and July 2015, the following remedial and monitoring actions were performed:

1. Operation and Maintenance (O&M) of the installed pump and treat (P&T) system in the two source areas (Building 16 and WWTP). The source removal system was installed to reduce FC concentrations in groundwater and mitigate the FC migration in these two areas. Regular water samples are taken from the effluent of the pumping wells to monitor the efficiency of the P&T system.

2. Periodic monitoring of water quality at the following locations:

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

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

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

5. Final evaluation of the remedial monitoring results of the former BTEX contamination observed in groundwater near the UST area and FCs found in the groundwater near the Southern Border of the E34 motorway (Z-wells).

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

6. A geohydrological investigation in the 3M path area (located along the western border of the Blokkerdijk nature reserve) to determine if there is an additional contaminant source in the Blokkersdijk area that may explain the presence of FCs in the monitoring wells and the 3M pond.

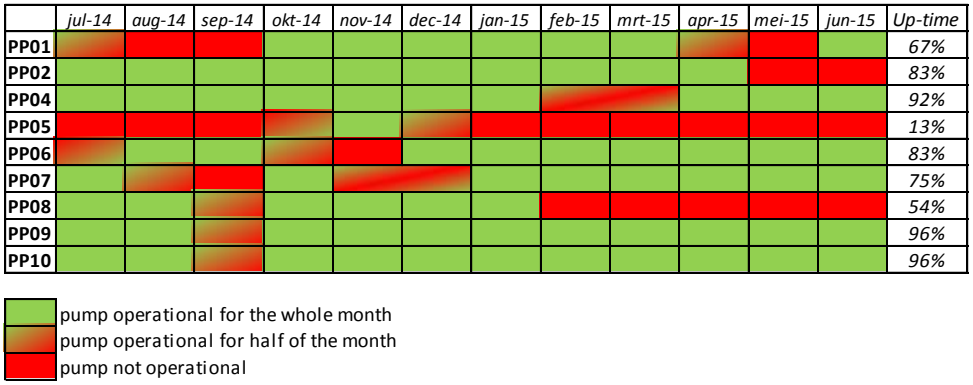
7. Environmental follow up of the following environmental incident at the site: 20% HF spill outside Building 017 (August 2014).

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. An additional five extraction wells were installed in the source areas in late 2010 and started fully operating in August 2011. In early 2012, two additional extraction wells were installed in the WWTP area. These wells became fully operational in May 2012. At present, the system comprises nine extraction wells, three wells to remediate groundwater in the Building 16 source area and six wells in the WWTP source area.

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

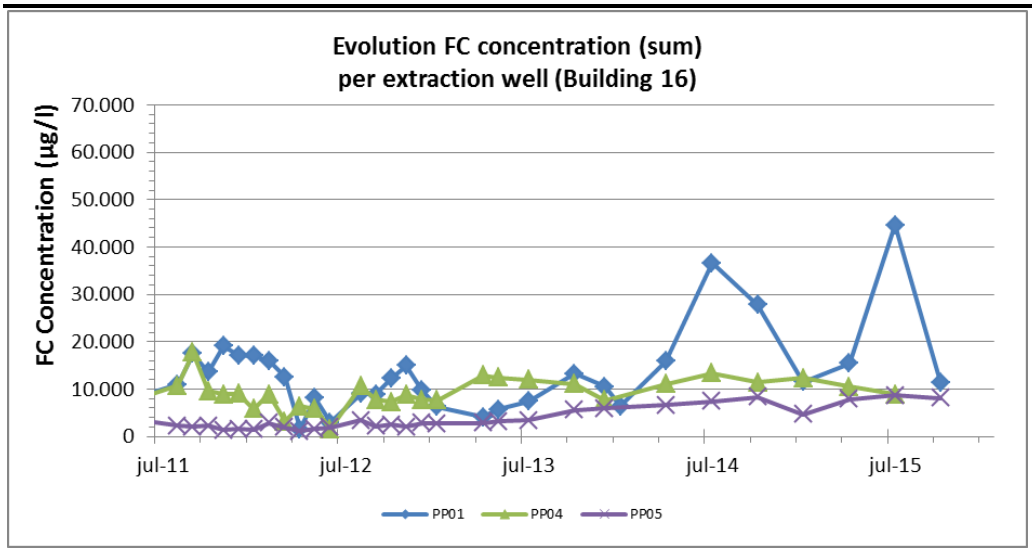
The ERM inspections show that the extraction system requires regular maintenance to remove sand and iron deposits accumulating in the wells. The average up-time of the remedial system is estimated to be 73%, but the up-time varies from well to well: minimum 13% for well PP05 and maximum 96% for wells PP09 and 10 as shown in the following table.

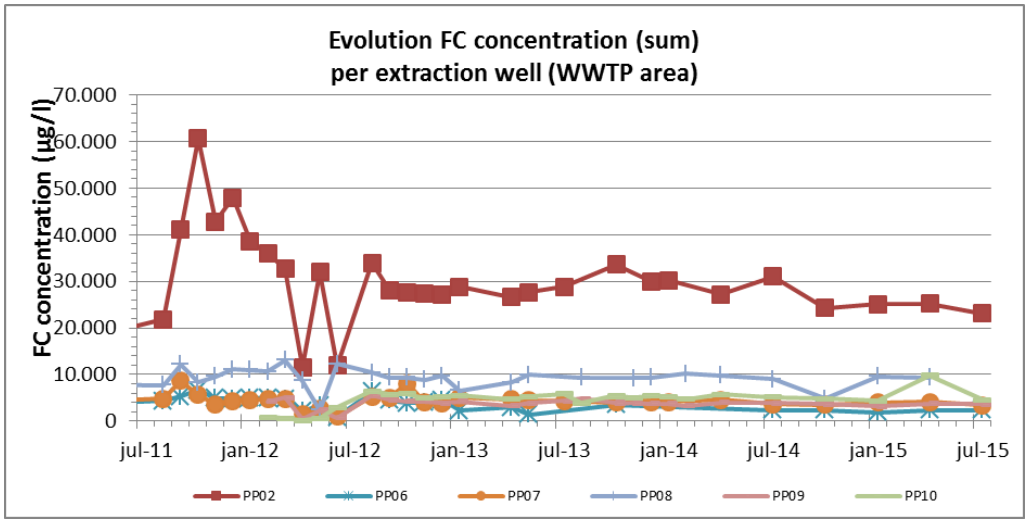
P&T system – Monthly summary of system up-time



The average flow rate of the system (excluding well PP05 that operates intermittently) varies between minimum 0,3 m^3/day (well PP02) and maximum 16,3 m^3/day (well PP10). The average groundwater FC concentration ranges between 2,7 mg/L (PP06) to 27,2 mg/L (PP01), depending on the extraction well, but the concentrations fluctuate significantly (see graph below) and further follow up is needed. The long-term trend in FC concentrations in most of the extraction wells is stable, although fluctuations can occur.

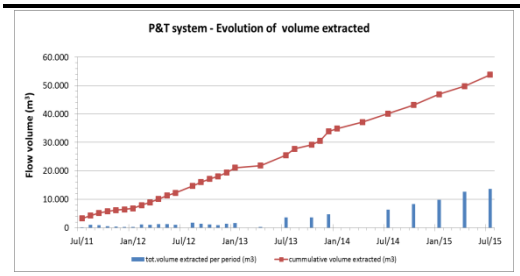
P&T system – Concentration Trends in Building 16 and WWTP area

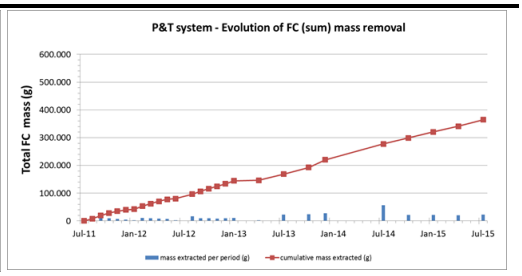




Between July 2011 and June 2015 approximately 52.700 m^3 of contaminated groundwater was extracted and approximately 362 kg of FCs were removed, as shown in the graphs below.

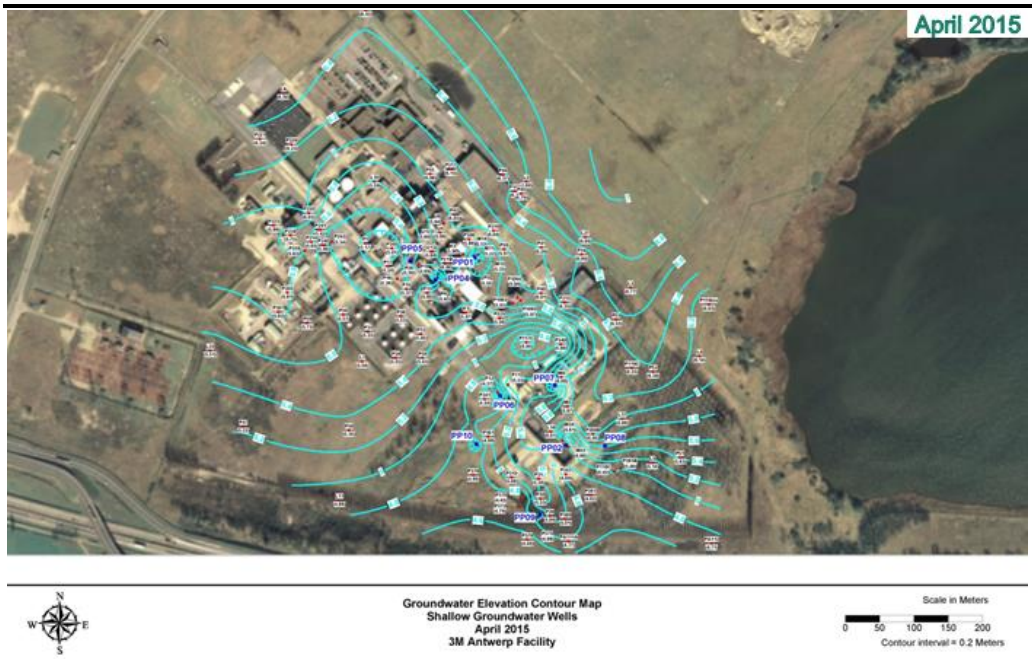
P&T system – Efficiency Trends





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

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



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

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

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

Monitoring Groundwater, Surface Water and Storm Water Quality

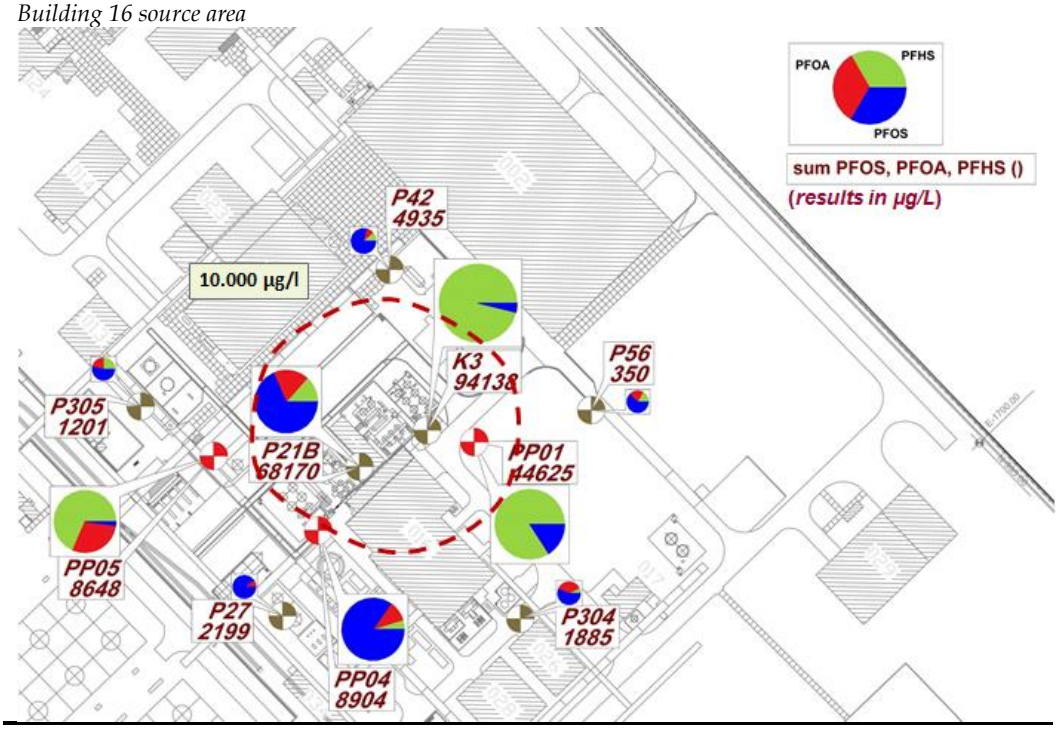
The remedial objectives of the monitoring program are to verify that the P&T system is mitigating the horizontal migration in the first aquifer in the areas of concern (Blokkersdijk nature reserve and the Paling ditch at the southern site boundary) as well as the vertical migration of contaminants from the first to

the second aquifer. As part of this monitoring program, ground-, surface and storm water samples are taken and analyzed for FCs.

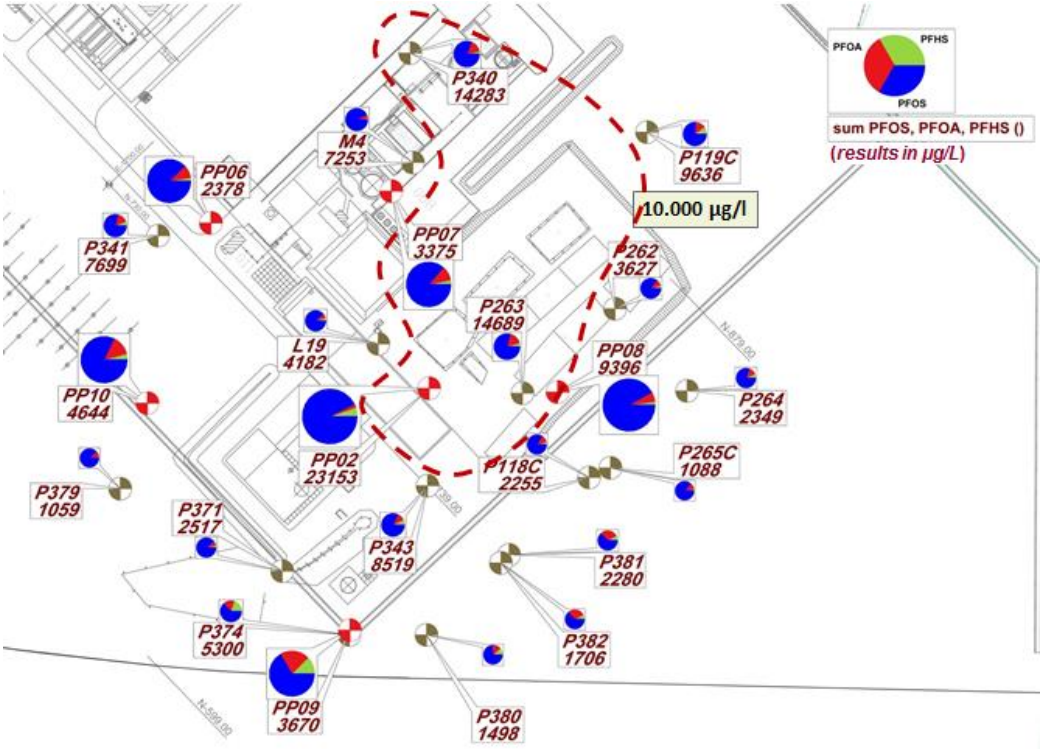
To evaluate the impact of the P&T system in the **FC source areas,** two groundwater wells are sampled annually and five groundwater wells are sampled semi-annually in the Building 16 area. Six groundwater wells are sampled annually, and ten wells sampled semi-annually in the WWTP area. The results of the July 2015 monitoring are shown in the figures below with the sum FC-concentration contour (10.000 µg/l).

FC Source Areas - Results July 2015 Monitoring

Building 16 source area



WWTP source area



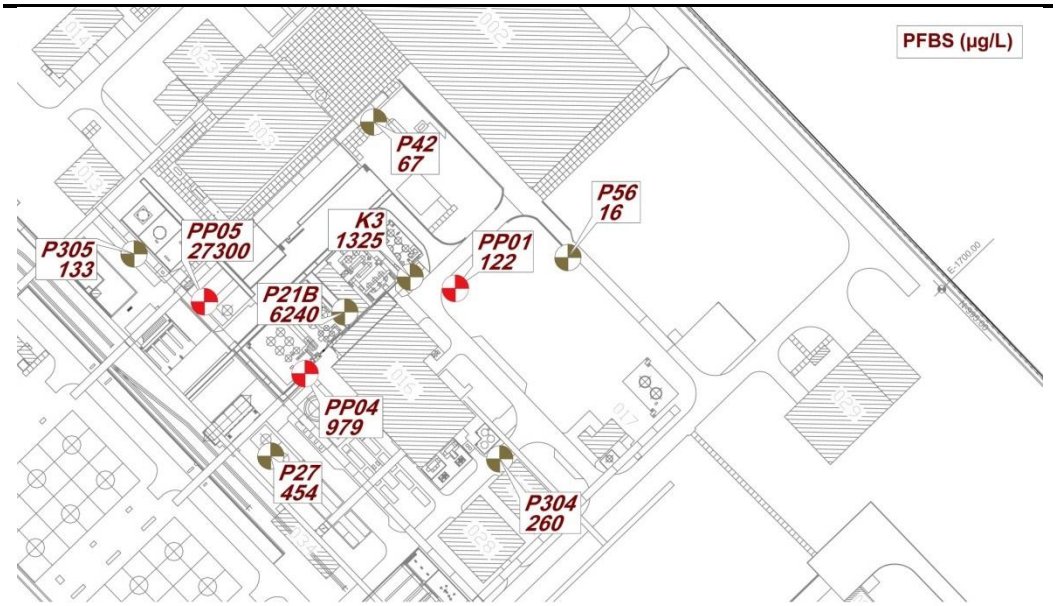
The general stable/decreasing trend in FC concentrations suggest that the shallow P&T system installed in the two source areas is reducing the concentrations of FCs in groundwater in the Building 16 and WWTP area.

Based on a review of the historic monitoring results, groundwater concentrations in the Building 16 area are stable or decreasing, except for the FC concentrations in well P21B. The extent of the sum FC concentration contour appears to be slightly smaller than the contour based on the results of the previous monitoring round (2013-14).

In the WWTP area, groundwater concentrations in most of the wells are stable or decreasing, except for wells P119C, P263 and P340. The PFOS concentration in these three wells has increased. The concentration increase in well P340 is not confirmed by the SGS analysis of the duplicate sample. The extent of the sum FC concentration contour appears to be increased to the contour based on the previous monitoring results (2013-14) mainly because the monitoring results of P119C and P340, which appear to have increased as compared to the last sampling round and need to be verified first.

The use of PFOS on-site was eliminated in 2002 and was replaced with PFBS which has a significantly lower toxicity than PFOS. Prior to the start of the production of PFBS, this compound was extensively screened by 3M for potential (eco)toxicological effects. PFBS replaced PFOS because it has a significantly shorter elimination half-life, is persistent but does not bio-accumulate, and has a high PNEC value (10.040 µg/L) compared to PFOS. Recent groundwater analytical data indicates the presence, and increase in concentrations, of PFBS in the area of Building 16. During the July 2015 monitoring, additional wells were sampled and analyzed for PFBS to assess the PFBS impact. The result of the monitoring is shown in the following figure. The highest groundwater concentrations are measured in near extraction well PP05 en monitoring well P21B.

FC Source Area near Building 16 – PFBS Results July 2015 Monitoring



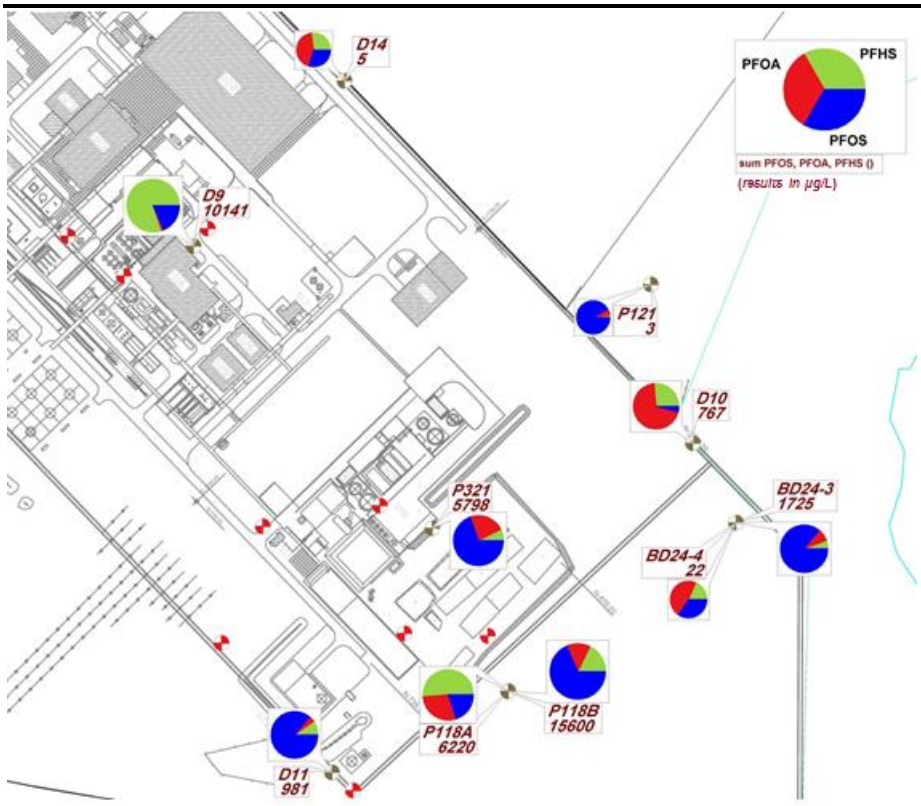
Building 16 source area

The cause of the PFBS detection and the observed concentration increase is, at present, unknown, but will continue to be investigated. PFBS concentrations will be monitored on a yearly basis.

ERM recommends to continue the current groundwater monitoring program for wells completed in the shallow (1st) aquifer, but to increase the monitoring frequency of wells P21B (Building 16 area) and P263 (WWTP area) to quarterly to allow a more frequent evaluation of the concentration trends in these wells. In addition ERM recommends assessing the need for further optimization of the P&T system in the first aquifer as some wells show increasing concentration trends.

To monitor the groundwater quality of the **second aquifer**, six wells are sampled annually, three wells are sampled on a semi-annual basis and one well on a quarterly basis. Two wells are located in, or downgradient, of the Building 16 source area and eight wells in, or downgradient, of the WWTP. The monitoring results are used to evaluate the effectiveness of the shallow P&T system in mitigating the vertical migration of contaminants from the first aquifer to the second aquifer. The results of the July 2015 monitoring are shown in the following figure.

Second Aquifer - Results July 2015 Monitoring



The evaluation of the monitoring results shows that the groundwater concentrations in the second aquifer fluctuate but in general are decreasing or stable over time, suggesting that the shallow P&T system in the first aquifer is achieving (partial) capture and is mitigating the downward migration of impacted groundwater. However, near Building 16, the PFOS concentration in well D9 appears to be increasing (more data is needed to confirm any

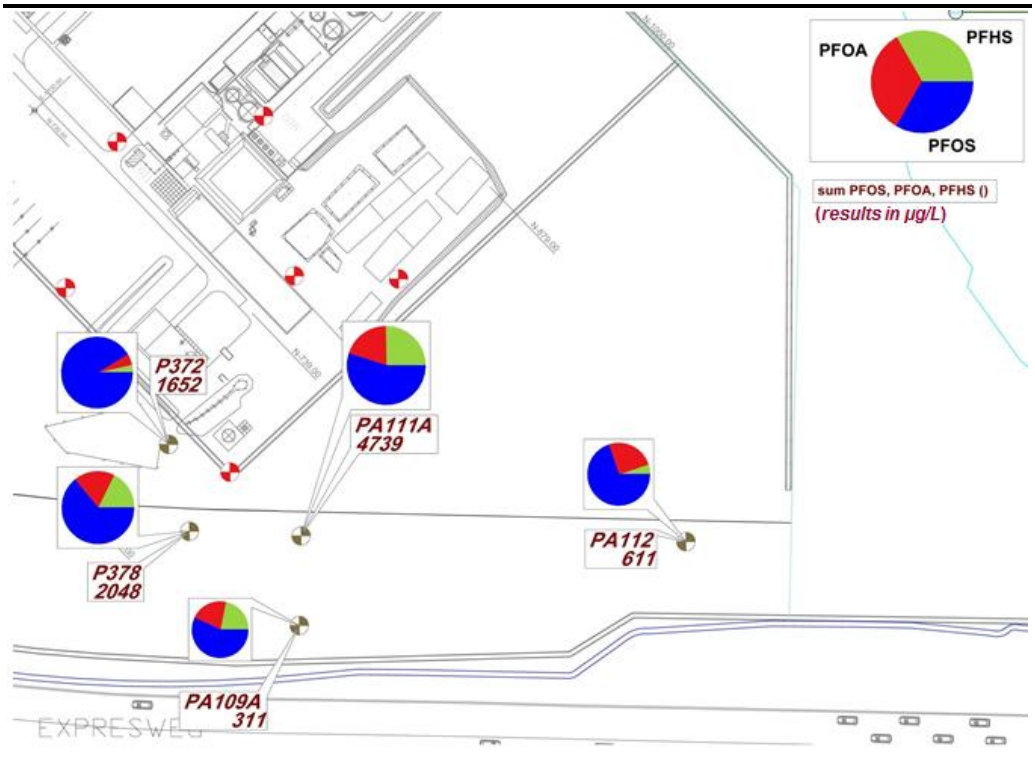
concentration trends in this well), and in the WWTP area, FC concentrations increased in 2015, in central well P321 and downgradient wells D10, BD24-3 and BD24-4. Continued monitoring of these wells is needed to assess the long-term trend as well as a potential optimization of the remedial system to further decrease the potential for downward contaminant migration.

The FC concentrations are below the five time limit value based on the concentrations measured during startup of the remediation system (mid 2011). At the northeast property boundary (downgradient from the source areas in the first aquifer) concentrations are below 10% of the solubility. Therefore, these conditions confirm 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 keeping the current monitoring frequency and assessing the need for further optimization of the P&T system in the first aquifer, as some wells show short-term increasing concentration trends.

The groundwater quality along the **southern site boundary** is currently being monitored semi-annually in seven wells. The results of the July 2015 monitoring are shown in the following figure. Well B3 could no longer be sampled (due to the excessive vegetation around the well and flooding because of high water levels) and will be replaced by a new well (B3-bis) close by.

Southern Site Boundary - Results July 2015 Monitoring

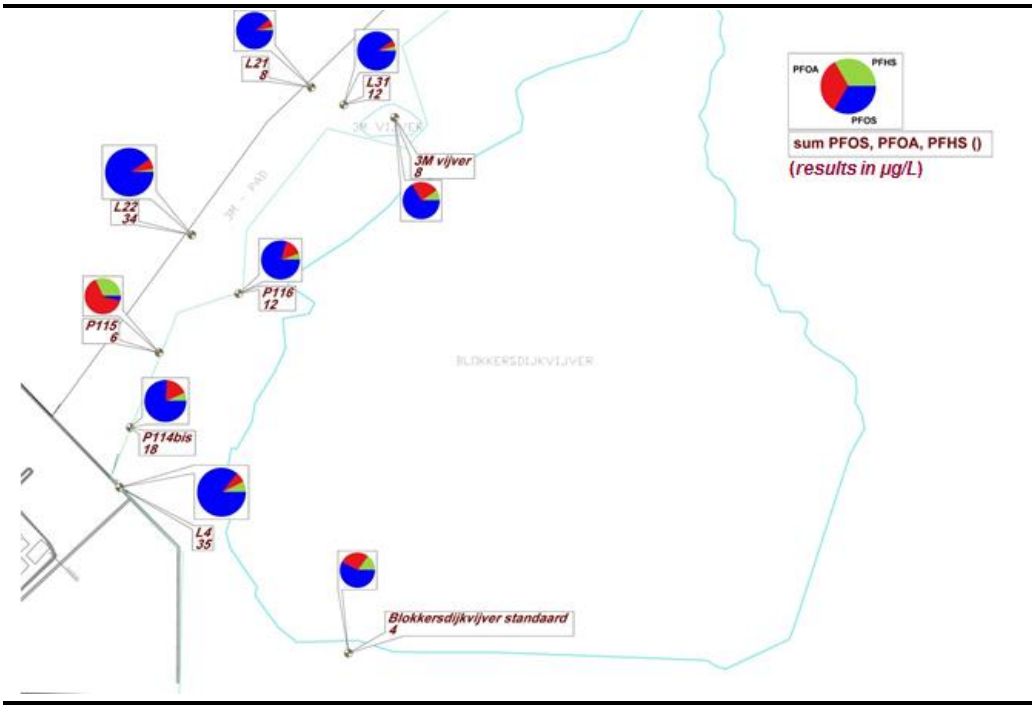


The evaluation of the monitoring results shows that, in general, the groundwater concentrations over time are decreasing or stable, but concentrations may fluctuate. The results suggest that the shallow P&T system installed at the WWTP source area is preventing contaminant migration to the southern site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

The groundwater quality in the **tank farm area** (near Building 3) is currently monitored annually for pH, conductivity and TPH. In 2014 a total of eight wells were sampled and analyzed to delineate the pH, conductivity and 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 highest TPH concentration is about twice the remedial norm (500 µg/l). The source of the TPH contamination is unknown but may be caused by interference from another impact; the elevated pH and conductivity are thought to be related to (minor) spills from the nearby KOH tank. Further historical and analytical investigation is needed. It is also recommended to continue the annual monitoring for TPH in wells P18 and P28.

Groundwater and surface water quality in, and close to, the **Blokkersdijk nature reserve** is monitored via eight wells and two ponds. Five groundwater wells are monitored semi-annually, and three monitoring wells and two surface water locations are monitored quarterly to statistically evaluate the trend of FC concentrations. The results of the July 2015 monitoring are shown in the figure below.

Blokkersdijk Nature Reserve - Results July 2015 Monitoring

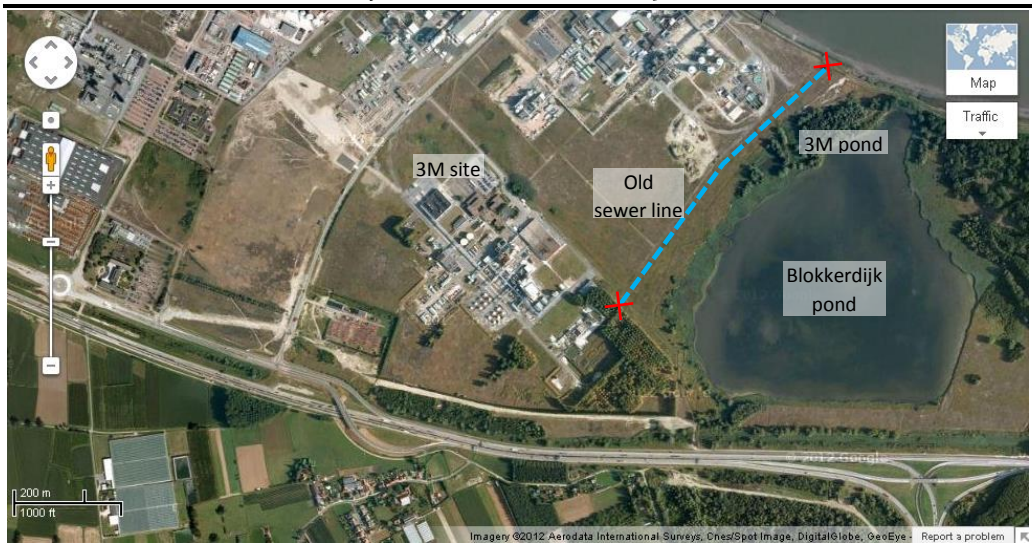


After the approval of the remedial action plan in 2009, periodic sampling has been conducted to collect water samples in the Blokkerdijk nature reserve pond, the 3M pond and selected wells adjacent to these two ponds. Analytical

results from these water samples have been used for the PFOS trend analysis as described in the approved remedial action plan.
Since the start of the remedial monitoring, different laboratories have been used to analyze the collected water samples (due to quality issues) and concern has been raised that the use of multiple laboratories over the period of data collection could have an impact on the results of the PFOS trend analysis. Since 2014 the analyses are being performed by the 3M Environmental Laboratory and based on an evaluation of the split sample data and the rigorous QA/QC program followed by the 3M lab, it has been determined that the analytical data generated by the 3M lab is the most representative data set. Therefore the initial assumptions on which the PFOS trend analysis was based in the 2008 RAP were evaluated taking into account the potential impact of using different labs by comparing the results of the statistical evaluation of the complete dataset to those of the 3M dataset only.
The evaluation showed that lab changes did confound the results of the PFOS trend analysis. The significant upward trends in three locations (L21, P114, and 3M vijver) appear to be due to changes in analytical laboratories. When the PFOS concentration trends were evaluated for only the data analyzed by 3M Environmental Lab, no significant trends were observed in those three locations. For this reason it is suggested that only data analyzed by 3M be utilized for future trend analysis.To populate the data set it is recommended that all the locations in the Blokkerdsijk area are sampled quarterly. The graphical and statistical trend analysis should be continued.

There is no need for additional remedial actions in this area as there is no significant increasing trend observed in the Blokkersdijk pond. However, ERM had recommended that the Blokkersdijk area be assessed for any potential FC sources that may explain the PFOS concentrations observed in the monitoring wells and the 3M pond, as the wells and 3M pond are located upgradient from the known nearby FC source area at the 3M site (WWTP). As a result, in late 2014, a geohydrological investigation was conducted in the area of the 3M path, which is located along the western border of the Blokkerdijk nature reserve. Underneath the 3M path, there is an old chemical sewer line that was abandoned by 3M in 1999 and replaced by a new, chemically resistant, sewer line. The location of the sewer line, 3M path, nature reserve and 3M site is shown in the aerial picture on the next page.

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



According to 3M, the old sewer line was not removed after abandonment; therefore, the old sewer may contain residual FCs that could leach from the sewer to the surrounding groundwater.
Based on the results of the geohydrological investigation, that was conducted in the area, the following observations can be made:

- Water levels in the old sewer line fluctuate even though the sewer line is blocked at the end and beginning. These water level fluctuations may be caused by infiltration of rain/groundwater into the sewer and drainage of water from the sewer to the aquifer.

- Water levels show that there is a gradient from the old sewer line towards the Blokkersdijk pond.

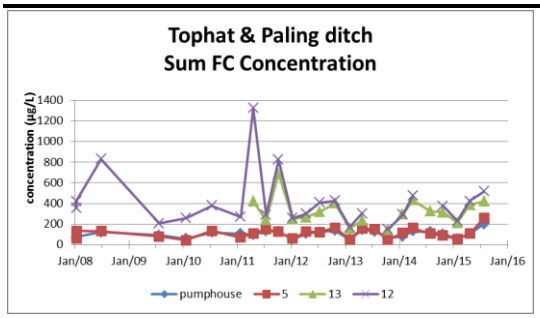
- Sediment and water samples show that residual FC mass is still present in the old sewer line.

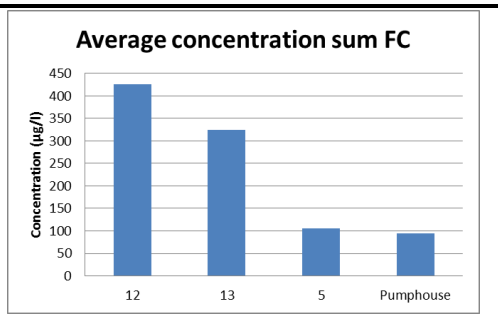
Based on these observations, it can be tentatively concluded that FC mass may leach from the old chemical sewer to the first phreatic aquifer and that the old sewer line could be a secondary contaminant source.
Further investigation (eg. mass calculations and groundwater modeling with contaminant migration) 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 FCs at four different locations. The evaluation of the monitoring results shows that FC concentrations in groundwater are stable at present, see graphs below, although concentrations can fluctuate.

Paling and Tophat Ditch - Monitoring Results since 2008



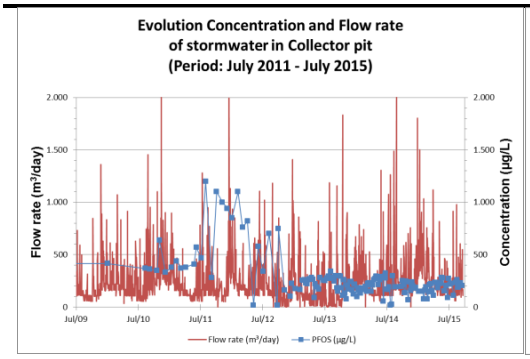


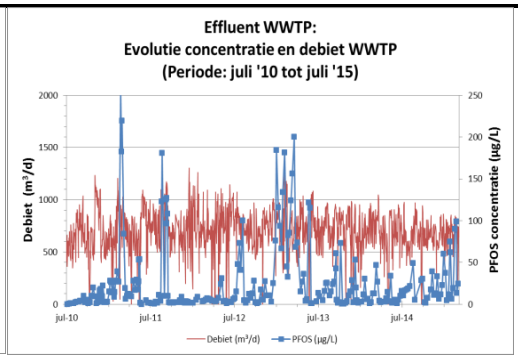
An inspection of the sampling results for the Paling and Tophat ditches shows less variability in concentration since 2012. FC concentrations in the Tophat ditch are lower than those measured in the Paling ditch.

ERM recommends continuing the groundwater 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, monthly samples are taken from the **WWTP** and **stormwater collector pit**. The monitoring results are also used to evaluate if the P&T system is effective in reducing the infiltration of contaminants into the stormwater sewer system. The collector pit results are less sensitive to fluctuations caused by rainwater and therefore more reliable.

Stormwater Collector Pit and WWTP - Monitoring Results



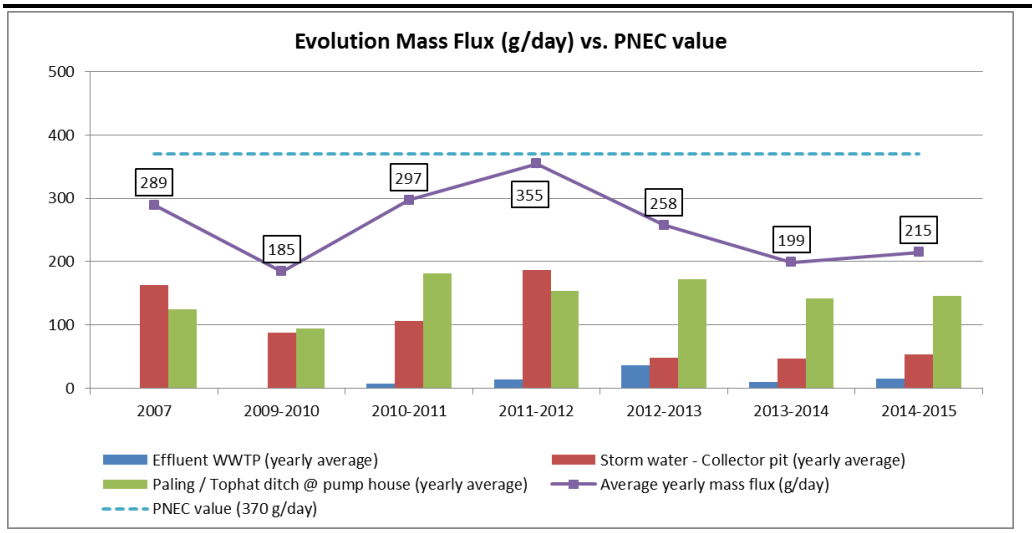


Since 2012-13, PFOS concentrations in the collector pit and waste water treatment plant have fluctuated, but show a stable trend (see figure above). The average PFOS concentration between July 2014 and July 2015 is 183 µg/l for the collector pit and 24 µg/l for the waste water treatment plant. ERM recommends continuation of the monthly monitoring of the stormwater collector pit and waste water treatment plant by 3M, and quarterly monitoring.

PFOS Mass Flux to the Schelde River

The daily average mass flux of PFOS from the 3M site to the Schelde River was calculated and compared to the maximum allowable mass flux value based on the analytical results of samples collected between July 2013 and June 2014 from the effluent of the WWTP, the storm water collector pit and the Tophat ditch near the pump station The calculated mass flux value (215 g/day) did not exceed the PNEC value (370 g/day). An overview of the calculated yearly mass flux of PFOS since 2007 is summarized in the following graph.

Average calculated Mass Flux of PFOS to the Schelde River



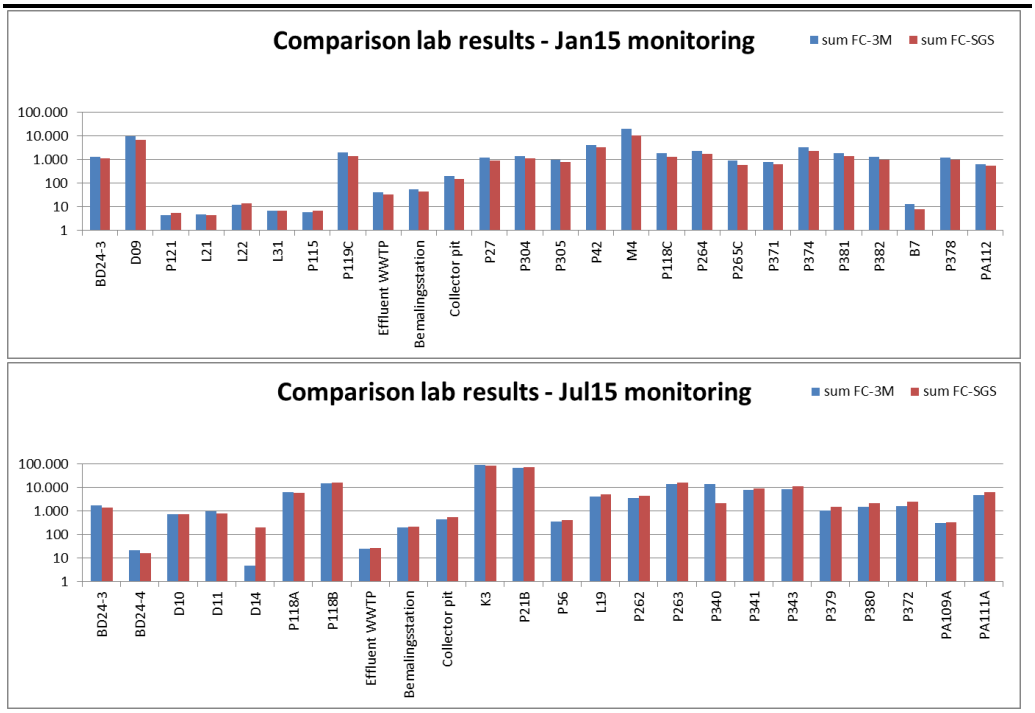
As discussed in previous reports, the higher mass flux calculated for the monitoring period 2011-2012 was likely caused by the accumulation of FC containing sludge at the bottom of the collector pit. This sludge was removed from the collector pit in September 2012. Since then, the FC concentrations in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

Evaluation analytical results water samples SGS vs 3M Environmental Laboratory

Per the agreement reached with OVAM (meeting dd. 22 October 2013) all water samples taken in 2015 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 event and analyzed by SGS nv (an OVAM certified lab) as well as the 3M Environmental Laboratory. At least one duplicate sample is taken during one of the four monitoring rounds. For the sake of consistency in this report, only the 3M results are shown.

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

Lab results comparison (SGS vs 3M Environmental Laboratory)



The average difference between the results is within the acceptable range of variation: in July 2015 the 3M results were 6% lower in April 2015 the results were 16% higher and in January 2015 results were 22% higher. The difference is likely due to different sample preparation techniques and analytical methods used by each laboratory (instrument parameters and calibration). However, the results of well P340 in July 2015 are an exception to the observation that the results are comparable: the sum FC-concentration measured by the 3M lab is 14283 µg/L, while the concentration measured by SGS is 2160 µg/L. The SGS result is in accordance with previous measured concentrations and further monitoring is needed to verify the results.

ERM recommends that in 2016 all water samples again be analyzed by the 3M Environmental Laboratory and the duplicate sampling to be spread out over the four monitoring events (similar to the monitoring in 2015). Duplicate water samples will be analyzed by the SGS laboratory during the monitoring period (January, April, July and October 2016).

End evaluation for BTEX at the tank farm area and FC's at the Zwijndrecht Z-wells

The groundwater monitoring for BTEX was discontinued last year as the concentrations were stable and below the remedial value. During the meeting with OVAM in October 2013, OVAM requested an end evaluation investigation for the (former) BTEX contamination in groundwater at this zone. The partial end evaluation report was submitted to OVAM for review on 24/Oct./2014 and approved by OVAM on 1st July 2015. ERM recommends abandoning all out of use monitoring wells in this area.

Groundwater monitoring for FCs in the four groundwater monitoring wells south of the E34 motorway, the Zwijndrecht Z-wells was discontinued last year as the remedial goal (i.e., stable concentrations that are comparable to the results prior to the start of the remedial monitoring) was met. During the meeting with OVAM in October 2013, OVAM requested that a partial end evaluation investigation be performed for FC-impact in the area of the Zwijndrecht Z-wells. The partial end evaluation report was submitted to OVAM for review on 24/Oct./2014 and approved by the OVAM on 1st July 2015. All remaining wells will be abandoned in accordance with the relevant regulations.

Environmental Follow-up of an Environmental Incident

On August 11th, 2014, a minor leak of a 20% HF Solutia was noticed in Building 017. The leak was closed and the inside of the building was decontaminated with water. Part of the solution and the cleaning water leaked outside to the soil. The incident was reported to OVAM in accordance with local regulations.

Two soil samples were taken to assess the impact of the leak: one sample of the impacted area and one sample of a nearby, undisturbed area. The analyses showed an elevated fluoride concentration in the impacted area. The measured concentration is below the EPA screening level for industrial use. The pH levels are normal.

Because it was a minor impact and the measured concentration of fluoride lie below the relevant screening level, ERM concludes that no further actions are needed. It is recommended that the area is included in the next periodic soil investigation of the site (to be conducted in 2017).

The results and conclusions were reported to OVAM in 2015 and the report and recommended actions were approved.

General Conclusions and Recommendations

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

The P&T system, which has been operational since 2011, currently consists of nine extraction wells. Its main purpose is to reduce FC concentrations and mitigate horizontal and vertical contaminant migration. Between July 2011 and June 2015 approximately 52.700 m^3 of contaminated groundwater was extracted and approximately 362 kg of FCs were removed.

The system had an average up-time of ca. 73%, but the up-time varies from well to well (min. 13% and max. 96%). The long-term trend in FC

concentrations in most of the extraction and monitoring wells is stable, although fluctuations can occur. Concentrations in some wells have increased and further follow up is needed.

The (potential) increasing trend in some of the Blokkersdijk monitoring wells and the 3M pond needs to be verified via monitoring and statistical evaluation. Based on the results of the geohydrological investigation in the Blokkersdijk nature reserve, it can be tentatively concluded that the sewer line may be a secondary contaminant source. However, further investigation is necessary to assess the impact of the FC mass present in the old chemical sewer on the water quality of the first phreatic aquifer and nearby ponds. At present, there is no need for additional remedial actions in this area as there is no increasing trend observed in the Blokkersdijk pond.

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

One minor incident occurred at the 3M site between July 2014 - July 2015. The sampling results identified no impact to soil and/or groundwater, therefore, no further action is required. The impacted area will need to be assessed during the next periodic soil investigation.

ERM recommends continuing the remediation and groundwater monitoring program as summarized in the following table to assess the long-term FC concentration trends. In addition ERM recommends evaluating the need for an extension of the remedial system to optimize the contaminant capitation. During this evaluation it should be investigated how the operation of the system can be improved. ERM also recommends continuing the investigation of the old chemical sewer.

The next interim report will be submitted to OVAM before the end of 2016, describing the remedial actions and monitoring results taken from July 2015 through July 2016.

■

Proposed Remedial Monitoring Program for 2016

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

Location	Number of Wells	Frequency	Well ID	Rationale	Remarks
Southern Site Boundary	7	Semi-annual	B3-bis, B7, PA111A, PA112, P378, PA109A, P372	-	Well B3 lost and replaced by well B3-bis in September 2015.
Tank Farm Area					
TPH	2	Annual	P18, P28	TPH monitoring at request of OVAM	Source investigation ongoing
Blokkersdijk Nature Reserve					
Wells	4	Quarterly	L21, L22, P115, L31	Increased frequency to populate data set for statistical evaluation	P119C was moved from the Blokkersdijk monitoring to the monitoring of the WWTP source zone because of its location (downgradient from 3M path and upgradient from source zone)
	3	Quarterly	L4, P114bis, P116	-	-
Surface water	2	Quarterly	Blokkersdijk standard, 3M pond	-	-
Paling and Tophat Ditch	4	Quarterly	12, 13, 5, Pumping House	-	-
Storm Water	1	Quarterly	Collector pit	Monthly samples analyzed by 3M internal lab	Quarterly verification samples by OVAM certified laboratory (eg. SGS)
Effluent WWTP	1	Quarterly	Discharge line	Monthly samples analyzed by 3M internal lab	Quarterly verification samples by OVAM certified laboratory (eg. SGS)

Legend

Increased monitoring frequency

Decreased monitoring frequency

No change

1. *INLEIDING*

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

Voorliggend verslag betreft het Zesde tussentijds verslag (TTV6) van de saneringswerken, zoals beschreven in het door de OVAM conform verklaarde bodemsaneringsproject (1e fase) (1) en de vijf voorgaande tussentijdse verslagen (2), (3), (4), (5) en (6). Het betreft de saneringswerken op het deel productiezone, voormalige slibbekkens/waterzuiveringsinstallatie, en het natuurgebied Blokkersdijk. De beschouwde saneringsperiode loopt van juli 2014 tot en met juli 2015.

De organofluorimpact ter hoogte van de zuidelijke perceelgrens en de Palingbeek wordt momenteel door middel van een grondwatermonitoring opgevolgd. Wanneer er meer duidelijkheid is over de impact van de werken in het kader van de Oosterweelverbinding op deze zone, kan de saneringsaanpak voor deze verontreiniging uitgewerkt worden in een tweede gefaseerd bodemsaneringsproject.

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

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

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

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

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

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

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 westen is het natuurgebied Blokkersdijk aanwezig.

Voor meer details in verband met de omgevingskenmerken wordt verwezen naar het eerste tussentijds verslag [2].

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 467d 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 volgende 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 [1].

2.3 GEOLOGISCHE EN HYDROGEOLOGISCHE GEGEVENS

Het geologisch kader is al uitvoerig beschreven in het eerste tussentijds verslag [2]. In het tweede tussentijds verslag [3] 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 (juni 2015) 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 vastgesteld (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 zone Gebouw 16;
- Bronzone FC verbindingen zone Waterzuiveringsinstallatie (WWTP);
- FC verontreiniging in grondwater 2e aquifer;
- FC verontreiniging in de regenwaterriolering door insijpelend verontreinigd grondwater;
- FC verontreiniging in het Blokkersdijk natuurgebied door atmosferische depositie;
- FC verontreiniging 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. Hieruit blijkt dat de vuilvracht aan FC verbindingen welke vóór de sanering naar de Schelde stroomde, aanvaardbaar was.

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

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

Tabel 2.1 Gemiddelde PFOS vuilvracht richting de Schelde (2007 tem. 2014)

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**

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

* Debiet in kader van pilootproef

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

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

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

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

3. ALGEMENE BESCHRIJVING SANERINGSWERKEN

3.1 ALGEMEEN

In het eerste gefaseerd bodemsaneringsproject [1] zijn de selectie en uitwerking beschreven van de saneringsaanpak voor de verontreinigingen met organofluorverbindingen (FC's) en vluchtige aromaten (BTEX) op de site van 3M, waarvoor een saneringsnoodzaak bestaat. De saneringswerken uitgevoerd tussen juli 2009 en juni 2014 zijn beschreven in de vijf voorgaande tussentijdse verslagen [2], [3], [4], [5] en [6].

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. Er zijn immers heel wat onduidelijkheden over de timing en uitvoeringsmodaliteiten van de werken welke ter hoogte van de Palingbeek voorzien zijn in het kader van de Oosterweelverbinding. Daar deze werken een belangrijke impact kunnen hebben op de waterhuishouding op deze locatie, dient een saneringsaanpak afgestemd te worden op deze werken.

3.2 SANERINGSWERKEN ZOALS VOORZIEN IN HET EERSTE GEFASEERD BODEMSANERINGSPROJECT

3.2.1 *Algemene saneringsdoelstelling*

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

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

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

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

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

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

3.2.2 Grondhoop verontreinigd met kwik en FC verbindingen

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

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

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

3.2.3.1 Doelstelling

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

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

3.2.3.2 Injectie

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

Volgende fases zijn bepaald:

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

3.2.3.3 Monitoring

Een maand na de injectie zullen wekelijks het zuurstofgehalte, de pH, EC en temperatuur bepaald worden ter hoogte van een zestal peilbuizen. Om de twee weken zullen eveneens de concentraties aan BTEX bepaald worden. Na

de eerste maand zullen de hoger vermelde veldparameters eenmaal per maand gemeten worden en de concentraties aan BTEX elke drie maanden.

3.2.4 Grondwaterextractie zone Gebouw 16

3.2.4.1 Doelstelling

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

3.2.4.2 Gefaseerde opbouw grondwaterextractiesysteem

Volgende fases zijn gedefinieerd in het bodemsaneringsproject:

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

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

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

3.2.4.3 Monitoring

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

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

Daarnaast zal in de eerste drie jaren maandelijks een staal genomen worden van het opgepompte grondwater uit de zone WWTP en de zone Gebouw 16, nadat deze stromen zijn samen gekomen en voordat ze over de grondwaterzuiveringsinstallatie worden geleid (zie ook paragraaf 3.2.5). De

frequentie zal worden afgebouwd tot een tweemaandelijkse bemonstering na drie jaar en driemaandelijks na 6 jaar werking. Deze informatie dient meer inzicht te geven in de vuilvracht welke door de actief koolfilters dient verwijderd te worden. De stalen worden geanalyseerd op SOF, individuele FC verbindingen (PFOS, PFOA), anorganisch fluoride, BTEX, arseen, chroom, COD en N. De bemonsteringsfrequentie alsook het analysepakket kan aangepast worden in functie van de bekomen resultaten.

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

3.2.5 Grondwaterextractie zone WWTP

3.2.5.1 Doelstelling

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

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

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

- De concentraties aan FC verbindingen ter hoogte van de terreingrenzen mogen de 10% oplosbaarheid niet overschrijden; en
- De concentraties aan FC verbindingen in de bronzone in de 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	-

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 m³/dag. Het

onttrokken grondwater zal over de grondwaterzuivering van 3M geleid worden;
- Fase 3: Evaluatie van de efficiëntie van het onttrekkingssysteem gedurende vijf jaar; en
- Fase 4: eventuele uitbreiding van het onttrekkingssysteem.

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

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

3.2.5.3 *Monitoring*

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

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

Daarnaast zal in de eerste drie jaren maandelijks een staal genomen worden van het opgepompte grondwater uit de zone WWTP en de zone gebouw 016, nadat deze stromen zijn samengekomen en voordat ze over de grondwaterzuiveringsinstallatie worden geleid (zie ook paragraaf 3.2.4). De frequentie zal worden afgebouwd tot een tweemaandelijkse bemonstering na drie jaar en driemaandelijks na 6 jaar werking. Deze informatie dient meer inzicht te geven in de vuilvracht welke door de actief koolfilters dient verwijderd te worden. De stalen worden geanalyseerd op SOF (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 hun milieuvergunning.

3.2.6 Monitoring 2e aquifer

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 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 vastgesteld, 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 vastgesteld 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 (1).

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

3.2.9 Zuidelijke terreingrens, Palingbeek en Tophatgracht

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

In afwachting van het tweede gefaseerd bodemsaneringsproject is voorzien om een 7-tal peilbuizen jaarlijks te bemonsteren. Daarnaast dient elke 6 maanden een oppervlaktewaterstaal genomen te worden ter hoogte van de

Palingbeek, Tophatgracht en het pompstation ter hoogte van de Schelde. De stalen dienen te worden geanalyseerd op FC verbindingen.

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

Om de evolutie van de FC concentraties in het landbouwgebied ten zuiden van de Expresweg na te gaan, zullen 4 peilbuizen halfjaarlijks bemonsterd worden. De stalen zullen geanalyseerd worden op FC verbindingen.

3.2.11 Vuilvracht richting de Schelde

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

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

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

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

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

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

3.3 WIJZIGINGEN IN HET BODEMSANERINGSPROJECT VOLGENS VOORGAANDE TUSSENTIJDSE VERSLAGEN

Ten opzichte van de saneringsaanpak zoals voorzien in het eerste gefaseerd bodemsaneringsproject (1) zijn in de voorgaande tussentijdse verslagen (2), (3), (4), (5) en (6) een aantal wijzigingen voorgesteld. Deze wijzigingen zijn toegelicht in onderstaande paragrafen. In paragraaf 3.3.11 wordt een overzicht gegeven van het monitoringsprogramma voor 2016.

3.3.1 Grondhoop verontreinigd met kwik en FC verbindingen

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

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

In het eerste tussentijds verslag (TTV1[(2)]) was al beslist dat een actieve saneringsaanpak voor de BTEX verontreiniging in het grondwater niet noodzakelijk was aangezien de concentraties in het grondwater sterk waren afgenomen en natuurlijke afbraak plaatsvindt. Bijgevolg werd een monitoring voorgesteld om de daling van de BTEX grondwaterconcentraties te bevestigen.

Uit de monitoring blijkt dat de concentraties blijven dalen, de concentraties liggen onder de relevante richtwaardes en dus is ook de saneringsdoelstelling behaald. Daarom is in het vierde tussentijds verslag voorgesteld om de monitoring van de BTEX concentraties in het grondwater stop te zetten (zie TTV4 [(5)]).

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 1/07/2015 door de OVAM conform verklaard (ref. BB-O-DS-20150289277).

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

3.3.3 Grondwaterextractie zone Gebouw 16 en zone WWTP

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

3.3.3.1 Fasering

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

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

3.3.3.2 Design en opvolging grondwateronttrekkingssysteem

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

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

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

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

Tabel 3.3 Overzicht geraamde onttrekkingsdebieten

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

3.3.3.3 *Grondwatermonitoring*

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

Aangezien de grondwatermonitoring ter hoogte van zone WWTP intussen zes jaar loopt, is, in overeenstemming met het conform verklaarde bodemsaneringsproject, sinds 2013 de monitoringsfrequentie van de meeste peilbuizen verlaagd tot jaarlijks. Om een beeld te krijgen van de verontreinigingstoestand stroomopwaarts van de bronzone zijn twee nieuwe peilbuizen geplaatst (P381 en P382) en is peilbuis P264 toegevoegd aan het programma. De monitoringsfrequentie van peilbuizen P118C, P264, P265C, P381 en P382 is sinds 2013 halfjaarlijks. Sinds 2014 is de monitoringsfrequentie van peilbuizen P371 en P374 eveneens halfjaarlijks om een stijgende trend voor bepaalde FC's beter te kunnen opvolgen. Eerder zijn drie peilbuizen (P11, P265B en P361) geschrapt uit het monitoringsprogramma omdat de concentraties ter hoogte van deze peilbuizen een dalende trend vertonen en niet meer noodzakelijk zijn voor de monitoring van de WWTP bronzone.
In het vijfde tussentijds verslag (TTV5[6]) 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.

3.3.4 *Monitoring 2e aquifer*

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

3.3.5 *Reductie van de FC impact in de regenwaterriolering*

Omdat de concentraties gemeten in de regenwaterriolering zeer gevoelig zijn aan fluctuaties als gevolg van neerslag is, in overleg met OVAM, besloten om vanaf 2014 de driemaandelijkse monitoring van de regenwaterriolering (RW3,

RW4, RW12 en RW13) te schrappen en te vervangen door de maandelijkse monitoring van de collectorput.

3.3.6 Natuurgebied Blokkersdijk

In het vierde tussentijds verslag (TTV4[5]) 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[6]) is aanbevolen om peilbuis P119C toe te voegen aan de monitoring van de WWTP bronzone, aangezien deze peilbuis stroomafwaarts ligt van het natuurgebied maar stroomopwaarts van de WWTP bronzone.
De monitoringsfrequentie van de overige peilbuizen en van de Blokkersdijkvijver en de 3M blijft ongewijzigd (halfjaarlijks respectievelijk driemaandelijks). Omwille van de slechte toegankelijkheid van het staalnamepunt "Blokkersdijk oost" is in overleg met OVAM (dd. 22 oktober 2013) besloten om dit staalnamepunt uit de monitoring te schrappen.

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

3.3.7 Zuidelijke terreingrens

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

3.3.8 Palingbeek en Tophatgracht

De monitoringsfrequentie is in 2011 verhoogd naar driemaandelijks.

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

In het eerste gefaseerd bodemsaneringsproject, dat in 2008 door Arcadis [1] 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 [5]).

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 1/07/2015 door de OVAM conform verklaard (ref. BB-O-DS-20150289432). Alle peilbuizen in deze zone zullen definitief buitengebruik worden gesteld conform de wettelijke voorschriften in de periode juli 2015-juni 2016.

De peilbuizen in deze zone zullen definitief buiten gebruik gesteld worden in de periode juli 2015 – juli 2016 conform de wettelijke voorschriften.

3.3.10 Vuilvracht richting de Schelde

Geen wijzigingen.

3.3.11 Monitoringsprogramma 2015

Op basis van de resultaten uit de periode juli 2011 tot en met juli 2014 is het monitoringsprogramma zoals opgenomen in het conform verklaarde BSP fase 1 waar nodig of wenselijk aangepast. In de volgende tabel is een overzicht gegeven van het gewijzigde monitoringsprogramma voor de periode juli 2014 – juli 2015, met verantwoording. Voor meer details wordt verwezen naar het vijfde TTV[6].

Tabel 3.4 Monitoringsprogramma 2015

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
P&T systeem	9	Driemaandelijks	PP01, PP02,PP04, PP05, PP06, PP07, PP08, PP09, PP10	-	Minstens maandelijkse inspectie van de installatie en de onttrekkingsputten
Stabiliteit	-	Eenmaal (stopzetting indien akkoord stabiliteitsdeskundige)	-	Geen onaanvaardbare zettingen en een technische evaluatie door onafhankelijke stabiliteitsdeskundige geeft aan dat er geen verder noodzaak is voor monitoring. Te bevestigen door eenmalige controle.	-
Grondhoop	-	Vijfjaarlijks	Visuele inspectie van grondhoop	Bovenafdek vervangen in 2012	Vanaf 2013 vijfjaarlijkse monitoring, aangezien bovenafdek in 2012 vervangen is; eerst volgende controle: 2018
FC Bronzones					
Gebouw 16	2	Jaarlijks	K3, P56	-	PFBS zal worden toegevoegd aan het analyse-pakket om de concentraties verder op te volgen
	5	Halfjaarlijks	P21B, P27, P42, P304, P305	Verhoogde frequentie om de evolutie van de som FC concentratiecontour beter te kunnen opvolgen (halfjaarlijks, ipv. jaarlijks)	P27 ter vervanging van K1 omdat de concentraties in peilbuis K1 sinds de start van de monitoring in 2008 stabiel en laag liggen
WWTP	6	Jaarlijks	P262, P340, P341, P343, P379, P380	-	-
	7	Halfjaarlijks	P118C, P264, P265C, P371, P374, P381, P382	-	-
	4	Halfjaarlijks	L19, M4, P119C, P263	Verhoogde frequentie om de evolutie van de som FC concentratiecontour beter te kunnen opvolgen (halfjaarlijks, ipv. jaarlijks)	P119C van Blokkersdijk monitoring naar bronzone monitoring gezien de locatie (stroomafwaarts van 3M pad, maar stroomopwaarts van bronzone)
Tweede aquifer	4	Jaarlijks	BP24-4, D11, D14, P118B	-	-
		Jaarlijks	D10, P118A	Stijgende trend niet bevestigd	
	1	Halfjaarlijks	D9	D9 zal halfjaarlijks bemonsterd worden (opvolging mogelijke stijgende trend)	Indien de stijgende trend niet wordt bevestigd bij twee opeenvolgende monitoringsrondes, zal de monitoringsfrequentie terug worden verlaagd naar jaarlijks
	4	Halfjaarlijks	BD24-3, P121	-	-
	1	Driemaandelijks	P321	-	-

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
Zuidelijke terreingrens	7	Halfjaarlijks	B3, B7, P372, P378, PA109A, PA111A, PA112	-	-
Ten zuiden van expresweg (Z-peilbuizen)	-	-	-	Monitoring stopgezet aangezien concentraties stabiel en laag zijn.	Er is een eindevaluatieonderzoek uitgevoerd worden voor deze zone. Na conformiteit van dit onderzoek zullen alle Z-peilbuizen buiten gebruik gesteld worden volgens de wettelijke voorschriften.
Ondergrondse tanken					
BTEX	-	-	-	BTEX monitoring is begin 2013 stopgezet vermits saneringsdoel is behaald	Er zal een eindevaluatieonderzoek uitgevoerd worden voor deze zone. Na conformiteit van dit onderzoek zullen alle Z-peilbuizen buiten gebruik gesteld worden volgens de wettelijke voorschriften.
Minerale olie (MO)	2	Jaarlijks	P18, P28	Monitoring op vraag van OVAM	Brononderzoek lopende
Blokkersdijk Natuurgebied					
Peilbuizen	4	Halfjaarlijks	L21, L22, P115, L31	-	P119C van Blokkersdijk monitoring naar bronzone monitoring gezien de locatie (stroomafwaarts van 3M pad, maar stroomopwaarts van bronzone)
	3	Driemaandelijks	L4, P114bis, P116	-	-
Oppervlaktewater	2	Driemaandelijks	Blokkersdijk standaard, 3M vijver	-	-
Palingbeek en Tophat gracht	4	Driemaandelijks	12, 13, 5, Bemalingsstation	-	-
Regenwater	1	Maandelijks	Collector put	Maandelijkse stalen geanalyseerd door 3M labo te Zwijndrecht	Driemaandelijkse verificatie van de stalen door SGS
Effluent WWTP	1	Driemaandelijks	Lozingspijp	Maandelijkse stalen geanalyseerd door 3M labo te Zwijndrecht	Driemaandelijkse verificatie van de stalen door SGS

Legende

Verhoogde monitoringsfrequentie
Verlaagde monitoringsfrequentie
Geen wijzigingen

4. UITGEVOERDE MONITORING EN RESULTATEN

4.1 ALGEMEEN

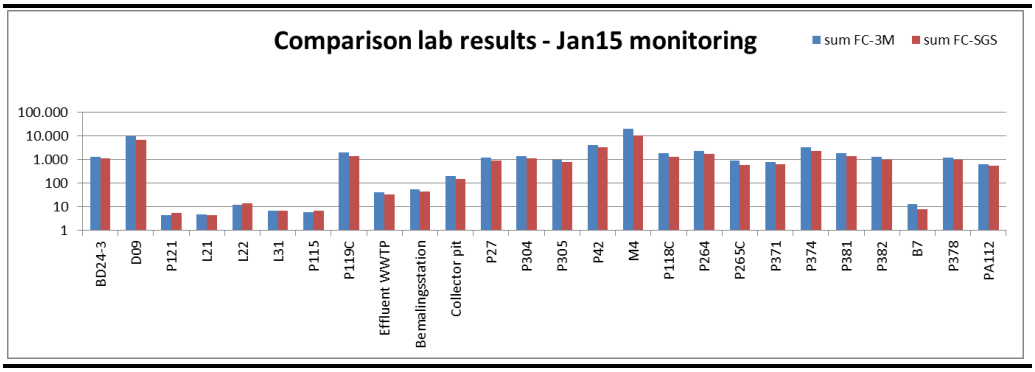
De monsternamewerkzaamheden zijn door ERM uitgevoerd conform de CMA-procedures (Compendium voor monsterneming en analyse, meest recente versie). Tijdens de grondwaterbemonstering zijn temperatuur, zuurtegraad (pH) en elektrische geleidbaarheid (conductiviteit) gemeten en zijn de grondwaterstanden in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

Zoals overeengekomen met OVAM tijdens het overleg van 12 november 2014 is besloten om in 2015 de wateranalyses van alle monitoringsrondes opnieuw te laten analyseren door het 3M Environmental Laboratory en alle waterstalen minstens éénmaal door het SGS nv geanalyseerd dienen te worden. Overeengekomen is de controle staalname en analyse te spreiden over de vier monitoringsperiodes (in plaats van één controle tijdens de monitoring van juli).

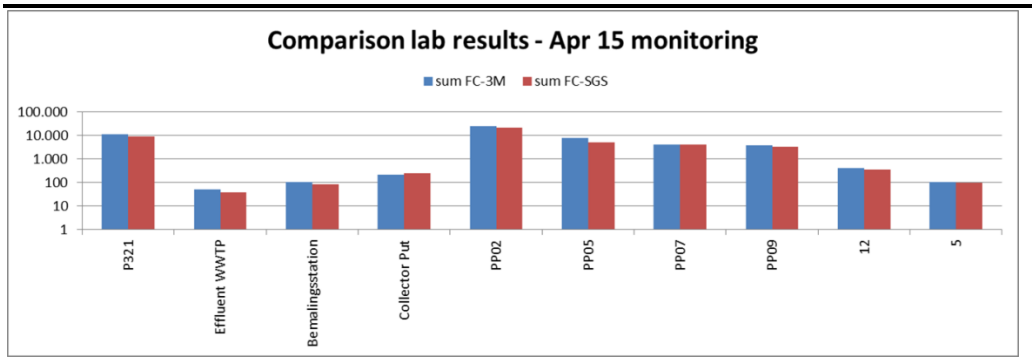
In de figuren en grafieken opgenomen in dit verslag zijn, omwille van consistentie, enkel de analyseresultaten van het 3M Environmental Laboratory gebruikt.

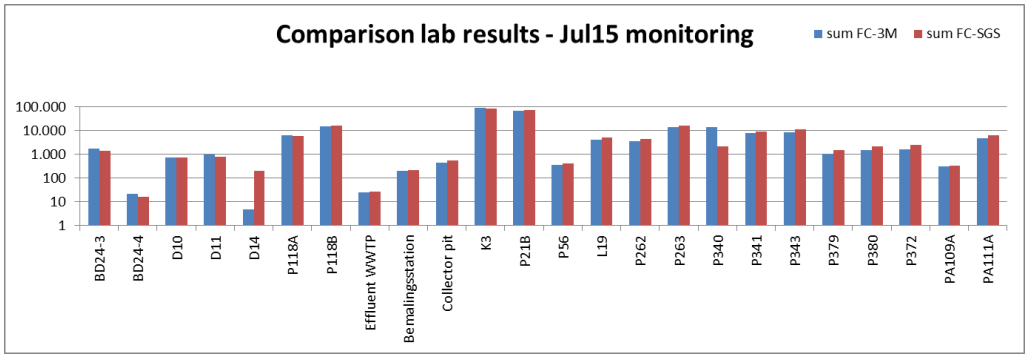
Uit een evaluatie van de datasets voor 2015 (SGS vs. 3M labo) volgt dat de resultaten van beide labo's in de meeste gevallen vergelijkbaar zijn (zie Grafiek 4.1). Gemiddeld ligt het verschil tussen de resultaten van beide labo's binnen de normale analysevariatie: in juli lagen de 3M resultaten 6% lager, in april 16% hoger en in januari 22% hoger. Een uitzondering hierop zijn de resultaten voor P340 gemeten in juli 2015: de concentratie aan som FC's gemeten door 3M is 14.283 µg/L, bij SGS is de concentratie som FC's 2160 µg/L. De concentratie gemeten door SGS stemt overeen met de historisch gemeten waarden en er wordt aangenomen dat deze waarde meer accuraat is. Bijkomende monitoring is nodig om de gemeten concentraties te controleren. 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 2015



Vervolg Grafiek 4.1





ERM stelt voor dat in 2016 opnieuw alle waterstalen geanalyseerd worden door het 3M Environmental Laboratory, en dat de duplicaatstaalname en evaluatie verdeeld wordt over de vier monitoringsrondes net als in 2015. Alle waterstalen zullen gedurende de 2016 monitoringsperiode eenmaal door het SGS laboratorium geanalyseerd worden.

4.2 *GRONDHOOP VERONTREINIGD MET KWIK EN FC VERBINDINGEN*

4.2.1 *Achtergrond*

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

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

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

Tijdens een inspectie in augustus 2011 bleek dat de afdekfolie over de hoop grond in slechte staat was en daarom is in maart 2012 door de saneringsaannemer DEC nv, onder supervisie van ERM, een nieuwe

afdekking aangebracht op de grondhoop. De nieuwe afdekking minimaliseert het risico op uitloging door infiltrerend regenwater.

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

4.2.2 Uitgevoerde werken

In 2015 zijn, conform de in TTV4 gewijzigde periodiciteit (vijfjaarlijkse monitoring), geen controles uitgevoerd.

4.2.3 Conclusies en aanbevelingen

In 2013 is de controlefrequentie verlaagd van jaarlijks naar vijfjaarlijks. De volgende controle zal plaatsvinden in 2018.

4.3 FC BRONZONES: GEBOUW 16 EN WWTP

4.3.1 Uitgevoerd veldwerk

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

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

Tabel 4.1 Overzicht bemonsterde peilbuizen bronzones

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum januari 2015	Bemonsteringsdatum juli 2015	Analyse
Zone Gebouw 16				
K3	3,6-5,6		X	FC inclusief PFBS
P21B	3,5-5,5	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	FC

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum januari 2015	juli 2015	Analyse
P264	5,0-5,5	X	X	FC
P265C	4,5-5,5	X	X	FC
P340	4,0-6,0		X	FC
P341	3,5-5,5		X	FC
P343	4,8-6,8		X	FC
P371	1,5-3,5	X	X	FC
P374	5,0-6,0	X	X	FC
P379	3,5-5,5		X	FC
P380	3,5-5,5		X	FC
P381	5,0-6,0	X	X	FC
P382	2,5-3,5	X	X	FC

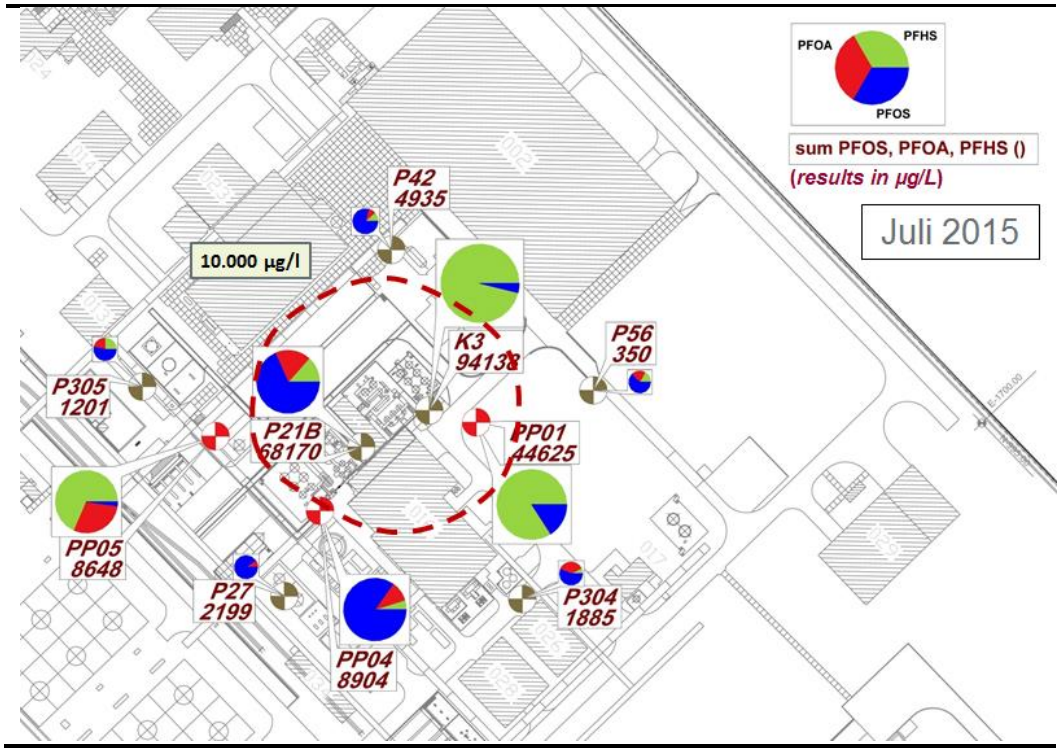
4.3.2 *Bespreking resultaten*

De analyseverslagen zijn toegevoegd in Bijlage 2. Een toetsingstabel met deze resultaten is opgenomen in Bijlage 3 samen met de resultaten van de veldmetingen.

4.3.2.1 *Gebouw 16*

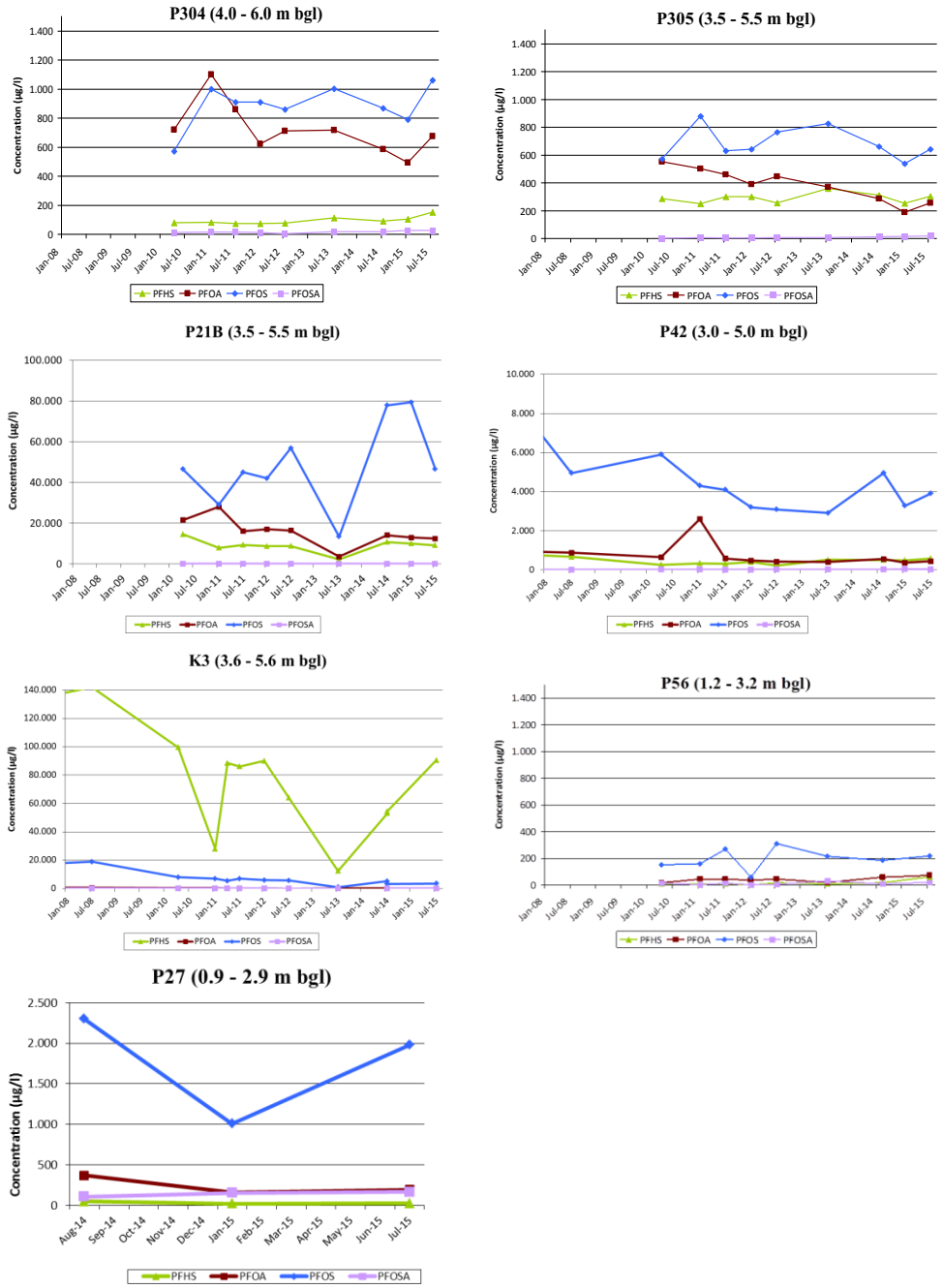
De grondwateranalyseresultaten voor de som van PFOS, PFHS en PFOA van juli 2014 ter hoogte van gebouw 016 zijn op de volgende figuur voorgesteld. De rode lijn stelt de contour van 10.000 µg/l voor de som van PFOS, PFHS en PFOA voor.

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



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

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



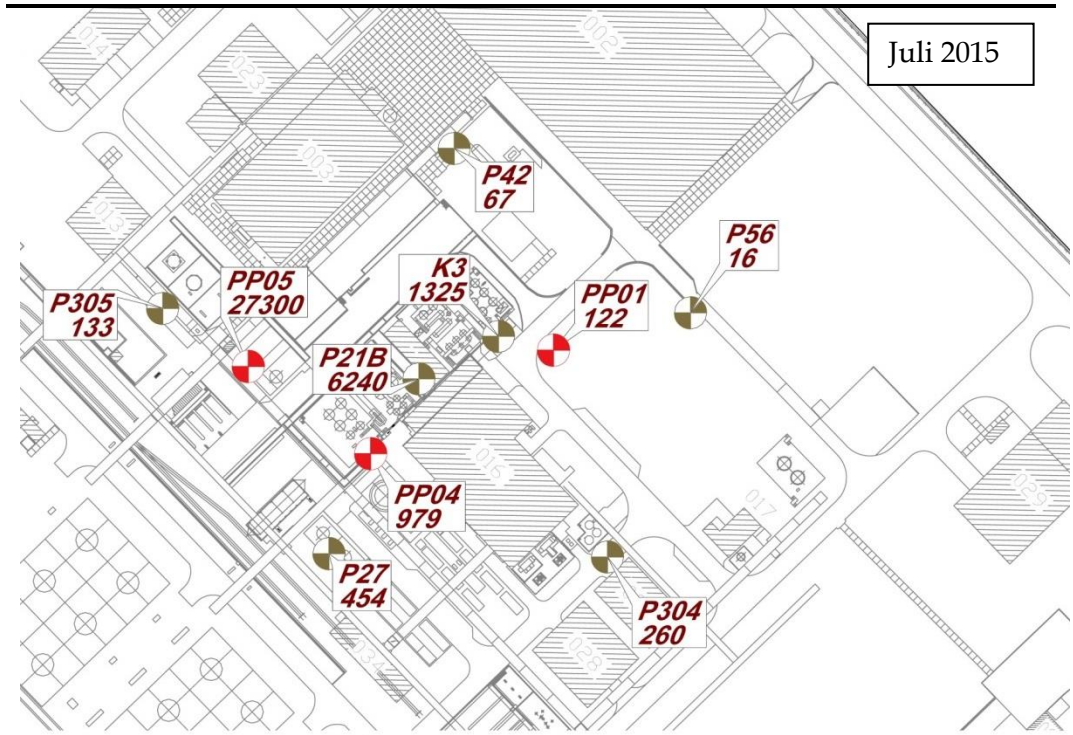
In het algemeen kan gesteld worden dat de FC concentraties in het grondwater ter hoogte van gebouw 016 dalen of stabiel zijn in de tijd, met uitzondering van de peilbuizen P21B waar algemeen een stijging van de PFOS concentratie wordt vastgesteld. Deze stijging dient verder opgevolgd te worden. In vergelijking met de vorige monitoringsperiode (2013-14) lijkt de omvang van de som FC concentratiecontour (10.000 µg/l) licht te zijn afgenomen.

De algemene stabiele/dalende trend in FC-concentraties lijkt erop te duiden dat het onttrekkingssysteem de FC-concentraties in het grondwater in de bronzone van gebouw 016 effectief capteert.

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

De resultaten voor PFBS van juli 2015 in het grondwater ter hoogte van gebouw 016 zijn op de volgende figuur voorgesteld.

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

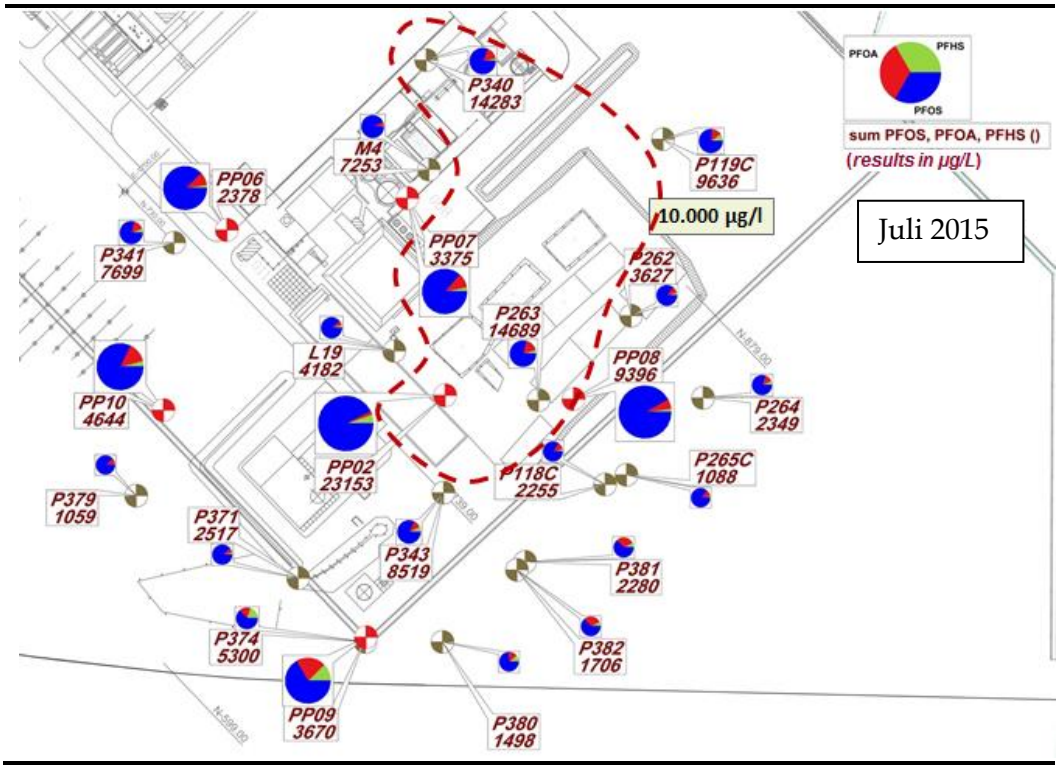


De hoogst gemeten concentraties worden gemeten ter hoogte van pompput PP05 en peilbuis P21B. De oorzaak van de verhoogde PFBS detectie is momenteel onvoldoende gekend, en wordt verder onderzocht.

4.3.2.2 *Zone WWTP*

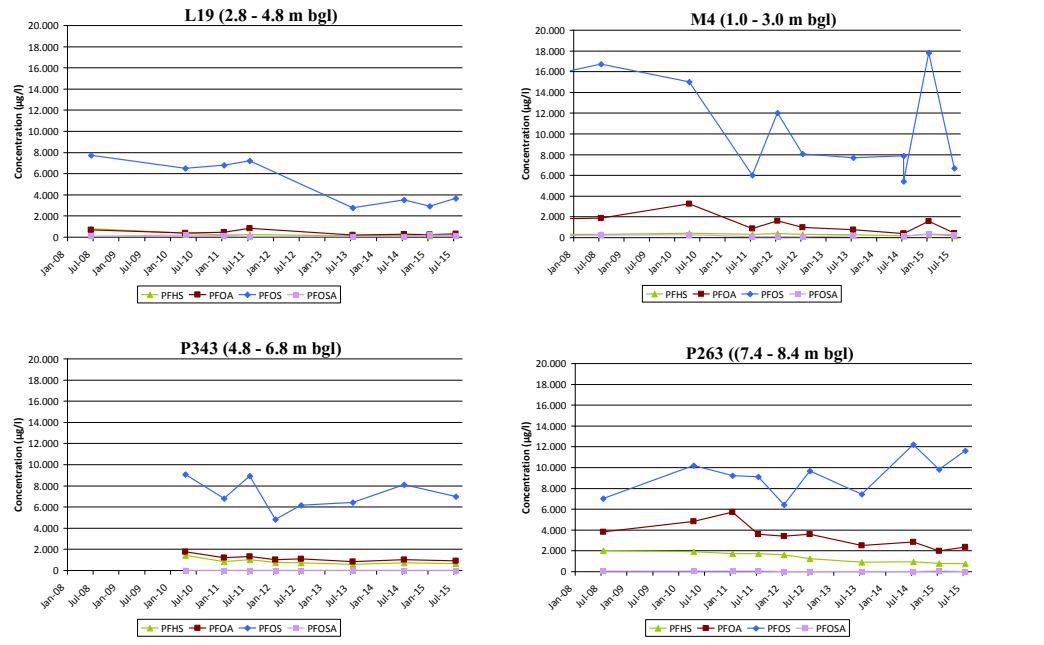
De grondwateranalyseresultaten voor de som van PFOS, PFHS en PFOA van juli 2015 ter hoogte van de WWTP zijn op de volgende figuur voorgesteld. De rode lijn stelt de concentratiecontour van 10.000 µg/l voor de som van PFOS, PFHS en PFOA voor.

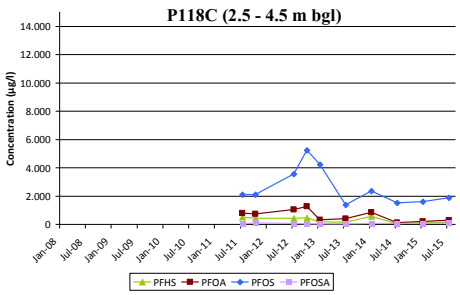
Figuur 4.3 Schematische voorstelling FC-concentraties in grondwater thv. WWTP

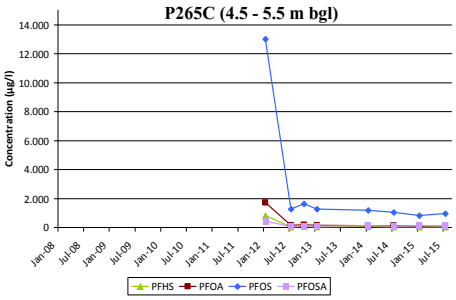


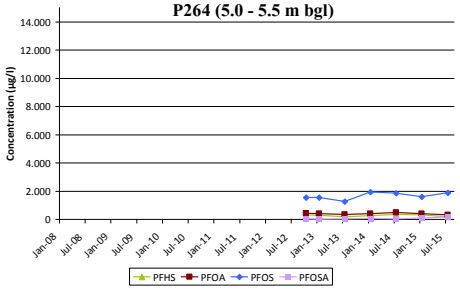
De evolutie van de concentraties aan PFOS, PFHS, PFOA en PFOSA in het grondwater ter hoogte van zone WWTP is voorgesteld in de volgende grafieken.

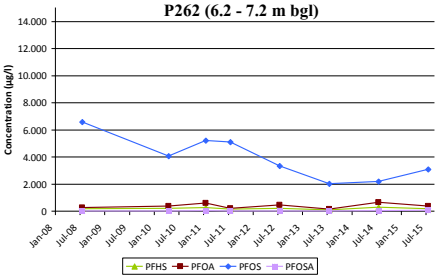
Grafiek 4.3 Evolutie FC concentraties t.h.v. zone WWTP

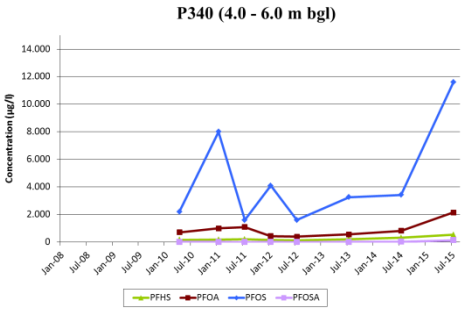


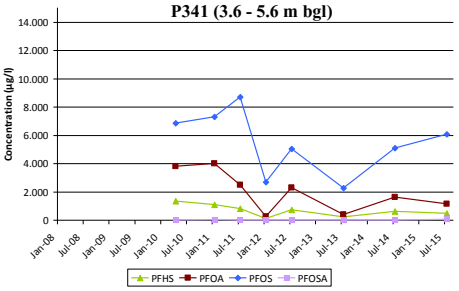

P118C (2.5 - 4.5 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA

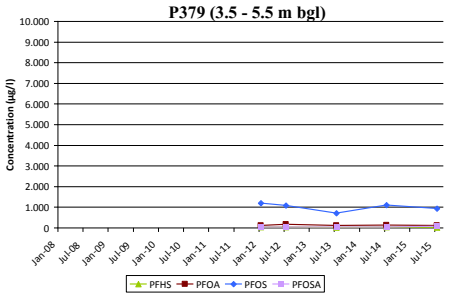

P265C (4.5 - 5.5 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA

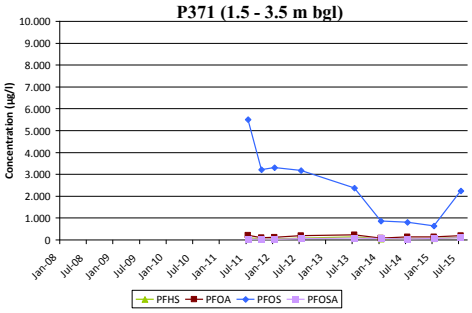

P264 (5.0 - 5.5 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA

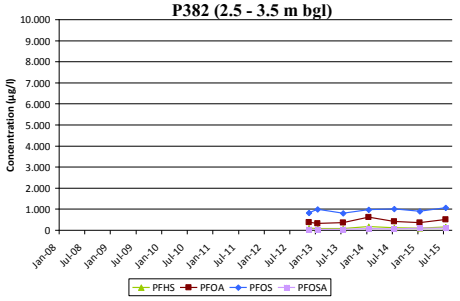

P262 (6.2 - 7.2 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA

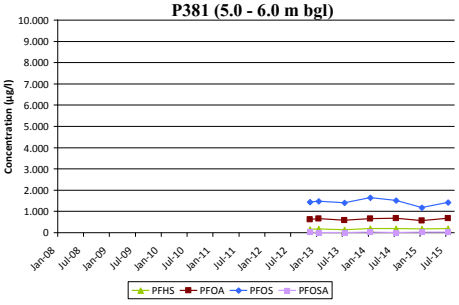

P340 (4.0 - 6.0 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA

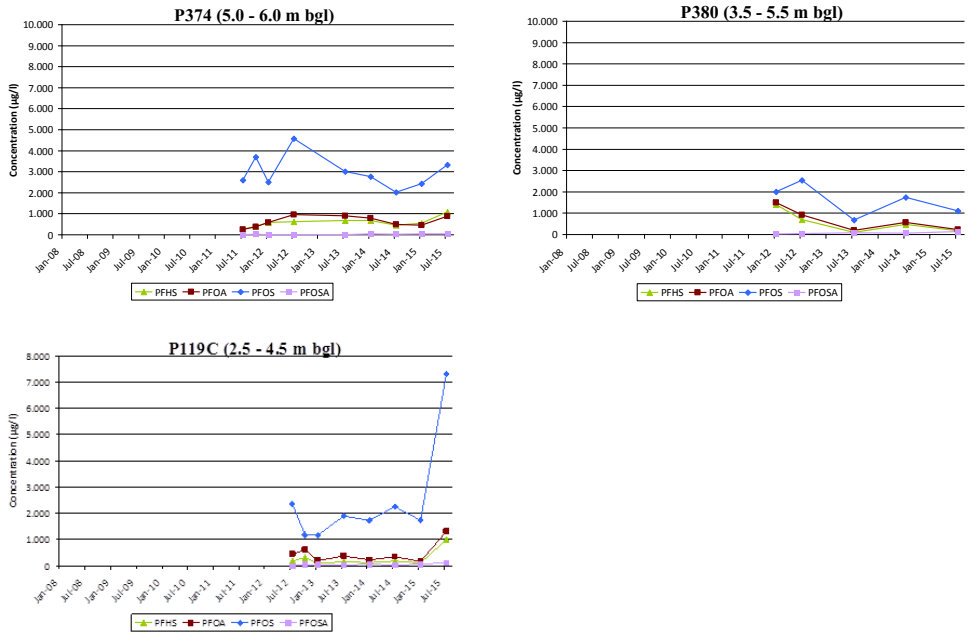

P341 (3.6 - 5.6 m bgl)
Concentration (µg/l)
PFHS PFOA PFOS PFOSA


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


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


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


P381 (5.0 - 6.0 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, met uitzondering van de concentraties ter hoogte van peilbuizen P119C, P263 en P340 waar een stijgende PFOS concentratie kan worden vastgesteld. De concentratiestijging in peilbuis P340 wordt echter niet bevestigd door de analyseresultaten van het duplicaatstaal dat door SGS geanalyseerd is. De potentiele stijging dient dus geverifieerd te worden door een volgende staalname.
De gemeten concentraties ter hoogte van peilbuis P119C worden voortaan opgenomen in de evaluatie van de evolutie van FC concentraties ter hoogte van de WWTP zone bronzone (ipv. het Blokkersdijk natuurgebied), aangezien de peilbuis stroomopwaarts ligt van de WWTP bronzone en stroomafwaarts van het natuurgebied. Tijdens de uitgevoerde metingen in juli 2015 is ter hoogte van deze peilbuis een sterk verhoogde PFOS concentratie gemeten, die niet in lijn ligt met de historisch gemeten waarden en dus geverifieerd moet worden door een volgende staalname.
De concentratiepiek gemeten ter hoogte van peilbuis M4 in januari 2015 (17800 µg/l) is niet bevestigd door de resultaten van juli 2015 (6690 µg/l) die in lijn liggen met de resultaten van vorige jaren (2012-13-14).

De omvang van de som FC concentratiecontour (10.000 µg/l) is toegenomen in vergelijking met de contour van vorige monitoringsperiode (2013-14) omwille van de verhoogde concentraties gemeten ter hoogte van peilbuis P340 en peilbuis P119C (deze is vanaf deze monitoringsperiode mee opgenomen in de monitoringszone van de WWTP). In beide peilbuizen is een sterk gestegen concentratie gemeten die niet overeenstemt met de historisch gemeten waarden en die eerst geverifieerd moeten worden. Als gevolg hiervan moet ook de observatie over de toename van de omvang van de FC contour eerst geverifieerd worden door de resultaten van de volgende staalname.

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 potentiele 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.

Om de concentratiestijging in peilbuis P21B nabij Gebouw 016 nader op te volgen wordt aanbevolen om deze peilbuis voortaan elke drie maanden te bemonsteren (in plaats van halfjaarlijks).

Om de concentratiestijging in peilbuizen P340 en P119C en de toename van de FC contour in Zone WWTP te verifiëren wordt aanbevolen om in de peilbuizen tijdens de monitoring in oktober 2015 opnieuw te bemonsteren. Verder wordt aanbevolen om de concentratiestijging in peilbuis P263 nader op te volgen en de peilbuis voortaan elke drie maanden te bemonsteren (in plaats van halfjaarlijks).

De volgende volledige monitoring in de twee bronzones zal plaatsvinden **in juli 2016**.

4.4 *TWEEDE AQUIFER*

4.4.1 *Uitgevoerd veldwerk*

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

Tabel 4.2 Overzicht bemonsterde peilbuizen tweede aquifer

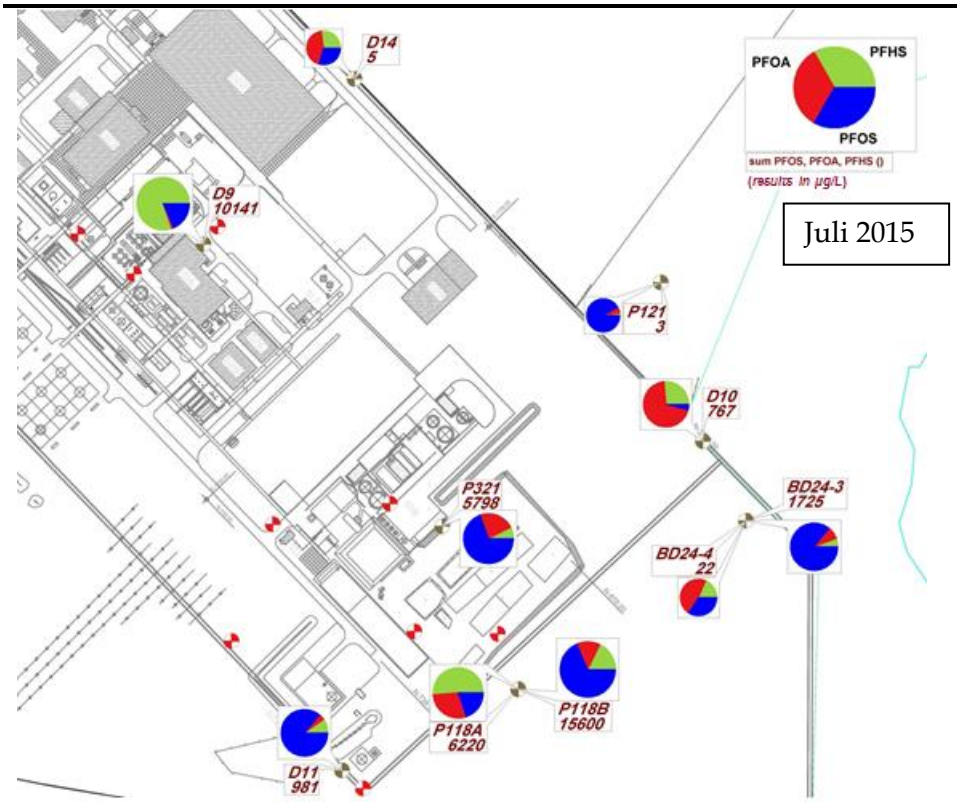
Peilbuis	Filterdiepte	Bemonsteringsdatum				Analyse
	(m-mv)	Okt. 2014	Jan. 2015	Apr. 2015	Jul. 2015	
Zone Gebouw 16						
D9	13,0-15,0		X		X	FC
D14	15,5-16,5				X	FC
Zone WWTP						
D10	15,0-16,0				X	FC
D11	14,9-15,9				X	FC
BD24-3	17,0-18,0		X		X	FC
BD24-4	22,0-24,0				X	FC
P118A	22,5-23,5				X	FC
P118B	13,0-14,0				X	FC
P121	23,5-24,5		X		X	FC
P321	15,5-15,6	X	X	X	X	FC

4.4.2 *Bespreking resultaten*

De analyseverslagen zijn toegevoegd in Bijlage 2. Een toetsingstabel met deze resultaten is opgenomen in Bijlage 3 samen met de resultaten van de veldmetingen.

De grondwateranalyseresultaten voor de som van PFOS, PFHS en PFOA van juli 2015 in de tweede aquifer zijn op de onderstaande figuur voorgesteld. De analyses uitgevoerd rond de opstart van de grondwaterextractie in de 1e aquifer (medio 2011) vormen de basis voor de verdere opvolging van de evolutie van de concentraties in de tweede aquifer.

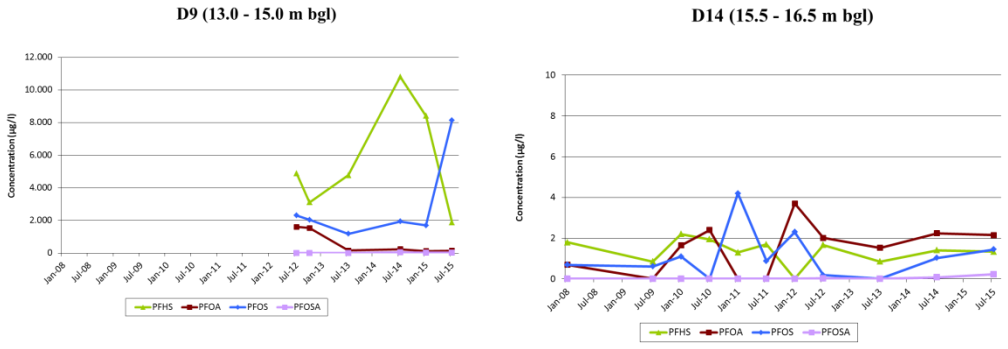
Figuur 4.4 Schematische voorstelling FC-concentraties in grondwater tweede aquifer



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

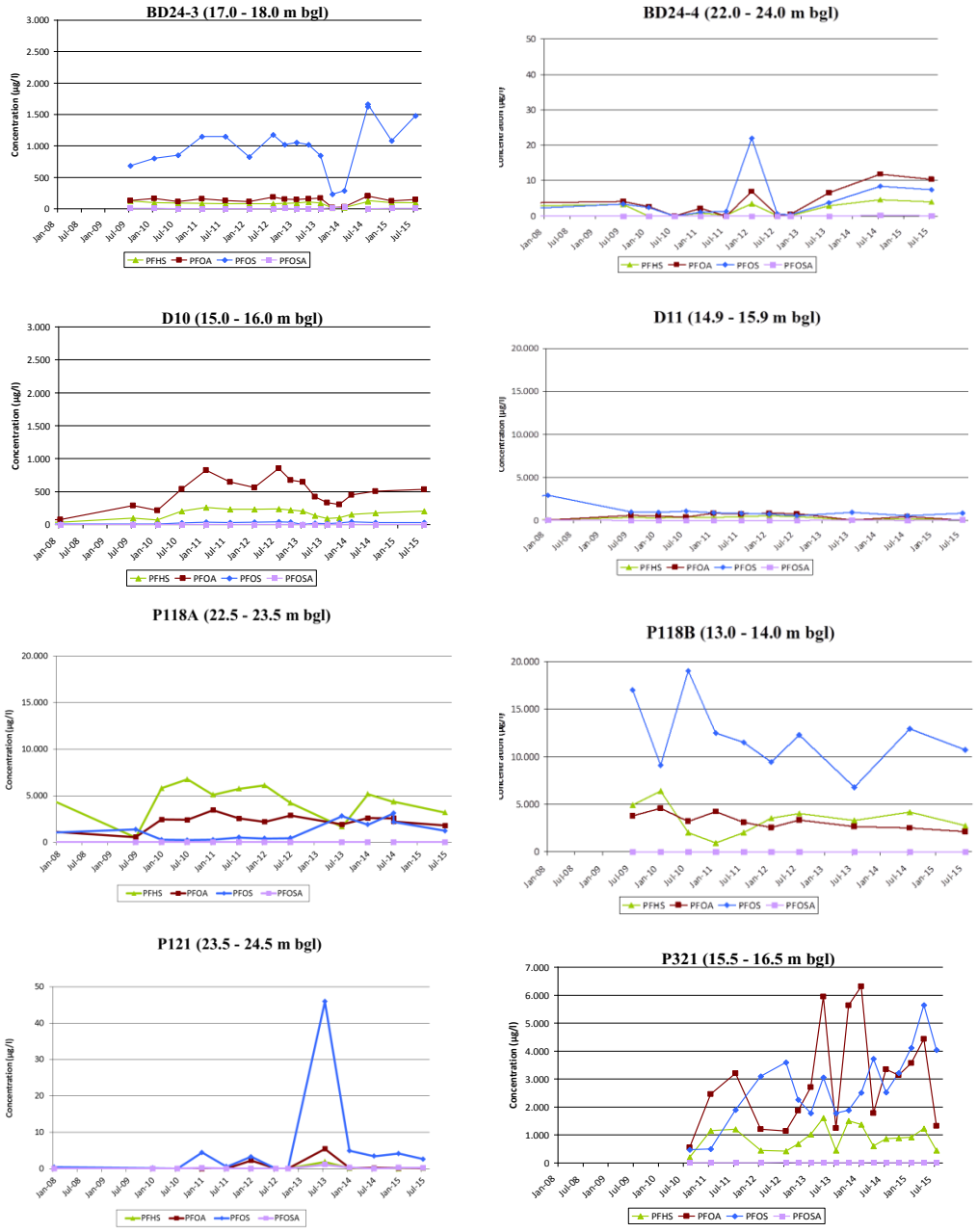
Grafiek 4.4 Evolutie FC concentraties in tweede aquifer t.h.v. Gebouw 16 en zone WWTP

Bronzone Gebouw 16



Vervolg Grafiek 4.4

Bronzone WWTP



Ter hoogte van gebouw 016 (peilbuis D9) is er een stijging van de PFOS concentraties, terwijl de PFOSA en PFOA concentraties stabiel of dalend lijken te zijn. Verhoogde PFHS concentraties gemeten in juli 2014 en januari 2015 zijn niet bevestigd tijdens de recente meetcampagne van juli 2015. Er zijn echter meer data nodig om een duidelijke uitspraak te doen voor peilbuis D9. In het grondwater ter hoogte van de stroomafwaartse peilbuis D14 zijn de FC concentraties stabiel in de tijd.

Ter hoogte van de WWTP vertonen de FC-concentraties in de meeste peilbuizen een stabiele of dalende trend met fluctuaties in de tijd. Een uitzondering hierop zijn de peilbuizen P321, BD24-3, BD24-4 en D10, waar de fluctuerende concentraties een stijgende trend lijken te tonen.

In de overige peilbuizen lijken de concentraties aan FC verbindingen in het grondwater in de tweede aquifer stabiel te zijn (P121, D11, D14, P118A) of te dalen (P118B) in de tijd, maar fluctuaties zijn mogelijk.

4.4.3 Conclusies en aanbevelingen

De gemeten concentraties in de onderzochte peilbuizen in de tweede aquifer zijn lager dat het vijfvoud van de concentraties bij de opstart van de sanering. Ter hoogte van de noordoostelijke perceelgrens (stroomafwaarts van de bronzones in de eerste aquifer) zijn de concentraties aan FC verbindingen lager dan 10% van de oplosbaarheid. Hieruit volgt dat wordt voldaan aan de saneringsdoelstellingen voor de tweede aquifer.

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

Verder wordt aanbevolen om de periodieke monitoring van peilbuizen in de tweede aquifer te behouden om het effect van het onttrekkingssysteem op de FC concentraties in de tweede aquifer te kunnen blijven evalueren. Om de concentratiestijging in peilbuis D9 te verifiëren wordt aanbevolen om in de peilbuis tijdens de monitoring in oktober 2015 opnieuw te bemonsteren.

De eerst volgende volledige monitoring zal plaatsvinden **in juli 2016**.

4.5 ZUIDELIJKE TERREINGRENS

4.5.1 Uitgevoerd veldwerk

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

Peilbuis B3 is niet bemonsterd in januari 2015 omdat de omgeving van de peilbuis onder water stond. Tijdens de uitvoering van de monitoringsronde in juli 2015 bleek het niet mogelijk deze peilbuis te bemonsteren vanwege van de weelderige vegetatie in de directe omgeving.

Om in de toekomst deze locatie te behouden in het bemonsteringsprogramma is de peilbuis op 7 oktober 2015 door Servaco nv (onder begeleiding van ERM) herplaatst op een hoger gelegen deel iets ten noorden van de locatie van peilbuis B3. De nieuwe peilbuis is B3bis genoemd.

Tabel 4.3 ***Overzicht bemonsterde peilbuizen zuidelijke terreingrens***

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum januari 2015	Bemonsteringsdatum juli 2015	Analyse
B3	0,4-1,4	- (1)	- (2)	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

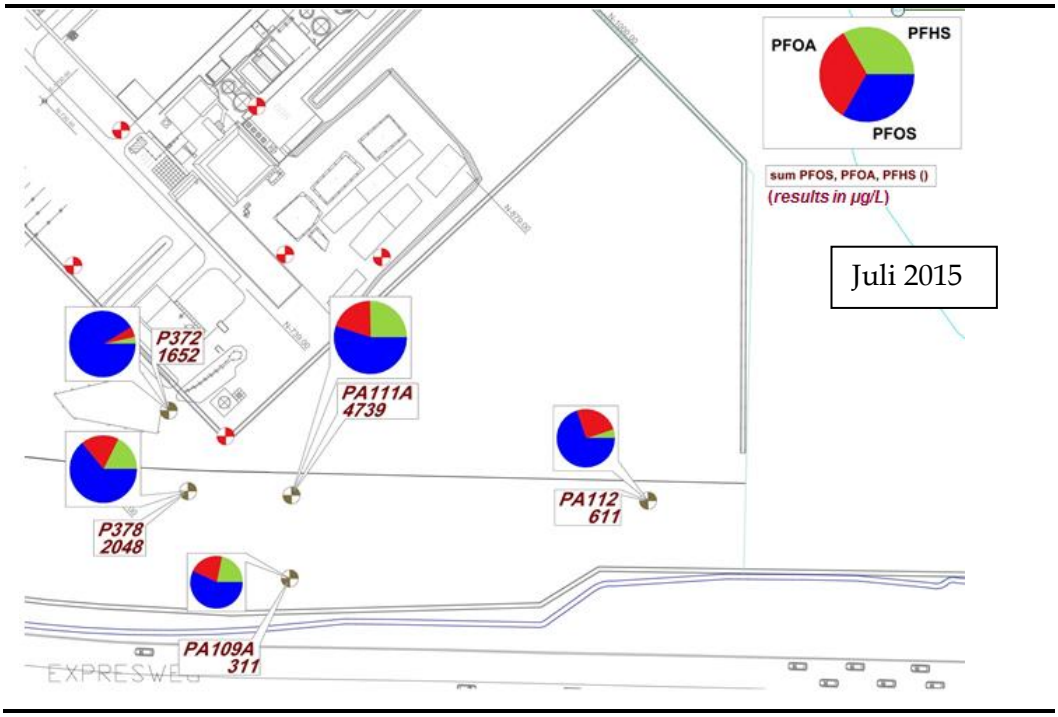
(1) *Omgeving peilbuis onder water*
(2) *Peilbuis onbereikbaar door begroeiing*

4.5.2 *Bespreking resultaten*

De analyseverslagen zijn toegevoegd in Bijlage 2. Een toetsingstabel met deze resultaten is opgenomen in Bijlage 3 samen met de resultaten van de veldmetingen.

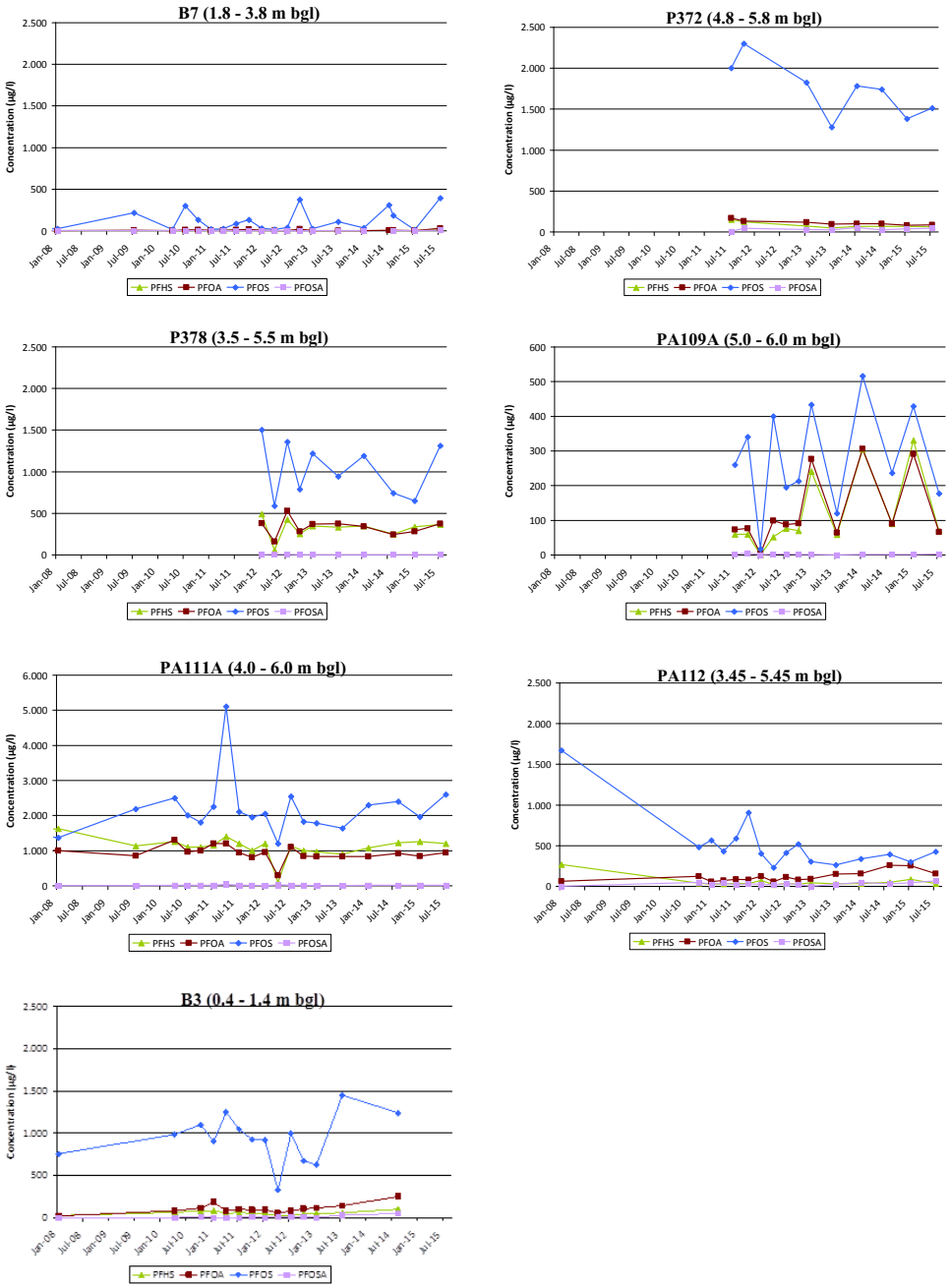
De grondwateranalyseresultaten voor de som van PFOS, PFHS en PFOA van juli 2014 ter hoogte van de zuidelijke perceelgrens zijn op de onderstaande figuur voorgesteld.

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 4.5 Evolutie FC concentraties t.h.v. zuidelijke perceelgrens



In het algemeen vertonen de FC-concentraties in het grondwater ter hoogte van de zuidelijke perceelgrens een stabiele of dalende trend met fluctuaties in de tijd. Een uitzondering hierop zijn peilbuizen B3 en PA111A, waar de fluctuerende concentraties een stijgende trend lijken te tonen. Verdere metingen dienen deze mogelijk stijgende trend nog te bevestigen.

4.5.3 Conclusies en aanbevelingen

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

de WWTP bronzone, de migratie van de verontreiniging naar de zuidelijke perceelgrens beperkt.

De halfjaarlijkse monitoring blijft behouden en de volgende monitoring zal plaatsvinden **in januari 2016**.

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

Het doel van de monitoring was om na te gaan of er een verdere toename van de organofluorconcentraties in het grondwater plaatsvindt in het gebied ten zuiden van de Expresweg. Op basis van de resultaten van de monitoring die tussen juli 2009 en januari 2014 zijn bekomen is besloten dat er geen indicaties zijn dat de organofluorconcentraties in het grondwater ter hoogte van de monitoringspeilbuizen toenemen met de tijd. De gemeten concentraties schommelen gedurende de periode van de monitoring, zonder een duidelijke trend aan te geven en zijn vergelijkbaar met de concentraties gemeten in de periode 2002 – 2008. De saneringsdoelstelling is bijgevolg behaald.

Tijdens het overleg met OVAM in oktober 2013 heeft OVAM de opmaak van een gefaseerd eindevaluatieonderzoek opgelegd voor de zone ten zuiden van de Expresweg (Z-peilbuizen). Dit tweede gefaseerd eindevaluatieonderzoek met referentie R002-0243999-v2.0 is op 1/07/2015 door de OVAM conform verklaard. Alle peilbuizen in deze zone zullen definitief buitengebruik worden gesteld conform de wettelijke voorschriften in de periode juli 2015-juni 2016.

4.7 ZONE ONDERGRONDSE TANKEN

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

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

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

4.7.1 Uitgevoerd veldwerk

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

Tabel 4.4 Overzicht bemonsterde peilbuizen zone ondergrondse tanks

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum Juli 2015	Analyses
P18	1,4-3,4	X	MO
P28	1,1-3,1	X	*MO

* Staal kon niet worden geanalyseerd in het labo omwille van matrix interferenties.

4.7.2 Bespreking resultaten

De analyseverslagen zijn toegevoegd in Bijlage 2. Een toetsingstabel met deze resultaten is opgenomen in Bijlage 3 samen met de resultaten van de veldmetingen.

4.7.2.1 Veldparameters

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

4.7.2.2 Minerale olie

In het grondwater ter hoogte van de peilbuizen P18 en P28 is een verhoogde concentratie aan minerale olie gemeten. Deze concentraties schommelen tussen 500 en 2100 µg/l (max. 2x de bodemsaneringsnorm van 500 µg/l). De dominante olie fracties zijn de C12-C20 en C20-C30 fracties. Het staal genomen in peilbuis P28 in juli 2015 kon niet worden geanalyseerd omwille van niet nader gedefinieerde interferenties.

4.7.3 Conclusies en aanbevelingen

De verontreiniging met minerale olie, pH en geleidbaarheid is afgeperkt en gelokaliseerd rond peilbuizen P18 en P28. De hoogst gemeten concentratie minerale olie bedraagt ca. twee maal de bodemsaneringsnorm.
De verhoogde waarden voor pH en geleidbaarheid zijn mogelijk gerelateerd aan (kleine) incidenten ter hoogte van de nabijgelegen KOH tank.

Momenteel kan geen verklaring voor de gemeten verhoogde waarden aan minerale olie in het grondwater worden gegeven. Interferentie door een andere verontreiniging kan niet worden uitgesloten. Verder historisch en analytisch brononderzoek ter hoogte van P18 en P28 is in uitvoering. De jaarlijkse monitoring van minerale olie in peilbuizen P18 en P28 blijft behouden en de volgende monitoring zal plaatsvinden **in juli 2016**.

4.8 NATUURGEBIED BLOKKERSDIJK

De kwaliteit van het grond- en oppervlaktewater in het natuurgebied de Blokkersdijk wordt gemonitoord via de periodieke bemonstering van acht peilbuizen en twee vijvers. Door middel van een statistische analyse van de PFOS-concentraties wordt nagegaan of er een concentratietrend aanwezig is.

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

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

4.8.1 Monitoring grond- en oppervlaktewater

4.8.1.1 *Uitgevoerd veldwerk*

De peilbuizen langs het 3M pad ten westen van het natuurgebied Blokkersdijk en het oppervlaktewater van de 3M-vijver worden halfjaarlijks bemonsterd. Het oppervlaktewater van de Blokkersdijkvijver wordt driemaandelijks bemonsterd om de evolutie in FC concentraties in deze zones na te gaan. Sinds april 2014 wordt het oppervlaktewater ter hoogte van de staalnamelocatie Blokkersdijk Oost niet meer bemonsterd omwille van de slechte toegankelijkheid.

In 2014 (zie TTV5 (6)) werd besloten om peilbuis P119C voortaan op te nemen in de evaluatie van de evolutie van FC concentraties ter hoogte van de WWTP zone bronzone, aangezien deze peilbuis stroomopwaarts ligt van de bronzone en stroomafwaarts van het natuurgebied Blokkersdijk.

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

Tabel 4.5 Overzicht staalnamelocaties natuurgebied Blokkersdijk

Locatie	Filterdiepte	Bemonsteringsdatum				Analyse
	(m-mv)	okt 2014	jan 2015	apr 2015	jul 2015	
L21	3,0-5,0		X		X	FC
L22	2,0-4,0		X		X	FC
L31	1,2-3,2		X		X	FC

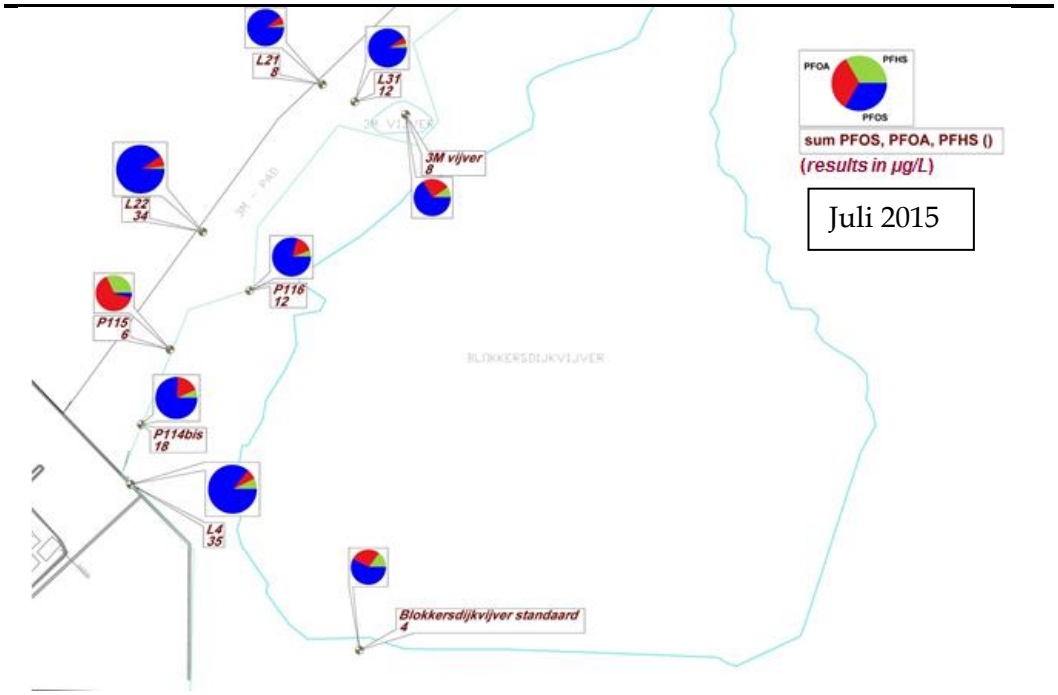
Locatie	Filterdiepte (m-mv)	Bemonsteringsdatum okt 2014	jan 2015	apr 2015	jul 2015	Analyse
L4	2,0-4,0	X	X	X	X	FC
P114bis	3,6-4,6	X	X	X	X	FC
P115	3,3-4,3		X		X	FC
P116	2,5-3,5	X	X	X	X	FC
3M vijver	-	X	X	X	X	FC
Blokkersdijk standaard	-	X	X	X	X	FC

4.8.1.2 *Resultaten*

De analyseverslagen zijn toegevoegd in Bijlage 2. Een toetsingstabel met deze resultaten is opgenomen in Bijlage 3 samen met de resultaten van de veldmetingen.

Op onderstaande figuren zijn de meest recent (juli 2015) gemeten concentraties voor de som van PFOS, PFHS en PFOA in het grondwater langs de westkant van het natuurgebied Blokkersdijk en in het oppervlaktewater in de vijvers weergegeven.

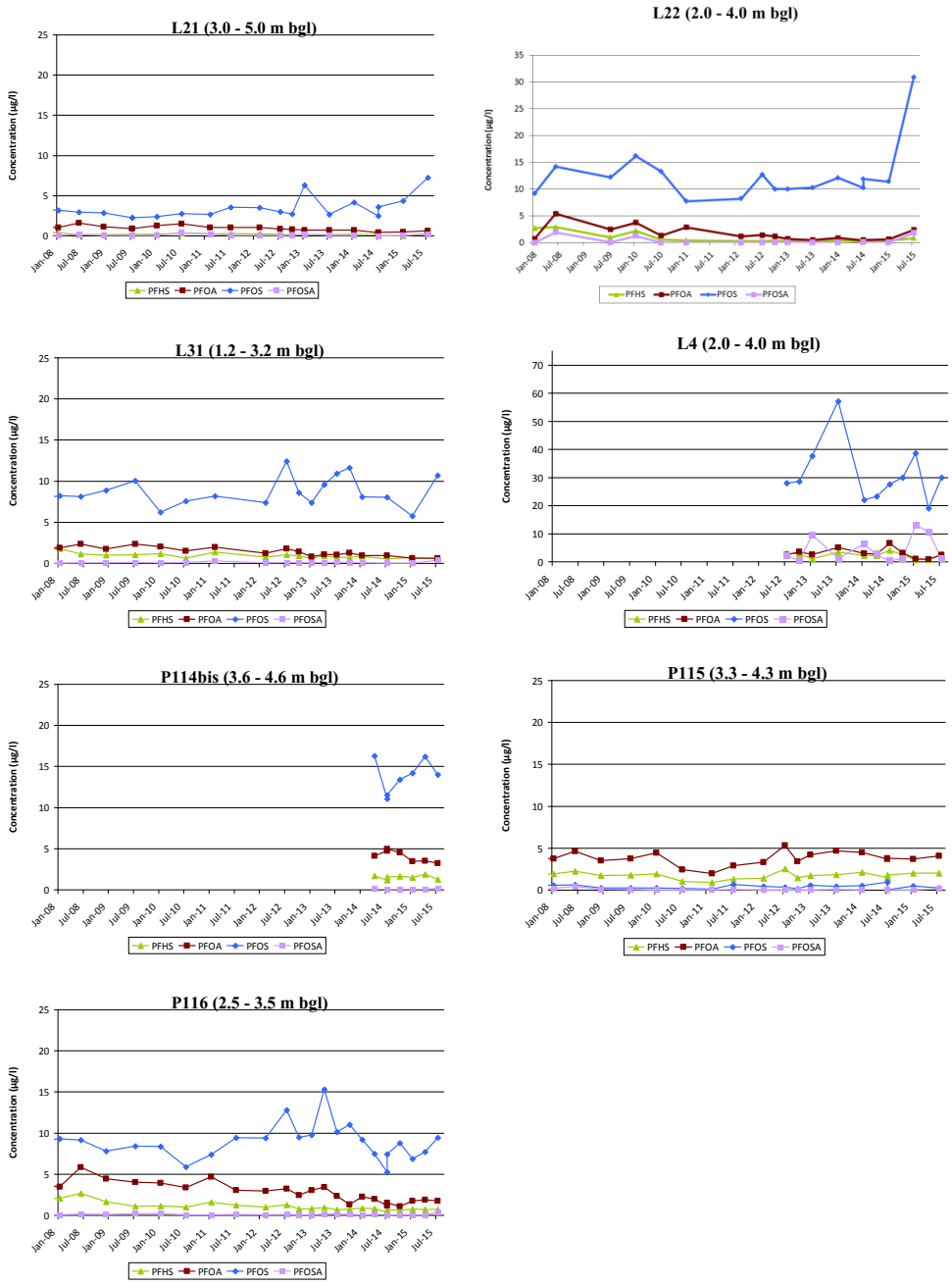
Figuur 4.6 ***Schematische voorstelling FC-concentraties in grondwater thv. natuurgebied Blokkersdijk***



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

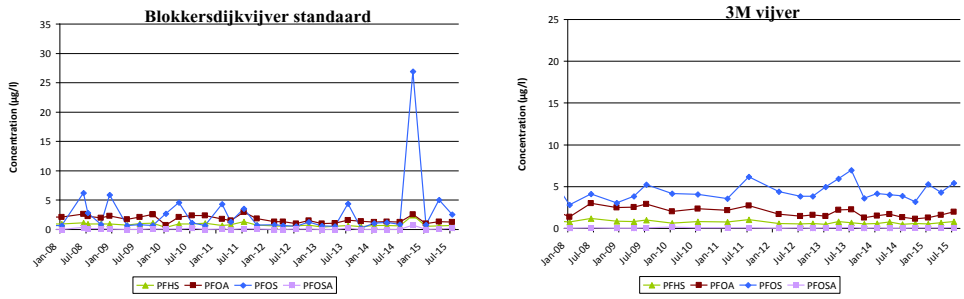
De evolutie van de FC concentraties in de peilbuizen in het 3M pad, dat langs de westkant van het natuurgebied loopt, is voorgesteld in onderstaande grafieken.

Grafiek 4.6 *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 4.7 Evolutie FC concentraties in oppervlaktewater Blokkersdijk natuurgebied



4.8.2 Statistische evaluatie PFOS-concentraties

Na de goedkeuring van het bodemsaneringsproject (1e fase) (1) 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.
Mogelijk heeft het gebruik van verschillende labo's een impact gehad op het resultaat van de PFOS trend analyse. Daarom zijn de initiele aannames waarop de trend analyse is gebaseerd (zie BSP (1)) opnieuw geëvalueerd rekening houdend met de mogelijke impact van het gebruik van verschillende labo's. De evaluatie is uitgevoerd door de resultaten van de statistische evaluatie van de volledige dataset te vergelijken met de resultaten van enkel de dataset van het 3M lab. De bevindingen van de evaluatie zijn beschreven in de memo die is opgenomen in Bijlage 4.

Uit de evaluatie blijkt dat de wisseling van labo's invloed heeft gehad op de PFOS trend analyse: De concentraties in de drie staalnamelocaties (L21, P114 en 3M vijver) tonen enkel een statistisch significante stijging als de volledige dataset gebruikt wordt voor de trend analyse en niet als enkel de data van het 3M lab dat sinds 2014 wordt gebruikt.

In Tabel 4.6 is een overzicht gegeven van de resultaten van de statistische analyse uitgevoerd in de evaluatie.

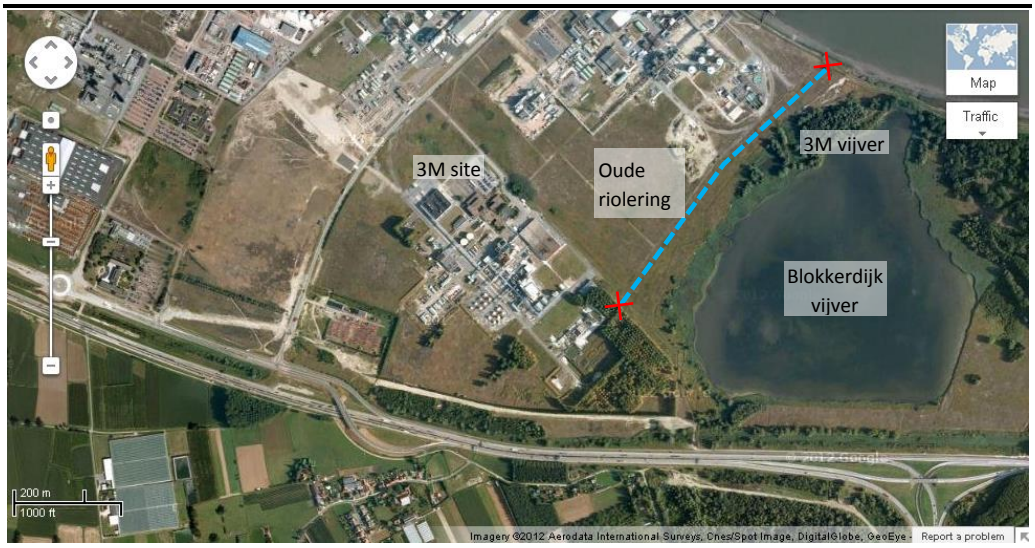
Tabel 4.6 ***Overzicht resultaten statistische analyse PFOS concentraties (enkel 3M data)***

Locatie	Mann-Kendall	Lineaire regressie test
L21	geen trend	geen trend
L22	geen trend	geen trend
L31	geen trend	geen trend
L4	geen trend	geen trend
P114(bis)	geen trend	geen trend
P115	geen trend	geen trend
P116	geen trend	geen trend
P119C	geen trend	geen trend
Blokkersdijk vijver (standaard locatie)	geen trend	geen trend
3M vijver	geen trend	geen trend

4.8.3 Nader brononderzoek Blokkersdijk natuurgebied

In deze paragraaf worden de resultaten beschreven van het geohydrologisch onderzoek ter hoogte van de oude chemische riolering. Deze oude riolering loopt onder het 3M pad, dat gelegen is ten westen van het natuurgebied Blokkersdijk (zie onderstaande Figuur 4.7, de riolering is aangeduid met een blauwe stippellijn).

Figuur 4.7 ***Ligging Blokkersdijk natuurgebied, het 3M pad, de 3M site en de Schelde***



4.8.3.1 Achtergrond

Uit de periodiek grondwatermonitoring van peilbuizen langs het 3M pad blijkt dat de PFOS concentraties in het grondwater op drie locaties (t.h.v. peilbuizen L4, P114bis en P116) een stijgende trend tonen. Ook de oppervlaktewater concentraties van de 3M vijver, een kleine vijver die ten noordwesten ligt van de Blokkersdijk vijver en er mee in connectie staat, vertonen een stijgende trend. Omdat het 3M pad hydraulisch gezien stroomopwaarts ligt van de 3M site is verder onderzoek nodig om na te gaan of er een bijkomende bron is in het gebied van de Blokkersdijk die de stijgende concentratie trend in de drie peilbuizen en de 3M vijver kan verklaren.

Uit historisch onderzoek blijkt dat de oude chemische riolering nog steeds aanwezig is onder het 3M pad. De oude riolering (betonnen pijp met een

diameter van 700 mm) is in 1999 buiten gebruik gesteld en vervangen door een nieuwe, chemisch resistente riolering. Volgens 3M is de oude riolering na buiten gebruikstelling niet verwijderd, noch gereinigd of opgevuld. De rioleringspijp is afgesloten aan het begin (ter hoogte van de bedrijfswaterzuivering) en het einde (lozingspunt ter hoogte van de Schelde). De enige zichtbare delen van de riolering zijn de betonnen structuren die het mogelijk maken om toegang te krijgen tot de riolering via de mangaten boven op de structuren (zie foto's in Figuur 4.8). De ligging van deze drie toegangspunten (verder 'mangaten' genoemd) is aangegeven op plan in Figuur 4.9.

Figuur 4.8 ***De eerste drie toegangspunten naar de oude chemische riolering (de ligging van deze punten is weergegeven op plan in Figuur 4.9)***



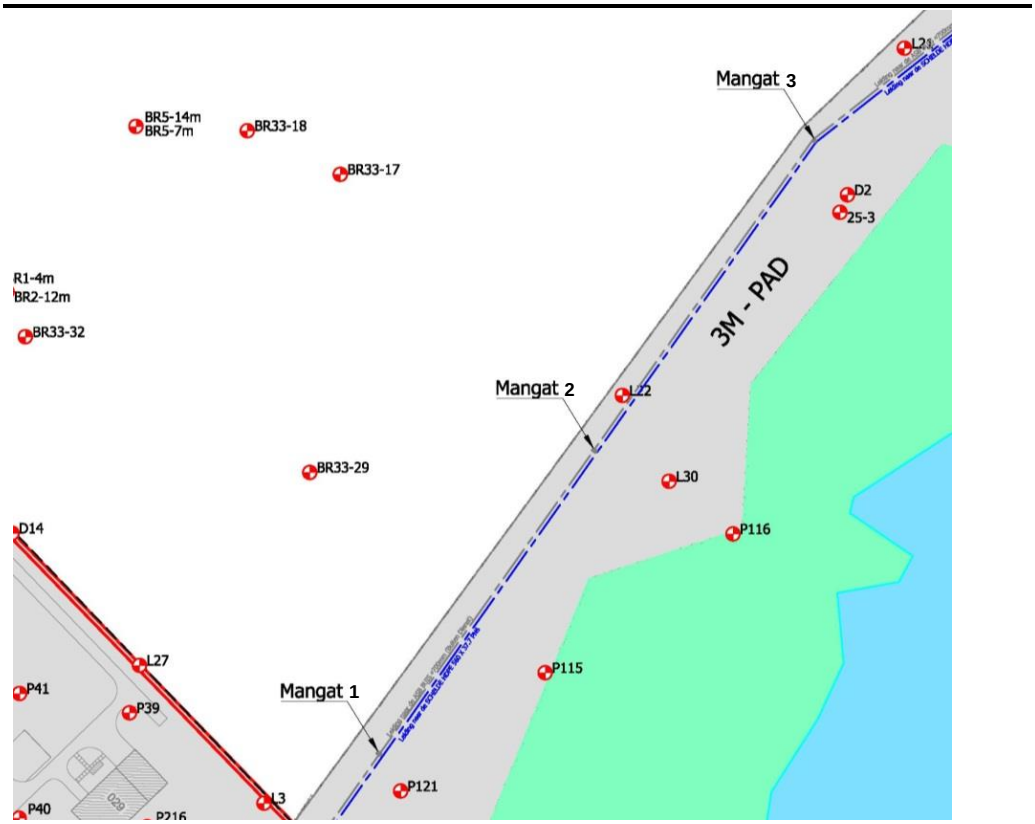
Er wordt vermoed dat de oude riolering FC-houdend slib bevat. Als de oude riolering beschadigd is, kan dit slib vanuit de riolering naar het omringende grondwater lekken. Indien deze veronderstelling correct is, kan de oude riolering een secundaire verontreinigingsbron zijn en de oorzaak van de stijgende concentraties die waargenomen zijn in enkele peilbuizen en de 3M vijver.

4.8.3.2 *Uitgevoerd veldwerk*

Om na te gaan of grondwater naar de oude riolering draineert of dat water vanuit de riolering lekt naar de aquifer zijn dataloggers in de eerste drie mangaten geïnstalleerd. Deze dataloggers registeren periodiek (elke 15 minuten) het waterniveau in de riolering over een periode van 43 dagen (van 20 oktober 2014 tot en met 2 december 2014). Toen de dataloggers werden opgehaald, zijn de grondwaterstanden in zes ondiepe, nabijgelegen peilbuizen gemeten.

De drie mangaten zijn opgemeten door een landmeter zodat de waterniveau's in de riolering vergeleken kunnen worden met de grondwaterniveau's gemeten in de nabijgelegen peilbuizen (zie plan in Figuur 4.9).

Figuur 4.9 Ligging mangaten/dataloggers vs. peilbuizen

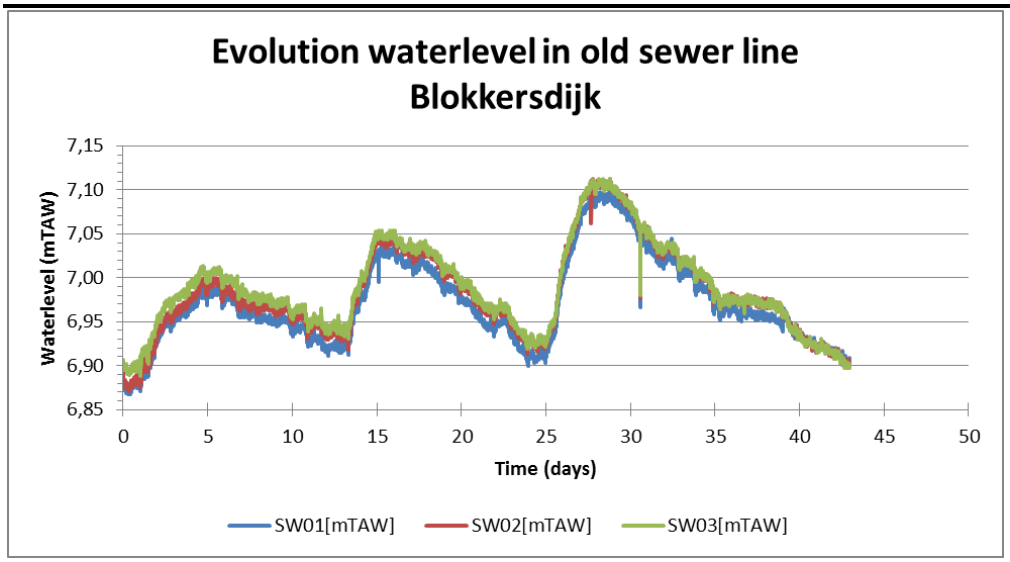


Verder zijn er slibstalen en watermonsters genomen in de oude riolering. Deze stalen zijn geanalyseerd op de drie relevantste FC-componenten (PFOS, PFOA en PFHS) door het 3M Environmental Lab in de VS.

4.8.3.3 *Bespreking resultaten*

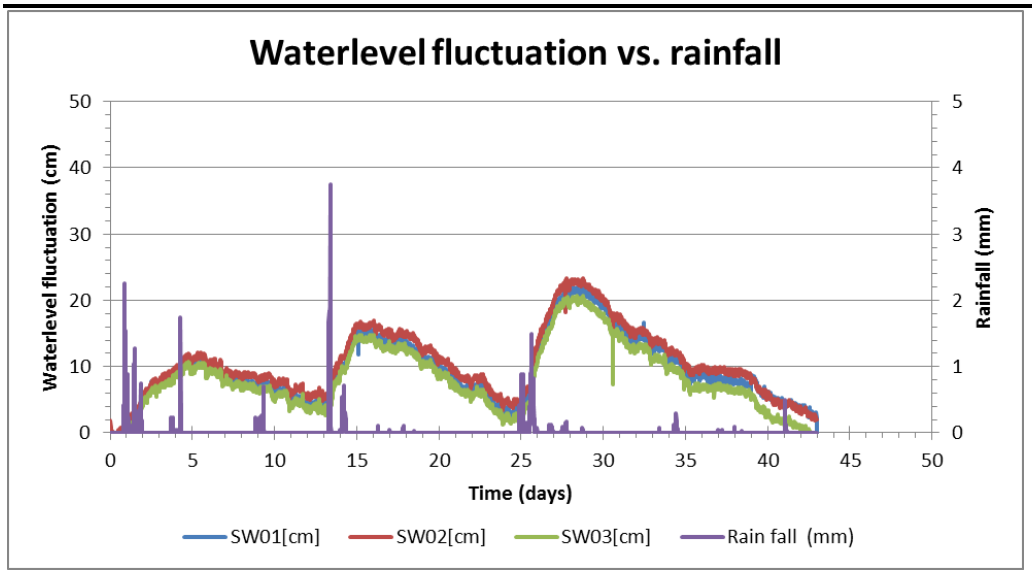
Het resultaat van de dataloggers die geïnstalleerd werden in de oude chemische riolering wordt getoond in onderstaande Figuur 4.10 en beschreven in de volgende paragraaf.

Figuur 4.10 Resultaat dataloggers geïnstalleerd in oude chemische riolering



Uit de grafieken blijkt dat het waterniveau in de riolering fluctueert. Echter, aangezien de rioleringspijp aan het begin en het einde is afgesloten, is er niet langer een verbinding met de site of de Schelde die deze fluctuaties kan verklaren. Om na te gaan of de fluctuaties van het waterniveau veroorzaakt kunnen worden door neerslag, is het waterpeil in de oude riolering uitgezet tegen de neerslag die in de meetperiode is gevallen (zie Figuur 4.11).

Figuur 4.11 Waterniveau gemeten in de oude chemische riolering vs. neerslag

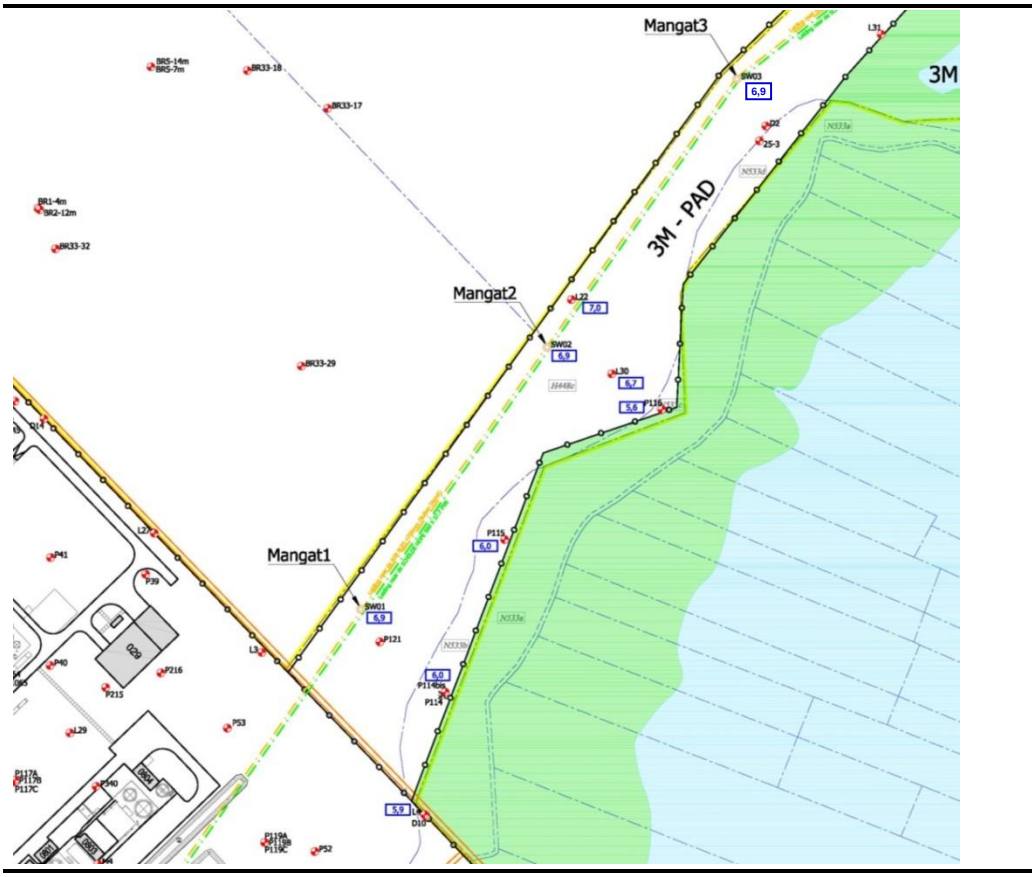


NB. De hoeveelheid neerslag is weergegeven in millimeter: 1 millimeter komt overeen met 1 liter per vierkante meter.

Bovenstaande grafiek toont dat de drie stijgingen van het waterniveau in de riolering (rond dag 5, 15 en 27) voorafgaan door een relatief langere periode van meer neerslag (rond dagen 1-2, 13-14 en 25-26). Tijdens de eerste neerslag periode viel er ongeveer 16,9 L/m^2 in ca. 1 dag, tijdens de tweede periode viel er 20,7 L/m^2 regen in ca. 1 dag en tijdens de periode viel er 23 L/m^2 regen in ca. 1 dag.

De waterpeilen gemeten in de drie mangaten en de nabijgelegen peilbuizen op 2 december 2014 zien weergegeven op plan in Figuur 4.12.

Figuur 4.12 Waterniveaus in de mangaten vs. grondwaterpeilen in de peilbuizen



NB. Peilbuizen P121, D2 en 24-3 hebben een filterscherm in de tweede aquifer en kunnen daarom niet gebruikt worden voor deze evaluatie

Uit de metingen weergegeven in Figuur 4.10 en Figuur 4.12 blijkt dat het waterniveau in de oude chemische riolering varieert tussen 6,9 en 7,1 m-TAW, terwijl het grondwaterpeil in de peilbuizen nabij de Blokkersdijk vijver ligt tussen 5,6 en 6,7 m-TAW.

De analyses op de slibstalen en de watermonsters genomen in de riolering zijn weergegeven in de volgende tabel.

- Resultaten slibstalen:

	Manhole 01	Manhole 02	Manhole 03
PFHS (mg/kg (dry-weight))	0,35	1,59	10,8
PFOA (mg/kg (dry-weight))	1,71	7,29	26,8
PFOS (mg/kg (dry-weight))	26,9	191	464
PFOSA (mg/kg (dry-weight)	1,8	5,24	11,6

- Resultaten watermonsters:

	Manhole 01	Manhole 02	Manhole 03
PFOS (µg/L)	3.387	4.103	6.130
PFOA (µg/L)	1.397	1.780	5.908

PFOS lijkt de belangrijkste component te zijn in de slib en waterstalen (ca. 90% in de slibstalen). De hoogste concentraties in het water (PFOS: 6,1 mg/L) en het

slib (PFOS: 464 mg/kg) worden gemeten in mangat 3, het mangat dat het verst verwijderd is van de 3M site.

4.8.4 Conclusies en aanbevelingen

Conform de evaluatiecriteria opgenomen in het eerste gefaseerd bodemsaneringsproject is er geen noodzaak om de actieve saneringsvariant op te starten aangezien er geen statistisch significante stijging van de PFOS concentraties gemeten is in de Blokkersdijkvijver.

Uit de herevaluatie van de statistische analyses blijkt dat de wisseling van labo's tijdens de monitoringsperiode een invloed heeft gehad op het resultaat van de PFOS trend analyse: in geen enkele locatie wordt een significante trend vastgesteld indien enkel de data van het het 3M lab, dat sinds 2014 de analyses uitvoerd, wordt gebruikt. Daarom wordt aanbevolen de statistische trend analyses enkel uit te voeren met de data van het 3M Environmental lab. De grafische en statistische trend analyse dienen voortgezet te worden. Verder wordt aanbevolen om de FC concentraties in de 3M vijver, de Blokkersdijkvijver en alle peilbuizen L4, L21, L22, L31, P114bis, P115 en P116 driemaandelijks op te volgen om de dataset verder aan te vullen. De volgende volledige monitoring zal plaats vinden **in januari 2016**.

Op basis van de resultaten van het geohydrologisch onderzoek ter hoogte van de oude chemische riolering uitgevoerd eind 2014 kunnen de volgende observaties gemaakt worden:

- Uit de meetgegevens van de dataloggers blijkt dat het waterpeil in de riolering fluctueert ondanks dat de rioleringspijp is afgesloten aan het begin en eind. Wanneer het waterpeil vergeleken wordt met neerslag metingen dan blijkt dat alle drie de stijgingen van het waterpeil vooraf gaan aan een periode van neerslag, wat er mogelijks op duidt dat de fluctuaties van het waterpeil in de riolering veroorzaakt worden door infiltratie van regen/grondwater in de riolering en drainage van water van de riolering naar de aquifer.

- De (grond)waterpeilen gemeten op 2 december 2014 duiden erop dat er een gradiënt is van de oude riolering naar de Blokkersdijk vijver.

- Uit de analyses van de slibstalen en watermonsters blijkt dat er nog residuele FC massa aanwezig is in de oude riolering.

Op basis van deze observaties kan voorzichtig geconcludeerd worden dat de oude chemische riolering een secundaire verontreinigingsbron kan zijn. Verder onderzoek (o.a. massa berekeningen en grondwatermodellering met stoftransport) is noodzakelijk om na te gaan wat de werkelijke impact is van de residuele FC massa (aanwezig in de oude riolering) op de waterkwaliteit van de freatische aquifer en de nabij gelegen vijvers.

4.9 PALINGBEEK EN TOPHAT GRACHT

4.9.1 Uitgevoerd veldwerk

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

Tabel 4.7 Overzicht staalnamelocaties Palingbeek en Tophatgracht

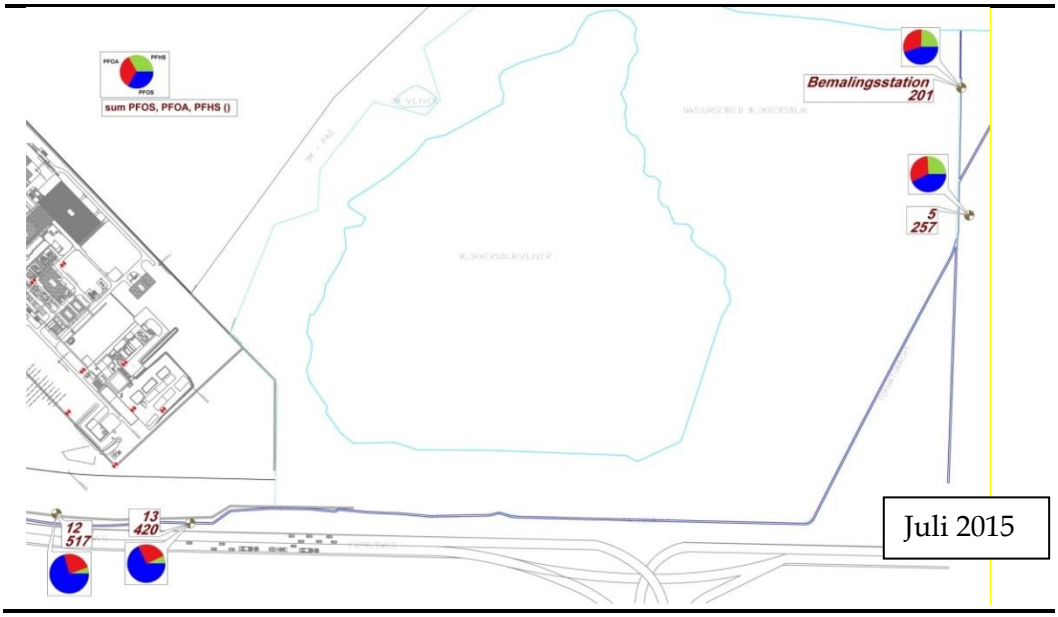
Locatie	Bemonsteringsdatum				Analyse
	Okt. 2014	Jan. 2015	Apr. 2015	Jul. 2015	
12	X	X	X	X	FC
13	X	X	X	X	FC
5	X	X	X	X	FC
Bemalingsstation	X	X	X	X	FC

4.9.2 Bespreking resultaten

De analyseverslagen zijn toegevoegd in Bijlage 2. In de toetsingstabellen in Bijlage 3 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 2015 zijn op de onderstaande figuur voorgesteld.

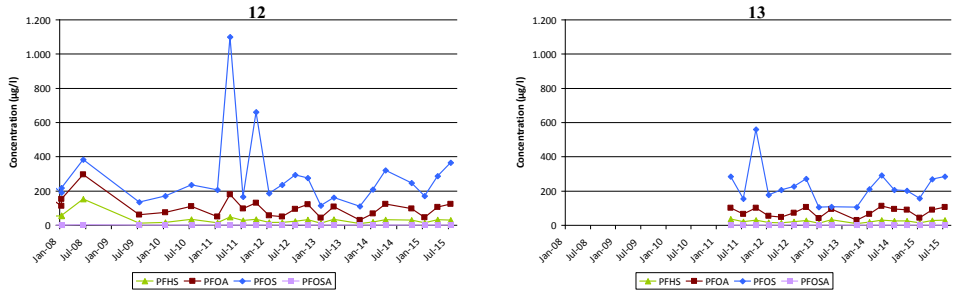
Figuur 4.13 Schematische voorstelling FC-concentraties in grondwater t.h.v. Palingbeek en de Tophat gracht



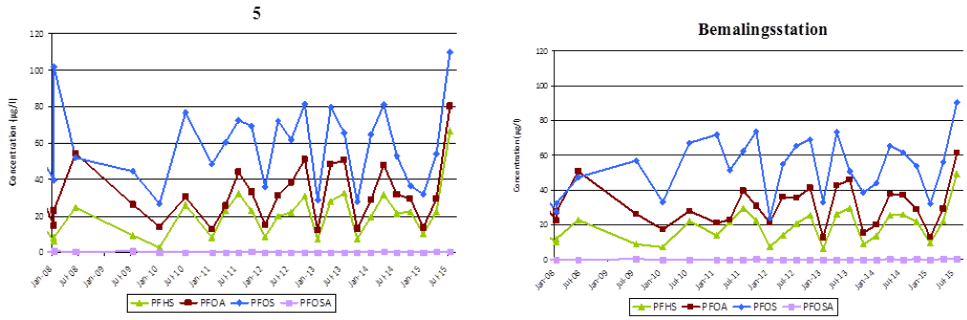
De evolutie van de FC concentraties in het oppervlaktewater van de Palingbeek en de Tophatgracht wordt weergegeven in onderstaande grafieken.

Grafiek 4.8 ***Evolutie FC concentraties in oppervlaktewater Palingbeek en de Tophat gracht***

Staalnamepunten Palingbeek



Staalnamepunten Tophat gracht



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

4.9.3 Vuilvrachtberekening

De dagelijkse vuilvracht aan PFOS welke, berekend op basis van het jaarlijks gemiddelde van de analyseresultaten ter hoogte van het bemalingsstation, via de Palingbeek en Tophat gracht in de Schelde terecht komt, bedraagt 146 g voor de periode 1 juli 2014 tot 30 juni 2015. In onderstaande tabel is een overzicht gegeven van de berekende vuilvracht per jaar sinds de start van de monitoring in 2009.

Tabel 4.8 ***Gemiddelde PFOS vuilvrachtberekening in oppervlaktewater***

Periode	Debiet (m^3/dag)	PFOS concentratie (µg/l)	Vuilvracht (g/dag)
juli 2009 tem. juni 2010	2 865	33,0	95
juli 2010 tem. juni 2011	2 865	63,5	182
juli 2011 tem. juni 2012	2 865	53,7	154
juli 2012 tem. juni 2013	2 865	60,3	173
juli 2013 tem. juni 2014	2 865	49,8	143
juli 2013 tem. juni 2014	**2 865**	**51,0**	**146**

4.9.4 Conclusies en aanbevelingen

De FC concentraties in de grachten zijn het hoogst ter hoogte van het 3M terrein (Palingbeek, locaties 12 en 13) en nemen af naarmate de afstand tot het 3M terrein groter wordt (Tophat gracht). Dit is vermoedelijk het gevolg van

verdunning door het instromen van minder of niet-verontreinigd grond- of oppervlaktewater. De concentraties in het oppervlaktewater van de Tophat gracht (locatie 5) en het bemalingsstation zijn vergelijkbaar en lijken licht te stijgen.

Aanbevolen wordt om de driemaandelijkse bemonsteringsfrequentie te behouden en de potentiele stijging in de Tophat gracht verder op te volgen. De volgende monitoring zal plaats vinden **in oktober 2015**.

4.10 *REGENWATERCOLLECTORPUT EN BEDRIJFSWATERZUIVERING*

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

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

4.10.1 *Uitgevoerd veldwerk*

Om de invloed van de lopende grondwatersanering op de concentraties in de regenwaterriolering na te gaan, zijn door 3M maandelijks stalen genomen en geanalyseerd van de regenwatercollectorput. Ook van de bedrijfswaterzuivering worden maandelijks stalen genomen en geanalyseerd. Het debiet van de regenwatercollectorput en de bedrijfswaterzuivering wordt dagelijks door 3M opgevolgd.
Daarnaast worden elk kwartaal waterstalen genomen door ERM, die geanalyseerd worden op de FC verbindingen PFOS en PFOA door het laboratorium van SGS.

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

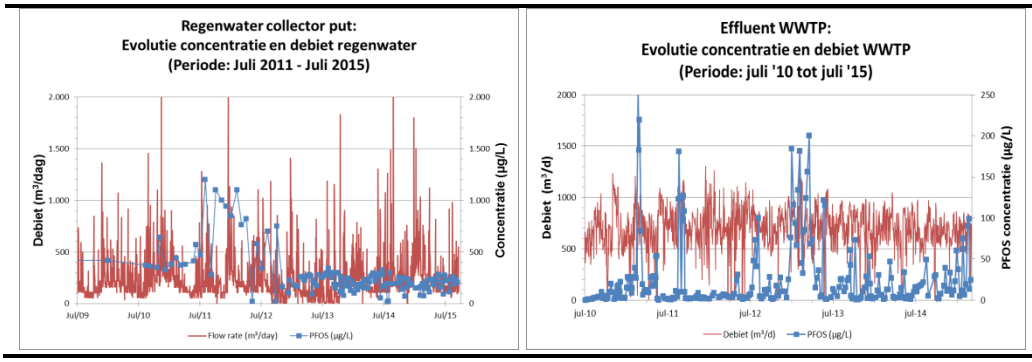
4.10.2 *Bespreking resultaten, conclusies en aanbevelingen*

De analyseverslagen zijn toegevoegd in Bijlage 2. Een toetsingstabel met deze resultaten is opgenomen in Bijlage 3.

4.10.2.1 *Debiet regenwatercollectorput versus PFOS concentratie*

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

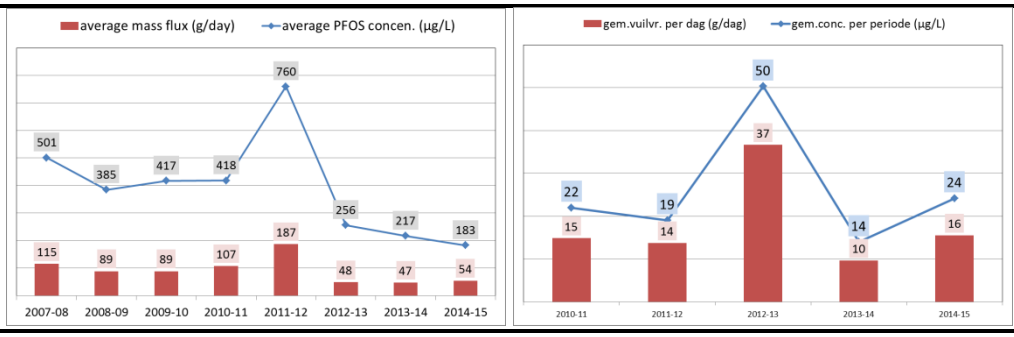
Grafiek 4.9 Evolutie PFOS concentratie in collector put en bedrijfswaterzuivering



Sinds november 2012 vertonen de PFOS concentraties in de collectorput een stabiel verloop. De concentraties kunnen echter fluctueren. De gemiddelde concentratie PFOS in de periode juli 2014 – juni 2015 bedraagt 183 µg/l. De gemiddelde PFOS concentratie in de bedrijfsriolering in dezelfde periode bedraagt 24 µg/l.

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

Grafiek 4.10 Vuilvrachtberekening regenwatercollectorput en bedrijfswaterzuivering voor PFOS



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

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

4.11 PFOS VUILVRACHT RICHTING SCHELDE

De vermoedelijke PFOS vuilvracht van het 3M terrein naar de Schelde is in het eerste gefaseerd bodemsaneringsproject geraamd op 290 g/dag. De veilige

vuilvracht voor PFOS (PNEC waarde) is vastgelegd op 370 g/dag en wordt gebruikt als toetsingswaarde om de gemiddelde dagelijks PFOS vuilvracht te evalueren.

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

Op basis van de gegevens bekomen uit de monitoringscampagne van 1 juli 2014 tot 30 juni 2015 is de vuilvracht die dagelijks naar de Schelde stroomt berekend (zie
Tabel 4.9).

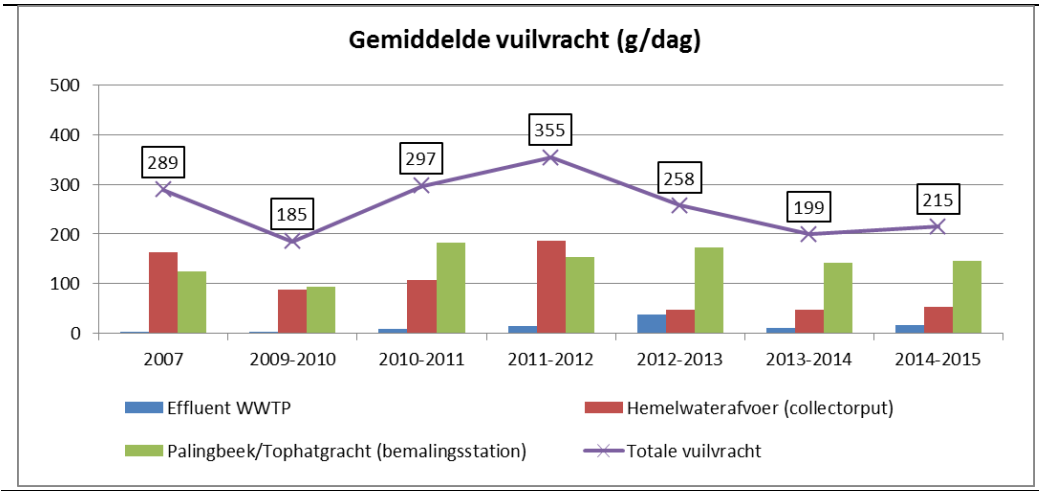
Tabel 4.9 Gemiddelde PFOS vuilvracht richting de Schelde (juli 2014 tem. juni 2015)

Influx naar de Schelde	Gemiddeld debiet (m^3/dag)	Gemiddelde PFOS concentratie (µg/L)	Gemiddelde vuilvracht (g/dag)
Effluent van het bedrijfsafvalwater (incl. opgepompt grondwater)	643	24	15,6
Regenwaterriolering	293	183	53,6
Palingbeek/Tophatgracht***	2.865	51	145,5
Totale vuilvracht			**214,7**

* *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*

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

Grafiek 4.11 Evolutie gemiddelde PFOS concentratie en vuilvracht naar de Schelde



Voor de periode juli 2014 tot en met juni 2015 bedraagt de berekende totale PFOS vuilvracht naar de Schelde 215 g/dag: de berekende vuilvracht overschrijdt de PNEC waarde (370 g/dag) niet.

4.12 VOORSTEL MONITORINGSPROGRAMMA 2015-16

In de volgende tabel is een overzicht gegeven van het voorgestelde monitoringsprogramma voor de periode oktober 2015 – juli 2016.

Tabel 4.10 Monitoringsprogramma 2016

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
P&T systeem	9	Driemaandelijks	PP01, PP02,PP04, PP05, PP06, PP07, PP08, PP09, PP10	-	Minstens maandelijkse inspectie van de installatie en de onttrekkingsputten
Stabiliteit	-	Eenmaal (stopzetting indien akkoord stabiliteitsdeskundige)	-	-	Geen onaanvaardbare zettingen en een technische evaluatie door onafhankelijke stabiliteitsdeskundige geeft aan dat er geen verder noodzaak is voor monitoring. Te bevestigen door eenmalige controle.
Grondhoop	-	Vijfjaarlijks	Visuele inspectie van grondhoop	Bovenafdek vervangen in 2012.	Vanaf 2013 vijfjaarlijkse monitoring, aangezien bovenafdek in 2012 vervangen is; eerst volgende controle: 2018
FC Bronzones					
Gebouw 16	2	Jaarlijks	K3, P56	-	PFBS zal worden toegevoegd aan het analysepakket om de concentraties verder op te volgen
	4	Halfjaarlijks	P27, P42, P304, P305	-	P27 ter vervanging van K1 omdat de concentraties in peilbuis K1 sinds de start van de monitoring in 2008 stabiel en laag liggen
	1	Driemaandelijks	P21B	Verhoogde frequentie om de concentratietrend beter te kunnen opvolgen	Stijgende concentratietrend ter hoogte van P21B waargenomen.
WWTP	6	Jaarlijks	P262, P340, P341, P343, P379, P380	-	-
	10	Halfjaarlijks	P118C, P264, P265C, P371, P374, P381, P382, L19, M4, P119C	-	P119C van Blokkersdijk monitoring naar bronzone monitoring gezien de locatie (stroomafwaarts van 3M pad, maar stroomopwaarts van bronzone)
	1	Driemaandelijks	P263	Verhoogde frequentie om de concentratietrend beter te kunnen opvolgen	-
Tweede aquifer	6	Jaarlijks	BD24-4, D11, D14, P118B, D10, P118A	-	-
	3	Halfjaarlijks	D9, BD24-3, P121	D9 zal halfjaarlijks bemonsterd worden (opvolging mogelijke stijgende trend)	Indien de stijgende trend niet wordt bevestigd bij twee opeenvolgende monitoringsrondes, zal

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
					de monitoringsfrequentie terug worden verlaagd naar jaarlijks
	1	Driemaandelijks	P321	-	-
Zuidelijke terreingrens	7	Halfjaarlijks	B3-bis, B7, P372, P378, PA109A, PA111A, PA112	-	Peilbuis B3 is beschadigd en vervangen door peilbuis B3-bis in september 2015.
Ondergrondse tanken					
Minerale olie (MO)	2	Jaarlijks	P18, P28	Monitoring op vraag van OVAM	Brononderzoek lopende
Blokkersdijk Natuurgebied					
Peilbuizen	4	Driemaandelijks	L21, L22, P115, L31	Verhoogde frequentie om de data set voor de statistische evaluatie sneller aan te vullen	-
	3	Driemaandelijks	L4, P114bis, P116	-	-
Oppervlaktewater	2	Driemaandelijks	Blokkersdijk standaard, 3M vijver	-	-
Palingbeek en Tophat gracht	4	Driemaandelijks	12, 13, 5, Bemalingsstation	-	-
Regenwater	1	Maandelijks	Collector put	Maandelijkse stalen geanalyseerd door 3M labo te Zwijndrecht	Driemaandelijkse verificatie van de stalen door SGS.
Effluent WWTP	1	Driemaandelijks	Lozingspijp	Maandelijkse stalen geanalyseerd door 3M labo te Zwijndrecht	Driemaandelijkse verificatie van de stalen door SGS.

Legende

Verhoogde monitoringsfrequentie

Verlaagde monitoringsfrequentie

Geen wijzigingen

5. GRONDWATERONTTREKKING BRONZONE

5.1 OPVOLGING ONTTREKKINGSSYSTEEM

De hieronder besproken resultaten hebben betrekking op de periode van juli 2014 tot juli 2015.

5.1.1 Beschrijving onttrekkingssysteem

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

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

Tabel 5.1 Technisch overzicht pompputten

Pompput	Locatie	Diepte (m-mv)	Gemiddeld jaardebiet (l/min)
PP01	Gebouw 16	2,0-5,5	1,8
PP04	Gebouw 16	2,0-6,0	0,6
PP05	Gebouw 16	1,7-5,7	0,8
PP02	WWTP	3,2-6,2	0,6
PP06	WWTP	1,3-5,3	3,7
PP07	WWTP	2,0-6,0	4,6
PP08	WWTP	3,2-7,2	2,5
PP09	WWTP	3,0-6,0	2,1
PP10	WWTP	3,0-6,0	5,8

* *Geen constant debiet door softwareproblemen*

Elke pompput beschikt over een niveausensor (zie schematisch overzicht in Bijlage 5). De niveausensor is net boven de inlaat van de pomp gehangen. De inlaat van de pomp mag nooit droog komen, daarom wordt steeds een minimale waterkolom van 30 cm aangehouden. De onttrekking in de betreffende pompput wordt stilgelegd als het niveau in de pompput onder dit laagst toelaatbare niveau komt te liggen. Van zodra het water terug voldoende gestegen is, start de onttrekking automatisch terug op. In onderstaande tabel is een overzicht gegeven van de huidige ingestelde schakelniveaus. In de pompputten die veel water geven, wordt het niveau waarop de pompen terug

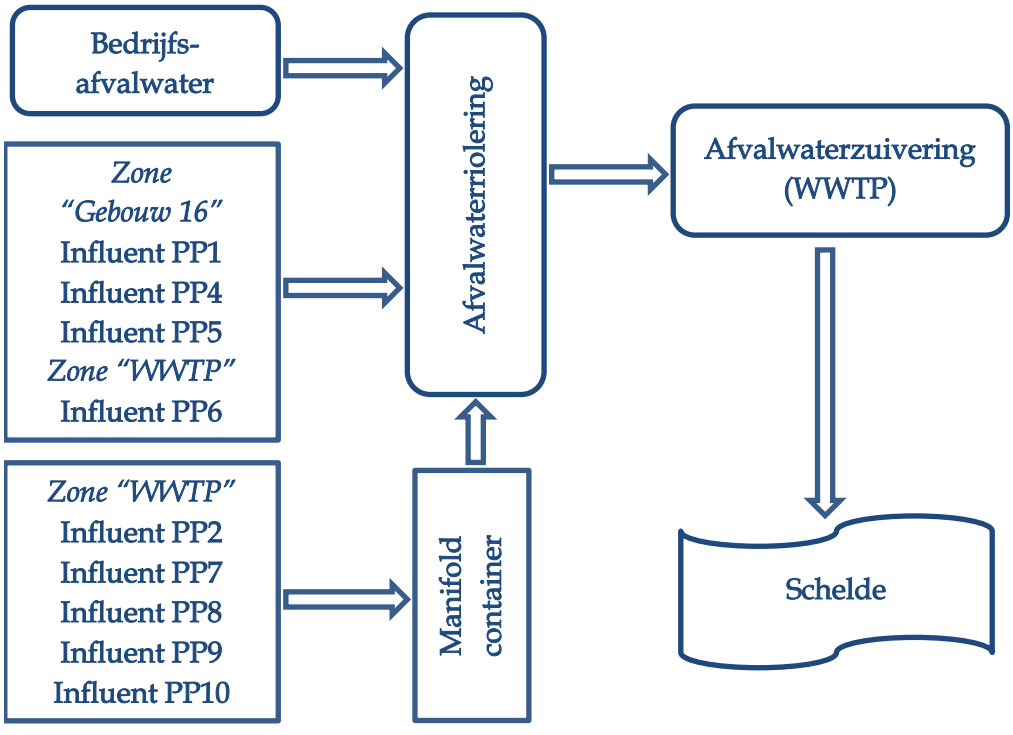
aangeschakeld worden iets hoger ingesteld. In onderstaande tabel worden deze schakelniveaus weergegeven.

Tabel 5.2 ***Schakelniveaus pompputten***

Naam pompput	Hoogste Schakelniveau (m-mv)	Laagste schakelniveau (m-mv)
PP01	0,3	1,4
PP02	0,3	1,2
PP04	0,3	1,5
PP05	0,3	1,2
PP06	0,3	1,2
PP07	0,3	1,50
PP08	0,3	1,5
PP09	0,3	1,2
PP10	0,3	1,2

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

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



Naast de niveausensoren, beschikt elke pompput over een debietmeter en een drukmeter. Alle instrumentatie wordt ingemeten via het centrale systeem van 3M. Op termijn zullen de geregistreerde data automatisch verzonden worden naar de consultant.

5.1.2 Opvolging sanering

Er worden verschillende aspecten van het onttrekkingssysteem opgevolgd:

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

5.1.2.1 Opvolging onttrokken debieten

Op regelmatige tijdstippen gaat ERM ter plaatse om de werking van de installatie te controleren en om meterstanden te noteren. Tijdens deze bezoeken worden ook grondwaterstanden genoteerd in omliggende peilbuizen (zie paragraaf 5.2). In de periode van 1 juli 2014 tot 1 juli 2015 heeft ERM bezoeken uitgevoerd op de data vermeld in onderstaande tabel. ERM komt ook steeds ter plaatse tijdens of kort na interventies door de aannemer, deze data zijn eveneens mee opgenomen in onderstaande tabel.

Tabel 5.3 Data controlebezoeken

Controlebezoeken ERM	Controlebezoeken ERM	Controlebezoeken ERM
01-07-14	28-10-14	13-03-15
08-07-14	03-11-14	16-03-15
14-07-14	13-11-14	03-04-15
22-07-14	17-11-14	09-04-15
29-07-14	03-12-14	16-04-15
05-08-14	09-12-14	24-04-15
13-08-14	16-12-14	28-04-15
18-08-14	08-01-15	05-05-15
26-08-14	14-01-15	13-05-15
28-08-14	21-01-15	22-05-15
03-09-14	28-01-15	28-05-15
18-09-14	05-02-15	05-06-15
23-09-14	13-02-15	12-06-15
03-10-14	18-02-15	17-06-15
07-10-14	26-02-15	26-06-15
16-10-14	06-03-15	
23-10-14	12-03-15	

5.1.2.2 Opvolging concentraties in het onttrokken grondwater

Het onttrokken grondwater in de pompputten wordt (twee)maandelijks 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

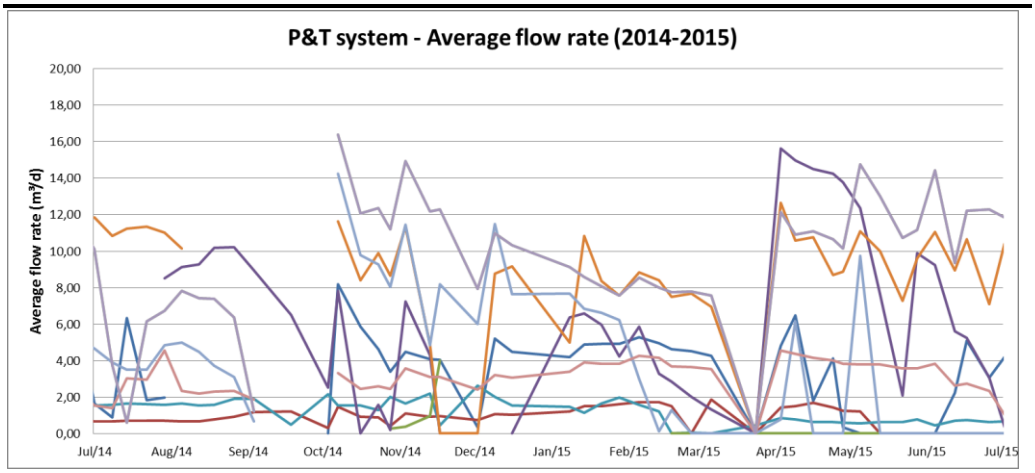
Datum	Bemonsterde pompputten
20/10/2014	PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 & PP10
28/01/2015	PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 & PP10
28/04/2015	PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 & PP10
29/07/2015	PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 & PP10

5.1.3 *Resultaten*

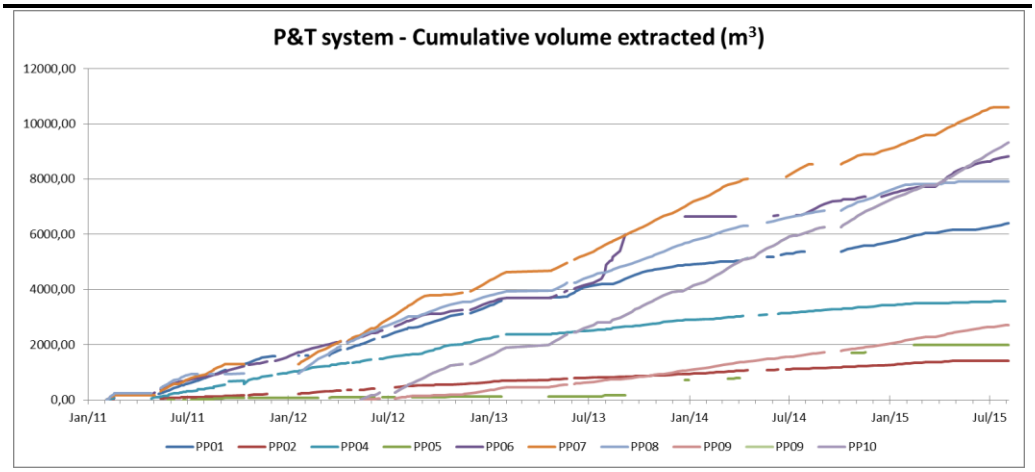
5.1.3.1 *De onttrokken debieten*

In onderstaande grafieken wordt getoond wat de gemiddelde verpompte debieten zijn en de cumulatieve verpompte debieten zijn van 1 juli 2014 tot 1 juli 2015.

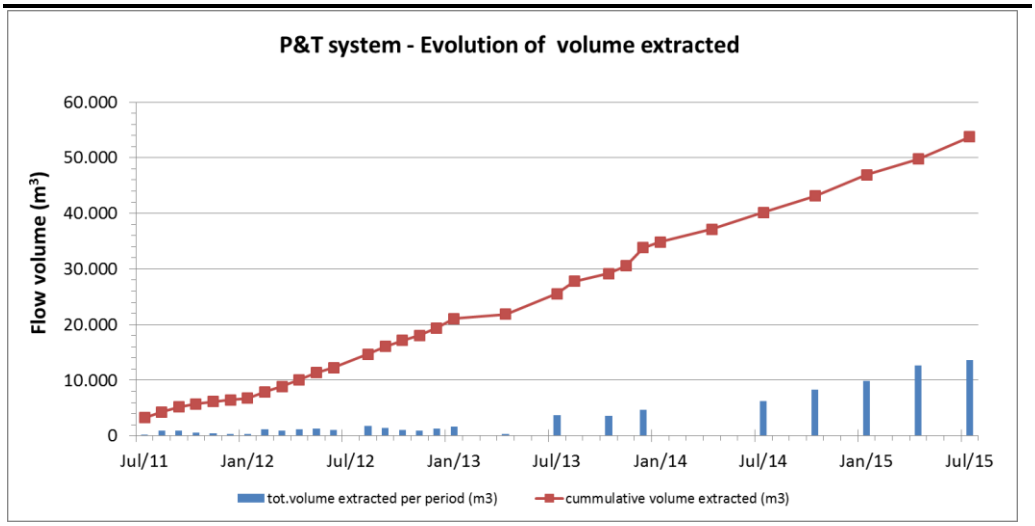
Grafiek 5.1 Gemiddeld debiet per onttrekkingsput (juli 2014 tot juli 2015)



Grafiek 5.2 Cumulatief debiet per onttrekkingsput



Grafiek 5.3 Cumulatief debiet van het totaal saneringssysteem



Uit hoger getoonde grafieken kunnen volgende zaken worden afgeleid:

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

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

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

  Op basis van de wekelijkse inspecties is geraamd dat de installatie een up-time heeft van gemiddeld circa 73%. Echter, zoals uit onderstaande Tabel 5.5 blijkt is er een groot verschil tussen de werking van de verschillende pompen. Pompput 5 heeft slechts een up-time van 13%, terwijl pompputten PP09 en PP10 een up-time hebben van 96%.

Tabel 5.5 *Werking pompputten*

	jul-14	aug-14	sep-14	okt-14	nov-14	dec-14	jan-15	feb-15	mrt-15	apr-15	mei-15	jun-15	Up-time
PP01													67%
PP02													83%
PP04													92%
PP05													13%
PP06													83%
PP07													75%
PP08													54%
PP09													96%
PP10													96%

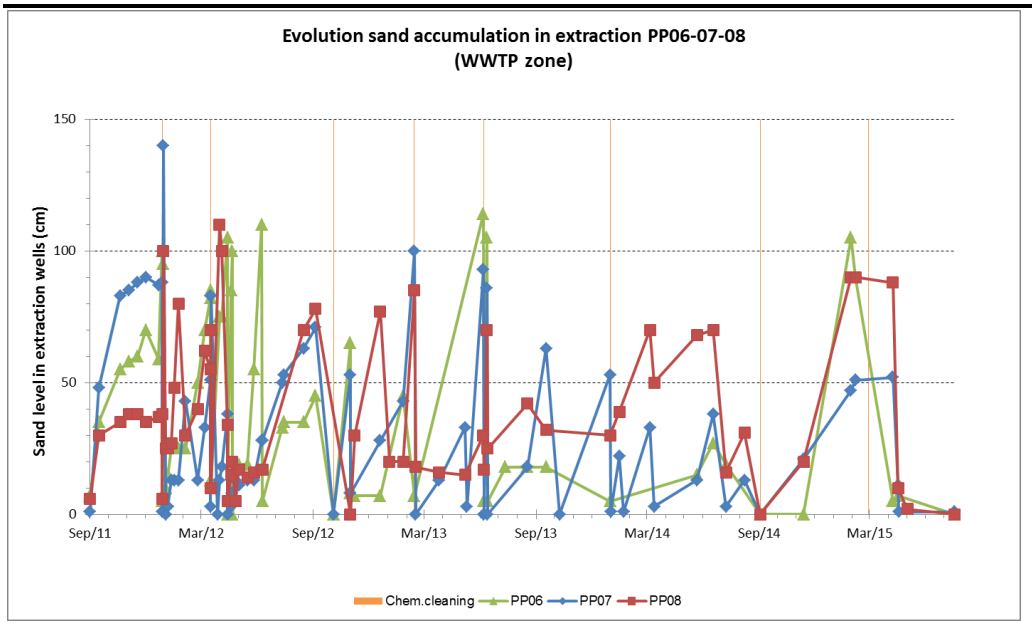
pomp operationeel voor de hele maand
pomp operationeel voor de helft van de maand
pomp niet operational

- Tussen juli 2014 en juli 2015 is ongeveer 12.535 m³ verontreinigd grondwater onttrokken (tegen circa 14.625 m³ vorige periode). In totaal is sinds juli 2011 circa 52.700 m³ grondwater onttrokken.

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

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

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.

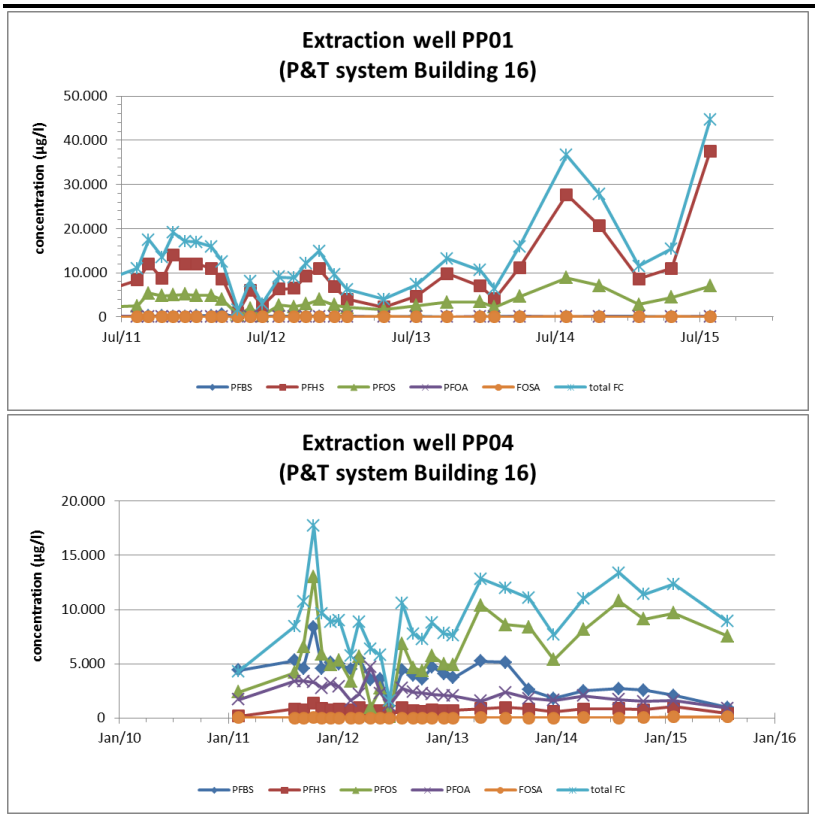
- Bij elektrische problemen of software problemen wordt de technische dienst van 3M gecontacteerd en gevraagd om de betreffende problemen op te lossen. Bij 3M is een vaste contactpersoon/technieker aangesteld om eventuele problemen op te lossen.

- Indien blijkt uit de data-opvolging dat de rendementen van een bepaalde onttrekkingsput dalen en er is geen aantoonbare technische reden, wordt er van uit gegaan dat het filterpakket verstopt begint te raken met ijzerslib. Er wordt dan over gegaan tot een chemische reiniging van de onttrekkingsput. Deze chemische reiniging wordt voorafgegaan door een reiniging met een vacuümtruck. In september 2014 en maart 2015 werd een chemische reiniging uitgevoerd.
  Het protocol van de reiniging is aangepast, zo worden alle putten nu eerst mechanisch en daarna chemisch gereinigd (nadat eerst de pompen zijn verwijderd). Na de chemische reiniging worden de putten opnieuw gespoeld en worden de pompen teruggehangen.

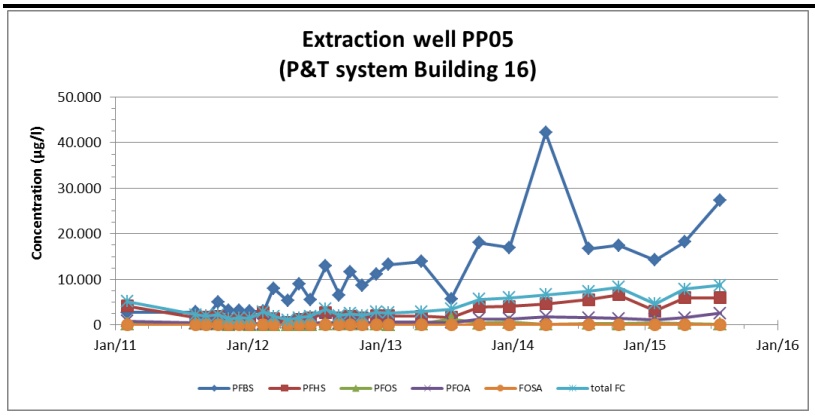
5.1.3.2 *De concentraties in het onttrokken grondwater*

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

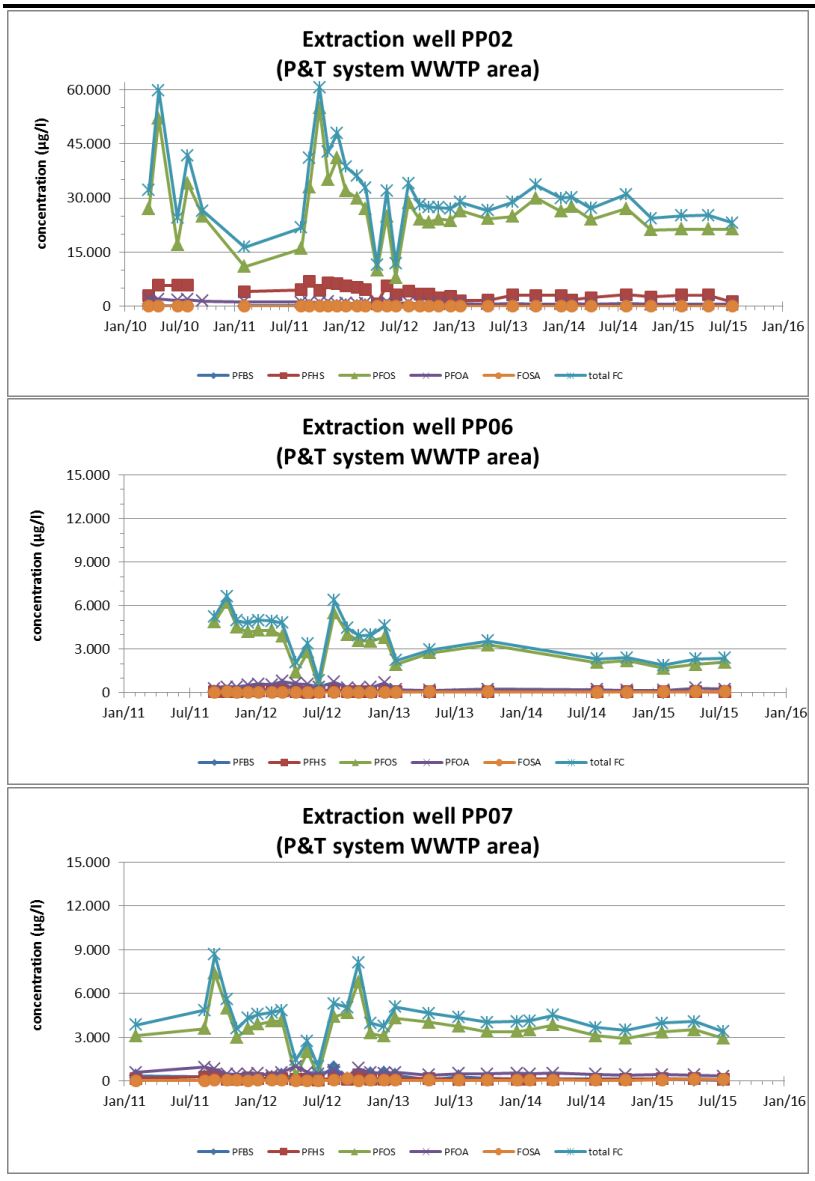
Grafiek 5.5 Evolutie FC concentraties t.h.v. gebouw 016 (pompput PP01, PP04 en PP05)



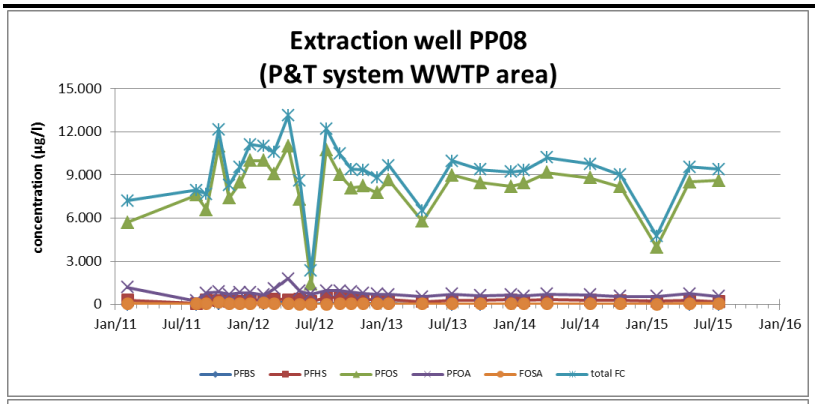
Vervolg Grafiek 5.5

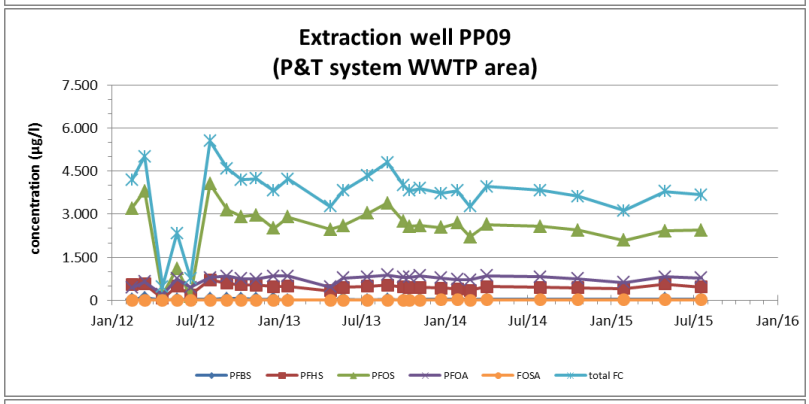


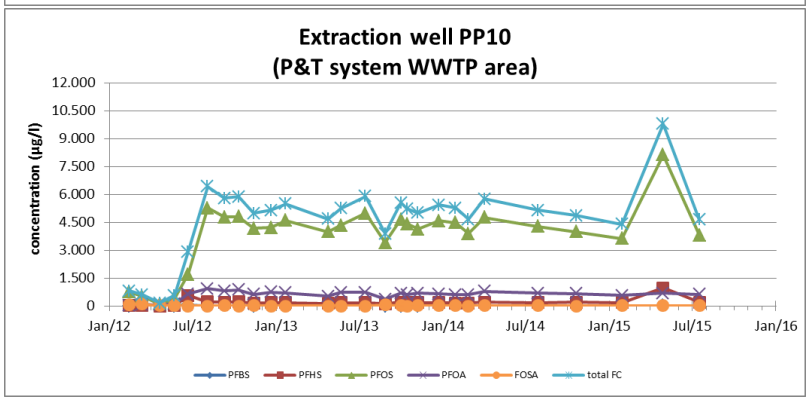
Grafiek 5.6 Evolutie FC concentraties t.h.v. WWTP zone (pompput PP02, PP06, PP07, PP08, PP09 en PP10)



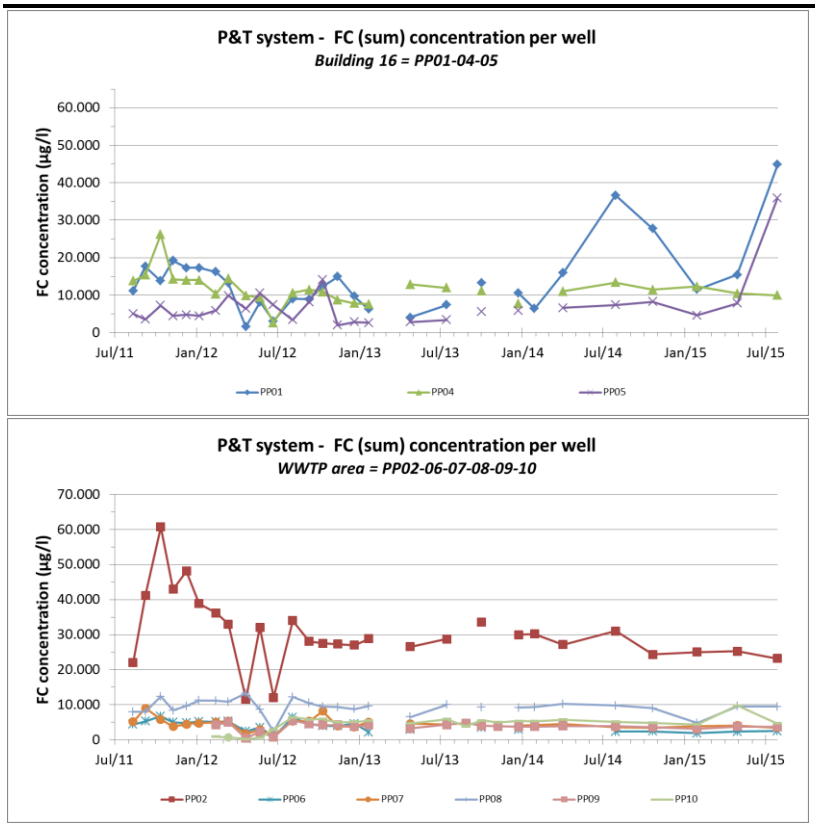
Vervolg Grafiek 5.6



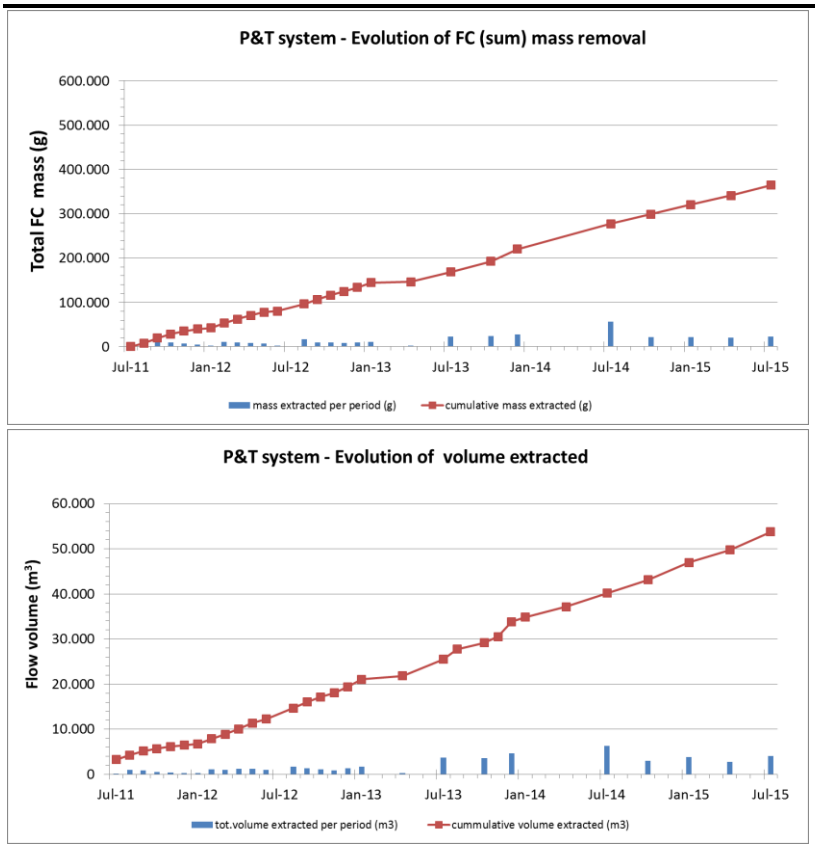




Grafiek 5.7 Overzicht evolutie som FC concentraties (PFOS, PFOA en PFHS) t.h.v. gebouw 016 en WWTP zone



Grafiek 5.8 Cumulatieve vrachtverwijdering per onttrekkingsput en van het totaal saneringssysteem



Uit bovenstaande grafieken kunnen volgende zaken afgeleid worden:

- Er is een verschil in samenstelling van het met FC's verontreinigde grondwater in de zone rond gebouw 016 (PP01, PP04 en PP05) en van het grondwater in de zone WWTP (overige pompputen):
  - In PP01 is PFHS de component met de hoogste concentraties.
  - In de overige pompputen wordt voornamelijk PFOS gemeten.
  - In pompput PP05 wordt naast de "standaard FC's" (PFOS, PFOA, PFHS en FOSA) ook een verhoogde concentratie PFBS (perfluorbutaansulfonzuur) gemeten.

- De hoogste concentraties aan totale FC's (PFOS, PFOA en PFHS) worden gemeten in PP01. Ter hoogte van PP01 is in juli 2015 een sterk verhoogde concentratie aan PFHS gemeten en ter hoogte van PP05 is eveneens in juli 2015 een sterk verhoogde concentratie aan PFBS gemeten.

- De opgepompte concentraties aan totale FC's (PFOS, PFOA en PFHS) fluctueren, maar in de meeste pompputten zijn de concentratietrends min of meer stabiel:
  - De gemeten concentraties aan totale FC's (PFOS, PFOA en PFHS) in PP06, PP07, PP09 en PP10 liggen doorgaans rond 5.000 µg/l.
  - Voor PP04 en PP08 ligt de gemeten concentratie aan totale FC's rond 10.000 µg/l.
  - In pompput PP02 ligt de totale FC concentratie rond de 30.000 µg/l.
  - Enkel in PP01 en PP05 is een stijging van de concentratie gemeten. In PP01 bedraagt de concentratie in juli 2015 circa 44.800 µg/l en in pompput PP05 circa 36.000 µg/l.

- Tussen juli 2014 en juni 2015 is ongeveer 84 kg FC's verwijderd. In totaal is sinds juli 2011 circa 362 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 (6)). 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 2014 tot juli 2015 ongeveer 12.535 m^3 verontreinigd grondwater onttrokken en circa 84 kg FC verwijderd. Uit de analyses blijkt dat in de meeste pompputten PFOS de belangrijkste component is. Uitzonderingen hierop zijn pompput PP01, waar PFHS de hoogste gemeten concentratie is en PP05 waar PFBS de belangrijkste FC parameter is. Over het algemeen zijn de concentraties in de pompputten stabiel wat erop duidt dat de verontreiniging globaal gezien gecapteerd wordt door het P&T systeem. Echter, in enkele pompputten worden stijgende concentraties gemeten wat erop kan duiden dat de captatie lokaal niet volledig is. Een mogelijke uitbreiding van het saneringssysteem om de captatie te optimaliseren moet worden geëvalueerd.

Tijdens de meetcampagne in juli 2015 is ter hoogte van pompput PP01 een sterk verhoogde concentratie aan PFHS gemeten en ter hoogte van pompput PP05 een sterk verhoogde concentratie aan PFBS. Deze verhoogde concentraties dienen verder te worden opgevolgd.

Het saneringssysteem is storingsgevoelig. 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.

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 de velddata blijkt dat de up-time van het saneringssysteem gemiddeld 73% bedraagt, maar dat er een groot verschil is tussen de putten onderling: min. 13% in PP05 en max. 96% in PP09 en PP10. Bij de evaluatie van de potentiele optimalisatie van het systeem moeten worden nagegaan hoe de werking van het systeem verbeterd kan worden.

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

5.2 MONITORING GRONDWATERSTANDEN

Om de seizoenale fluctuaties van het grondwaterpeil en de invloed van de pompputten beter te kunnen opvolgen, is opgenomen in TTV5 (6) dat vanaf 2015 elk kwartaal een dipronde zal worden uitgevoerd samen met de bemonsteringscampagnes voorzien in januari, april, juli en oktober.

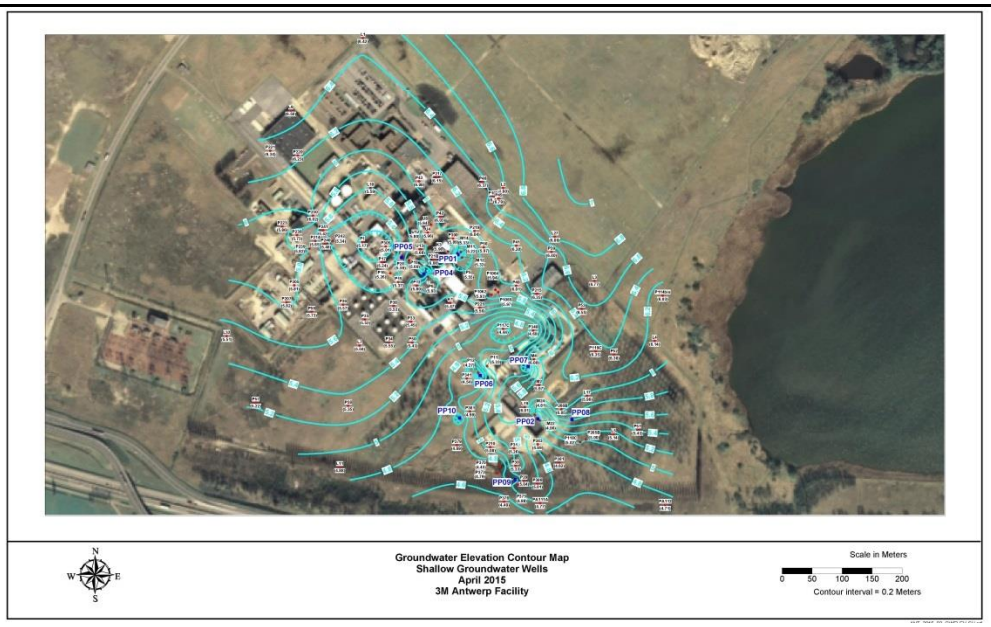
In de periode besproken in dit rapport zijn dan ook de volgende diprondes uitgevoerd:

- op 20 april 2015, en;
- op 17 juli 2015.

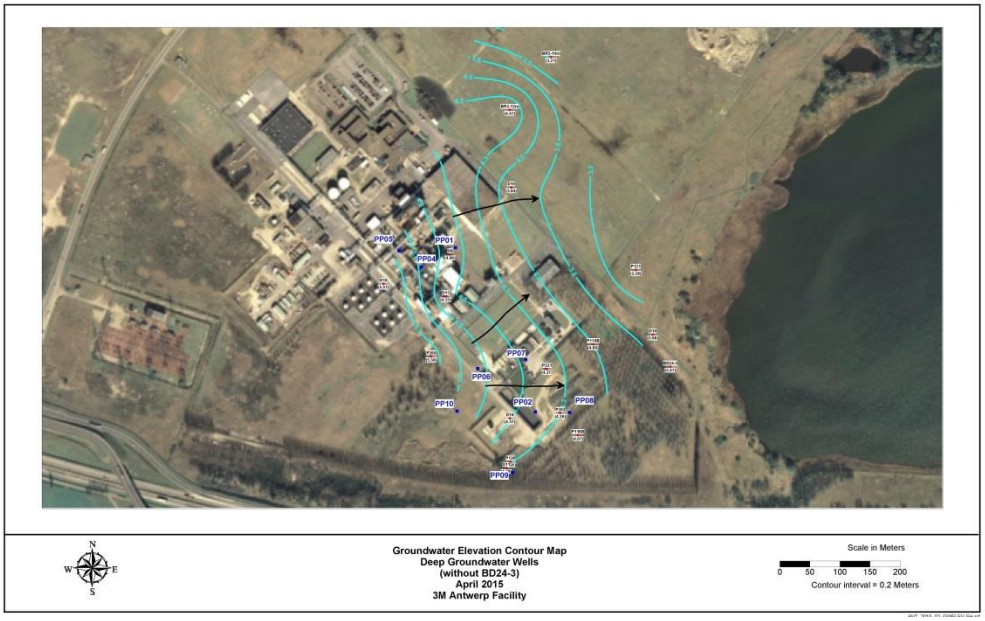
Op 20 april 2015 is een algemene dipronde uitgevoerd waarbij de grondwaterstand in een groot gedeelte van de peilbuizen op en in de onmiddellijke nabijheid van de site is opgemeten. De peilbuizen hebben een filter in of de eerste freatische aquifer of de tweede aquifer.
In juli 2015 is een beperkte ronde uitgevoerd waarbij enkel peilbuizen in de eerste freatische aquifer zijn opgemeten.

De grondwatercontouren die opgemaakt werden op basis van de meetresultaten van de twee diprondes (april en juli) zijn weergegeven in Figuur 5.2.

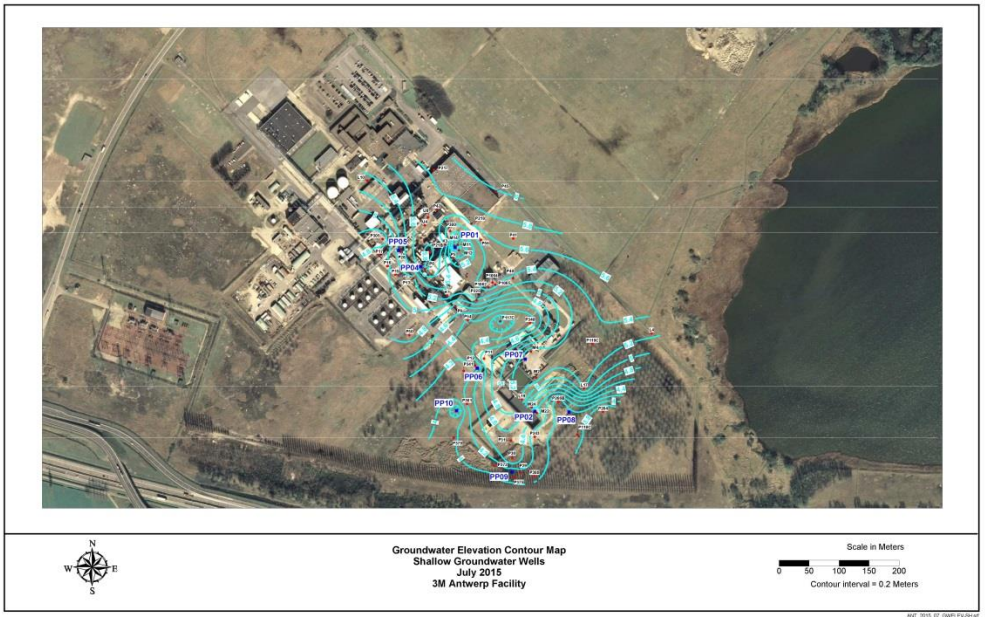
Figuur 5.2 Grondwatercontouren april en juli 2015



Grondwatercontour eerste freatische aquifer april 2015



Grondwatercontour tweede aquifer april 2015



Grondwatercontour eerste freatische aquifer juli 2015

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 (5)).

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

5.3 VOORZIENE WERKEN VOOR VOLGEND JAAR

5.3.1 *Grondwateronttrekking*

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

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

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

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

- Opvolgen van de opgepompte concentraties in het grondwater, zodat een vuilvracht bepaald kan worden.

- 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 na te gaan of een mogelijke uitbreiding van het saneringssysteem nodig is om de captatie van de verontreiniging te optimaliseren. Bij deze evaluatie moet ook worden nagegaan hoe de werking van het systeem verbeterd kan worden (minder storingen en hogere up-time).

6. VOORZIENE WERKEN IN VOLGENDE JAAR VAN DE SANERING

6.1 GRONDWATERSANERING

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

Er wordt voorgesteld om de viermaandelijkse 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 4.10 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 monitoringsronde uitgevoerd worden waarbij de grondwaterstand in een geselecteerd aantal peilbuizen gemeten zal worden om zo de grondwaterstromingsrichting en -contouren te bepalen en de mogelijke evolutie ervan op te volgen.

6.3 PLANNING

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

- Afronden van de evaluatie over de noodzaak van een optimalisatie van het saneringssysteem;
- Voortzetten van een grondwateronttrekking in de twee bronzones;
- Monitoring grondwaterconcentraties conform frequentie vermeld in Tabel 4.10;
- Viermaandelijkse opvolging van de grondwaterpeilen; en,
- September - november 2016: Opstellen en indienen 7e 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 2014) wordt verwezen naar paragraaf 3.3.

De wijzigingen in de saneringsaanpak van juli 2014 tot en met juni 2015 zijn in detail toegelicht in hoofdstuk 4 en 5. Algemeen kan gesteld worden dat het aantal monitoringspeilbuizen en de keuze ervan voor verschillende zones in beperkte mate is gewijzigd. Dit was meestal een gevolg van het aanpassen van de frequentie in functie van de concentratietrends. Een overzicht van het nieuw voorgestelde monitoringsprogramma is gegeven in Tabel 4.10.

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

8. CALAMITEITEN

8.1 20% HF INCIDENT TER HOOGTE VAN GEBOUW 017

Op 11 augustus 2014 is in gebouw 017 een lek van een 20% HF waterige oplossing vastgesteld. Een deel van deze HF oplossing is tussen de betonnen muur en de buitenste plaat van het gebouw (golfplaat) naar buiten gelopen en is daar op de kiezels en het gras terecht gekomen. Geschat wordt dat zo een tiental liter op de bodem terecht gekomen is. De kiezels ter hoogte van de geïmpacteerde zone zijn deels opgelost en zwart gekleurd.
Het lek is onmiddellijk gedicht en de muur afgespoten met water. Dit water is grotendeels in de chemische riolering terecht gekomen. Een klein deel is ook buiten op de bodem terecht gekomen.

Om de impact naar de bodem van de eerder beperkte lekkage van 20% HF te onderzoeken zijn op 26 augustus 2014 door ERM twee mengstalen van de grond genomen tot een diepte van max. 0,5 m-mv: het eerste staal ter hoogte van de geïmpacteerde zone, en het tweede staal in een onverdachte zone.
Uit de analyseresultaten blijkt dat er in de grond ter hoogte van de geïmpacteerde zone een verhoogde fluorideconcentratie meetbaar is. De gemeten concentratie ligt echter ruim onder de relevante (EPA) norm voor industriegebied. De pH waarde in beide grondstalen is normaal.

Aangezien het om een beperkte impact gaat, is ERM van oordeel dat er geen verdere maatregelen (zoals een grondwateronderzoek of ontgraving) noodzakelijk zijn. Er wordt wel aanbevolen de zone op te nemen in het volgende oriënterend bodemonderzoek (voorzien voor medio 2017).

Het incident is op 19 augustus 2014 (binnen de 14 dagen na vaststelling) via het standaardformulier "Melding schadegeval bij klasse I-inrichtingen" gemeld aan OVAM en de milieu-inspectie.

De resultaten en besluiten zijn in een verkennend onderzoek aan OVAM gerapporteerd (referentie R004-0243999-v1.0). Dit onderzoek is op 24 augustus 2015 door OVAM conform verklaard (referentie BVC-20150358804). In de conformverklaring is opgenomen dat "*het incident met waterstoffluoride dient te worden opgenomen in de lopende bodemsanering of eerstvolgende (periodieke) bodemonderzoek*".

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. Deze tweede fase van bodemsaneringsproject zal opgemaakt worden zodra er meer duidelijkheid is over de uitvoering van de infrastructuurwerken in het kader van de Oosterweelverbinding. Er zijn immers heel wat onduidelijkheden over zowel de timing als de uitvoeringsmodaliteiten van de werken welke ter hoogte van de Palingbeek voorzien zijn in het kader van de Oosterweelverbinding. Daar deze werken een belangrijke impact kunnen hebben op de waterhuishouding op deze locatie, dient de saneringsaanpak afgestemd te worden op deze werken.

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

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 zesde tussentijds verslag (TTV6) van de saneringswerken, zoals beschreven in het door de OVAM conform verklaarde bodemsaneringsproject (1e fase) en de vijf voorgaande tussentijdse verslagen. Het betreft de saneringswerken op het deel productiezone, voormalige slibbekkens/waterzuiveringsinstallatie, natuurgebied Blokkersdijk en de 2e aquifer. De beschouwde saneringsperiode loopt van juli 2014 tot en met juli 2015.

Het doel van het eerste gefaseerd bodemsaneringsproject is om de vastgestelde grond- en grondwaterverontreinigingen te monitoring, 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 2014 tot en met juni 2015 de volgende zaken uitgevoerd:

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

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

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

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

- Opmaak van een gedeeltelijk eindevaluatieonderzoek voor de FC-verontreiniging gemeten ter hoogte van de Z-peilbuizen gelegen aan de overkant van de E34 snelweg (Expresweg) en de voormalige BTEX verontreiniging in de zone van de ondergrondse tanken. Deze twee partiële eindevaluatieonderzoeken zijn op 1 juli 2015 door OVAM conform verklaard;

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

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

- Geohydrologisch onderzoek ter hoogte van de oude chemische riolering onder het 3M pad (gelegen ten westen van de Blokkersdijk vijver) om na te gaan of deze riolering mogelijk een bijkomende verontreinigingsbron die de stijgende concentratie trend in de drie peilbuizen en de 3M vijver kan verklaren; en

- Begeleiden van de mitigerende maatregelen genomen naar aanleiding van het incident waarbij op 11 augustus 2014 in gebouw 017 een beperkte hoeveelheid 20% HF waterige oplossing is weggelekt, en het opstellen van een rapport verkennend onderzoek voor OVAM na afloop van de werken.

Uit de onderzoeksverrichtingen en saneringswerken uitgevoerd in de voorbije periode (juli 2014-juli 2015) kunnen volgende conclusies getrokken worden:

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

- Het P&T system, dat operationeel is sinds 2010, bestaat momenteel uit negen pompputten. In totaal is in de periode juli 2014 tot juli 2015 ongeveer 12.535 m^3 verontreinigd grondwater onttrokken en circa 84 kg FC verwijderd. In totaal is sinds juli 2011 circa 362 kg FC verwijderd. Over het algemeen zijn de concentraties in de peilbuizen en pompputten stabiel wat erop duidt dat de verontreiniging globaal gezien gecapteerd wordt door het P&T systeem. Echter, in enkele pompputten en peilbuizen worden stijgende concentraties gemeten, wat erop kan duiden dat de captatie lokaal niet volledig is. Uit veldobservaties blijkt verder dat het saneringssysteem storingsgevoelig is en een up-time heeft van gemiddeld 73%. Er is wel een groot verschil tussen de putten onderling: min. 13% in PP05 en max. 96% in PP09 en PP10.
  ERM raadt aan om de saneringsacties en regelmatige monitoring voort te zetten zoals samengevat in Tabel 4.10 (p.53). De potentiële stijgende concentraties moeten verder geverifieerd worden door monitoring.

Verder wordt aanbevolen om te evalueren of een uitbreiding van het saneringssysteem nodig is om de captatie van de verontreiniging te optimaliseren. Bij deze evaluatie moet ook worden nagegaan hoe de werking van het systeem verbeterd kan worden.

- In geen enkele locatie wordt een significante trend vastgesteld indien enkel de data van het het 3M Environmental lab, dat sinds 2014 de analyses uitvoerd, wordt gebruikt. De PFOS concentratie trend in de Blokkersdijk peilbuizen, de 3M vijver en de Blokkersdijk vijver moet verder opgevolgd worden via regelmatige monitoring en statistische evaluatie voort te zetten zoals samengevat in Tabel 4.10 (p.53). De statistische trend analyses zullen enkel gebeuren 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.
  Op basis van de resultaten van het geohydrologisch onderzoek uitgevoerd ter hoogte van een oude chemische riolering gelegen in de Blokkersdijk kan voorzichtig geconcludeerd worden dat deze riolering mogelijk een secundaire verontreinigingsbron kan zijn. Verder onderzoek (o.a. massa berekeningen en grondwatermodellering met stoftransport) is noodzakelijk om de impact van het FC-houdend slib in deze riolering op de waterkwaliteit van de eerste aquifer en de nabij gelegen vijvers na te gaan.
  Momenteel is geen noodzaak voor bijkomende saneringsacties in deze zone aangezien er geen stijgende concentratietrend is waargenomen in de Blokkersdijk vijver.

- De gemiddelde berekende vuilvracht van PFOS naar de Schelde voor de periode 2014 – 2015 ligt in lijn met eerder berekende vuilvrachten. De hogere vuilvracht voor de periode 2011 – 2012 was waarschijnlijk het gevolg van het slib dat onderin de collectorput van de regenwaterriolering aanwezig was. Intussen is dit slib geruimd en zijn de PFOS concentraties in de regenwaterriolering en de berekende gemiddelde vuilvracht opnieuw gedaald.
  De berekende gemiddelde vuilvrachten zijn steeds onder de maximaal toegelaten vuilvracht van 370 g/dag gebleven.

- In de periode juli 2014 - juli 2015 heeft zich één milieu-incident voorgedaan op de site van 3M. De analyseresultaten toonden geen impact in deze zone. Bijgevolg zijn er geen verdere acties vereist, de zone moet wel worden opgenomen in de lopende sanering of het eerst volgende (periodieke) bodemonderzoek.

- In afwachting van het tweede gefaseerd bodemsaneringsproject worden de FC-concentraties ter hoogte van de zuidelijke perceelgrens opgevolgd door middel van regelmatige grond- en oppervlaktestaalnames. Deze tweede fase van bodemsaneringsproject zal opgemaakt worden zodra er

meer duidelijkheid is over de uitvoering van de infrastructuurwerken in het kader van de Oosterweelverbinding.

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

Het volgende interim rapport met de bespreking van de saneringswerken en monitoringsresultaten van de periode juli 2015 tot en met juli 2016 zal aan OVAM overgemaakt worden voor eind 2016.

■

BIJLAGEN

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		Afdeling	Bodembeheer
Uw Kenmerk		Dienst	Bodemonderzoek en -sanering Oost
Bijlagen	1 bundel	Contactpersoon	Dominique Suys [redacted]
Mechelen	09 FEB. 2009	Ons Kenmerk	BB-O-DS/2009500832 (Dossiernummer: 732)

Kennisgeving van het conformiteitsattest voor het gefaseerd bodemsaneringsproject

Geachte heer,
Geachte mevrouw,

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

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

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

Nulheffing

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

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

OVAM
de heer Filip De Naeyer
Diensthoofd
Stationsstraat 110
2800 MECHELEN

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

Beroep

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

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

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

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

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

Hoogachtend,

Eddy Van Dyck
Afdelingshoofd

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

Conformiteitsattest gefaseerd bodemsaneringsproject

OPENBARE VLAAMSE AFVALSTOFFENMAATSCHAPPIJ

Contactpersoon:Dominique Suys

Referentie:CBSP-nr-4177

09 FEB. 2009

1 Identificatie van het gefaseerd bodemsaneringsproject

Informatie over het gefaseerd bodemsaneringsproject:

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

Informatie over de locatie:

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

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

11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0496 C
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0496 D
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0496 E
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0499 B
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0502 C
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0511 A
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0769 K
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0775 P
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0775 R
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0779 E
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0782 B
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0784 B
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0785 B
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0786 A
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0786/02 G
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0786/02 H
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0789/02 K
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	0789/02 L
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	Canadastraat
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	N49
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	A	Neerstraat
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	H	0353 Z
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	H	0448 D
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	H	0325 E
11056 ZWIJNDRECHT 1 AFD/ZWIJNDRECHT/	H	0505 D
11813 ANTWERPEN 13 AFD	N	0477 A
11813 ANTWERPEN 13 AFD	N	0478 A
11813 ANTWERPEN 13 AFD	N	0479 A
11813 ANTWERPEN 13 AFD	N	0480 A
11813 ANTWERPEN 13 AFD	N	0481 A
11813 ANTWERPEN 13 AFD	N	0482 B
11813 ANTWERPEN 13 AFD	N	0483 A
11813 ANTWERPEN 13 AFD	N	0484
11813 ANTWERPEN 13 AFD	N	0485 A
11813 ANTWERPEN 13 AFD	N	0486 A
11813 ANTWERPEN 13 AFD	N	0487
11813 ANTWERPEN 13 AFD	N	0488 A
11813 ANTWERPEN 13 AFD	N	0490 A
11813 ANTWERPEN 13 AFD	N	0491
11813 ANTWERPEN 13 AFD	N	0492
11813 ANTWERPEN 13 AFD	N	0493
11813 ANTWERPEN 13 AFD	N	0494
11813 ANTWERPEN 13 AFD	N	0495
11813 ANTWERPEN 13 AFD	N	0496
11813 ANTWERPEN 13 AFD	N	0497
11813 ANTWERPEN 13 AFD	N	0498
11813 ANTWERPEN 13 AFD	N	0499
11813 ANTWERPEN 13 AFD	N	0500
11813 ANTWERPEN 13 AFD	N	0501
11813 ANTWERPEN 13 AFD	N	0502
11813 ANTWERPEN 13 AFD	N	0503

11813 ANTWERPEN 13 AFD	N	0504
11813 ANTWERPEN 13 AFD	N	0505
11813 ANTWERPEN 13 AFD	N	0506
11813 ANTWERPEN 13 AFD	N	0507
11813 ANTWERPEN 13 AFD	N	0508
11813 ANTWERPEN 13 AFD	N	0509
11813 ANTWERPEN 13 AFD	N	0510
11813 ANTWERPEN 13 AFD	N	0511
11813 ANTWERPEN 13 AFD	N	0512
11813 ANTWERPEN 13 AFD	N	0513
11813 ANTWERPEN 13 AFD	N	0514
11813 ANTWERPEN 13 AFD	N	0515
11813 ANTWERPEN 13 AFD	N	0516
11813 ANTWERPEN 13 AFD	N	0517
11813 ANTWERPEN 13 AFD	N	0518
11813 ANTWERPEN 13 AFD	N	0519
11813 ANTWERPEN 13 AFD	N	0520
11813 ANTWERPEN 13 AFD	N	0521
11813 ANTWERPEN 13 AFD	N	0522
11813 ANTWERPEN 13 AFD	N	0523
11813 ANTWERPEN 13 AFD	N	0524
11813 ANTWERPEN 13 AFD	N	0525
11813 ANTWERPEN 13 AFD	N	0526
11813 ANTWERPEN 13 AFD	N	0527
11813 ANTWERPEN 13 AFD	N	0528
11813 ANTWERPEN 13 AFD	N	0529
11813 ANTWERPEN 13 AFD	N	0530
11813 ANTWERPEN 13 AFD	N	0531
11813 ANTWERPEN 13 AFD	N	0532
11813 ANTWERPEN 13 AFD	N	0533 B
11813 ANTWERPEN 13 AFD	N	0533 C
11813 ANTWERPEN 13 AFD	N	0533 D
11813 ANTWERPEN 13 AFD	N	0533 E
11813 ANTWERPEN 13 AFD	N	0534 A
11813 ANTWERPEN 13 AFD	N	0534/02 A
11813 ANTWERPEN 13 AFD	N	0543 A
11813 ANTWERPEN 13 AFD	N	0568
11813 ANTWERPEN 13 AFD	N	0569
11813 ANTWERPEN 13 AFD	N	0570
11813 ANTWERPEN 13 AFD	N	0571
11813 ANTWERPEN 13 AFD	N	0572
11813 ANTWERPEN 13 AFD	N	0573
11813 ANTWERPEN 13 AFD	N	0574
11813 ANTWERPEN 13 AFD	N	0575
11813 ANTWERPEN 13 AFD	N	0576
11813 ANTWERPEN 13 AFD	N	0577
11813 ANTWERPEN 13 AFD	N	0578
11813 ANTWERPEN 13 AFD	N	0579
11813 ANTWERPEN 13 AFD	N	0590 A
11813 ANTWERPEN 13 AFD	N	0610
11813 ANTWERPEN 13 AFD	N	0611 A

11813 ANTWERPEN 13 AFD	N	0613 A
11813 ANTWERPEN 13 AFD	N	0614 A
11813 ANTWERPEN 13 AFD	N	0615
11813 ANTWERPEN 13 AFD	N	0616
11813 ANTWERPEN 13 AFD	N	0617 A
11813 ANTWERPEN 13 AFD	N	0619 A
11813 ANTWERPEN 13 AFD	N	0620 A
11813 ANTWERPEN 13 AFD	N	0621 A
11813 ANTWERPEN 13 AFD	N	0622
11813 ANTWERPEN 13 AFD	N	0623 A
11813 ANTWERPEN 13 AFD	N	0624 A
11813 ANTWERPEN 13 AFD	N	0625 A
11813 ANTWERPEN 13 AFD	N	0626 A
11813 ANTWERPEN 13 AFD	N	0627 B
11813 ANTWERPEN 13 AFD	N	0628 E
11813 ANTWERPEN 13 AFD	N	0629 D
11813 ANTWERPEN 13 AFD	N	0630 C
11813 ANTWERPEN 13 AFD	N	0632 A
11813 ANTWERPEN 13 AFD	N	0633 B
11813 ANTWERPEN 13 AFD	N	0634 A
11813 ANTWERPEN 13 AFD	N	0636 A
11813 ANTWERPEN 13 AFD	N	0638 A
11813 ANTWERPEN 13 AFD	N	0640 A
11813 ANTWERPEN 13 AFD	N	0641 A
11813 ANTWERPEN 13 AFD	N	0642 C
11813 ANTWERPEN 13 AFD	N	Blockerdweirweg
11813 ANTWERPEN 13 AFD	N	Rotbeek

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

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

- Andere dan te saneren percelen:

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

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

Informatie over de geplande werkzaamheden:

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

2 Uiteenzetting

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

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

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

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

Ontvankelijk en volledig gefaseerd bodemsaneringsproject

De OVAM heeft het gefaseerd bodemsaneringsproject ontvankelijk en volledig bevonden.

Kennisgeving

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

Advies en openbaar onderzoek

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

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

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

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

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

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

De onderstaande overheden of besturen hebben gunstig advies verleend:

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

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

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

- Alle wettelijke maatregelen om de milieuveiligheid en de arbeidsveiligheid te verzekeren bij de uitvoering van de bodemsaneringswerken, en de maatregelen zoals beschreven in Achilles (veiligheid, gezondheid en milieupreventiesysteem voor on-site bodemsaneringswerken) moeten strikt worden gevolgd.
- Tijdens de sanering moeten regelmatig analyses worden uitgevoerd en ter beschikking gehouden van de controlerende overheden.
- De voorziene grondwateronttrekking past in een bodemsanering met als doel de aanwezige bodemverontreiniging te saneren. De duur van de grondwateronttrekking is slechts richtinggevend en afhankelijk van het verloop van de bodemsanering. Als uit de monitoring gedurende de bodemsaneringswerken blijkt dat de saneringsobjectieven niet worden gehaald binnen de vooropgestelde termijnen, moeten de saneringswerken worden voortgezet.
- Het effluent van de grondwaterzuiveringsinstallatie dat geloosd wordt in het oppervlaktewater, moet voldoen aan de lozingsnormen zoals opgenomen in de vigerende milieuvergunning van 3M van 20 maart 2008.

- De start en het einde van elke fase van de grondwatersanering, moet worden gemeld aan de VMM op het volgende centrale e-mailadres: bodemsanering@VMM.be. Vermeld daarbij de naam van het gefaseerd bodemsaneringsproject, de gemeente, de start- of einddatum en bemaling of sanering.

- Het influent van de waterzuiveringsinstallatie moet periodiek worden gecontroleerd. Als de bodemsaneringsdeskundige op basis van een voldoende aantal representatieve analyses over een voldoende lange periode kan aantonen dat er een stabiele eindsituatie werd verkregen kan de grondwaterzuiveringsinstallatie worden verwijderd.

- De VMM heeft volgende bedenking : "Dat wat de organofluorverontreiniging ter hoogte van het natuurreservaat Blokkersdijk betreft recent onderzoek heeft uitgewezen (Universiteit Antwerpen) dat PFOS (perfluoroctaan sulfaat) werd aangetroffen in vogeleieren en –veren. Deze studie stelde vast dat "de PFOS-concentraties in de eieren van drie vogelsoorten de hoogste waren die ooit werden gemeten bij wilde dieren" ; Dat de voorgestelde maatregelen dan ook ondermaats zijn gezien de grote kwetsbaarheid van dit gebied ;Dat de vaststelling dat er geen negatief effect is waar te nemen gezien aan de vooropstelling (aantal soorten en aantal broedjaren) wordt voldaan, wetenschappelijk zwak is ; Dat zich hier een veel dieper wetenschappelijke studie opdringt teneinde na te gaan welke gevolgen deze hoge concentraties aan PFOS bij dieren (inzonder vogels) hebben, nu en in de toekomst en wat de mogelijke saneringsremedies zijn ; Dat voorgesteld wordt deze studie op te maken in een termijn van 3 jaar na bekomen van de conformverklaring van de bodemsanering. Een exemplaar van deze studie te bezorgen aan VMM, OVAM, AMV en ANB.
- Het agentschap voor Natuur en Bos heeft een opmerking in dezelfde richting : "ANB pleit voor één of andere vorm van biomonitoring om na te gaan of er door accumulatie geen steeds hogere concentraties aan PFOS in de lokale voedselketen geraken, zelfs bij een stabiele concentratie in het oppervlaktewater. Monitoring van de aantallen en soorten van de vogels gebeurt reeds, en wordt hiermee niet bedoeld.
  Wel blijft dus voor ANB wenselijk te meten of er geen verdere accumulatie optreedt in het ecosysteem, hetzij via concentraties in algen, onderwatervegetatie, ongewervelden en vissen. De frequentie hoeft ook niet jaarlijks te zijn."

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

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

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

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

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

Als back-up scenario wordt ter hoogte van Blokkersdijk een beheersing van de verontreiniging voorzien door middel van onttrekking op horizontale drains ter hoogte van het 3M pad. Het gezuiverde grondwater wordt geherinfiltreerd via een open gracht om een negatief effect op het oppervlaktewaterpeil in de Blokkersdijk tegen te gaan. Op deze manier zal een zelfde hoeveelheid water in Blokkersdijk terechtkomen. De reliëfwijziging heeft dus net tot doel om geen effect op de waterhuishouding in Blokkersdijk te veroorzaken.
De back-upvariant wordt uitgevoerd op eigendom van 3M, meer bepaald de verbindingsweg tussen de 3M hoofd-site en de Schelde. De administratieve grenzen van het natuurreservaat (Vogelrichtlijngebied) liggen eenduidig meer naar het oosten. Er valt geen eigendom van 3M binnen het natuurreservaat.
Op basis van het K.B. van 12 mei 1980 vallen 3 percelen van 3M binnen het 'beschermd landschap Blokkersdijk'. Er zijn geen indicaties dat de reliëfwijziging op deze percelen plaatsvindt en bijgevolg binnen het 'beschermd landschap Blokkersdijk' wordt doorgevoerd:

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

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

In het kader van het bodemsaneringsproject werd nagegaan of MER-plichtige activiteiten werden uitgevoerd. Op de voorgestelde saneringswerken is geen MER-plicht van toepassing. Wel was een passende beoordeling noodzakelijk door de nabije ligging van het natuurreservaat Blokkersdijk en deze werd ook uitgevoerd.
In deze fase is er eveneens geen aanvraag noodzakelijk bij de Provincie-Dienst Water, gezien de Palingbeek niet beïnvloed wordt door de voorgestelde werken. De onttrekking in de bronzones bevindt zich op grote afstand van de Palingbeek en piloottesten hebben eveneens uitgewezen dat de invloedsstraal minimaal is. De Provincie-Dienst Water zal echter wel geïnformeerd moeten worden bij opmaak van het gefaseerd bodemsaneringsproject dat werken voorziet in de nabijheid van de Palingbeek.

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

  Momenteel zijn er geen risico's verbonden aan de verhoogde organofluorconcentraties in de Palingbeek. Gezien de Palingbeek in industriegebied gelegen is en deze geen natuurfunctie heeft, is de ecotoxicologische veilige concentratie (PNEC) van weinig belang. Rekening houdende met de verschillende influxen van PFOS in de Schelde (waaronder ook een gedeelte van de Palingbeek) wordt de ecotoxicologisch veilige PFOS-dagvracht voor de Schelde niet overschreden.
  Aangezien het een historische verontreiniging betreft moeten er cfr het bodemdecreet vanuit risicogebaseerd standpunt niet onmiddellijk maatregelen voor de Palingbeek genomen worden. In het kader van de Oosterweelverbinding wordt de Palingbeek waarschijnlijk verlegd en krijgt ze een ecocorridorfunctie. Op dat moment kan er niet uitgesloten worden dat er risico's verbonden zijn aan de fluorimpact en dient overeenkomstig het bodemdecreet overgegaan te worden naar een BATNEEC-sanering.
  Door de onzekerheden met betrekking tot de werken aan de Palingbeek in het kader van de Oosterweelverbinding kan momenteel geen finale saneringsvariant voorgesteld worden. Een gefaseerd bodemsaneringsproject zal bijgevolg opgesteld worden nadat de werken aan de Palingbeek zijn uitgevoerd en de organofluorimpact in de nieuwe Palingbeek gekend is.

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

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

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

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

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

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

Watertoets

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

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

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

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

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

Bepalingen van de bodemsaneringswerken

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

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

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

3 Besluit

Artikel 1 - Conformiteit

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

Art. 2 – Voorwaarden voor de uitvoering van de bodemsaneringswerken

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

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

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

- De verantwoordelijke of bij contract aangestelde verantwoordelijke van de bodemsaneringswerken moet op eenvoudig verzoek van de toezichthoudende ambtenaren of een door hen aangestelde onafhankelijke deskundige alle nodige gegevens verstrekken om een degelijke evaluatie en controle van de voortgang van de werken te kunnen uitvoeren.
- Alle wettelijke maatregelen om de milieuveiligheid en de arbeidsveiligheid te verzekeren bij de uitvoering van de bodemsaneringswerken en de maatregelen zoals beschreven in Achilles, moeten strikt worden gevolgd,
- Het verloop en de resultaten van de bodemsaneringswerken moet **jaarlijks** aan de OVAM worden gerapporteerd volgens de standaardprocedure 'Bodemsaneringswerken, Eindevaluatieonderoek en Nazorg'.
- De grondwateronttrekking is gekoppeld aan de strikte naleving van de eigen milieuvergunningsvoorwaarden van 3M.
- Het effluent van de grondwaterzuiveringsinstallatie dat geloosd wordt in het oppervlaktewater, moet voldoen aan de lozingsnormen zoals opgenomen in de vigerende milieuvergunning van 3M van 20 maart 2008.

- De start en het einde van elke fase van de grondwatersanering, moet worden gemeld aan de VMM op het volgende centrale e-mailadres: bodemsanering@VMM.be. Vermeld daarbij de naam, de gemeente, de start- of einddatum en bemaling of sanering.

- De monitoring van de peilbuizen bij het beëindigen van de saneringswerken moet gebeuren nadat de evenwichtsituatie in de bodem is bereikt. Ter controle van de efficiëntie van de saneringsmaatregelen moeten minimaal de peilbuizen stroomafwaarts worden gecontroleerd alsook de peilbuizen met de hoogste concentratie aan verontreinigende stoffen, in de kern van de verontreiniging.
- De saneringswerken mogen pas worden stilgelegd na het bereiken van de vooropgestelde doelstellingen, of na onderling overleg met de OVAM, rekening houdend met de efficiëntie van de saneringstechniek (BATNEEC-principe) en de kwaliteit van het vaste deel van de aarde en van het grondwater. De vooropgestelde termijn van de bodemsanering is niet bindend en moet, gebaseerd op de bodemkwaliteit, op eenvoudig verzoek van de OVAM worden voortgezet.
- De uitvoering van bodemsaneringswerken is gebonden aan de regels die zijn opgenomen in het meest recente Achillespreventiesysteem. Dat betekent dat de opdrachtgever de bodemsaneerder, voor aanvang van de bodemsaneringswerken, een certificatie-instelling moet aanstellen. Op die manier kan worden nagegaan of de bodemsaneerder het Achillespreventiesysteem naleeft. Indien de bodemsaneerder lid is van de OVB heeft de hij een milieubeleidsovereenkomst getekend en is hij gebonden aan het het Achilleszorgsysteem. In dat geval is het niet nodig dat er een certificatie-instelling wordt aangeschreven. De audit in het kader van het Achillespreventiesysteem moet worden uitgevoerd overeenkomstig de bepalingen van deel B van het Achillespreventiesysteem.
- De OVAM is van mening dat het aangewezen is om op korte termijn met de verschillende betrokken partijen de relevantie en de opzet van dergelijke wetenschappelijke studie te bespreken. Er moet onder meer uitgemaakt worden hoe dergelijke studie er zou moeten uitzien, op welke factoren dan gemeten moet worden, op welke targetsoorten gefocust moet worden, hoe bepaald moet worden of een bepaalde fluorconcentratie een risico inhoudt, en welke gevolgen aan de resultaten van de studie gegeven worden.
- Aangezien het een bedrijf in exploitatie betreft, zijn de mogelijke saneringstechnieken beperkt. De OVAM wil hierbij als randvoorwaarde stellen dat de in uitvoering zijnde saneringstechnologie op jaarlijkse basis geherevalueerd moet worden. De evolutie in nieuwe saneringstechnieken moet opgevolgd worden. Indien er een overdracht of sluiting gepland is, is een herevaluatie van de saneringstechniek eveneens noodzakelijk. Indien infrastructuurwerken op de site gepland zijn, dient bekeken te worden of andere actieve saneringsmaatregelen zoals ontgraving mogelijk zijn.
- Bij de uitvoering van de sanering dient rekening gehouden te worden met de aanwezigheid van de andere aanwezige verontreinigingen in het grondwater.
- De monitoringspeilbuizen dienen met een bepaalde frequentie op een uitgebreider parameterpakket (bv met TFA, ...) geanalyseerd te worden.

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

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

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

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

Art. 3 – Termijn aanvang van de bodemsaneringswerken

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

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

te Mechelen, 09 FEB. 2009

Eddy Van Dyck
Afdelingshoofd

Bijlage 2

Analysecertificaten

Final Report

Analysis of Groundwater, Surface Water, and Sludge Samples for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Belgium October 2014 Sampling

Laboratory Request Number: ISO14-0028

Report Date – Date of Last Signature

Testing Laboratory

3M EHS Operations
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 ISO14-0028

Analysis of Groundwater, Surface Water, and Sludge Samples for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Belgium – October 2014 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M Environmental Laboratory prepared and analyzed groundwater and surface water samples and three sludge samples collected by Environmental Resources Management (ERM) personnel at the 3M Belgium facility. Samples were collected on October 20-21, 2014. Samples were returned to the 3M Environmental Laboratory on October 28, 2014 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 ISO14-0028.

The 3M Environmental Laboratory prepared sample containers for nineteen water sampling locations. Each sample set for the water collection locations consisted of a field sample, field sample duplicate, and a target analyte field matrix spike. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 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 internal standards prior to being sent to the field for sample collection. Three empty bottles were sent for the three sludge locations along with one soil trip blank.

Water samples were prepared and analyzed using method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Sludge samples were prepared and analyzed using method ETS-8-042.0 "Solvent Extraction and Isocratic LC/MS/MS Analysis of Soils for C4-C12 Perfluorinated Carboxylic Acids and Perfluorobutane Sulfonate, Perfluorohexane Sulfonate and Perfluorooctane Sulfonate". Select internal standards were used to aid in the data quality objectives. Sludge sample results reported by method ETS-8-042.0 are not covered under the laboratory's current A2LA accreditation.

Table 1 summarizes the water sample results with Table 2 for sludge 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**
ISO14-0028-001	P321; Sample	3170	890	3180	0.465
ISO14-0028-002	P321; Sample Dup	3110	893	3240	0.411
	Average	**3140**	**892**	**3210**	**0.438** [2]
	%RPD Sample/Sample Dup	**1.9**	**0.34**	**1.9**	**12**
ISO14-0028-004	Blokkersdijkvijver std; Sample	3.89	3.68	41.1	1.02
ISO14-0028-005	Blokkersdijkvijver std; Sample Dup	1.24	1.19	12.6	0.439
	Average	**2.57**	**2.44**	**26.9**	**0.730**
	%RPD Sample/Sample Dup	**103** [3]	**102** [3]	**106** [3]	**80** [3]
ISO14-0028-007	3M Pond; Sample	1.09	0.541	3.31	0.0476
ISO14-0028-008	3M Pond; Sample Dup	1.10	0.562	3.05	0.0414
	Average	**1.10**	**0.552**	**3.18** [4]	**0.0445** [2]
	%RPD Sample/Sample Dup	**0.91**	**3.8**	**8.2**	**14**
ISO14-0028-010	L4; Sample	3.13	2.39	30.4	1.04
ISO14-0028-011	L4; Sample Dup	3.15	2.38	29.3	1.05
	Average	**3.14**	**2.39**	**29.9**	**1.05**
	%RPD Sample/Sample Dup	**0.64**	**0.42**	**3.7**	**0.96**
ISO14-0028-013	P114bis; Sample	4.62	1.63	13.7	0.0306
ISO14-0028-014	P114bis; Sample Dup	4.43	1.62	13.0	0.0261
	Average	**4.53**	**1.63**	**13.4** [2]	**0.0284** [2]
	%RPD Sample/Sample Dup	**4.2**	**0.62**	**5.2**	**16**
ISO14-0028-016	P116; Sample	1.13	0.626	8.54	0.0651
ISO14-0028-017	P116; Sample Dup	1.05	0.612	8.99	0.0695
	Average	**1.09**	**0.619**	**8.77** [2]	**0.0673** [2]
	%RPD Sample/Sample Dup	**7.3**	**2.3**	**5.1**	**6.5**
ISO14-0028-046	12; Sample	100	33.3	249	0.985
ISO14-0028-047	12; Sample Dup	94.0	26.0	244	0.296
	Average	**97.0**	**29.7**	**247**	**0.641** [2]
	%RPD Sample/Sample Dup	**6.2**	**25** [3]	**2.0**	**108** [3]
ISO14-0028-049	13; Sample	89.3	25.9	199	0.303
ISO14-0028-050	13; Sample Dup	87.6	24.9	202	0.278
	Average	**88.5**	**25.4**	**201**	**0.291** [2]
	%RPD Sample/Sample Dup	**1.9**	**3.9**	**1.5**	**8.6**
ISO14-0028-052	5; Sample	30.5	22.9	34.5	0.143
ISO14-0028-053	5; Sample Dup	28.4	21.7	38.8	0.152
	Average	**29.5**	**22.3**	**36.7**	**0.148** [2]
	%RPD Sample/Sample Dup	**7.1**	**5.4**	**12**	**6.1**
ISO14-0028-055	Bemlingsstation; Sample	29.0	21.7	53.7	0.190
ISO14-0028-056	Bemlingsstation; Sample Dup	28.9	21.9	53.7	0.165
	Average	**29.0**	**21.8**	**53.7**	**0.178** [2]
	%RPD Sample/Sample Dup	**0.35**	**0.92**	**0.0**	**14**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 17%, PFBS ± 6.7%, PFHS ± 11%, PFOS ± 19%, and PFOSA ± 22%.

(2) Samples reported using internal calibration. The analytical method uncertainty associated with the reported results are as follows: PFOS ± 24% and PFOSA ± 18%.

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

(4) The method uncertainty has been expanded based on field matrix spike or laboratory 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**
ISO14-0028-019	PP01; Sample	86.8	90.2	19000	6440	29.4
ISO14-0028-020	PP01; Sample Dup	92.1	96.3	22100	7690	48.8
	Average	**89.5**	**93.3**	**20600**	**7070**	**39.1**
	%RPD Sample/Sample Dup	**5.9**	**6.5**	**15**	**18**	**50 [(3)]**
ISO14-0028-022	PP02; Sample	586	27.1	2640	20700	23.3
ISO14-0028-023	PP02; Sample Dup	585	26.7	2640	21400	28.4
	Average	**586**	**26.9**	**2640**	**21100**	**25.9**
	%RPD Sample/Sample Dup	**0.17**	**1.5**	**0.0**	**3.3**	**20**
ISO14-0028-025	PP04; Sample	1520	2580	758	9460	46.6
ISO14-0028-026	PP04; Sample Dup	1560	2600	773	8780	37.8
	Average	**1540**	**2590**	**766**	**9120**	**42.2**
	%RPD Sample/Sample Dup	**2.6**	**0.77**	**2.0**	**7.5**	**21 [(3)]**
ISO14-0028-028	PP05; Sample	1440	17200	6510	214	25.3
ISO14-0028-029	PP05; Sample Dup	1420	17600	6770	187	23.8
	Average	**1430**	**17400**	**6640**	**201**	**24.6**
	%RPD Sample/Sample Dup	**1.4**	**2.3**	**3.9**	**13**	**6.1**
ISO14-0028-031	PP06; Sample	159	24.7	46.0	2200	41.2
ISO14-0028-032	PP06; Sample Dup	157	20.8	44.3	2210	35.7
	Average	**158**	**22.8**	**45.2**	**2210**	**38.5 [(4)]**
	%RPD Sample/Sample Dup	**1.3**	**17**	**3.8**	**0.45**	**14**
ISO14-0028-034	PP07; Sample	412	127	132	2950	53.7
ISO14-0028-035	PP07; Sample Dup	431	126	132	2910	50.4
	Average	**422**	**127**	**132**	**2930**	**52.1**
	%RPD Sample/Sample Dup	**4.5**	**0.79**	**0.0**	**1.4**	**6.3**
ISO14-0028-037	PP08; Sample	572	34.7	282	8430	47.3
ISO14-0028-038	PP08; Sample Dup	553	34.1	268	7910	45.3
	Average	**563**	**34.4**	**275**	**8170**	**46.3**
	%RPD Sample/Sample Dup	**3.4**	**1.7**	**5.1**	**6.4**	**4.3**
ISO14-0028-040	PP09; Sample	743	30.9	438	2440	11.6
ISO14-0028-041	PP09; Sample Dup	747	30.7	441	2430	11.0
	Average	**745**	**30.8**	**440**	**2440**	**11.3**
	%RPD Sample/Sample Dup	**0.54**	**0.65**	**0.68**	**0.41**	**5.3**
ISO14-0028-043	PP10; Sample	663	21.5	201	4000	11.5
ISO14-0028-044	PP10; Sample Dup	659	20.9	204	4000	10.8
	Average	**661**	**21.2**	**203**	**4000**	**11.2**
	%RPD Sample/Sample Dup	**0.61**	**2.8**	**1.5**	**0.0**	**6.3**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 17%, PFBS ± 6.7%, PFHS ± 11%, PFOS ± 19%, and PFOSA ± 22%.

(2) Samples reported using internal calibration. The analytical method uncertainty associated with the reported results are as follows: PFOS ± 24% and PFOSA ± 18%.

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

(4) The method uncertainty has been expanded based on field matrix spike or laboratory matrix spike recovery. See section 4 for details.

Table 2. Sludge Sample Results Summary [1,2]

		Concentration (ng/g), dry wt.			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
ISO14-0028-062	Manhole 1; Sample	1100	251	18900	1250
ISO14-0028-062 Dup	Manhole 1; Sample Dup	2330	449	34900	2350
	Average	**1710 [3]**	**350**	**26900**	**1800 [4]**
	%RPD Sample/Sample Dup	**72 [5]**	**57 [5]**	**59 [5]**	**61 [5]**
ISO14-0028-063	Manhole 2; Sample	5420	1280	127000	3270
ISO14-0028-063 Dup	Manhole 2; Sample Dup	9170	1900	254000	7210
	Average	**7290**	**1590**	**191000**	**5240**
	%RPD Sample/Sample Dup	**51 [5]**	**39**	**66 [5]**	**75 [5]**
ISO14-0028-064	Manhole 3; Sample	27100	10500	505000	13100
ISO14-0028-064 Dup	Manhole 3; Sample Dup	26500	11100	424000	10100
	Average	**26800 [4]**	**10800**	**464000**	**11600**
	%RPD Sample/Sample Dup	**2.5**	**5.5**	**18**	**26**
ISO14-0028-065	Trip Blank, Soil; Sample	<0.435	<0.0908	<0.168	<0.0908
ISO14-0028-065 Dup	Trip Blank, Soil; Sample Dup	<0.433	<0.0904	<0.167	<0.0904
	Average	<0.434	<0.0906	<0.168	<0.0906

(1) The method used to analyze the sludge samples is not covered by the laboratory's current A2LA accreditation.
(2) Samples were reported using internal calibration. The analytical method uncertainties associated with the reported results are based on the laboratory control sample recoveries and are as follows: PFOA ± 45%, PFHS ± 65%, PFOS ± 80%, and PFOSA ± 61%.
(3) The analytical method uncertainty has been expanded for PFOA to ± 56% based on the LMS recovery.
(4) Laboratory matrix spike recovery (LMS) did not meet acceptance criteria of 100 ± 30%, but was within the analytical method uncertainty.
(5) Sample/sample duplicate RPD ≥50%.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M Environmental Laboratory method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis" and method ETS-8-042.0 "Solvent Extraction and Isocratic LC/MS/MS Analysis of Soils for C4-C12 Perfluorinated Carboxylic Acids and Perfluorobutane Sulfonate, Perfluorohexane Sulfonate and Perfluorooctane Sulfonate". Sampling locations P321, L4, P114bis, P116, 3M Pond, Blokkersdijkvijver – standard, 12, 13, 5, and Bemalingsstation and sludge samples were reported for PFOA, PFHS, PFOS, and PFOSA. Sampling locations PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09, and PP10 were reported for PFOA, PFBS, PFHS, PFOS, and PFOSA.

Table 3. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected on October 20-21, 2014 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 water sample locations were spiked with a mixture of mass-labeled internal standards at a nominal concentration of 1 ng/mL prior to sample collection. Collected sample bottles were returned to the laboratory at ambient temperature on October 28, 2014.

2.3 Sample Preparation

Water 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 were also fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL.

Samples re-analyzed with internal standard: Samples were prepared by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2).

During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 ng/mL). The sample bottles were spiked with an internal standard mix at a nominal concentration of 1 ng/mL prior to being sent to the field for sample collection. The laboratory control samples were then diluted with methanol in the same manner as the samples.

Sludge Analysis:
Sample preparation involved the removal of approximately a 1 cc aliquot of the well mixed sludge sample or trip blank, which typically weighs nominal 1.0 gram (dry weight), in duplicate. A third and fourth aliquot of taken of each sludge sample and fortified with known quantities of target analytes as a laboratory matrix spike (LMS). The level of LMS was determined based on a sample screening run. Only one LMS was prepared for the trip blank at 1 ng/g.

Each sample aliquot was fortified with a fixed quantity of stable isotope labeled internal standards and surrogate recovery standards. Then 8 mL of a 4:1 acetonitrile:water extraction solution was added, followed by sonication for one hour, and followed by centrifugation to pellet out the solids. An aliquot of the supernatant from the trip blank was transferred to an autovial for analysis. For the sludge analysis, the supernatant was further diluted by a factor of 1:500 by adding a 0.01 mL aliquot of the sample to 4.99 mL of dilution solvent or a factor of 1:1000 by adding a 0.01 mL aliquot of the samples to 9.99 mL of dilution solvent.

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). 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 4. Instrument Parameters.

Instrument Name	ETS Kirk	ETS MaryAnn
Liquid Chromatograph	Agilent 1260	Agilent 1200
Analysis Method	ETS-8-044.1 (water analysis)	ETS-8-042.0 (sludge analysis)
Analysis Date	11/3/14, 11/6/14, 11/12/14, 11/18/14	11/14/14
Guard column	Betasil C18 (4.6 mm X 100 mm), 5 µ	Not Applicable
Analytical column	Betasil C18 (4.6 mm X 100 mm), 5 µ	Betasil C8 (4.6 mm X 150 mm), 5 µ
Injection Volume	5 µL	10 µL
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex API 5000
Ion Source	Turbo Spray	Turbo Spray
Polarity	Negative	Negative
Software	Analyst 1.6.2	Analyst 1.6.2

Table 5. Liquid Chromatography Gradient Program.

ETS-8-044.1 Water Analysis				
Step Number	**Total Time (min)**	**Flow Rate (µL/min)**	**Percent A (2 mM ammonium acetate)**	**Percent B (Methanol)**
0	0.00	750	90.0	10.0
1	0.50	750	90.0	10.0
2	4.00	750	70.0	30.0
3	6.00	750	70.0	30.0
4	11.0	750	20.0	80.0
5	13.0	750	20.0	80.0
6	13.5	750	10.0	90.0
7	16.0	750	10.0	90.0
8	16.5	750	90.0	10.0
9	19.0	750	90.0	10.0
ETS-8-042.0 Sludge Analysis				
Step Number	**Total Time (min)**	**Flow Rate (µL/min)**	**Percent A (1% acetic acid in water)**	**Percent B (10mM Ammonium Acetate in 80:20 Methanol: Acetonitrile)**
0	0.00	1000	15.0	85.0
1	4.00	1000	15.0	85.0

Table 6. 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

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 used for water samples re-analyzed on 11/6/14 and 11/18/14 and for the sludge analysis.

3 Data Analysis

3.1 Calibration

11/3/14 and 11/12/14 Water Analysis: 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. A total of eleven spiked standards ranging from 0.02 ng/mL to 50 ng/mL (nominal) were analyzed. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL to 25 ng/mL nominal. A quadratic, 1/x weighted, calibration curve of the 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.

11/6/14 and 11/18/14 Water Analysis with Internal Standard: Samples were analyzed for all analytes against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50 mL of 50:50 methanol:laboratory reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. Calibration standards ranging from 0.0125 ng/mL or 1.00 ng/mL to 100 ng/mL (nominal) were analyzed. 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.

For both analyses, 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.

Sludge Analysis: Samples were analyzed for all analytes against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared with the samples using a control soil TCR-456 as the control matrix. A 1 cc aliquot of soil was weighed out, which typically weighs nominal 1.0 gram (dry weight). The instrument response is calibrated for each analyte based on the mass of analyte added to the standard calibration soil matrix prior to extraction. Fourteen calibration standards were prepared ranging from 0.1 ng to 1000 ng (nominal) with nine calibration standards containing surrogate recovery standards ranging from 0.1 ng to 25 ng (nominal).

Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±30% (100±35% for the lowest curve point) were met for all analytes. The correlation coefficient (r) was greater than 0.990 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times (three times for the sludge analysis) at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes except for PFOS analyzed on 11/18/14, which had an area ratio RSD of 16%. A method deviation is included with the raw data.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in methods ETS-8-044.1 and ETS-8-042.0 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 **Table 7** below.

Table 7. LOQ

Analyte	LOQ, ng/mL (1) 11/3/14 Water Analysis	LOQ, ng/mL (2) 11/6/14 Water Analysis	LOQ, ng/mL (1) 11/12/14 Water Analysis	LOQ, ng/mL (2) 11/18/14 Water Analysis	LOQ, ng (1) 11/14/14 Sludge Analysis
PFOA	0.0192	0.0240	NA	0.0240	0.479
PFBS	0.0200	NA	0.250	NA	NA
PFHS	0.0200	0.0250	0.250	0.0250	0.100
PFOS	0.0464	0.0232	0.232	1.85	0.185
PFOSA	0.0200	0.0250	NA	0.0250	0.100

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

3.4 Continuing Calibration

During the course of the analytical sequence, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve were still in control. All reported results were bracketed by CCVs that met method acceptance criteria of 100%±25% (100%±30% for sludge samples) except for one CCV for PFOSA for the sludge analysis on 11/14/14, which had a recovery of 67.8%. A method deviation is included with the raw data.

3.5 Blanks

Water Analysis: 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.

Sludge Analysis: Three types of blanks were prepared and analyzed with the samples: a soil trip blank, method blanks consisting of extracted soil matrix TCR-456, and extraction solvent blanks. The method blanks were reviewed and used to evaluate method performance to determine the LOQ of 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. For the water sample analysis, LCSs were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. If applicable, the LCSs were then diluted in the same manner as the samples. Method ETS-8-044.1 states that the average recovery of LCSs at each spiking level must be within 80%-120% with a RSD ≤20%. All LCSs prepared with the water samples met method acceptance criteria.

For the sludge analysis, the low, mid, and high LCS samples were prepared in control soil TCR-456 and extracted in the same manner as the samples. Method ETS-8-042.0 for sludge analysis states that the average recovery of LCSs must be within 70%-130% with a RSD ≤50%. All LCSs prepared with the sludge samples met acceptance criteria with the exception of low level LCSs PFHS which had an average recovery of 134%, and PFOS which had an average recovery of 136%. A Dixon's Q-test was performed on the LCSs for all analytes to determine if any of the LCS sample results were outliers. One low level LCS for PFOA was determined to be an outlier and as such, was excluded from statistical analysis of the low level LCSs for PFOA. A method deviation is included with the raw data. All LCS samples, unless excluded as an outlier by the Q test, 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 8**.

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

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

Table 8. Laboratory Control Spike Results.

ETS-8-044.1 External Calibration Analyzed 11/3/14	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-141030-1	0.191	0.170	89.3	0.199	0.197	98.9
LCS-141030-2	0.191	0.167	87.4	0.199	0.200	101
LCS-141030-3	0.191	0.177	92.8	0.199	0.205	103
Average ± %RSD	**89.8% ± 3.0%**			**101% ± 2.0%**		
LCS-141030-4	19.1	18.9	98.8	19.9	20.4	102
LCS-141030-5	19.1	18.5	96.8	19.9	20.0	100
LCS-141030-6	19.1	19.0	99.5	19.9	20.0	101
Average ± %RSD	**98.4% ± 1.4%**			**101% ± 0.99%**		
LCS-141030-7	76.0	73.5	96.7	79.4	77.1	97.1
LCS-141030-8	76.0	74.1	97.6	79.4	79.0	99.5
LCS-141030-9	76.0	73.2	96.3	79.4	78.0	98.2
Average ± %RSD	**96.9% ± 0.69%**			**98.3% ± 1.2%**		

ETS-8-044.1 External Calibration Analyzed 11/3/14	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-141030-1	0.199	0.206	103	0.185	0.175	94.6
LCS-141030-2	0.199	0.195	98.1	0.185	0.179	96.6
LCS-141030-3	0.199	0.207	104	0.185	0.232	125
Average ± %RSD	**102% ± 3.1%**			**105% ± 16%**		
LCS-141030-4	19.9	20.0	100	18.5	17.8	96.1
LCS-141030-5	19.9	19.8	99.7	18.5	17.5	94.4
LCS-141030-6	19.9	20.2	102	18.5	18.0	97.2
Average ± %RSD	**101% ± 1.2%**			**95.9% ± 1.5%**		
LCS-141030-7	79.4	>ULOQ	NA	73.6	68.9	93.6
LCS-141030-8	79.4	>ULOQ	NA	73.6	70.2	95.3
LCS-141030-9	79.4	>ULOQ	NA	73.6	69.3	94.1
Average ± %RSD	**NA (1)**			**94.3% ± 0.93%**		

LLOQ = Lower Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the acceptable calibration range.
(2) LCS calculated as an outlier by Dixon's Q-test. Not included in statistical calculations.
(3) LCSs did not meet ETS-8-042 method acceptance criteria of 100 ± 30%.

Table 8 continued. Laboratory Control Spike Results.

ETS-8-044.1 External Calibration Analyzed 11/3/14	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-141030-1	0.199	0.205	103
LCS-141030-2	0.199	0.201	101
LCS-141030-3	0.199	0.208	104
Average ± %RSD	**103% ± 1.5%**		
LCS-141030-4	19.9	20.8	105
LCS-141030-5	19.9	20.6	104
LCS-141030-6	19.9	20.6	104
Average ± %RSD	**104% ± 0.55%**		
LCS-141030-7	79.4	>ULOQ	NA
LCS-141030-8	79.4	>ULOQ	NA
LCS-141030-9	79.4	>ULOQ	NA
Average ± %RSD	**NA (1)**		

ETS-8-044.1 External Calibration Analyzed 11/3/14	$^{13}C_4$-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-141030-1	0.199	0.219	110	0.190	0.203	107
LCS-141030-2	0.199	0.223	112	0.190	0.213	112
LCS-141030-3	0.199	0.234	118	0.190	0.225	119
Average ± %RSD	**113% ± 3.7%**			**113% ± 5.4%**		
LCS-141030-4	1.99	2.16	109	1.90	2.15	113
LCS-141030-5	1.99	2.18	110	1.90	2.10	110
LCS-141030-6	1.99	2.14	108	1.90	2.17	114
Average ± %RSD	**109% ± 0.92%**			**112% ± 1.9%**		

LLOQ = Lower Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the acceptable calibration range.
(2) LCS calculated as an outlier by Dixon's Q-test. Not included in statistical calculations.
(3) LCSs did not meet ETS-8-042 method acceptance criteria of 100 ± 30%.

Table 8 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal Calibration Analyzed 11/6/14	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-141106-1	0.190	0.208	109	0.199	0.220	111
LCS-141106-2	0.190	0.194	102	0.199	0.214	107
LCS-141106-3	0.190	0.189	99.5	0.199	0.202	101
Average ± %RSD	**104% ± 4.8%**			**106% ± 4.7%**		
LCS-141106-4	9.52	9.31	97.8	9.94	10.1	101
LCS-141106-5	9.52	9.24	97.1	9.94	10.2	103
LCS-141106-6	9.52	9.64	101	9.94	10.6	106
Average ± %RSD	**98.6% ± 2.1%**			**103% ± 2.4%**		
LCS-141106-7	75.7	67.6	89.3	79.0	75.3	95.3
LCS-141106-8	75.7	69.8	92.1	79.0	74.8	94.7
LCS-141106-9	75.7	70.4	93.0	79.0	75.1	95.1
Average ± %RSD	**91.5% ± 2.1%**			**95.0% ± 0.32%**		

ETS-8-044.1 Internal Calibration Analyzed 11/6/14	PFOS (Linear + Branched)			PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-141106-1	0.184	0.269	146	0.199	0.238	120
LCS-141106-2	0.184	0.193	105	0.199	0.230	115
LCS-141106-3	0.184	0.196	106	0.199	0.229	115
Average ± %RSD	**119% ± 20%**			**117% ± 2.5%**		
LCS-141106-4	9.21	9.07	98.5	9.94	11.1	111
LCS-141106-5	9.21	8.97	97.4	9.94	11.1	112
LCS-141106-6	9.21	9.16	99.5	9.94	11.6	116
Average ± %RSD	**98.5% ± 1.1%**			**113% ± 2.3%**		
LCS-141106-7	73.2	64.6	88.3	79.0	>ULOQ	NA
LCS-141106-8	73.2	68.3	93.2	79.0	>ULOQ	NA
LCS-141106-9	73.2	67.6	92.4	79.0	>ULOQ	NA
Average ± %RSD	**91.3% ± 2.9%**			**NA (1)**		

LLOQ = Lower Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the acceptable calibration range.
(2) LCS calculated as an outlier by Dixon's Q-test. Not included in statistical calculations.
(3) LCSs did not meet ETS-8-042 method acceptance criteria of 100 ± 30%.

Table 8 continued. Laboratory Control Spike Results.

ETS-8-044.1 External Calibration Analyzed 11/12/14	PFBS			PFHS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-141112-1	1.99	2.03	102	1.99	2.02	102
LCS-141112-2	1.99	1.99	100	1.99	2.01	101
LCS-141112-3	1.99	2.04	102	1.99	2.08	105
Average ± %RSD	**101% ± 1.1%**			**103% ± 2.0%**		
LCS-141112-4	9.98	10.4	104	9.98	10.3	103
LCS-141112-5	9.98	10.2	102	9.98	10.2	102
LCS-141112-6	9.98	10.1	102	9.98	10.2	103
Average ± %RSD	**103% ± 1.1%**			**103% ± 0.56%**		
LCS-141112-7	79.4	>ULOQ	NA	79.4	>ULOQ	NA
LCS-141112-8	79.4	>ULOQ	NA	79.4	>ULOQ	NA
LCS-141112-9	79.4	>ULOQ	NA	79.4	>ULOQ	NA
Average ± %RSD	**NA (1)**			**NA (1)**		

ETS-8-044.1 External Calibration Analyzed 11/12/14	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-141112-1	1.85	1.75	94.5	1.90	1.95	102
LCS-141112-2	1.85	1.75	94.6	1.90	1.94	102
LCS-141112-3	1.85	1.81	97.7	1.90	1.97	104
Average ± %RSD	**95.6% ± 1.9%**			**103% ± 1.1%**		
LCS-141112-4	9.25	9.03	97.6	9.54	10.6	111
LCS-141112-5	9.25	8.77	94.9	9.54	9.57	100
LCS-141112-6	9.25	8.86	95.7	9.54	9.41	98.7
Average ± %RSD	**96.1% ± 1.4%**			**103% ± 6.5%**		
LCS-141112-7	73.6	69.3	94.2			
LCS-141112-8	73.6	69.6	94.5			
LCS-141112-9	73.6	68.5	93.1			
Average ± %RSD	**93.9% ± 0.78%**					

LLOQ = Lower Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the acceptable calibration range.
(2) LCS calculated as an outlier by Dixon's Q-test. Not included in statistical calculations.
(3) LCSs did not meet ETS-8-042 method acceptance criteria of 100 ± 30%.

Table 8 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal Calibration Analyzed 11/18/14	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-141118-1	0.190	0.160	84.3	0.199	0.193	97.1
LCS-141118-2	0.190	0.167	87.8	0.199	0.201	101
LCS-141118-3	0.190	0.161	85.0	0.199	0.194	97.6
Average ± %RSD	**85.7% ± 2.2%**			**98.6% ± 2.2%**		
LCS-141118-4	9.52	10.3	108	9.94	10.9	110
LCS-141118-5	9.52	10.2	107	9.94	10.9	110
LCS-141118-6	9.52	10.4	110	9.94	11.2	113
Average ± %RSD	**108% ± 1.4%**			**111% ± 1.6%**		
LCS-141118-7	38.0	34.0	89.4	39.6	36.4	92.0
LCS-141118-8	38.0	34.5	90.8	39.6	36.7	92.7
LCS-141118-9	38.0	34.8	91.7	39.6	37.1	93.7
Average ± %RSD	**90.6% ± 1.3%**			**92.8% ± 0.92%**		

ETS-8-044.1 Internal Calibration Analyzed 11/18/14	PFOS (Linear + Branched)			PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-141118-1	0.184	<LLOQ	NA	0.199	0.209	105
LCS-141118-2	0.184	<LLOQ	NA	0.199	0.208	105
LCS-141118-3	0.184	<LLOQ	NA	0.199	0.209	105
Average ± %RSD	**NA (1)**			**105% ± 0.0%**		
LCS-141118-4	9.21	9.68	105	9.94	11.4	115
LCS-141118-5	9.21	9.58	104	9.94	11.2	113
LCS-141118-6	9.21	9.61	104	9.94	11.3	114
Average ± %RSD	**104% ± 0.55%**			**114% ± 0.88%**		
LCS-141118-7	36.7	33.6	91.5	39.6	41.1	104
LCS-141118-8	36.7	33.0	89.8	39.6	40.5	102
LCS-141118-9	36.7	32.7	89.0	39.6	40.2	102
Average ± %RSD	**90.1% ± 1.4%**			**103% ± 1.1%**		

LLOQ = Lower Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the acceptable calibration range.
(2) LCS calculated as an outlier by Dixon's Q-test. Not included in statistical calculations.
(3) LCSs did not meet ETS-8-042 method acceptance criteria of 100 ± 30%.

Table 8 continued. Laboratory Control Spike Results.

ETS-8-042.0 Internal Calibration Analyzed 11/14/14	PFOA (Linear + Branched)			PFHS		
Lab ID	**Spiked Concentration (ng)**	**Calculated Concentration (ng)**	**%Recovery**	**Spiked Concentration (ng)**	**Calculated Concentration (ng)**	**%Recovery**
LCS-141113-1-01	1.44	2.81	195 (2)	1.50	2.20	147
LCS-141113-1-02	1.44	1.67	116	1.50	1.92	128
LCS-141113-1-03	1.44	1.39	96.8	1.50	1.90	126
Average ± %RSD	**106% ± 13%**			**134% ± 8.7% (3)**		
LCS-141113-1-04	14.4	14.1	98.1	15.0	13.2	87.7
LCS-141113-1-05	14.4	10.3	71.4	15.0	10.5	69.8
LCS-141113-1-06	14.4	14.0	97.4	15.0	14.9	99.5
Average ± %RSD	**89.0% ± 17%**			**85.7% ± 17%**		
LCS-141113-1-07	144	87.5	60.7	150	99.5	66.4
LCS-141113-1-08	144	159	110	150	165	110
LCS-141113-1-09	144	130	90.1	150	137	91.4
Average ± %RSD	**86.9% ± 29%**			**89.3% ± 25%**		

ETS-8-042.0 Internal Calibration Analyzed 11/14/14	PFOS (Linear + Branched)			PFOSA		
Lab ID	**Spiked Concentration (ng)**	**Calculated Concentration (ng)**	**%Recovery**	**Spiked Concentration (ng)**	**Calculated Concentration (ng)**	**%Recovery**
LCS-141113-1-01	1.39	2.50	180	1.50	2.21	147
LCS-141113-1-02	1.39	1.73	124	1.50	1.83	122
LCS-141113-1-03	1.39	1.43	103	1.50	1.60	107
Average ± %RSD	**136% ± 29% (3)**			**125% ± 16%**		
LCS-141113-1-04	13.9	12.0	86.1	15.0	12.5	83.2
LCS-141113-1-05	13.9	10.0	72.1	15.0	10.7	71.2
LCS-141113-1-06	13.9	13.7	98.5	15.0	14.8	98.6
Average ± %RSD	**85.6% ± 15%**			**84.3% ± 16%**		
LCS-141113-1-07	139	91.7	66.0	150	98.2	65.5
LCS-141113-1-08	139	153	110	150	163	109
LCS-141113-1-09	139	125	89.8	150	134	89.6
Average ± %RSD	**88.6% ± 25%**			**88.0% ± 25%**		

LLOQ = Lower Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the acceptable calibration range.
(2) LCS calculated as an outlier by Dixon's Q-test. Not included in statistical calculations.
(3) LCSs did not meet ETS-8-042 method acceptance criteria of 100 ± 30%.

Table 8 continued. Laboratory Control Spike Results.

ETS-8-042.0 Internal Calibration Analyzed 11/14/14	$^{13}C_4$-PFOA			$^{13}C_4$-PFOS		
Lab ID	**Spiked Concentration (ng)**	**Calculated Concentration (ng)**	**%Recovery**	**Spiked Concentration (ng)**	**Calculated Concentration (ng)**	**%Recovery**
LCS-141113-1-01	5.00	5.60	112	4.78	4.88	102
LCS-141113-1-02	5.00	5.07	101	4.78	4.15	86.8
LCS-141113-1-03	5.00	5.19	104	4.78	5.00	105
LCS-141113-1-04	5.00	4.66	93.2	4.78	4.31	90.2
LCS-141113-1-05	5.00	2.42	48.4 (3)	4.78	2.62	54.8 (3)
LCS-141113-1-06	5.00	4.76	95.2	4.78	4.59	96.0
LCS-141113-1-07	5.00	2.95	59.0 (3)	4.78	2.92	61.1 (3)
LCS-141113-1-08	5.00	4.45	89.0	4.78	3.88	81.2
LCS-141113-1-09	5.00	4.04	80.8	4.78	3.52	73.6
Average ± %RSD	**87.0% ± 24%**			**83.4 ± 21%**		

LLOQ = Lower Limit of Quantitation
ULOQ = Upper Limit of Quantitation
(1) LCSs spiked outside the acceptable calibration range.
(2) LCS calculated as an outlier by Dixon's Q-test. Not included in statistical calculations.
(3) LCSs did not meet ETS-8-042 method acceptance criteria of 100 ± 30%.

3.7 Analytical Method Uncertainty

Water Analysis: 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-12-012.2. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.1, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

Table 9. Water Analysis Analytical Method Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	8.65	±17%
PFBS	External	3.37	±6.7%
PFHS	External	5.66	±11%
PFOS	External	9.39	±19%
PFOSA	External	10.8	±22%
PFOA	Internal	11.0	±22%
PFHS	Internal	6.23	±12%
PFOS	Internal	11.8	±24%
PFOSA	Internal	9.08	±18%

Sludge Analysis: Laboratory control samples (LCS) were prepared and analyzed with the sludge analysis batch and the statistical evaluation of the LCS recovery results were used to estimate the analytical uncertainty. Since there are significantly less than 30 samples for each set of LCS data, the degrees of freedom (*n*-1) was used to determine *k* for estimating the method uncertainty. Value for *k* was taken from "G104-A2LA Guide for Estimation of Measurement Uncertainty in Testing, July 2002; Appendix 2 (Students t-distribution table)" for a *p* of 95.45%. The method uncertainty about the mean is calculated by multiplying the standard deviation by the *k value*, which corresponds to a confidence interval of 95%.

Table 10. Sludge Analysis Analytical Method Uncertainty.

Analyte	Degrees of Freedom (n-1)	Standard Deviation (%)	Method Uncertainty
PFOA	7	18	±45%
PFHS	8	27	±65%
PFOS	8	34	±80%
PFOSA	8	26	±61%

3.8 Field Matrix Spikes (FMS)

A target analyte field matrix spike sample was collected at each sampling point 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. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, each diluted sample was fortified with stable isotope surrogate recovery spikes of $^{13}C_4$-PFOA and $^{13}C_4$-PFOS, which were added at a nominal concentration of 1.0 ng/mL to following sample collection. The $^{13}C_4$- PFOA was selected to represent the C8 perfluorocarboxylic acid. The $^{13}C_4$-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids and PFOSA. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

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

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

Table 11. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
P114bis, P116	FMS	9.99	10.0	10.0	9.99	10.0
3M Pond (1)	FMS	9.04	9.05	9.05	9.04	9.05
Blokkersdijkvijver – standard (1)	FMS	8.22	8.23	8.23	8.22	8.23
L4	FMS	20.0	20.0	20.0	20.0	20.0
12, 5	FMS	99.9	100	100	99.9	10.0
13 (1)	FMS	90.4	90.5	90.5	90.4	9.05
Bemalingsstation (1)	FMS	89.2	89.3	89.3	89.2	8.93
P321	FMS	2000	10.0	1990	2000	10.0
PP02, PP05, PP06, PP07, PP08	FMS	996	100	993	996	100
PP04, PP09, PP10	FMS	1990	100	1990	1990	100
Trip Blank	Low	9.99	10.0	10.0	9.99	10.0
	Mid	99.9	100	100	99.9	10.0
	High	1990	100	1990	1990	100

(1) Concentration of FMS was adjusted due to the sample bottle being overfilled by more than 10% at the time of sample collection.

3.9 Lab Matrix Spikes (LMS)

Water Samples:
Due to the non-compliant field matrix spike for PFOS for location 3M Pond, a lab matrix spike was generated by adding a measured volume of standard spiking solution with the target analytes to an aliquot of the initial sample. Lab matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Lab 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 lab matrix spike was prepared for PFOS at 4.61 ng/mL for the 3M Pond location. The lab matrix spike result is presented in section 4 of the report.

Sludge Samples:
A low-level and high-level laboratory matrix spike (LMS) was prepared for each sampling location by fortifying with a known quantity of each target analyte in effort to achieve a fortification concentration that was within 0.5-times and 20-times the determined endogenous analyte level. For locations Manhole 1 and Manhole 2, only a single LMS spike level was prepared for PFOA, PFHS, and PFOSA. For PFOS, a low-level and high-level LMS was prepared for each location, however; only the most appropriate LMS was used to evaluate recovery and data accuracy. Lab 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 lab matrix spike results for the sludge samples are shown in **Tables 33-36**.

In addition to target analyte lab matrix spikes, each sample was fortified with stable isotope surrogate recovery spikes of $^{13}C_4$-PFOA and $^{13}C_4$-PFOS, which were added at a nominal concentration of 5.0 ng (nominal). The $^{13}C_4$- PFOA was selected to represent the C8 perfluorocarboxylic acid. The $^{13}C_4$-labeled PFOS was selected to represent the 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 for the sludge samples are included in **Tables 33-36**.

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

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

3.10 Percent Moisture Determination (Sludge)

Percent moisture data were used for calculating results on a dry weight basis and determined from separate sample aliquots than those used for analysis. The percent moisture was determined in duplicate for each sample. In summary, each sample aliquot is accurately weighed to determine the "wet" weight. Then the aliquot is dried at >100°C until no further loss in mass occurs (assumed loss due to water evaporation), and then the dried weight is accurately determined. The average percent moisture and RPD of the duplicate determinations for each is reported in **Table 12**. The percent moisture was calculated by the following equation:

$$\text{Percent Moisture} = \frac{(\text{Original Soil Wet Mass} - \text{Dried Soil Mass})}{\text{Original Soil Wet Mass}} * 100\%$$

Table 12. Percent Moisture

LIMS ID	Sample ID	Average Percent Moisture (%, w/w)	RPD
ISO14-0028-062	Manhole 01	51.3	0.0%
ISO14-0028-063	Manhole 02	58.1	0.86%
ISO14-0028-064	Manhole 03	72.5	5.5%
ISO14-0028-065	Trip Blank; Soil	2.3	4.4%

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.

For those analytes where the field matrix spike level was not appropriate as compared to the sample concentration, the surrogate recovery standards were used to assess method accuracy. All surrogate recovery standards met method acceptance criteria except for select sludge samples. The following sampling locations had field QC samples outside method acceptance criteria:

3M Pond: The FMS recovery for PFOS was 209% and the LMS recovery was 146%. The method uncertainty has been expanded for PFOS to ±46%, based on the recovery of the LMS sample.

Blokkersdijkvijver std: Sample/sample duplicate RPD was 103% for PFOA, 102% for PFHS, 106% for PFOS, and 80% for PFOSA. Samples were re-prepared and analyzed for all analytes, confirming the results from the initial analysis. The results from the initial analysis are reported.

12: Sample/sample duplicate RPD was 25% for PFHS and 108% for PFOSA. Samples were re-prepared and analyzed for PFOSA, confirming the results from the initial analysis. The results from the initial analysis are reported.

PP01: Sample/sample duplicate RPD was 50% for PFOSA.

PP04: Sample/sample duplicate RPD was 21% for PFOSA.

PP05: Surrogate recovery standards had recoveries greater than 100±30% for the sample duplicate. The average SRS recovery for the sample set was within acceptance criteria; therefore, the method uncertainty was not expanded.

PP06: The FMS recovery for PFOSA was 63.6%. The method uncertainty has been expanded for PFOSA to ±36%, based on the recovery of the FMS sample.

Trip Blank: The FMS High recovery for PFOSA was 67.6%.

Sludge Samples: Due to the number of high RPDs that were seen, the samples were re-prepared and analyzed using an aliquot of the dried sludge. No improvements were seen in the RPD values or LMS recoveries. The initial sample results are reported.

Manhole 01: The LMS Low sample recovery was 156% for PFOA and 146% for PFOSA. The LMS High sample recovery for PFOA was 93.0% and 87.1% for PFOSA. Since the low level LMS is the most appropriate spike level to assess sample recovery, the method uncertainty will be based on the LMS Low recovery. The method uncertainty has been expanded for PFOA to ±56% based on the %bias. Since the %bias for PFOSA (±46%) is lower than the analytical method uncertainty for PFOSA (±61%), the method uncertainty will not be expanded for PFOSA for the sample location. Also, while no acceptance criteria is set in ETS-8-042 for sample/sample duplicate RPDs, the RPDs were greater than 50% for all analytes and were flagged as such.

Manhole 02: Similar to the Manhole 01 location, the sample/sample duplicate RPDs were flagged as greater than 50% for PFOA, PFOS, and PFOSA.

Manhole 03: The LMS Low sample recovery was 68.6% for PFOA. Since the %bias (±31%) is lower than the analytical method uncertainty (±45%), the method uncertainty will not be expanded for the sample location. The SRS recoveries for the sample set were below 70%, but within the overall method uncertainty. The method uncertainty was not expanded further.

Table 13. Location ID: P321

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-001	P321; Sample	3170	NA	890	NA
ISO14-0028-002	P321; Sample Dup	3110	NA	893	NA
ISO14-0028-003	P321; FMS	4810	83.5	2890	100
Average Concentration (ng/mL) ± %RPD		**3140 ng/mL ± 1.9%**		**892 ng/mL ± 0.34%**	

		PFOS [1]		PFOSA [2]		$^{13}C_4$-PFOA [1]	$^{13}C_4$-PFOS [1]
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-001	P321; Sample	3180	NA	0.465	NA	114	121
ISO14-0028-002	P321; Sample Dup	3240	NA	0.411	NA	112	117
ISO14-0028-003	P321; FMS	5490	114	11.6	112	114	118
Average Concentration (ng/mL) ± %RPD		**3210 ng/mL ± 1.9%**		**0.438 ng/mL ± 12%**		**114% ± 1.0%**	**119% ± 1.8%**

NA = Not Applicable
(1) Samples analyzed using external calibration.
(2) Samples analyzed using internal calibration.

Table 14. Location ID: Blokkersdijkvijver std

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-004	Blokkersdijkvijver std; Sample	3.89	NA	3.68	NA
ISO14-0028-005	Blokkersdijkvijver std;Sample Dup	1.24	NA	1.19	NA
ISO14-0028-006	Blokkersdijkvijver std;FMS	8.61	73.5	8.83	77.7
Average Concentration (ng/mL) ± %RPD		**2.57 ng/mL ± 103% [1]**		**2.44 ng/mL ± 102% [1]**	

		PFOS		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-004	Blokkersdijkvijver std; Sample	41.1	NA	1.02	NA	119	122
ISO14-0028-005	Blokkersdijkvijver std;Sample Dup	12.6	NA	0.439	NA	123	121
ISO14-0028-006	Blokkersdijkvijver std;FMS	23.4	NC	6.81	73.9	121	124
Average Concentration (ng/mL) ± %RPD		**26.9 ng/mL ± 106% [1]**		**0.730 ng/mL ± 80% [21**		**121% ± 1.7%**	**123% ± 1.3%**

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

Table 15. Location ID: 3M Pond

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-007	3M pond; Sample	1.09	NA	0.541	NA
ISO14-0028-008	3M pond; Sample Dup	1.10	NA	0.562	NA
ISO14-0028-009	3M pond; FMS	10.0	98.5	10.5	110
Average Concentration (ng/mL) ± %RPD		**1.10 ng/mL ± 0.91%**		**0.552 ng/mL ± 3.8%**	

		PFOS [2]		PFOSA [2]		$^{13}C_4$-PFOA [1]	$^{13}C_4$-PFOS [1]
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-007	3M pond; Sample	3.31	NA	0.0476	NA	118	121
ISO14-0028-008	3M pond; Sample Dup	3.05	NA	0.0414	NA	118	120
ISO14-0028-009	3M pond; FMS	22.1	209 [3]	10.3	113	116	119
ISO14-0028-007; LMS	3M pond; Sample LMS	9.92	146 [4]	NA [5]	NA [5]	NA [5]	NA [5]
Average Concentration (ng/mL) ± %RPD		**3.18 ng/mL ± 8.2%**		**0.0445 ng/mL ± 14%**		**118% ± 0.98%**	**120% ± 0.87%**

NA = Not Applicable
(1) Samples analyzed using external calibration.
(2) Samples analyzed using internal calibration.
(3) FMS recovery did not meet acceptance criteria of 100 ± 30%.
(4) LMS recovery did not meet acceptance criteria of 100 ± 30%. The method uncertainty has been expanded to ± 46% for PFOS.
(5) LMS sample only analyzed for PFOS.

Table 16. Location ID: L4

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-010	L4; Sample	3.13	NA	2.39	NA
ISO14-0028-011	L4; Sample Dup	3.15	NA	2.38	NA
ISO14-0028-012	L4; FMS	21.0	89.4	21.1	93.6
Average Concentration (ng/mL) ± %RPD		**3.14 ng/mL ± 0.64%**		**2.39 ng/mL ± 0.42%**	

		PFOS		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-010	L4; Sample	30.4	NA	1.04	NA	121	124
ISO14-0028-011	L4; Sample Dup	29.3	NA	1.05	NA	120	121
ISO14-0028-012	L4; FMS	46.5	83.3	16.6	77.8	117	119
Average Concentration (ng/mL) ± %RPD		**29.9 ng/mL ± 3.7%**		**1.05 ng/mL ± 0.96%**		**120% ± 1.7%**	**122% ± 2.2%**

NA = Not Applicable
Samples analyzed using external calibration.

Table 17. Location ID: P114bis

		PFOA [1]		PFHS [1]	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0028-013	P114bis; Sample	4.62	NA	1.63	NA
ISO14-0028-014	P114bis; Sample Dup	4.43	NA	1.62	NA
ISO14-0028-015	P114bis; FMS	14.6	101	11.9	103
Average Concentration (ng/mL) ± %RPD		**4.53 ng/mL ± 4.2%**		**1.63 ng/mL ± 0.62%**	

		PFOS [2]		PFOSA [2]		$^{13}C_4$-PFOA [1]	$^{13}C_4$-PFOS [1]
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0028-013	P114bis; Sample	13.7	NA	0.0306	NA	127	128
ISO14-0028-014	P114bis; Sample Dup	13.0	NA	0.0261	NA	118	122
ISO14-0028-015	P114bis; FMS	22.7	93.6	11.1	111	121	121
Average Concentration (ng/mL) ± %RPD		**13.4 ng/mL ± 5.2%**		**0.0284 ng/mL ± 16%**		**122% ± 3.8%**	**124% ± 2.7%**

NA = Not Applicable
(1) Samples analyzed using external calibration.
(2) Samples analyzed using internal calibration.

Table 18. Location ID: P116

		PFOA [1]		PFHS [1]	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0028-016	P116; Sample	1.13	NA	0.626	NA
ISO14-0028-017	P116; Sample Dup	1.05	NA	0.612	NA
ISO14-0028-018	P116; FMS	11.4	103	10.8	102
Average Concentration (ng/mL) ± %RPD		**1.09 ng/mL ± 7.3%**		**0.619 ng/mL ± 2.3%**	

		PFOS [2]		PFOSA [2]		$^{13}C_4$-PFOA [1]	$^{13}C_4$-PFOS [1]
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0028-016	P116; Sample	8.54	NA	0.0651	NA	122	122
ISO14-0028-017	P116; Sample Dup	8.99	NA	0.0695	NA	123	122
ISO14-0028-018	P116; FMS	18.7	99.4	11.2	111	122	120
Average Concentration (ng/mL) ± %RPD		**8.77 ng/mL ± 5.1%**		**0.0673 ng/mL ± 6.5%**		**123% ± 0.47%**	**122% ± 0.99%**

NA = Not Applicable
(1) Samples analyzed using external calibration.
(2) Samples analyzed using internal calibration.

Table 19. Location ID: 12

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-046	12; Sample	100	NA	33.3	NA
ISO14-0028-047	12; Sample Dup	94.0	NA	26.0	NA
ISO14-0028-048	12; FMS	178	81.1	116	86.4
Average Concentration (ng/mL) ± %RPD		**97.0 ng/mL ± 6.2%**		**29.7 ng/mL ± 25% [2]**	

		PFOS [1]		PFOSA [3]		$^{13}C_4$-PFOA [1]	$^{13}C_4$-PFOS [1]
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-046	12; Sample	249	NA	0.985	NA	119	118
ISO14-0028-047	12; Sample Dup	244	NA	0.296	NA	117	120
ISO14-0028-048	12; FMS	336	NC	11.7	111	118	119
Average Concentration (ng/mL) ± %RPD		**247 ng/mL ± 2.0%**		**0.641 ng/mL ± 108% [2]**		**118% ± 0.85%**	**119% ± 0.88%**

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Samples analyzed using external calibration.
(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
(3) Samples analyzed using internal calibration.

Table 20. Location ID: 13

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-049	13; Sample	89.3	NA	25.9	NA
ISO14-0028-050	13; Sample Dup	87.6	NA	24.9	NA
ISO14-0028-051	13; FMS	175	95.7	121	106
Average Concentration (ng/mL) ± %RPD		**88.5 ng/mL ± 1.9%**		**25.4 ng/mL ± 3.9%**	

		PFOS [1]		PFOSA [2]		$^{13}C_4$-PFOA [1]	$^{13}C_4$-PFOS [1]
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-049	13; Sample	199	NA	0.303	NA	117	123
ISO14-0028-050	13; Sample Dup	202	NA	0.278	NA	119	119
ISO14-0028-051	13; FMS	259	NC	10.3	111	118	120
Average Concentration (ng/mL) ± %RPD		**201 ng/mL ± 1.5%**		**0.291 ng/mL ± 8.6%**		**118% ± 0.85%**	**121% ± 1.8%**

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Samples analyzed using external calibration.
(2) Samples analyzed using internal calibration.

Table 21. Location ID: 5

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-052	5; Sample	30.5	NA	22.9	NA
ISO14-0028-053	5; Sample Dup	28.4	NA	21.7	NA
ISO14-0028-054	5; FMS	118	88.6	117	94.7
Average Concentration (ng/mL) ± %RPD		**29.5 ng/mL ± 7.1%**		**22.3 ng/mL ± 5.4%**	

		PFOS [1]		PFOSA [2]		$^{13}C_4$-PFOA [1]	$^{13}C_4$-PFOS [1]
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-052	5; Sample	34.5	NA	0.143	NA	121	122
ISO14-0028-053	5; Sample Dup	38.8	NA	0.152	NA	118	121
ISO14-0028-054	5; FMS	121	84.4	11.3	112	118	120
Average Concentration (ng/mL) ± %RPD		**36.7 ng/mL ± 12%**		**0.148 ng/mL ± 6.1%**		**119% ± 1.5%**	**121% ± 0.86%**

NA = Not Applicable
(1) Samples analyzed using external calibration.
(2) Samples analyzed using internal calibration.

Table 22. Location ID: Bemlingsstation

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-055	Bemalingsstation; Sample	29.0	NA	21.7	NA
ISO14-0028-056	Bemalingsstation; Sample Dup	28.9	NA	21.9	NA
ISO14-0028-057	Bemalingsstation; FMS	115	96.5	114	103
Average Concentration (ng/mL) ± %RPD		**29.0 ng/mL ± 0.35%**		**21.8 ng/mL ± 0.92%**	

		PFOS [1]		PFOSA [2]		$^{13}C_4$-PFOA [1]	$^{13}C_4$-PFOS [1]
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-055	Bemalingsstation; Sample	53.7	NA	0.190	NA	119	123
ISO14-0028-056	Bemalingsstation; Sample Dup	53.7	NA	0.165	NA	120	124
ISO14-0028-057	Bemalingsstation; FMS	134	90.0	10.1	111	122	126
Average Concentration (ng/mL) ± %RPD		**53.7 ng/mL ± 0.0%**		**0.178 ng/mL ± 14%**		**121% ± 1.3%**	**124% ± 0.84%**

NA = Not Applicable
(1) Samples analyzed using external calibration.
(2) Samples analyzed using internal calibration.

Table 23. Location ID: PP01

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-019	PP01; Sample	86.8	NA	90.2	NA	19000	NA
ISO14-0028-020	PP01; Sample Dup	92.1	NA	96.3	NA	22100	NA
ISO14-0028-021	PP01; FMS	177	87.6	188	94.8	NA (1)	NA (1)
Average Concentration (ng/mL) ± %RPD		**89.5 ng/mL ± 5.9%**		**93.3 ng/mL ± 6.5%**		**20600 ng/mL ± 15%**	

		PFOS		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-019	PP01; Sample	6440	NA	29.4	NA	127	122
ISO14-0028-020	PP01; Sample Dup	7690	NA	48.8	NA	119	113
ISO14-0028-021	PP01; FMS	6710	NC	112	72.9	128	122
Average Concentration (ng/mL) ± %RPD		**7070 ng/mL ± 18%**		**39.1 ng/mL ± 50% (2)**		**125% ± 4.0%**	**119% ± 4.6%**

NA = Not Applicable
Samples analyzed using external calibration.
(1) Sample concentration exceeded calibration range; sample was not re-analyzed since field matrix spike level was not appropriate.
(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 24. Location ID: PP02

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-022	PP02; Sample	586	NA	27.1	NA	2640	NA
ISO14-0028-023	PP02; Sample Dup	585	NA	26.7	NA	2640	NA
ISO14-0028-024	PP02; FMS	1480	89.8	121	94.1	3710	NC
Average Concentration (ng/mL) ± %RPD		**586 ng/mL ± 0.17%**		**26.9 ng/mL ± 1.5%**		**2640 ng/mL ± 0.0%**	

		PFOS		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS)
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-022	PP02; Sample	20700	NA	23.3	NA	122	94.9
ISO14-0028-023	PP02; Sample Dup	21400	NA	28.4	NA	124	93.4
ISO14-0028-024	PP02; FMS	NA (1)	NA (1)	131	105	123	NA (1)
Average Concentration (ng/mL) ± %RPD		**21100 ng/mL ± 3.3%**		**25.9 ng/mL ± 20%**		**123% ± 0.81%**	**94.1% ± 1.1%**

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
Samples analyzed using external calibration.
(1) Sample concentration exceeded calibration range; sample was not re-analyzed since field matrix spike level was not appropriate.

Table 25. Location ID: PP04

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-025	PP04; Sample	1520	NA	2580	NA	758	NA
ISO14-0028-026	PP04; Sample Dup	1560	NA	2600	NA	773	NA
ISO14-0028-027	PP04; FMS	3250	85.9	2690	NC	2690	96.7
Average Concentration (ng/mL) ± %RPD		**1540 ng/mL ± 2.6%**		**2590 ng/mL ± 0.77%**		**766 ng/mL ± 2.0%**	

		PFOS		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-025	PP04; Sample	9460	NA	46.6	NA	120	99.1
ISO14-0028-026	PP04; Sample Dup	8780	NA	37.8	NA	101	103
ISO14-0028-027	PP04; FMS	NA (1)	NA (1)	130	87.8	116	NA (1)
Average Concentration (ng/mL) ± %RPD		**9120 ng/mL ± 7.5%**		**42.2 ng/mL ± 21% (2)**		**113% ± 8.9%**	**101% ± 3.1%**

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
Samples analyzed using external calibration.
(1) Sample concentration exceeded calibration range; sample was not re-analyzed since field matrix spike level was not appropriate.
(2) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 26. Location ID: PP05

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-028	PP05; Sample	1440	NA	17200	NA	6510	NA
ISO14-0028-029	PP05; Sample Dup	1420	NA	17600	NA	6770	NA
ISO14-0028-030	PP05; FMS	2250	82.3	NA (1)	NA (1)	NA (1)	NA (21
Average Concentration (ng/mL) ± %RPD		**1430 ng/mL ± 1.4%**		**17400 ng/mL ± 2.3%**		**6640 ng/mL ± 3.9%**	

		PFOS		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-028	PP05; Sample	214	NA	25.3	NA	123	124
ISO14-0028-029	PP05; Sample Dup	187	NA	23.8	NA	131 (2)	133 (2)
ISO14-0028-030	PP05; FMS	996	79.8	104	79.5	120	122
Average Concentration (ng/mL) ± %RPD		**201 ng/mL ± 13%**		**24.6 ng/mL ± 6.1%**		**125% ± 4.6%**	**127% ± 4.4%**

NA = Not Applicable
Samples analyzed using external calibration.
(1) Sample concentration exceeded calibration range; sample was not re-analyzed since field matrix spike level was not appropriate.
(2) Surrogate recovery standards did not meet acceptance criteria of 100 ± 30%.

Table 27. Location ID: PP06

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-031	PP06; Sample	159	NA	24.7	NA	46.0	NA
ISO14-0028-032	PP06; Sample Dup	157	NA	20.8	NA	44.3	NA
ISO14-0028-033	PP06; FMS	1070	91.5	117	94.3	1010	97.2
Average Concentration (ng/mL) ± %RPD		**158 ng/mL ± 1.3%**		**22.8 ng/mL ± 17%**		**45.2 ng/mL ± 3.8%**	

		PFOS		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-031	PP06; Sample	2200	NA	41.2	NA	122	126
ISO14-0028-032	PP06; Sample Dup	2210	NA	35.7	NA	122	123
ISO14-0028-033	PP06; FMS	3190	NC	102	63.6 (1)	122	126
Average Concentration (ng/mL) ± %RPD		**2210 ng/mL ± 0.45%**		**38.5 ng/mL ± 14%**		**122% ± 0.0%**	**125% ± 0.97%**

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
Samples analyzed using external calibration.
(1) FMS recovery did not meet acceptance criteria of 100 ± 30%. The method uncertainty has been expanded to ± 36% for PFOSA.

Table 28. Location ID: PP07

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-034	PP07; Sample	412	NA	127	NA	132	NA
ISO14-0028-035	PP07; Sample Dup	431	NA	126	NA	132	NA
ISO14-0028-036	PP07; FMS	1340	92.2	225	98.5	1090	96.5
Average Concentration (ng/mL) ± %RPD		**422 ng/mL ± 4.5%**		**127 ng/mL ± 0.79%**		**132 ng/mL ± 0.0%**	

		PFOS		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-034	PP07; Sample	2950	NA	53.7	NA	122	123
ISO14-0028-035	PP07; Sample Dup	2910	NA	50.4	NA	126	127
ISO14-0028-036	PP07; FMS	3890	NC	132	80.0	124	122
Average Concentration (ng/mL) ± %RPD		**2930 ng/mL ± 1.4%**		**52.1 ng/mL ± 6.3%**		**124% ± 1.6%**	**124% ± 1.8%**

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
Samples analyzed using external calibration.

Table 29. Location ID: PP08

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-037	PP08; Sample	572	NA	34.7	NA	282	NA
ISO14-0028-038	PP08; Sample Dup	553	NA	34.1	NA	268	NA
ISO14-0028-039	PP08; FMS	1490	93.1	129	94.6	1250	98.2
Average Concentration (ng/mL) ± %RPD		**563 ng/mL ± 3.4%**		**34.4 ng/mL ± 1.7%**		**275 ng/mL ± 5.1%**	

		PFOS		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-037	PP08; Sample	8430	NA	47.3	NA	124	120
ISO14-0028-038	PP08; Sample Dup	7910	NA	45.3	NA	125	122
ISO14-0028-039	PP08; FMS	NA (1)	NA (1)	141	94.7	126	117
Average Concentration (ng/mL) ± %RPD		**8170 ng/mL ± 6.4%**		**46.3 ng/mL ± 4.3%**		**125% ± 0.80%**	**120% ± 2.2%**

NA = Not Applicable
Samples analyzed using external calibration.
(1) Sample concentration exceeded calibration range; sample was not re-analyzed since field matrix spike level was not appropriate.

Table 30. Location ID: PP09

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-040	PP09; Sample	743	NA	30.9	NA	438	NA
ISO14-0028-041	PP09; Sample Dup	747	NA	30.7	NA	441	NA
ISO14-0028-042	PP09; FMS	2590	92.7	130	99.2	2480	103
Average Concentration (ng/mL) ± %RPD		**745 ng/mL ± 0.54%**		**30.8 ng/mL ± 0.65%**		**440 ng/mL ± 0.68%**	

		PFOS		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-040	PP09; Sample	2440	NA	11.6	NA	124	126
ISO14-0028-041	PP09; Sample Dup	2430	NA	11.0	NA	119	119
ISO14-0028-042	PP09; FMS	4470	102	88.3	77.0	122	120
Average Concentration (ng/mL) ± %RPD		**2440 ng/mL ± 0.41%**		**11.3 ng/mL ± 5.3%**		**122% ± 2.1%**	**122% ± 2.8%**

NA = Not Applicable
Samples analyzed using external calibration.

Table 31. Location ID: PP10

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-043	PP10; Sample	663	NA	21.5	NA	201	NA
ISO14-0028-044	PP10; Sample Dup	659	NA	20.9	NA	204	NA
ISO14-0028-045	PP10; FMS	2510	92.9	116	94.8	2140	97.4
Average Concentration (ng/mL) ± %RPD		**661 ng/mL ± 0.61%**		**21.2 ng/mL ± 2.8%**		**203 ng/mL ± 1.5%**	

		PFOS		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-043	PP10; Sample	4000	NA	11.5	NA	120	121
ISO14-0028-044	PP10; Sample Dup	4000	NA	10.8	NA	118	117
ISO14-0028-045	PP10; FMS	6090	NC	82.9	71.8	116	113
Average Concentration (ng/mL) ± %RPD		**4000 ng/mL ± 0.0%**		**11.2 ng/mL ± 6.3%**		**118% ± 1.7%**	**117% ± 3.6%**

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
Samples analyzed using external calibration.

Table 32. Trip Blank (1)

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0028-058	Trip Blank; Sample	<0.0240 (2)	NA	<0.200	NA	<0.0250 (2)	NA
ISO14-0028-059	Trip Blank; FMS Low	9.41	94.2	10.1	101	10.0	99.9
ISO14-0028-060	Trip Blank; FMS Mid	94.9	95.0	101	101	101	101
ISO14-0028-061	Trip Blank; FMS High	1800	90.5	96.5	96.5	1950	98.0

		PFOS		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**%Recovery**	**%Recovery**
ISO14-0028-058	Trip Blank; Sample	<0.464	NA	<0.0250 (2)	NA	125	124
ISO14-0028-059	Trip Blank; FMS Low	9.45	94.6	7.19	71.9	124	127
ISO14-0028-060	Trip Blank; FMS Mid	96.4	96.5	7.37	73.7	128	129
ISO14-0028-061	Trip Blank; FMS High	1820	91.5	67.6	67.6 (3)	120	120

NA = Not Applicable
(1) Samples analyzed using external calibration unless noted otherwise.
(2) Samples analyzed using internal calibration.
(3) FMS recovery did not meet acceptance criteria of 100 ± 30%.

Table 33. PFOA Sludge Sample LMS Recoveries

PFOA results for Antwerp Manhole Sludge Samples						
3M LIMS ID	**Sample Description**	**Surrogate ($^{13}C_4$-PFOA) % Recovery**	**LMS Spike Concentration ng/g; dry weight**	**Measured PFOA Concentration (ng/g; dry weight)**	**LMS % Recovery**	**Average PFOA Concentration (ng/g; dry wt. and RPD)**
ISO14-0028-062	Manhole 01	84.8	NA	1100	NA	1710 ± 72% (3, 4)
ISO14-0028-062	Manhole 01 Duplicate	84.0	NA	2330	NA	
ISO14-0028-062	Manhole 01 LMS Low	84.0	3020	6430	156 (1)	
ISO14-0028-062	Manhole 01 LMS High	81.8	75900	72300	93.0 (2)	
ISO14-0028-063	Manhole 02	83.2	NA	5420	NA	7290 ± 51% (3)
ISO14-0028-063	Manhole 02 Duplicate	83.6	NA	9170	NA	
ISO14-0028-063	Manhole 02 LMS Low	83.4	18100	23300	88.7	
ISO14-0028-063	Manhole 02 LMS High	83.8	NA	4680	NC	
ISO14-0028-064	Manhole 03	68.8 (1)	NA	27100	NA	26800 ± 2.5%
ISO14-0028-064	Manhole 03 Duplicate	68.6 (1)	NA	26500	NA	
ISO14-0028-064	Manhole 03 LMS Low	67.4 (1)	61000	68600	68.6 (1)	
ISO14-0028-064	Manhole 03 LMS High	68.6 (1)	NA	29200	NC	
ISO14-0028-065	Trip Blank; Soil	91.2	NA	<0.435	NA	<0.434
ISO14-0028-065	Trip Blank; Soil Duplicate	86.2	NA	<0.433	NA	
ISO14-0028-065	Trip Blank; Soil LMS	84.2	0.911	1.06	116%	
Average SRS Recovery		80.2 %				
Standard Deviation		7.7%				
RSD		9.6 %				

NA = Not Applicable
NC = Not Calculated; Spike level did not include PFOA.
(1) LMS/SRS recovery does not meet acceptance criteria of 100 ± 30%. Analytical method uncertainty will be adjusted for those samples with LMS recoveries that exceeded the calculated overall uncertainty for the method.
(2) LMS spike level greater than 20 times the endogenous sample concentration.
(3) Sample/sample duplicate RPD ≤50%.
(4) The analytical method uncertainty is expanded for Manhole 1 to ± 56% based on the %bias of the most appropriate LMS.

Table 34. PFHS Sludge Sample LMS Recoveries

PFHS results for Antwerp Manhole Sludge Samples						
3M LIMS ID	**Sample Description**	**Surrogate ($^{13}C_4$-PFOS) % Recovery**	**LMS Spike Concentration ng/g; dry weight**	**Measured PFHS Concentration (ng/g; dry weight)**	**LMS % Recovery**	**Average PFHS Concentration (ng/g; dry wt. and RPD)**
ISO14-0028-062	Manhole 01	81.4	NA	251	NA	350 ± 57% (3)
ISO14-0028-062	Manhole 01 Duplicate	82.0	NA	449	NA	
ISO14-0028-062	Manhole 01 LMS Low	82.2	3020	3650	109	
ISO14-0028-062	Manhole 01 LMS High	82.0	75900	70500	92.4 (2)	
ISO14-0028-063	Manhole 02	80.5	NA	1280	NA	1590 ± 39%
ISO14-0028-063	Manhole 02 Duplicate	82.6	NA	1900	NA	
ISO14-0028-063	Manhole 02 LMS Low	84.3	18100	18800	95.2	
ISO14-0028-063	Manhole 02 LMS High	82.0	NA	1300	NC	
ISO14-0028-064	Manhole 03	67.6 (1)	NA	10500	NA	10800 ± 5.5%
ISO14-0028-064	Manhole 03 Duplicate	67.6 (1)	NA	11100	NA	
ISO14-0028-064	Manhole 03 LMS Low	68.4 (1)	61000	55800	73.8	
ISO14-0028-064	Manhole 03 LMS High	68.8 (1)	NA	11900	NC	
ISO14-0028-065	Trip Blank; Soil	94.6	NA	<0.0908	NA	<0.0906
ISO14-0028-065	Trip Blank; Soil Duplicate	86.6	NA	<0.0904	NA	
ISO14-0028-065	Trip Blank; Soil LMS	85.8	0.951	1.19	125	
Average SRS Recovery		79.8%				
Standard Deviation		8.0%				
RSD		10%				

NA = Not Applicable
NC = Not Calculated; Spike level did not include PFHS.
(1) SRS recovery does not meet acceptance criteria of 100 ± 30%.
(2) LMS spike level greater than 20 times the endogenous sample concentration.
(3) Sample/sample duplicate RPD ≤50%.

Table 35. PFOS Sludge Sample LMS Recoveries

PFOS results for Antwerp Manhole Sludge Samples						
3M LIMS ID	**Sample Description**	**Surrogate ($^{13}C_4$-PFOS) % Recovery**	**LMS Spike Concentration ng/g; dry weight**	**Measured PFOS Concentration (ng/g; dry weight)**	**LMS % Recovery**	**Average PFOS Concentration (ng/g; dry wt. and RPD)**
ISO14-0028-062	Manhole 01	81.4	NA	18900	NA	26900 ± 59% (2)
ISO14-0028-062	Manhole 01 Duplicate	82.0	NA	34900	NA	
ISO14-0028-062	Manhole 01 LMS Low	82.2	3020	54100	NC	
ISO14-0028-062	Manhole 01 LMS High	82.0	75900	95800	90.8	
ISO14-0028-063	Manhole 02	80.5	NA	127000	NA	191000 ± 66% (2)
ISO14-0028-063	Manhole 02 Duplicate	82.6	NA	254000	NA	
ISO14-0028-063	Manhole 02 LMS Low	84.3	18100	155000	NC	
ISO14-0028-063	Manhole 02 LMS High	82.0	663000	789000	90.3	
ISO14-0028-064	Manhole 03	67.6 (1)	NA	505000	NA	464000 ± 18%
ISO14-0028-064	Manhole 03 Duplicate	67.6 (1)	NA	424000	NA	
ISO14-0028-064	Manhole 03 LMS Low	68.4 (1)	61000	415000	NC	
ISO14-0028-064	Manhole 03 LMS High	68.8 (1)	1440000	1580000	77.4	
ISO14-0028-065	Trip Blank; Soil	94.6	NA	<0.168	NA	<0.168
ISO14-0028-065	Trip Blank; Soil Duplicate	86.6	NA	<0.167	NA	
ISO14-0028-065	Trip Blank; Soil LMS	85.8	0.882	1.08	123	
Average SRS Recovery		79.8%				
Standard Deviation		8.0%				
RSD		10%				

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) SRS recovery does not meet acceptance criteria of 100 ± 30%.
(2) Sample/sample duplicate RPD ≤50%.

Table 36. PFOSA Sludge Sample LMS Recoveries

PFOSA results for Antwerp Manhole Sludge Samples						
3M LIMS ID	**Sample Description**	**Surrogate ($^{13}C_4$-PFOS) % Recovery**	**LMS Spike Concentration ng/g; dry weight**	**Measured PFOSA Concentration (ng/g; dry weight)**	**LMS % Recovery**	**Average PFOSA Concentration (ng/g; dry wt. and RPD)**
ISO14-0028-062	Manhole 01	81.4	NA	1250	NA	1800 ± 61% (3)
ISO14-0028-062	Manhole 01 Duplicate	82.0	NA	2350	NA	
ISO14-0028-062	Manhole 01 LMS Low	82.2	3021	6220	146 (1)	
ISO14-0028-062	Manhole 01 LMS High	82.0	75900	67900	87.1 (2)	
ISO14-0028-063	Manhole 02	80.5	NA	3270	NA	5240 ± 75% (3)
ISO14-0028-063	Manhole 02 Duplicate	82.6	NA	7210	NA	
ISO14-0028-063	Manhole 02 LMS Low	84.3	18100	18400	73.0	
ISO14-0028-063	Manhole 02 LMS High	82.0	NA	2460	NC	
ISO14-0028-064	Manhole 03	67.6 (1)	NA	13100	NA	11600 ± 26%
ISO14-0028-064	Manhole 03 Duplicate	67.6 (1)	NA	10100	NA	
ISO14-0028-064	Manhole 03 LMS Low	68.4 (1)	61000	55800	72.5	
ISO14-0028-064	Manhole 03 LMS High	68.8 (1)	NA	10500	NC	
ISO14-0028-065	Trip Blank; Soil	94.6	NA	<0.0908	NA	<0.0906
ISO14-0028-065	Trip Blank; Soil Duplicate	86.6	NA	<0.0904	NA	
ISO14-0028-065	Trip Blank; Soil LMS	85.8	0.951	1.07	113	
Average SRS Recovery		79.8%				
Standard Deviation		8.0%				
RSD		10%				

NA = Not Applicable
NC = Not Calculated; Spike level did not include PFOSA.
(1) LMS/SRS recovery does not meet acceptance criteria of 100 ± 30%. Analytical method uncertainty will be adjusted for those samples with LMS recoveries that exceeded the calculated overall uncertainty for the method.
(2) LMS spike level greater than 20 times the endogenous sample concentration.
(3) Sample/sample duplicate RPD ≤50%.

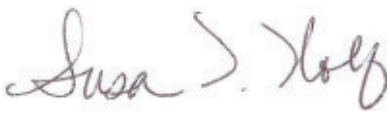
5 Conclusion

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 or lab 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-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis" for the water samples and method ETS-8-042.0 "Solvent Extraction and Isocratic LC/MS/MS Analysis of Soils for C4-C12 Perfluorinated Carboxylic Acids and Perfluorobutane Sulfonate, Perfluorohexane Sulfonate and Perfluorooctane Sulfonate" for the sludge samples. Analytical results are reported in Tables 1-2 and 13-36 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 Susan T. Wolf
DN: c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory - authenticated by LRA, [redacted] o=3M, cn=Susan T. Wolf
Reason: I have reviewed this document
Date: 2015.01.07 11:12:23 -06'00'

Susan T. Wolf, 3M Principal Analytical Investigator



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DN: c=US, st=MN, l=St. Paul, ou=Laboratory Director, ou=3M Environmental Laboratory - authenticated by LRA, [redacted]
o=3M, cn=William K. Reagen
Reason: I am approving this document
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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.



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DN: c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory - authenticated by LRA, [redacted] o=3M, cn=Daryl K. Peterson
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Final Report

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

January 2015 Sampling

Laboratory Request Number: ISO15-0001

Report Date – Date of Last Signature

Testing Laboratory

3M EHS Operations
3M Environmental Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

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

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

3M Environmental Laboratory

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

Analytical Report ISO15-0001

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at
3M Belgium
January 2015 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M Environmental Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Belgium facility. Samples were collected on January 26-28, 2015. Samples were returned to the 3M Environmental Laboratory on February 2, 2015 at ambient temperature for the analysis of Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO15-0001.

The 3M Environmental Laboratory prepared sample containers for fifty-one sampling locations. Each sample set consisted of a field sample, field sample duplicate, and a target analyte field matrix spike. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 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 internal standards prior to being sent to the field for sample collection. Sample location B3 was not collected.

Samples were prepared and analyzed using method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Select internal standards were used to aid in the data quality objectives.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.

ilac-MRA A2LA ACCREDITED Testing Cert. # 2052.01

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

Table 1. Sample Results Summary [1]

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA [2]**
ISO15-0001-001	BD24-3; Sample	122	93.9	1080	2.89
ISO15-0001-002	BD24-3; Sample Dup	124	93.6	1080	2.75
	Average	**123**	**93.8**	**1080**	**2.82**
	%RPD Sample/Sample Dup	**1.6**	**0.32**	**0.0**	**5.0**
ISO15-0001-003	D09; Sample	125	8330	1740	17.5
ISO15-0001-004	D09; Sample Dup	127	8490	1640	17.8
	Average	**126**	**8410**	**1690**	**17.7**
	%RPD Sample/Sample Dup	**1.6**	**1.9**	**5.9**	**1.7**
ISO15-0001-005	P121; Sample	0.212	<0.200	3.38	0.230
ISO15-0001-006	P121; Sample Dup	0.284	<0.200	4.99	0.285
	Average	**0.248**	**<0.200**	**4.19**	**0.258**
	%RPD Sample/Sample Dup	**29 [3]**	**NA**	**38 [3]**	**21 [3]**
ISO15-0001-008	P321; Sample	3630	939	4160	5.67
ISO15-0001-009	P321; Sample Dup	3500	891	4080	6.00
	Average	**3570**	**915**	**4120**	**5.84**
	%RPD Sample/Sample Dup	**3.6**	**5.2**	**1.9**	**5.7**
ISO15-0001-010	3M vijver; Sample	1.26	0.507	5.41	0.0515
ISO15-0001-011	3M vijver; Sample Dup	1.21	0.524	5.12	0.0486
	Average	**1.24**	**0.516**	**5.27**	**0.0501**
	%RPD Sample/Sample Dup	**4.0**	**3.3**	**5.5**	**5.8**
ISO15-0001-012	Blokkersdijkvijver std; Sample	0.958	0.543	0.789	<0.0250
ISO15-0001-013	Blokkersdijkvijver std; Sample Dup	0.972	0.547	0.794	<0.0250
	Average	**0.965**	**0.545**	**0.792**	**<0.0250**
	%RPD Sample/Sample Dup	**1.5**	**0.73**	**0.63**	**NA**
ISO15-0001-014	L21; Sample	0.448	<0.200	4.35	0.0412
ISO15-0001-015	L21; Sample Dup	0.477	<0.200	4.36	0.0383
	Average	**0.463**	**<0.200**	**4.36**	**0.0398**
	%RPD Sample/Sample Dup	**6.3**	**NA**	**0.23**	**7.3**
ISO15-0001-016	L22; Sample	0.685	0.366	12.3	0.0407
ISO15-0001-017	L22; Sample Dup	0.638	0.363	10.5	0.0483
	Average	**0.662**	**0.365**	**11.4**	**0.0445**
	%RPD Sample/Sample Dup	**7.1**	**0.82**	**16**	**17**
ISO15-0001-018	L31; Sample	0.585	0.679	5.82	<0.0250
ISO15-0001-019	L31; Sample Dup	0.617	0.677	5.62	<0.0250
	Average	**0.601**	**0.678**	**5.72**	**<0.0250**
	%RPD Sample/Sample Dup	**5.3**	**0.29**	**3.5**	**NA**
ISO15-0001-020	L4; Sample	1.02	0.653	38.5	12.9
ISO15-0001-021	L4; Sample Dup	1.01	0.709	38.6	13.1
	Average	**1.02**	**0.681**	**38.6**	**13.0**
	%RPD Sample/Sample Dup	**0.99**	**8.2**	**0.26**	**1.5**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 19%, PFBS ± 14%, PFHS ± 24%, and PFOS ± 17%.

(2) Samples reported using internal calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 28%.

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

(4) NR = Not reportable; see section 4 for details.

(5) The method uncertainty has been expanded to ± 33% for PFOSA. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA (2)**
ISO15-0001-022	P114bis; Sample	3.40	1.48	14.5	0.0472
ISO15-0001-023	P114bis; Sample Dup	3.46	1.45	13.9	0.0345
	Average	**3.43**	**1.47**	**14.2**	**0.0409**
	%RPD Sample/Sample Dup	**1.7**	**2.0**	**4.2**	**31 (3)**
ISO15-0001-025	P115; Sample	3.73	2.02	0.497	NR (4)
ISO15-0001-026	P115; Sample Dup	3.63	1.96	0.436	<0.0250
	Average	**3.68**	**1.99**	**0.467**	**<0.0250**
	%RPD Sample/Sample Dup	**2.7**	**3.0**	**13**	**NA**
ISO15-0001-027	P116; Sample	1.77	0.772	7.03	0.0568
ISO15-0001-028	P116; Sample Dup	1.71	0.697	6.70	0.0546
	Average	**1.74**	**0.735**	**6.87**	**0.0557**
	%RPD Sample/Sample Dup	**3.4**	**10**	**4.8**	**3.9**
ISO15-0001-029	P119C; Sample	174	105	1730	63.2
ISO15-0001-030	P119C; Sample Dup	164	99.3	1720	62.6
	Average	**169**	**102**	**1730**	**62.9**
	%RPD Sample/Sample Dup	**5.9**	**5.6**	**0.58**	**0.95**
ISO15-0001-031	Effluent WWTP; Sample	14.0	9.85	19.8	0.339
ISO15-0001-032	Effluent WWTP; Sample Dup	13.6	9.85	19.8	0.342
	Average	**13.8**	**9.85**	**19.8**	**0.341**
	%RPD Sample/Sample Dup	**2.9**	**0.0**	**0.0**	**0.88**
ISO15-0001-053	12; Sample	45.3	13.6	170	0.642
ISO15-0001-054	12; Sample Dup	44.5	13.5	171	0.638
	Average	**44.9**	**13.6**	**171**	**0.640**
	%RPD Sample/Sample Dup	**1.8**	**0.74**	**0.59**	**0.63**
ISO15-0001-055	13; Sample	43.4	13.6	156	0.622
ISO15-0001-056	13; Sample Dup	43.0	13.6	156	0.663
	Average	**43.2**	**13.6**	**156**	**0.643**
	%RPD Sample/Sample Dup	**0.93**	**0.0**	**0.0**	**6.4**
ISO15-0001-057	5; Sample	13.6	10.0	31.9	0.135
ISO15-0001-058	5; Sample Dup	13.7	10.2	31.8	0.129
	Average	**13.7**	**10.1**	**31.9**	**0.132**
	%RPD Sample/Sample Dup	**0.73**	**2.0**	**0.31**	**4.5**
ISO15-0001-060	Bemalingsstation; Sample	13.1	9.55	31.4	0.135
ISO15-0001-061	Bemalingsstation; Sample Dup	12.9	9.55	32.5	0.133
	Average	**13.0**	**9.55**	**32.0**	**0.134**
	%RPD Sample/Sample Dup	**1.5**	**0.0**	**3.4**	**1.5**
ISO15-0001-062	Collector pit; Sample	45.4	16.9	138	15.1
ISO15-0001-063	Collector pit; Sample Dup	44.7	16.8	134	15.0
	Average	**45.1**	**16.9**	**136**	**15.1**
	%RPD Sample/Sample Dup	**1.6**	**0.59**	**2.9**	**0.66**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 19%, PFBS ± 14%, PFHS ± 24%, and PFOS ± 17%.

(2) Samples reported using internal calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 28%.

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

(4) NR = Not reportable; see section 4 for details.

(5) The method uncertainty has been expanded to ± 33% for PFOSA. See section 4 for details.

Table 1 continued. Sample Results Summary [1]

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA [2]**
ISO15-0001-078	M4; Sample	1590	318	17500	341
ISO15-0001-079	M4; Sample Dup	1570	314	18000	341
	Average	**1580**	**316**	**17800**	**341 [5]**
	%RPD Sample/Sample Dup	**1.3**	**1.3**	**2.8**	**0.0**
ISO15-0001-080	P118C; Sample	214	125	1670	60.3
ISO15-0001-081	P118C; Sample Dup	198	114	1520	60.2
	Average	**206**	**120**	**1600**	**60.3**
	%RPD Sample/Sample Dup	**7.8**	**9.2**	**9.4**	**0.17**
ISO15-0001-082	P263; Sample	2010	772	10900	21.1
ISO15-0001-083	P263; Sample Dup	1970	767	8730	21.8
	Average	**1990**	**770**	**9820**	**21.5**
	%RPD Sample/Sample Dup	**2.0**	**0.65**	**22 [3]**	**3.3**
ISO15-0001-084	P264; Sample	398	319	1560	82.5
ISO15-0001-085	P264; Sample Dup	379	300	1630	86.9
	Average	**389**	**310**	**1600**	**84.7**
	%RPD Sample/Sample Dup	**4.9**	**6.1**	**4.4**	**5.2**
ISO15-0001-086	P265C; Sample	70.6	26.0	793	117
ISO15-0001-087	P265C; Sample Dup	72.9	26.5	831	119
	Average	**71.8**	**26.3**	**812**	**118**
	%RPD Sample/Sample Dup	**3.2**	**1.9**	**4.7**	**1.7**
ISO15-0001-088	P371; Sample	127	54.1	629	48.4
ISO15-0001-089	P371; Sample Dup	129	53.2	641	48.1
	Average	**128**	**53.7**	**635**	**48.3**
	%RPD Sample/Sample Dup	**1.6**	**1.7**	**1.9**	**0.62**
ISO15-0001-091	P374; Sample	422	524	2710	29.6
ISO15-0001-092	P374; Sample Dup	489	566	2140	33.1
	Average	**456**	**545**	**2430**	**31.4**
	%RPD Sample/Sample Dup	**15**	**7.7**	**24 [3]**	**11**
ISO15-0001-093	P381; Sample	557	174	1170	12.1
ISO15-0001-094	P381; Sample Dup	559	179	1170	12.9
	Average	**558**	**177**	**1170**	**12.5**
	%RPD Sample/Sample Dup	**0.36**	**2.8**	**0.0**	**6.4**
ISO15-0001-095	P382; Sample	359	99.3	913	85.1
ISO15-0001-096	P382; Sample Dup	345	97.2	872	86.9
	Average	**352**	**98.3**	**893**	**86.0**
	%RPD Sample/Sample Dup	**4.0**	**2.1**	**4.6**	**2.1**
ISO15-0001-099	B7; Sample	1.47	0.639	10.5	0.0840
ISO15-0001-100	B7; Sample Dup	1.50	0.654	11.3	0.0837
	Average	**1.49**	**0.647**	**10.9**	**0.0839**
	%RPD Sample/Sample Dup	**2.0**	**2.3**	**7.3**	**0.36**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 19%, PFBS ± 14%, PFHS ± 24%, and PFOS ± 17%.

(2) Samples reported using internal calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 28%.

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

(4) NR = Not reportable; see section 4 for details.

(5) The method uncertainty has been expanded to ± 33% for PFOSA. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA (2)**
ISO15-0001-101	P372; Sample	80.9	66.5	1380	38.3
ISO15-0001-102	P372; Sample Dup	80.3	66.3	1380	38.7
	Average	**80.6**	**66.4**	**1380**	**38.5**
	%RPD Sample/Sample Dup	**0.74**	**0.30**	**0.0**	**1.0**
ISO15-0001-103	P378; Sample	280	337	648	0.929
ISO15-0001-104	P378; Sample Dup	279	337	647	0.957
	Average	**280**	**337**	**648**	**0.943**
	%RPD Sample/Sample Dup	**0.36**	**0.0**	**0.15**	**3.0**
ISO15-0001-105	PA109A; Sample	288	328	421	1.27
ISO15-0001-106	PA109A; Sample Dup	293	332	436	1.24
	Average	**291**	**330**	**429**	**1.26**
	%RPD Sample/Sample Dup	**1.7**	**1.2**	**3.5**	**2.4**
ISO15-0001-108	PA111A; Sample	840	1260	1950	13.1
ISO15-0001-109	PA111A; Sample Dup	828	1240	1960	16.2
	Average	**834**	**1250**	**1960**	**14.7**
	%RPD Sample/Sample Dup	**1.4**	**1.6**	**0.51**	**21 (3)**
ISO15-0001-110	PA112; Sample	253	83.5	293	42.5
ISO15-0001-111	PA112; Sample Dup	251	84.5	300	46.1
	Average	**252**	**84.0**	**297**	**44.3**
	%RPD Sample/Sample Dup	**0.79**	**1.2**	**2.4**	**8.1**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 19%, PFBS ± 14%, PFHS ± 24%, and PFOS ± 17%.

(2) Samples reported using internal calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 28%.

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

(4) NR = Not reportable; see section 4 for details.

(5) The method uncertainty has been expanded to ± 33% for PFOSA. See section 4 for details.

Table 1 continued. Sample Results Summary [1]

		Concentration (ng/mL)				
3M LIMS ID	**Sample Description**	**PFOA**	**PFBS**	**PFHS**	**PFOS**	**PFOSA [2]**
ISO15-0001-034	PP01; Sample	66.7	72.2	9030	2980	33.9
ISO15-0001-035	PP01; Sample Dup	64.7	72.0	8130	2760	34.5
	Average	**65.7**	**72.1**	**8580**	**2870**	**34.2 [5]**
	%RPD Sample/Sample Dup	**3.0**	**0.28**	**10**	**7.7**	**1.8**
ISO15-0001-036	PP02; Sample	587	35.1	3120	20700	28.2
ISO15-0001-037	PP02; Sample Dup	574	33.3	3030	22000	27.7
	Average	**581**	**34.2**	**3080**	**21400**	**28.0 [5]**
	%RPD Sample/Sample Dup	**2.2**	**5.3**	**2.9**	**6.1**	**1.8**
ISO15-0001-038	PP04; Sample	1650	2110	1050	8830	81.7
ISO15-0001-039	PP04; Sample Dup	1560	2090	1040	10500	126
	Average	**1610**	**2100**	**1050**	**9670**	**104 [5]**
	%RPD Sample/Sample Dup	**5.6**	**0.95**	**0.96**	**17**	**43 [3]**
ISO15-0001-040	PP05; Sample	1110	14300	3010	481	117
ISO15-0001-041	PP05; Sample Dup	1120	14100	3090	419	100
	Average	**1120**	**14200**	**3050**	**450**	**109 [5]**
	%RPD Sample/Sample Dup	**0.90**	**1.4**	**2.6**	**14**	**16**
ISO15-0001-042	PP06; Sample	152	19.8	56.4	1670	63.2
ISO15-0001-043	PP06; Sample Dup	150	17.5	54.5	1690	62.8
	Average	**151**	**18.7**	**55.5**	**1680**	**63.0 [5]**
	%RPD Sample/Sample Dup	**1.3**	**12**	**3.4**	**1.2**	**0.63**
ISO15-0001-044	PP07; Sample	440	116	162	3400	88.7
ISO15-0001-045	PP07; Sample Dup	432	116	162	3330	88.8
	Average	**436**	**116**	**162**	**3370**	**88.8 [5]**
	%RPD Sample/Sample Dup	**1.8**	**0.0**	**0.0**	**2.1**	**0.11**
ISO15-0001-047	PP08; Sample	571	21.3	224	3760	28.1
ISO15-0001-048	PP08; Sample Dup	546	22.1	228	4190	32.9
	Average	**559**	**21.7**	**226**	**3980**	**30.5 [5]**
	%RPD Sample/Sample Dup	**4.5**	**3.7**	**1.8**	**11**	**16**
ISO15-0001-049	PP09; Sample	632	26.7	415	2080	19.5
ISO15-0001-050	PP09; Sample Dup	615	26.8	411	2100	18.3
	Average	**624**	**26.8**	**413**	**2090**	**18.9 [5]**
	%RPD Sample/Sample Dup	**2.7**	**0.37**	**0.97**	**0.96**	**6.3**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 19%, PFBS ± 14%, PFHS ± 24%, and PFOS ± 17%.

(2) Samples reported using internal calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 28%.

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

(4) NR = Not reportable; see section 4 for details.

(5) The method uncertainty has been expanded based on FMS recovery. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)				
3M LIMS ID	**Sample Description**	**PFOA**	**PFBS**	**PFHS**	**PFOS**	**PFOSA (2)**
ISO15-0001-051	PP10; Sample	575	18.7	191	3800	20.7
ISO15-0001-052	PP10; Sample Dup	562	18.5	186	3450	21.1
	Average	**569**	**18.6**	**189**	**3630**	**20.9 (5)**
	%RPD Sample/Sample Dup	**2.3**	**1.1**	**2.7**	**9.7**	**1.9**
ISO15-0001-065	P27; Sample	161	2430	20.2	1030	154
ISO15-0001-066	P27; Sample Dup	157	2360	19.5	988	151
	Average	**159**	**2400**	**19.9**	**1010**	**153**
	%RPD Sample/Sample Dup	**2.5**	**2.9**	**3.5**	**4.2**	**2.0**
ISO15-0001-067	P21B; Sample	12400	4520	9550	83800	32.2
ISO15-0001-068	P21B; Sample Dup	13400	4960	10500	74700	22.3
	Average	**12900**	**4740**	**10000**	**79300**	**27.3**
	%RPD Sample/Sample Dup	**7.8**	**9.3**	**9.5**	**11**	**36 (3)**
ISO15-0001-069	P304; Sample	493	164	104	792	25.1
ISO15-0001-070	P304; Sample Dup	493	164	103	787	24.8
	Average	**493**	**164**	**104**	**790**	**25.0**
	%RPD Sample/Sample Dup	**0.0**	**0.0**	**0.97**	**0.63**	**1.2**
ISO15-0001-071	P305; Sample	183	100	242	535	15.5
ISO15-0001-072	P305; Sample Dup	190	106	259	540	15.0
	Average	**187**	**103**	**251**	**538**	**15.3**
	%RPD Sample/Sample Dup	**3.8**	**5.8**	**6.8**	**0.93**	**3.3**
ISO15-0001-074	P42; Sample	367	42.0	493	3310	29.7
ISO15-0001-075	P42; Sample Dup	370	41.3	488	3250	28.3
	Average	**369**	**41.7**	**491**	**3280**	**29.0**
	%RPD Sample/Sample Dup	**0.81**	**1.7**	**1.0**	**1.8**	**4.8**
ISO15-0001-076	P19; Sample	233	31.2	102	2900	190
ISO15-0001-077	P19; Sample Dup	241	30.7	102	2900	186
	Average	**237**	**31.0**	**102**	**2900**	**188 (5)**
	%RPD Sample/Sample Dup	**3.4**	**1.6**	**0.0**	**0.0**	**2.1**

NA = Not Applicable
(1) Unless noted otherwise, samples were reported using external calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 19%, PFBS ± 14%, PFHS ± 24%, and PFOS ± 17%.
(2) Samples reported using internal calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 28%.
(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
(4) NR = Not reportable; see section 4 for details.
(5) The method uncertainty has been expanded based on FMS recovery. See section 4 for details.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M Environmental Laboratory method ETS-8-044.1 “Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis”. All sampling locations were reported for PFOA, PFHS, PFOS, and PFOSA. Select sampling locations were also reported for PFBS.

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected on January 26-28, 2015 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 mixture of mass-labeled internal standards at a nominal concentration of 1 ng/mL prior to sample collection. Collected sample bottles were returned to the laboratory at ambient temperature on February 2, 2015.

2.3 Sample Preparation

All samples were initially prepared using a direct injection by methanol dilution method. Samples that required a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples that required a 1:100 dilution were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol. Samples that required a 1:500 dilution were prepared by diluting 0.02 mL of a well-mixed sample with 9.9 mL of methanol. Diluted samples and LCSs were fortified with surrogate recovery standards at a nominal concentration of 1 ng/mL following dilution.

All samples were re-analyzed for PFOSA due to non-compliant LCS sample recoveries. Samples were prepared by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). Samples requiring further dilution were prepared by diluting 2 mL of a well-mixed sample with 8 mL of synthetic groundwater (1:5 dilution) and fortified with internal standards at a nominal concentration of 1 ng/mL The diluted samples were then diluted 1:1 with methanol as described above.

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.

2.4 Analysis

All samples and quality control samples were analyzed for five target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.1. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk	ETS Buster
Liquid Chromatograph	Agilent 1260	Agilent 1100
Analysis Method	ETS-8-044.1	ETS-8-044.1
Analysis Date	2/4/15, 2/9/15, 2/11/15	2/23/15
Guard column	Betasil C18 (4.6 mm X 100 mm), 5 µ	Betasil C18 (4.6 mm X 100 mm), 5 µ
Analytical column	Betasil C18 (4.6 mm X 100 mm), 5 µ	Betasil C18 (4.6 mm X 100 mm), 5 µ
Injection Volume	2 or 5 µL	5 µL
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex API 4000
Ion Source	Turbo Spray	Turbo Spray
Polarity	Negative	Negative
Software	Analyst 1.6.2	Analyst 1.6.2

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.1 Analysis				
Step Number	**Total Time (min)**	**Flow Rate (µL/min)**	**Percent A (2 mM ammonium acetate)**	**Percent B (Methanol)**
0	0.00	750	90.0	10.0
1	0.50	750	90.0	10.0
2	4.00	750	70.0	30.0
3	6.00	750	70.0	30.0
4	11.0	750	20.0	80.0
5	13.0	750	20.0	80.0
6	13.5	750	10.0	90.0
7	16.0	750	10.0	90.0
8	16.5	750	90.0	10.0
9	19.0	750	90.0	10.0

Table 5. Mass Transitions

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

NA = Not Applicable

Dwell time was 20 or 50 msec for each transition. The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

(1) Internal standards were used for samples re-analyzed for PFOSA on 2/11/15 and 2/23/15.

3 Data Analysis

3.1 Calibration

2/4/15 and 2/9/15 Analysis: Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL or 0.1 ng/mL to 100 ng/mL (nominal) were analyzed. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL or 0.1 ng/mL to 25 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.

2/11/15 Analysis of PFOSA: 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 50 mL of 50:50 methanol:laboratory reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. Calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed. 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.

2/23/15 Analysis of PFOSA: 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 50 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 5 ng/mL to 200 ng/mL (nominal) were analyzed. Prior to analysis, the calibration standards were diluted in the same manner as the samples 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-8-044.1 is the lowest non-zero calibration standard in the curve that meets linearity and accuracy requirements and for which the area counts are at least twice those of the appropriate blanks. The LOQs associated with the sample analysis are listed in the Table 6 below.

Table 6. LOQ

Analyte	LOQ, ng/mL [1] 2/4/15 Analysis	LOQ, ng/mL [1] 2/9/15 Analysis	LOQ, ng/mL [2] 2/11/15 Analysis	LOQ, ng/mL [1] 2/23/15 Analysis
PFOA	0.0192	0.0958	NA	NA
PFBS	0.0200	0.100	NA	NA
PFHS	0.0200	0.100	NA	NA
PFOS	0.0185	0.0927	NA	NA
PFOSA	0.0200	NA	0.0250	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/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. If applicable, the LCSs were then diluted in the same manner as the samples. Method ETS-8-044.1 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:

- 2/4/15 Analysis: High level LCSs were spiked above the resulting ULOQ (50 ng/mL) for PFHS.
- 2/9/15 Analysis: High level LCSs were not spiked within 80% of the ULOQ (100 ng/mL) for PFOS.
- 2/11/15 Analysis: High level LCSs were calculated above the ULOQ for PFOSA.
- 2/23/15 Analysis: Low, Mid, and High level LCSs diluted 1:5 had average recoveries <80% for PFOSA.

A method deviation is included with the raw data.

All LCS samples were used in the determination of the analytical method uncertainty in section 3.7 of the report.

The following calculations were used to generate data in Table 7.

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

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

Table 7. Laboratory Control Spike Results.

ETS-8-044.1 External Calibration Analyzed 2/4/15	PFOA (Linear + Branched)			PFBS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150203-1	1.91	2.11	111	2.00	2.13	106
LCS-150203-2	1.91	1.97	103	2.00	2.10	105
LCS-150203-3	1.91	2.03	107	2.00	2.14	107
Average ± %RSD	**107% ± 3.7%**			**106% ± 0.94%**		
LCS-150203-4	199	202	102	200	216	108
LCS-150203-5	199	201	101	200	218	109
LCS-150203-6	199	203	102	200	220	110
Average ± %RSD	**102% ± 0.57%**			**109% ± 0.92%**		
LCS-150203-7	793	673	84.8	794	827	104
LCS-150203-8	793	671	84.6	794	824	104
LCS-150203-9	793	668	84.2	794	823	104
Average ± %RSD	**84.5% ± 0.36%**			**104% ± 0.0%**		

ETS-8-044.1 External Calibration Analyzed 2/4/15	PFHS			PFOS (Linear + Branched)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150203-1	2.00	2.20	110	1.85	1.61	87.3
LCS-150203-2	2.00	2.06	103	1.85	1.64	88.5
LCS-150203-3	2.00	2.12	106	1.85	1.65	89.4
Average ± %RSD	**107% ± 1.9%**			**88.4% ± 1.2%**		
LCS-150203-4	200	230	115	199	203	102
LCS-150203-5	200	229	114	199	199	99.9
LCS-150203-6	200	232	116	199	205	103
Average ± %RSD	**106% ± 1.9%**			**102% ± 1.6%**		
LCS-150203-7	794	>ULOQ	NA	793	873	110
LCS-150203-8	794	>ULOQ	NA	793	854	108
LCS-150203-9	794	>ULOQ	NA	793	856	108
Average ± %RSD	**NA (1)**			**109% ± 1.1%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCSs quantitated above the ULOQ.
(2) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External Calibration Analyzed 2/4/15	$^{13}C_4$-PFOA			$^{13}C_4$-PFOS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150203-1	1.99	1.98	99.5	1.91	1.96	103
LCS-150203-2	1.99	2.02	102	1.91	1.97	103
LCS-150203-3	1.99	1.96	98.3	1.91	1.92	100
Average ± %RSD	**99.9% ±1.9%**			**102% ± 1.7%**		
LCS-150203-4	19.9	19.8	99.5	19.1	20.1	105
LCS-150203-5	19.9	19.8	99.7	19.1	19.8	103
LCS-150203-6	19.9	19.9	99.9	19.1	19.9	104
Average ± %RSD	**99.7% ± 0.20%**			**104% ± 0.96%**		

ETS-8-044.1 External Calibration Analyzed 2/9/15	PFOA (Linear + Branched)			PFBS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150209-1	498	511	103	498	516	104
LCS-150209-2	498	517	104	498	529	106
LCS-150209-3	498	514	103	498	515	103
Average ± %RSD	**103% ± 0.56%**			**104% ± 1.5%**		
LCS-150209-4	5000	5200	104	5000	5320	106
LCS-150209-5	5000	5250	105	5000	5280	106
LCS-150209-6	5000	5270	105	5000	5290	106
Average ± %RSD	**105% ± 0.55%**			**106% ± 0.0%**		
LCS-150209-7	20000	19500	97.5	20000	20400	102
LCS-150209-8	20000	19300	96.7	20000	20700	103
LCS-150209-9	20000	19700	98.6	20000	20600	103
Average ± %RSD	**97.6% ± 0.98%**			**103% ± 0.56%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCSs quantitated above the ULOQ.
(2) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External Calibration Analyzed 2/9/15	PFHS			PFOS (Linear + Branched)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150209-1	498	511	103	498	479	96.1
LCS-150209-2	498	525	105	498	472	94.8
LCS-150209-3	498	511	103	498	478	96.0
Average ± %RSD	**104% ± 1.1%**			**95.6% ± 0.76%**		
LCS-150209-4	5000	5310	106	5000	4970	99.4
LCS-150209-5	5000	5390	108	5000	4900	98.1
LCS-150209-6	5000	5380	108	5000	4990	99.8
Average ± %RSD	**107% ± 1.1%**			**99.1% ± 0.90%**		
LCS-150209-7	20000	21200	106	20000	20600	103
LCS-150209-8	20000	21200	106	20000	20300	102
LCS-150209-9	20000	21300	106	20000	20500	102
Average ± %RSD	**106% ± 0.0%**			**102% ± 0.56%**		

ETS-8-044.1 External Calibration Analyzed 2/9/15	$^{13}C_4$-PFOA			$^{13}C_4$-PFOS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150209-1	99.4	97.0	97.5	95.2	94.9	99.7
LCS-150209-2	99.4	103	104	95.2	95.7	101
LCS-150209-3	99.4	99.8	100	95.2	96.2	101
Average ± %RSD	**101% ± 3.3%**			**101% ± 0.75%**		
LCS-150209-4	994	1060	107	952	1020	107
LCS-150209-5	994	1050	105	952	1030	108
LCS-150209-6	994	1030	104	952	991	104
Average ± %RSD	**105% ± 1.5%**			**106% ± 2.0%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCSs quantitated above the ULOQ.
(2) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal Calibration Analyzed 2/11/15	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150211-1	0.199	0.232	116
LCS-150211-2	0.199	0.232	117
LCS-150211-3	0.199	0.232	116
Average ± %RSD	**116% ± 0.50%**		
LCS-150211-4	19.9	24.4	123
LCS-150211-5	19.9	25.0	126
LCS-150211-6	19.9	22.3	112
Average ± %RSD	**120% ± 6.1%**		
LCS-150211-7	160	>ULOQ	NA
LCS-150211-8	160	>ULOQ	NA
LCS-150211-9	160	>ULOQ	NA
Average ± %RSD	**NA (1)**		

ETS-8-044.1 Internal Calibration Analyzed 2/23/15	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150223-1	7.98	8.10	101
LCS-150223-2	7.98	8.17	102
LCS-150223-3	7.98	8.09	101
Average ± %RSD	**101% ± 0.57%**		
LCS-150223-4	39.6	38.7	97.8
LCS-150223-5	39.6	38.6	97.6
LCS-150223-6	39.6	38.9	98.2
Average ± %RSD	**97.9% ± 0.31%**		
LCS-150223-7	160	156	97.3
LCS-150223-8	160	157	97.9
LCS-150223-9	160	155	97.0
Average ± %RSD	**97.4% ± 0.47%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCSs quantitated above the ULOQ.
(2) LCSs did not meet acceptance criteria of 100 ± 20%.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal Calibration Analyzed 2/23/15	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150225-1	39.6	30.3	76.6
LCS-150225-2	39.6	30.7	77.6
LCS-150225-3	39.6	29.8	75.2
Average ± %RSD	**76.5% ± 1.6% (2)**		
LCS-150225-4	199	148	74.3
LCS-150225-5	199	146	73.3
LCS-150225-6	199	144	72.3
Average ± %RSD	**73.3% ± 1.4% (2)**		
LCS-150225-7	798	611	76.6
LCS-150225-8	798	613	76.8
LCS-150225-9	798	611	76.6
Average ± %RSD	**76.7% ± 0.15% (2)**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCSs quantitated above the ULOQ.
(2) LCSs did not meet acceptance criteria of 100 ± 20%.

3.7 Analytical Method Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.2. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.1, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

Table 8. Analytical Method Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	9.59	±19%
PFBS	External	7.11	±14%
PFHS	External	11.8	±24%
PFOS	External	8.55	±17%
PFOSA	Internal	13.9	±28%

3.8 Field Matrix Spikes (FMS)

A target analyte field matrix spike sample(s) was collected for nine 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. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, each sample was fortified with stable isotope surrogate recovery spikes of $^{13}C_4$-PFOA and $^{13}C_4$-PFOS, which were added at a nominal concentration of 1.0 ng/mL to all diluted samples following sample collection. The $^{13}C_4$- PFOA was selected to represent the C8 perfluorocarboxylic acid. The $^{13}C_4$-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids and PFOSA. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

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

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

Table 9. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
P121, P114bis	FMS	9.99	10.0	10.0	9.99	10.0
Effluent WWTP, 5, Collector Pit, PA109A	FMS	99.9	100	100	99.9	10.0
P305, P371	FMS	996	100	993	996	100
PP07	FMS	1990	100	1980	1990	100
Trip Blank	Low	9.99	10.0	10.0	9.99	10.0
	High	1990	100	1980	1990	100

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. The field matrix spike concentration was selected based on the expected concentration of PFOA and/or PFOS, based on previous results for the samples. 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 those analytes where the field matrix spike level was not appropriate as compared to the sample concentration, the surrogate recovery standards were used to assess method accuracy. All surrogate recovery standards met method acceptance criteria. The following sampling locations had field QC samples outside method acceptance criteria:

P121: Sample/sample duplicate RPD was 29% for PFOA, 38% for PFOS, and 21% for PFOSA.

P374 and P263: Sample/sample duplicate RPD was >20% for PFOS.

P114bis, PA111A, PP04, P21B: Sample/sample duplicate RPD was >20% for PFOSA.

M4, PP01-PP10, P19: The PP07 sample location had a FMS recovery of 67.3% for PFOSA. The method uncertainty has been expanded for PFOSA to ± 33%, based on the %bias of the FMS recovery. Since sample locations M4 and P19 did not contain a FMS sample and the samples were prepared in the same manner as PP07, the method uncertainty was also expanded for M4 and P19 based on the PP07 FMS recovery. Location PP07 was the only location with a FMS for the extraction wells PP01-PP10, therefore; the method uncertainty has been expanded for all extraction well sites for PFOSA to ± 33%.

The PFOSA sample result for location **P115**; Sample (ISO15-0001-025) analyzed by internal standard calibration was ‘not reportable’ (NR). The area ratio (target analyte peak are counts over the internal standard area counts) for this sample was within the calibration range, however; the sample target analyte peak area counts for PFOSA was less than the peak area counts for the LOQ standard and less than twice the average area counts for the method blanks, making the sample result not reportable according to method reporting requirements.

Table 10. Location ID: P121

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-005	P121; Sample	0.212	NA	<0.200	NA
ISO15-0001-006	P121; Sample Dup	0.284	NA	<0.200	NA
ISO15-0001-007	P121; FMS	9.69	94.5 [3]	10.9	109
Average Concentration (ng/mL) ± %RPD		**0.248 ng/mL ± 29% [4]**		**<0.200 ng/mL**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-005	P121; Sample	3.38	NA	0.230	NA
ISO15-0001-006	P121; Sample Dup	4.99	NA	0.285	NA
ISO15-0001-007	P121; FMS	12.5	83.2	11.8	115 [3]
Average Concentration (ng/mL) ± %RPD		**4.19 ng/mL ± 38% [4]**		**0.258 ng/mL ± 21% [4]**	

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.
(3) Spike level greater than 10x the endogenous sample concentration.
(4) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 11. Location ID: P114bis

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-022	P114bis; Sample	3.40	NA	1.48	NA
ISO15-0001-023	P114bis; Sample Dup	3.46	NA	1.45	NA
ISO15-0001-024	P114bis; FMS	12.1	86.8	11.2	97.4
Average Concentration (ng/mL) ± %RPD		**3.43 ng/mL ± 1.7%**		**1.47 ng/mL ± 2.0%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-022	P114bis; Sample	14.5	NA	0.0472	NA
ISO15-0001-023	P114bis; Sample Dup	13.9	NA	0.0345	NA
ISO15-0001-024	P114bis; FMS	22.1	79.1	10.9	109 [3]
Average Concentration (ng/mL) ± %RPD		**14.2 ng/mL ± 4.2%**		**0.0409 ng/mL ± 31% [4]**	

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.
(3) Spike level greater than 10x the endogenous sample concentration.
(4) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 12. Location ID: Effluent WWTP

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-031	Effluent WWTP; Sample	14.0	NA	9.85	NA
ISO15-0001-032	Effluent WWTP; Sample Dup	13.6	NA	9.85	NA
ISO15-0001-033	Effluent WWTP; FMS	91.5	77.8	114	104 [3]
Average Concentration (ng/mL) ± %RPD		**13.8 ng/mL ± 2.9%**		**9.85 ng/mL ± 0.0%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-031	Effluent WWTP; Sample	19.8	NA	0.339	NA
ISO15-0001-032	Effluent WWTP; Sample Dup	19.8	NA	0.342	NA
ISO15-0001-033	Effluent WWTP; FMS	110	90.3	11.9	116 [3]
Average Concentration (ng/mL) ± %RPD		**19.8 ng/mL ± 0.0%**		**0.341 ng/mL ± 0.88%**	

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.
(3) Spike level greater than 10x the endogenous sample concentration.

Table 13. Location ID: 5

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-057	5; Sample	13.6	NA	10.0	NA
ISO15-0001-058	5; Sample Dup	13.7	NA	10.2	NA
ISO15-0001-059	5; FMS	97.0	83.4	108	97.9
Average Concentration (ng/mL) ± %RPD		**13.7 ng/mL ± 0.73%**		**10.1 ng/mL ± 2.0%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-057	5; Sample	31.9	NA	0.135	NA
ISO15-0001-058	5; Sample Dup	31.8	NA	0.129	NA
ISO15-0001-059	5; FMS	116	84.2	11.5	114 [3]
Average Concentration (ng/mL) ± %RPD		**31.9 ng/mL ± 0.31%**		**0.132 ng/mL ± 4.5%**	

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.
(3) Spike level greater than 10x the endogenous sample concentration.

Table 14. Location ID: Collector pit

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-062	Collector pit; Sample	45.4	NA	16.9	NA
ISO15-0001-063	Collector pit; Sample Dup	44.7	NA	16.8	NA
ISO15-0001-064	Collector pit; FMS	126	81.0	114	97.2
Average Concentration (ng/mL) ± %RPD		**45.1 ng/mL ± 1.6%**		**16.9 ng/mL ± 0.59%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-062	Collector pit; Sample	138	NA	15.1	NA
ISO15-0001-063	Collector pit; Sample Dup	134	NA	15.0	NA
ISO15-0001-064	Collector pit; FMS	219	83.1	26.9	119
Average Concentration (ng/mL) ± %RPD		**136 ng/mL ± 2.9%**		**15.1 ng/mL ± 0.66%**	

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.

Table 15. Location ID: P371

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-088	P371; Sample	127	NA	54.1	NA
ISO15-0001-089	P371; Sample Dup	129	NA	53.2	NA
ISO15-0001-090	P371; FMS	963	83.8	1030	98.3 [3]
Average Concentration (ng/mL) ± %RPD		**128 ng/mL ± 1.6%**		**53.7 ng/mL ± 1.7%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-088	P371; Sample	629	NA	48.4	NA
ISO15-0001-089	P371; Sample Dup	641	NA	48.1	NA
ISO15-0001-090	P371; FMS	1480	84.8	143	94.8
Average Concentration (ng/mL) ± %RPD		**635 ng/mL ± 1.9%**		**48.3 ng/mL ± 0.62%**	

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.
(3) Spike level greater than 10x the endogenous sample concentration.

Table 16. Location ID: PA109A

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-105	PA109A; Sample	288	NA	328	NA
ISO15-0001-106	PA109A; Sample Dup	293	NA	332	NA
ISO15-0001-107	PA109A; FMS	375	NC	425	NC
Average Concentration (ng/mL) ± %RPD		**291 ng/mL ± 1.7%**		**330 ng/mL ± 1.2%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-105	PA109A; Sample	421	NA	1.27	NA
ISO15-0001-106	PA109A; Sample Dup	436	NA	1.24	NA
ISO15-0001-107	PA109A; FMS	499	NC	12.6	113
Average Concentration (ng/mL) ± %RPD		**429 ng/mL ± 3.5%**		**1.26 ng/mL ± 2.4%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.

Table 17. Location ID: PP07

		PFOA [1]		PFBS [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-044	PP07; Sample	440	NA	116	NA	162	NA
ISO15-0001-045	PP07; Sample Dup	432	NA	116	NA	162	NA
ISO15-0001-046	PP07; FMS	2100	83.6	206	90.0	2100	97.9 [3]
Average Concentration (ng/mL) ± %RPD		**436 ng/mL ± 1.8%**		**116 ng/mL ± 0.0%**		**162 ng/mL ± 0.0%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-044	PP07; Sample	3400	NA	88.7	NA
ISO15-0001-045	PP07; Sample Dup	3330	NA	88.8	NA
ISO15-0001-046	PP07; FMS	5050	84.7	156	67.3 [4]
Average Concentration (ng/mL) ± %RPD		**3370 ng/mL ± 2.1%**		**88.8 ng/mL ± 0.11% [5]**	

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.
(3) Spike level greater than 10x the endogenous sample concentration.
(4) Field matrix spike recovery did not meet acceptance criteria of 100 ± 30%.
(5) Method uncertainty has been expanded to ± 33%.

Table 18. Location ID: P305

		PFOA (1)		PFBS (1)		PFHS (1)	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-071	P305; Sample	183	NA	100	NA	242	NA
ISO15-0001-072	P305; Sample Dup	190	NA	106	NA	259	NA
ISO15-0001-073	P305; FMS	1040	85.7	195	92.0	1240	99.7
Average Concentration (ng/mL) ± %RPD		**187 ng/mL ± 3.8%**		**103 ng/mL ± 5.8%**		**251 ng/mL ± 6.8%**	

		PFOS (1)		PFOSA (2)	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-071	P305; Sample	535	NA	15.5	NA
ISO15-0001-072	P305; Sample Dup	540	NA	15.0	NA
ISO15-0001-073	P305; FMS	1520	98.6	114	98.8
Average Concentration (ng/mL) ± %RPD		**538 ng/mL ± 0.93%**		**15.3 ng/mL ± 3.3%**	

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.

Table 19. Trip Blank

		PFOA (1)		PFBS (1)		PFHS (1)	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-112	Trip Blank; Sample	<0.192	NA	<0.200	NA	<0.200	NA
ISO15-0001-113	Trip Blank; FMS Low	8.60	86.1	9.09	90.9	9.73	97.3
ISO15-0001-114	Trip Blank; FMS High	1690	84.9	93.5	93.5	1960	99.0

		PFOS (1)		PFOSA (2)	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0001-112	Trip Blank; Sample	<0.185	NA	<0.0250	NA
ISO15-0001-113	Trip Blank; FMS Low	8.59	86.0	11.2	112
ISO15-0001-114	Trip Blank; FMS High	1750	87.9	113	113

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.

Table 20. Surrogate Recovery (1)

3M LIMS ID	Description	$^{13}C_4$-PFOA %Recovery	$^{13}C_4$-PFOS %Recovery
ISO15-0001-001	BD24-3; Sample	93.1	97.6
ISO15-0001-002	BD24-3; Sample Dup	98.9	102
ISO15-0001-003	D09; Sample	94.4	94.1
ISO15-0001-004	D09; Sample Dup	94.6	98.3
ISO15-0001-005	P121; Sample	101	102
ISO15-0001-006	P121; Sample Dup	97.3	97.9
ISO15-0001-007	P121; FMS	95.7	95.2
ISO15-0001-008	P321; Sample	88.7	94.0
ISO15-0001-009	P321; Sample Dup	94.5	95.7
ISO15-0001-010	3M vijver; Sample	95.1	99.9
ISO15-0001-011	3M vijver; Sample Dup	95.8	98.4
ISO15-0001-012	Blokkersdijkvijver std; Sample	95.9	96.8
ISO15-0001-013	Blokkersdijkvijver std; Sample Dup	95.8	96.1
ISO15-0001-014	L21; Sample	96.2	100
ISO15-0001-015	L21; Sample Dup	98.5	98.4
ISO15-0001-016	L22; Sample	100	101
ISO15-0001-017	L22; Sample Dup	98.6	102
ISO15-0001-018	L31; Sample	97.7	97.2
ISO15-0001-019	L31; Sample Dup	97.7	101
ISO15-0001-020	L4; Sample	93.1	97.2
ISO15-0001-021	L4; Sample Dup	94.9	99.8
ISO15-0001-022	P114bis; Sample	92.5	98.1
ISO15-0001-023	P114bis; Sample Dup	93.0	96.8
ISO15-0001-024	P114bis; FMS	92.0	94.3
ISO15-0001-025	P115; Sample	95.1	97.3
ISO15-0001-026	P115; Sample Dup	92.7	93.4
ISO15-0001-027	P116; Sample	97.6	97.5
ISO15-0001-028	P116; Sample Dup	89.7	95.1
ISO15-0001-029	P119C; Sample	94.5	95.8
ISO15-0001-030	P119C; Sample Dup	93.1	93.9
ISO15-0001-031	Effluent WWTP; Sample	88.1	99.2
ISO15-0001-032	Effluent WWTP; Sample Dup	83.7	96.2
ISO15-0001-033	Effluent WWTP; FMS	81.6	97.8
ISO15-0001-034	PP01; Sample	91.0	88.8
ISO15-0001-035	PP01; Sample Dup	87.9	87.7
ISO15-0001-036	PP02; Sample	87.8	108
ISO15-0001-037	PP02; Sample Dup	87.9	103
ISO15-0001-038	PP04; Sample	85.9	104
ISO15-0001-039	PP04; Sample Dup	83.2	102
ISO15-0001-040	PP05; Sample	88.4	92.7
ISO15-0001-041	PP05; Sample Dup	87.2	92.3
ISO15-0001-042	PP06; Sample	87.3	87.6
ISO15-0001-043	PP06; Sample Dup	89.5	92.1
ISO15-0001-044	PP07; Sample	88.4	89.3

(1) Surrogate recovery standards were added during sample preparation.

Table 20 continued. Surrogate Recovery (1)

3M LIMS ID	Description	$^{13}C_4$-PFOA %Recovery	$^{13}C_4$-PFOS %Recovery
ISO15-0001-045	PP07; Sample Dup	90.3	87.2
ISO15-0001-046	PP07; FMS	85.5	86.6
ISO15-0001-047	PP08; Sample	88.6	87.6
ISO15-0001-048	PP08; Sample Dup	86.9	88.2
ISO15-0001-049	PP09; Sample	87.2	88.3
ISO15-0001-050	PP09; Sample Dup	87.8	90.2
ISO15-0001-051	PP10; Sample	86.7	86.4
ISO15-0001-052	PP10; Sample Dup	85.6	85.5
ISO15-0001-053	12; Sample	92.8	94.2
ISO15-0001-054	12; Sample Dup	94.8	95.8
ISO15-0001-055	13; Sample	89.6	89.8
ISO15-0001-056	13; Sample Dup	90.6	94.9
ISO15-0001-057	5; Sample	106	105
ISO15-0001-058	5; Sample Dup	89.6	93.0
ISO15-0001-059	5; FMS	89.8	93.5
ISO15-0001-060	Bemalingsstation; Sample	89.8	93.0
ISO15-0001-061	Bemalingsstation; Sample Dup	89.7	95.1
ISO15-0001-062	Collector pit; Sample	94.0	95.3
ISO15-0001-063	Collector pit; Sample Dup	92.3	95.1
ISO15-0001-064	Collector pit; FMS	89.8	92.3
ISO15-0001-065	P27; Sample	88.7	93.3
ISO15-0001-066	P27; Sample Dup	88.4	90.4
ISO15-0001-067	P21B; Sample	97.1	104
ISO15-0001-068	P21B; Sample Dup	102	109
ISO15-0001-069	P304; Sample	87.9	89.7
ISO15-0001-070	P304; Sample Dup	85.2	89.1
ISO15-0001-071	P305; Sample	86.3	90.3
ISO15-0001-072	P305; Sample Dup	87.6	91.1
ISO15-0001-073	P305; FMS	85.9	89.0
ISO15-0001-074	P42; Sample	84.4	84.2
ISO15-0001-075	P42; Sample Dup	86.4	86.3
ISO15-0001-076	P19; Sample	84.5	81.4
ISO15-0001-077	P19; Sample Dup	89.3	87.5
ISO15-0001-078	M4; Sample	91.4	111
ISO15-0001-079	M4; Sample Dup	86.7	105
ISO15-0001-080	P118C; Sample	90.2	94.6
ISO15-0001-081	P118C; Sample Dup	89.6	91.7
ISO15-0001-082	P263; Sample	84.8	109
ISO15-0001-083	P263; Sample Dup	83.5	107
ISO15-0001-084	P264; Sample	92.3	95.1
ISO15-0001-085	P264; Sample Dup	87.6	90.3
ISO15-0001-086	P265C; Sample	89.7	92.9
ISO15-0001-087	P265C; Sample Dup	92.0	95.2

(1) Surrogate recovery standards were added during sample preparation.

Table 20 continued. Surrogate Recovery (1)

3M LIMS ID	Description	$^{13}C_4$-PFOA %Recovery	$^{13}C_4$-PFOS %Recovery
ISO15-0001-088	P371; Sample	89.2	93.3
ISO15-0001-089	P371; Sample Dup	85.7	88.8
ISO15-0001-090	P371; FMS	87.7	89.3
ISO15-0001-091	P374; Sample	87.3	89.2
ISO15-0001-092	P374; Sample Dup	87.5	88.9
ISO15-0001-093	P381; Sample	89.7	92.4
ISO15-0001-094	P381; Sample Dup	89.8	94.1
ISO15-0001-095	P382; Sample	88.3	93.2
ISO15-0001-096	P382; Sample Dup	86.6	90.1
ISO15-0001-099	B7; Sample	89.2	91.6
ISO15-0001-100	B7; Sample Dup	90.4	95.4
ISO15-0001-101	P372; Sample	88.3	90.9
ISO15-0001-102	P372; Sample Dup	90.5	93.6
ISO15-0001-103	P378; Sample	86.1	87.6
ISO15-0001-104	P378; Sample Dup	92.9	95.6
ISO15-0001-105	PA109A; Sample	88.0	93.3
ISO15-0001-106	PA109A; Sample Dup	88.2	93.4
ISO15-0001-107	PA109A; FMS	88.6	91.9
ISO15-0001-108	PA111A; Sample	86.5	91.2
ISO15-0001-109	PA111A; Sample Dup	89.7	93.5
ISO15-0001-110	PA112; Sample	88.3	92.1
ISO15-0001-111	PA112; Sample Dup	89.7	95.5
ISO15-0001-112	Trip Blank; Sample	89.8	91.3
ISO15-0001-113	Trip Blank; FMS Low	89.7	93.3
ISO15-0001-114	Trip Blank; FMS High	83.2	88.6

(1) Surrogate recovery standards were added during sample preparation.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M Environmental Laboratory method ETS-8-044.1 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Analytical results are reported in Tables 1 and 10-20 of this report.

6 Data / Sample Retention

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

7 Signatures



Digitally signed by Susan T. Wolf
DN: c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory - authenticated by LRA, [redacted] o=3M, cn=Susan T. Wolf
Reason: I have reviewed this document
Date: 2015.04.08 13:49:41 -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: 2015.04.09 10:33:17 -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 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: 2015.04.09 07:46:05 -05'00'

Quality Assurance Representative

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Final Report

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

April 2015 Sampling

Laboratory Request Number: ISO15-0007

Report Date – Date of Last Signature

Testing Laboratory

3M EHS Operations
3M Environmental Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

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

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

3M Environmental Laboratory

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

Analytical Report ISO15-0007

Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at
3M Belgium
April 2015 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M Environmental Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Belgium facility. Samples were collected on April 21 and April 28, 2015. Samples were returned to the 3M Environmental Laboratory on May 1, 2015 at ambient temperature for the analysis of Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO15-0007. PFBS was only reported for extraction wells PP01-PP10.

The 3M Environmental Laboratory prepared sample containers for twenty-one sampling locations. A total of eleven sample sets consisted of a field sample, field sample duplicate, and a field matrix spike. All other locations included a field sample and a field sample duplicate only. 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 internal standards prior to being sent to the field for sample collection.

Samples were prepared and analyzed using method ETS-8-044.2 “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. The GPO initiated on 4/13/15 and approved on 4/21/15, indicated that samples would be prepared and analyzed using ETS-8-044.1. The most recent revision of the method, ETS-8-044.2, was effective on 4/9/15 and as such was used for the analysis of the samples for this project.

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]**
ISO15-0007-001	P321; Sample	4400	1220	5720	0.391
ISO15-0007-002	P321; Sample Dup	4450	1230	5560	0.523
	Average	**4430**	**1230**	**5640**	**0.457**
	%RPD Sample/Sample Dup	**1.1**	**0.82**	**2.8**	**29 [3]**
ISO15-0007-004	3M vijver; Sample	1.51	0.628	4.42	0.0597
ISO15-0007-005	3M vijver; Sample Dup	1.64	0.707	4.18	0.0384
	Average	**1.58**	**0.668**	**4.30**	**0.0491**
	%RPD Sample/Sample Dup	**8.3**	**12**	**5.6**	**43 [3]**
ISO15-0007-007	Blokkersdijkvijver std; Sample	1.22	0.594	5.27	0.0616
ISO15-0007-008	Blokkersdijkvijver std; Sample Dup	1.36	0.634	4.75	0.0539
	Average	**1.29**	**0.614**	**5.01**	**0.0578**
	%RPD Sample/Sample Dup	**11**	**6.5**	**10**	**13**
ISO15-0007-009	L4; Sample	0.850	0.661	19.2	10.6
ISO15-0007-010	L4; Sample Dup	0.829	0.656	18.8	10.6
	Average	**0.840**	**0.659**	**19.0**	**10.6**
	%RPD Sample/Sample Dup	**2.5**	**0.76**	**2.1**	**0.0**
ISO15-0007-012	P114bis; Sample	3.45	1.82	16.5	0.0678
ISO15-0007-013	P114bis; Sample Dup	3.50	1.87	15.9	0.0542
	Average	**3.48**	**1.85**	**16.2**	**0.0610**
	%RPD Sample/Sample Dup	**1.4**	**2.7**	**3.7**	**22 [3]**
ISO15-0007-014	P116; Sample	1.91	0.727	7.83	0.0649
ISO15-0007-015	P116; Sample Dup	1.79	0.717	7.59	0.0585
	Average	**1.85**	**0.722**	**7.71**	**0.0617**
	%RPD Sample/Sample Dup	**6.5**	**1.4**	**3.1**	**10**
ISO15-0007-017	Effluent WWTP; Sample	16.0	19.2	16.3	0.328
ISO15-0007-018	Effluent WWTP; Sample Dup	15.6	18.4	16.2	0.300
	Average	**15.8 [4]**	**18.8**	**16.3**	**0.314**
	%RPD Sample/Sample Dup	**2.5**	**4.3**	**0.62**	**8.9**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 15%, PFBS ± 10%, PFHS ± 12%, and PFOS ± 12%.

(2) Samples reported using internal calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 34%.

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

(4) The method uncertainty has been expanded based on FMS recovery. See section 4 for details.

Table 1 continued. Sample Results Summary [1]

		Concentration (ng/mL)				
3M LIMS ID	**Sample Description**	**PFOA**	**PFBS**	**PFHS**	**PFOS**	**PFOSA [2]**
ISO15-0007-020	PP01; Sample	44.5	30.3	10700	4400	75.9
ISO15-0007-021	PP01; Sample Dup	44.2	29.2	11300	4410	73.2
	Average	**44.4**	**29.8**	**11000**	**4410**	**74.6**
	%RPD Sample/Sample Dup	**0.68**	**3.7**	**5.5**	**0.23**	**3.6**
ISO15-0007-022	PP02; Sample	654	37.8	3140	21000	30.4
ISO15-0007-023	PP02; Sample Dup	669	37.7	3200	21800	31.2
	Average	**662**	**37.8**	**3170**	**21400**	**30.8**
	%RPD Sample/Sample Dup	**2.3**	**0.26**	**1.9**	**3.7**	**2.6**
ISO15-0007-024	PP04; Sample	1310	1310	716	8360	77.9
ISO15-0007-025	PP04; Sample Dup	1340	1350	717	8480	53.6
	Average	**1330**	**1330**	**717**	**8420**	**65.8**
	%RPD Sample/Sample Dup	**2.3**	**3.0**	**0.14**	**1.4**	**37 [3]**
ISO15-0007-027	PP05; Sample	1610	18000	5750	307	58.2
ISO15-0007-028	PP05; Sample Dup	1560	18400	6100	300	61.6
	Average	**1590**	**18200**	**5930**	**304**	**59.9**
	%RPD Sample/Sample Dup	**3.2**	**2.2**	**5.9**	**2.3**	**5.7**
ISO15-0007-029	PP06; Sample	316	44.6	56.2	1990	57.8
ISO15-0007-030	PP06; Sample Dup	321	42.6	56.4	1900	59.6
	Average	**319**	**43.6**	**56.3**	**1950**	**58.7**
	%RPD Sample/Sample Dup	**1.6**	**4.6**	**0.36**	**4.6**	**3.1**
ISO15-0007-032	PP07; Sample	410	78.5	155	3510	135
ISO15-0007-033	PP07; Sample Dup	401	81.4	148	3520	136
	Average	**406**	**80.0**	**152**	**3520**	**136**
	%RPD Sample/Sample Dup	**2.2**	**3.6**	**4.6**	**0.28**	**0.74**
ISO15-0007-034	PP08; Sample	739	27.3	290	8210	61.0
ISO15-0007-035	PP08; Sample Dup	752	27.4	298	8830	61.1
	Average	**746**	**27.4**	**294**	**8520**	**61.1**
	%RPD Sample/Sample Dup	**1.7**	**0.37**	**2.7**	**7.3**	**0.16**
ISO15-0007-036	PP09; Sample	808	35.9	569	2370	14.6
ISO15-0007-037	PP09; Sample Dup	823	35.9	573	2450	13.7
	Average	**816**	**35.9**	**571**	**2410**	**14.2**
	%RPD Sample/Sample Dup	**1.8**	**0.0**	**0.70**	**3.3**	**6.4**
ISO15-0007-039	PP10; Sample	722	35.2	872	6510	23.9
ISO15-0007-040	PP10; Sample Dup	663	27.3	1070	9750	22.3
	Average	**693**	**31.3**	**971**	**8130**	**23.1**
	%RPD Sample/Sample Dup	**8.5**	**25 [3]**	**20**	**40 [3]**	**6.9**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 15%, PFBS ± 10%, PFHS ± 12%, and PFOS ± 12%.

(2) Samples reported using internal calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 34%.

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

(4) The method uncertainty has been expanded based on FMS recovery. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA (2)**
ISO15-0007-041	12; Sample	103	30.9	305	0.652
ISO15-0007-042	12; Sample Dup	109	31.5	269	0.633
	Average	**106**	**31.2**	**287**	**0.643 (4)**
	%RPD Sample/Sample Dup	**5.7**	**1.9**	**13**	**3.0**
ISO15-0007-044	13; Sample	89.6	27.3	269	0.573
ISO15-0007-045	13; Sample Dup	90.0	27.2	269	0.594
	Average	**89.8**	**27.3**	**269**	**0.584**
	%RPD Sample/Sample Dup	**0.45**	**0.37**	**0.0**	**3.6**
ISO15-0007-047	5; Sample	29.5	21.8	53.3	0.241
ISO15-0007-048	5; Sample Dup	29.4	22.3	54.4	0.254
	Average	**29.5**	**22.1**	**53.9**	**0.248**
	%RPD Sample/Sample Dup	**0.34**	**2.3**	**2.0**	**5.3**
ISO15-0007-049	Bemalingsstation; Sample	29.1	22.0	55.4	0.173
ISO15-0007-050	Bemalingsstation; Sample Dup	29.7	22.5	56.6	0.175
	Average	**29.4**	**22.3**	**56.0**	**0.174**
	%RPD Sample/Sample Dup	**2.0**	**2.2**	**2.1**	**1.1**
ISO15-0007-051	Collector put; Sample	57.1	16.3	158	12.1
ISO15-0007-052	Collector put; Sample Dup	52.5	15.4	143	11.0
	Average	**54.8**	**15.9**	**151**	**11.6 (4)**
	%RPD Sample/Sample Dup	**8.4**	**5.7**	**10**	**9.5**

NA = Not Applicable
(1) Unless noted otherwise, samples were reported using external calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 15%, PFBS ± 10%, PFHS ± 12%, and PFOS ± 12%.
(2) Samples reported using internal calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 34%.
(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
(4) The method uncertainty has been expanded based on FMS recovery. See section 4 for details.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M Environmental Laboratory method ETS-8-044.2 “Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis”. All sampling locations were reported for PFOA, PFBS, PFHS, PFOS, and PFOSA.

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected on April 21 and April 28, 2015 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 mixture of mass-labeled internal standards at a nominal concentration of 1 ng/mL prior to sample collection. Collected sample bottles were returned to the laboratory at ambient temperature on May 1, 2015.

2.3 Sample Preparation

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

All samples were prepared for PFOSA by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2). During the preparation of the laboratory control samples, an aliquot of a separate internal standard spiking solution was added to the laboratory control samples (nominal concentration of 1 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.

2.4 Analysis

All samples and quality control samples were analyzed for five target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.1. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk	ETS Buster
Liquid Chromatograph	Agilent 1260	Agilent 1100
Analysis Method	ETS-8-044.2	ETS-8-044.2
Analysis Date	5/8/15, 5/11/15	5/15/15
Guard column	Betasil C18 (4.6 mm X 100 mm), 5 μ	Betasil C18 (4.6 mm X 100 mm), 5 μ
Analytical column	Betasil C18 (4.6 mm X 100 mm), 5 μ	Betasil C18 (4.6 mm X 100 mm), 5 μ
Injection Volume	5 μL	5 μL
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex API 4000
Ion Source	Turbo Spray	Turbo Spray
Polarity	Negative	Negative
Software	Analyst 1.6.2	Analyst 1.6.2

Table 4. Liquid Chromatography Gradient Program.

Step Number	Total Time (min)	Flow Rate (µL/min)	Percent A (2 mM ammonium acetate)	Percent B (Methanol)
ETS-8-044.2 Analysis (5/8/15, 5/11/15)				
0	0.00	750	90.0	10.0
1	0.50	750	90.0	10.0
2	4.00	750	70.0	30.0
3	6.00	750	70.0	30.0
4	11.0	750	20.0	80.0
5	13.0	750	20.0	80.0
6	13.5	750	10.0	90.0
7	16.0	750	10.0	90.0
8	16.5	750	90.0	10.0
9	19.0	750	90.0	10.0
ETS-8-044.2 Analysis (5/15/15)				
0	0.00	750	97.0	3.0
1	0.50	750	97.0	3.0
2	4.00	750	70.0	30.0
3	6.00	750	70.0	30.0
4	11.0	750	20.0	80.0
5	13.0	750	20.0	80.0
6	13.5	750	10.0	90.0
7	16.0	750	10.0	90.0
8	16.5	750	97.0	3.0
9	19.0	750	97.0	3.0

Table 5. Mass Transitions

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

NA = Not Applicable

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

(1) Internal standards were used for samples analyzed for PFOSA on 5/15/15.

3 Data Analysis

3.1 Calibration

5/8/15 and 5/11/15 Analysis: Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL or 0.1 ng/mL to 100 ng/mL (nominal) were analyzed. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL or 0.1 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.

5/15/15 Analysis of PFOSA: 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 50 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 (nominal) were analyzed. Prior to analysis, the calibration standards were diluted in the same manner as the samples 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-8-044.2 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) 5/8/15 Analysis	LOQ, ng/mL (1) 5/11/15 Analysis	LOQ, ng/mL (1) 5/15/15 Analysis
PFOA	0.0192	NA	NA
PFBS	0.0200	0.100	NA
PFHS	0.0200	0.100	NA
PFOS	0.0185	0.0927	NA
PFOSA	NA	NA	0.0250

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

3.4 Continuing Calibration

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

3.5 Blanks

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

3.6 Lab Control Spikes (LCSs)

Low, mid, and high lab control spikes were prepared for the target analytes and analyzed in triplicate. LCSs were prepared by spiking known amounts of the analytes into 10 mL of laboratory reagent water to produce the desired concentration. The LCSs were then diluted in the same manner as the samples. Method ETS-8-044.2 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:

- 5/8/15 Analysis: High level LCSs were spiked above the resulting ULOQ (50 ng/mL) for PFHS.
- 5/11/15 Analysis: High level LCSs were spiked above the resulting ULOQ (50 ng/mL) for PFHS. LCS sample LCS-150511-9 quantitated above the ULOQ 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.

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

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

Table 7. Laboratory Control Spike Results.

ETS-8-044.2 External Calibration Analyzed 5/8/15	PFOA (Linear + Branched)			PFBS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150507-1	19.1	19.8	103	20.0	21.9	109
LCS-150507-2	19.1	20.8	109	20.0	22.8	114
LCS-150507-3	19.1	20.6	108	20.0	21.9	110
Average ± %RSD	**107% ± 3.0%**			**111% ± 2.4%**		
LCS-150507-4	1990	2230	112	1990	2290	115
LCS-150507-5	1990	2050	103	1990	2150	108
LCS-150507-6	1990	2030	102	1990	2110	106
Average ± %RSD	**106% ± 5.2%**			**110% ± 4.3%**		
LCS-150507-7	7950	7020	88.2	7950	8140	102
LCS-150507-8	7950	6950	87.4	7950	8100	102
LCS-150507-9	7950	7070	88.9	7950	8300	104
Average ± %RSD	**88.2% ± 0.85%**			**103% ± 1.1%**		

ETS-8-044.2 External Calibration Analyzed 5/8/15	PFHS			PFOS (Linear + Branched)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150507-1	20.0	22.6	113	18.5	19.5	105
LCS-150507-2	20.0	24.0	120	18.5	19.9	108
LCS-150507-3	20.0	22.7	113	18.5	19.7	106
Average ± %RSD	**115% ± 3.5%**			**106% ± 1.4%**		
LCS-150507-4	1990	2380	120	1990	2220	111
LCS-150507-5	1990	2200	111	1990	2040	102
LCS-150507-6	1990	2160	109	1990	2060	104
Average ± %RSD	**113% ± 5.2%**			**106% ± 4.5%**		
LCS-150507-7	7950	>ULOQ	NA	7950	8770	110
LCS-150507-8	7950	>ULOQ	NA	7950	8770	110
LCS-150507-9	7950	>ULOQ	NA	7950	8780	110
Average ± %RSD	**NA (1)**			**110% ± 0.0%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCSs quantitated above the ULOQ.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.2 External Calibration Analyzed 5/8/15	$^{13}C_4$-PFOA			$^{13}C_4$-PFOS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150507-1	19.9	23.8	119	19.1	22.1	115
LCS-150507-2	19.9	22.8	114	19.1	21.8	114
LCS-150507-3	19.9	23.8	119	19.1	23.1	121
Average ± %RSD	**117% ± 2.5%**			**117% ± 3.2%**		
LCS-150507-4	199	239	120	191	234	122
LCS-150507-5	199	233	117	191	228	119
LCS-150507-6	199	230	116	191	229	120
Average ± %RSD	**118% ± 1.8%**			**120% ± 1.3%**		

ETS-8-044.2 External Calibration Analyzed 5/11/15	PFBS			PFHS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150511-1	498	535	107	498	543	109
LCS-150511-2	498	541	109	498	531	107
LCS-150511-3	498	517	104	498	518	104
Average ± %RSD	**107% ± 2.4%**			**107% ± 2.4%**		
LCS-150511-4	5000	5740	115	5000	5820	116
LCS-150511-5	5000	5750	115	5000	5790	116
LCS-150511-6	5000	5770	115	5000	5800	116
Average ± %RSD	**115% ± 0.0%**			**116% ± 0.0%**		
LCS-150511-7	40000	40400	101	40000	>ULOQ	NA
LCS-150511-8	40000	41700	104	40000	>ULOQ	NA
LCS-150511-9	40000	43200	108	40000	>ULOQ	NA
Average ± %RSD	**104% ± 3.4%**			**NA (1)**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCSs quantitated above the ULOQ.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.2 External Calibration Analyzed 5/11/15	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-150511-1	498	489	98.2	95.6	106	111
LCS-150511-2	498	494	99.2	95.6	103	107
LCS-150511-3	498	461	92.5	95.6	103	108
Average ± %RSD	**96.6% ± 3.7%**			**109% ± 1.9%**		
LCS-150511-4	5000	5400	108	956	1100	115
LCS-150511-5	5000	5240	105	956	1060	110
LCS-150511-6	5000	5380	108	956	1050	110
Average ± %RSD	**107% ± 1.6%**			**112% ± 2.6%**		
LCS-150511-7	40000	43700	109			
LCS-150511-8	40000	44600	111			
LCS-150511-9	40000	>ULOQ	NA (1)			
Average ± %RPD	**110% ± 1.8%**					

ETS-8-044.2 Internal Calibration Analyzed 5/15/15	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150515-1	0.199	0.213	107
LCS-150515-2	0.199	0.203	102
LCS-150515-3	0.199	0.203	102
Average ± %RSD	**104% ± 2.8%**		
LCS-150515-4	39.6	38.1	96.2
LCS-150515-5	39.6	38.3	96.6
LCS-150515-6	39.6	39.1	98.7
Average ± %RSD	**97.2% ± 1.4%**		
LCS-150515-7	160	157	98.2
LCS-150515-8	160	155	96.8
LCS-150515-9	160	156	97.2
Average ± %RSD	**97.4% ± 0.74%**		

NA = Not Applicable
ULOQ = Upper Limit of Quantification
(1) LCSs quantitated above the ULOQ.

3.7 Analytical Method Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.2. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.2, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

Table 8. Analytical Method Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	7.49	±15%
PFBS	External	4.98	±10%
PFHS	External	5.94	±12%
PFOS	External	5.92	±12%
PFOSA	Internal	16.8	±34%

3.8 Field Matrix Spikes (FMS)

A target analyte field matrix spike sample(s) was collected for eleven 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. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, each sample was fortified with stable isotope surrogate recovery spikes of $^{13}C_4$-PFOA and $^{13}C_4$-PFOS, which were added at a nominal concentration of 1.0 ng/mL to all diluted samples following 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.

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

Table 9. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
3M vijver, P116, Effluent WWTP	FMS	11.0	11.0	11.0	10.9	1.00
L4	FMS	10.0	9.99	10.0	10.0	10.0
12	FMS	101	101	101	101	1.00
13 (1)	FMS	86.3	86.3	86.3	86.3	0.855
Collector Put	FMS	110	110	110	110	10.0
PP04, PP06, PP09	FMS	1480	99.9	1480	1470	100
P321	FMS	2520	9.99	2510	2510	10.0
Trip Blank	Low	11.0	11.0	11.0	10.9	1.00
	High	1480	99.9	1480	1470	100

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

4 Data Summary and Discussion

Tables 10-21 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. Field matrix spikes meeting the method acceptance criteria of ±30% demonstrate that the method is appropriate for the given matrix.

For those analytes where the field matrix spike level was not appropriate as compared to the sample concentration, the surrogate recovery standards were used to assess method accuracy. All surrogate recovery standards met method acceptance criteria (Table 22). The following sampling locations had field QC samples outside method acceptance criteria:

Effluent WWTP: FMS recovery was 67.5% for PFOA. The method uncertainty for this sample location has been expanded for PFOA to ±33%, based on the %bias of the FMS recovery.

PP10: Sample/sample duplicate RPD was >20% for PFBS and PFOS.

P321, 3M vijver, P114bis, PP04: Sample/sample duplicate RPD was >20% for PFOSA.

12: FMS recovery was 140% for PFOSA. The sample location was re-analyzed and the FMS recovery was verified. The method uncertainty for this sample location has been expanded for PFOSA to ±40%, based on the %bias of the FMS recovery.

Collector put: FMS recovery was 169% for PFOSA. The sample location was re-analyzed and the FMS recovery was verified. The FMS sample has an internal standard response 40% lower than that of the sample and sample duplicate resulting in an increase in sample concentration. Although the method ETS-8-044.2 states that FMS recoveries greater than 100±50% will not be reported, the sample results are consistent with PFOSA results from previous sampling events with acceptable FMS recovery. Since the sample concentration for PFOSA for this sampling round was similar to previous results, and the lower internal standard area response for the FMS sample would explain the high FMS recovery, the results will be reported with an expanded method uncertainty. The method uncertainty for this sample location has been expanded for PFOSA to ±69%, based on the %bias of the FMS recovery.

PFOSA Sample Method Uncertainty: Since only select sample locations contained a FMS sample and locations 12 and Collector put had non-compliant FMS recoveries, to determine whether or not to expand the overall method uncertainty for PFOSA, the average FMS recovery of all sample locations was calculated. The average FMS recovery is 110%, which meets acceptance criteria of 100±30%; therefore, the overall method uncertainty will not be expanded for PFOSA and was determined by ETS-12-012.

Table 10. Location ID: P321

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-001	P321; Sample	4400	NA	1220	NA
ISO15-0007-002	P321; Sample Dup	4450	NA	1230	NA
ISO15-0007-003	P321; FMS	6550	84.3	4020	111
Average Concentration (ng/mL) ± %RPD		**4430 ng/mL ± 1.1%**		**1230 ng/mL ± 0.82%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-001	P321; Sample	5720	NA	0.391	NA
ISO15-0007-002	P321; Sample Dup	5560	NA	0.523	NA
ISO15-0007-003	P321; FMS	8840	NC	10.7	102 [3]
Average Concentration (ng/mL) ± %RPD		**5640 ng/mL ± 2.8%**		**0.457 ng/mL ± 29% [4]**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.
(3) Spike level greater than 10x the endogenous sample concentration.
(4) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 11. Location ID: 3M vijver

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-004	3M vijver; Sample	1.51	NA	0.628	NA
ISO15-0007-005	3M vijver; Sample Dup	1.64	NA	0.707	NA
ISO15-0007-006	3M vijver; FMS	14.4	117	14.4	125 [3]
Average Concentration (ng/mL) ± %RPD		**1.58 ng/mL ± 8.3%**		**0.668 ng/mL ± 12%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-004	3M vijver; Sample	4.42	NA	0.0597	NA
ISO15-0007-005	3M vijver; Sample Dup	4.18	NA	0.0384	NA
ISO15-0007-006	3M vijver; FMS	14.5	93.3	1.11	106 [3]
Average Concentration (ng/mL) ± %RPD		**4.30 ng/mL ± 5.6%**		**0.0491 ng/mL ± 43% [4]**	

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.
(3) Spike level greater than 10x the endogenous sample concentration.
(4) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 12. Location ID: L4

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-009	L4; Sample	0.850	NA	0.661	NA
ISO15-0007-010	L4; Sample Dup	0.829	NA	0.656	NA
ISO15-0007-011	L4; FMS	11.5	107 [3]	11.9	112 [3]
Average Concentration (ng/mL) ± %RPD		**0.840 ng/mL ± 2.5%**		**0.659 ng/mL ± 0.76%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-009	L4; Sample	19.2	NA	10.6	NA
ISO15-0007-010	L4; Sample Dup	18.8	NA	10.6	NA
ISO15-0007-011	L4; FMS	30.3	113	20.5	99.0
Average Concentration (ng/mL) ± %RPD		**19.0 ng/mL ± 2.1%**		**10.6 ng/mL ± 0.0%**	

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.
(3) Spike level greater than 10x the endogenous sample concentration.

Table 13. Location ID: P116

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-014	P116; Sample	1.91	NA	0.727	NA
ISO15-0007-015	P116; Sample Dup	1.79	NA	0.717	NA
ISO15-0007-016	P116; FMS	13.7	108	13.1	113 [3]
Average Concentration (ng/mL) ± %RPD		**1.85 ng/mL ± 6.5%**		**0.722 ng/mL ± 1.4%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-014	P116; Sample	7.83	NA	0.0649	NA
ISO15-0007-015	P116; Sample Dup	7.59	NA	0.0585	NA
ISO15-0007-016	P116; FMS	16.7	82.3	1.03	96.8 [3]
Average Concentration (ng/mL) ± %RPD		**7.71 ng/mL ± 3.1%**		**0.0617 ng/mL ± 10%**	

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.
(3) Spike level greater than 10x the endogenous sample concentration.

Table 14. Location ID: Effluent WWTP

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-017	Effluent WWTP; Sample	16.0	NA	19.2	NA
ISO15-0007-018	Effluent WWTP; Sample Dup	15.6	NA	18.4	NA
ISO15-0007-019	Effluent WWTP; FMS	23.2	67.5 [3]	29.4	96.4
Average Concentration (ng/mL) ± %RPD		**15.8 ng/mL ± 2.5% [4]**		**18.8 ng/mL ± 4.3%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-017	Effluent WWTP; Sample	16.3	NA	0.328	NA
ISO15-0007-018	Effluent WWTP; Sample Dup	16.2	NA	0.300	NA
ISO15-0007-019	Effluent WWTP; FMS	24.2	72.8	1.39	108
Average Concentration (ng/mL) ± %RPD		**16.3 ng/mL ± 0.62%**		**0.314 ng/mL ± 8.9%**	

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.
(3) Field matrix spike recovery did not meet acceptance criteria of 100 ± 30%.
(4) Method uncertainty has been expanded to ± 33%.

Table 15. Location ID: PP04

		PFOA [1]		PFBS [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-024	PP04; Sample	1310	NA	1310	NA	716	NA
ISO15-0007-025	PP04; Sample Dup	1340	NA	1350	NA	717	NA
ISO15-0007-026	PP04; FMS	2760	97.0	1400	NC	2360	111
Average Concentration (ng/mL) ± %RPD		**1330 ng/mL ± 2.3%**		**1330 ng/mL ± 3.0%**		**717 ng/mL ± 0.14%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-024	PP04; Sample	8360	NA	77.9	NA
ISO15-0007-025	PP04; Sample Dup	8480	NA	53.6	NA
ISO15-0007-026	PP04; FMS	NA [3]	NA [3]	150	84.3
Average Concentration (ng/mL) ± %RPD		**8420 ng/mL ± 1.4%**		**65.8 ng/mL ± 37% [4]**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.
(3) Field matrix spike sample was not analyzed since the spike level was less than 0.5x the endogenous sample concentration.
(4) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 16. Location ID: PP06

		PFOA [1]		PFBS [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-029	PP06; Sample	316	NA	44.6	NA	56.2	NA
ISO15-0007-030	PP06; Sample Dup	321	NA	42.6	NA	56.4	NA
ISO15-0007-031	PP06; FMS	1820	101	147	104	1710	112 [3]
Average Concentration (ng/mL) ± %RPD		**319 ng/mL ± 1.6%**		**43.6 ng/mL ± 4.6%**		**56.3 ng/mL ± 0.36%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-029	PP06; Sample	1990	NA	57.8	NA
ISO15-0007-030	PP06; Sample Dup	1900	NA	59.6	NA
ISO15-0007-031	PP06; FMS	3340	94.9	156	97.3
Average Concentration (ng/mL) ± %RPD		**1950 ng/mL ± 4.6%**		**58.7 ng/mL ± 3.1%**	

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.
(3) Spike level greater than 10x the endogenous sample concentration.

Table 17. Location ID: PP09

		PFOA [1]		PFBS [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-036	PP09; Sample	808	NA	35.9	NA	569	NA
ISO15-0007-037	PP09; Sample Dup	823	NA	35.9	NA	573	NA
ISO15-0007-038	PP09; FMS	2210	94.2	136	100	2100	103
Average Concentration (ng/mL) ± %RPD		**816 ng/mL ± 1.8%**		**35.9 ng/mL ± 0.0%**		**571 ng/mL ± 0.70%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-036	PP09; Sample	2370	NA	14.6	NA
ISO15-0007-037	PP09; Sample Dup	2450	NA	13.7	NA
ISO15-0007-038	PP09; FMS	3840	97.3	111	96.9
Average Concentration (ng/mL) ± %RPD		**2410 ng/mL ± 3.3%**		**14.2 ng/mL ± 6.4%**	

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.

Table 18. Location ID: 12

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-041	12; Sample	103	NA	30.9	NA
ISO15-0007-042	12; Sample Dup	109	NA	31.5	NA
ISO15-0007-043	12; FMS	198	91.1	134	102
Average Concentration (ng/mL) ± %RPD		**106 ng/mL ± 5.7%**		**31.2 ng/mL ± 1.9%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-041	12; Sample	305	NA	0.652	NA
ISO15-0007-042	12; Sample Dup	269	NA	0.633	NA
ISO15-0007-043	12; FMS	400	NC	2.04	140 [3]
Average Concentration (ng/mL) ± %RPD		**287 ng/mL ± 13%**		**0.643 ng/mL ± 3.0% [4]**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.
(3) Field matrix spike recovery did not meet acceptance criteria of 100 ± 30%.
(4) Method uncertainty has been expanded to ± 40%.

Table 19. Location ID: 13

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-044	13; Sample	89.6	NA	27.3	NA
ISO15-0007-045	13; Sample Dup	90.0	NA	27.2	NA
ISO15-0007-046	13; FMS	177	101	123	111
Average Concentration (ng/mL) ± %RPD		**89.8 ng/mL ± 0.45%**		**27.3 ng/mL ± 0.37%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-044	13; Sample	269	NA	0.573	NA
ISO15-0007-045	13; Sample Dup	269	NA	0.594	NA
ISO15-0007-046	13; FMS	350	NC	1.68	128
Average Concentration (ng/mL) ± %RPD		**269 ng/mL ± 0.0%**		**0.584 ng/mL ± 3.6%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.

Table 20. Location ID: Collector put

		PFOA [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-051	Collector put; Sample	57.1	NA	16.3	NA
ISO15-0007-052	Collector put; Sample Dup	52.5	NA	15.4	NA
ISO15-0007-053	Collector put; FMS	160	95.6	132	106
Average Concentration (ng/mL) ± %RPD		**54.8 ng/mL ± 8.4%**		**15.9 ng/mL ± 5.7%**	

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-051	Collector put; Sample	158	NA	12.1	NA
ISO15-0007-052	Collector put; Sample Dup	143	NA	11.0	NA
ISO15-0007-053	Collector put; FMS	251	91.4	28.4	169 [3]
Average Concentration (ng/mL) ± %RPD		**151 ng/mL ± 10%**		**11.6 ng/mL ± 9.5% [4]**	

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.
(3) Field matrix spike recovery did not meet acceptance criteria of 100 ± 30%.
(4) Method uncertainty has been expanded to ± 69%.

Table 21. Trip Blank

		PFOA [1]		PFBS [1]		PFHS [1]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-054	Trip Blank; Sample	<0.192	NA	<0.200	NA	<0.200	NA
ISO15-0007-055	Trip Blank; FMS Low	10.8	98.6	11.0	100	11.4	104
ISO15-0007-056	Trip Blank; FMS High	1480	100	97.8	97.9	1560	105

		PFOS [1]		PFOSA [2]	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0007-054	Trip Blank; Sample	<0.185	NA	<0.0250	NA
ISO15-0007-055	Trip Blank; FMS Low	10.1	92.4	1.05	105
ISO15-0007-056	Trip Blank; FMS High	1420	96.6	98.3	98.3

NA = Not Applicable
(1) Samples analyzed by external calibration.
(2) Samples analyzed by internal calibration.

Table 22. Surrogate Recovery (1)

3M LIMS ID	Description	$^{13}C_4$-PFOA %Recovery 5/8/15 Analysis	$^{13}C_4$-PFOS %Recovery 5/8/15 Analysis	$^{13}C_4$-PFOS %Recovery 5/11/15 Analysis
ISO15-0007-001	P321; Sample	105	112	NA
ISO15-0007-002	P321; Sample Dup	107	111	NA
ISO15-0007-003	P321; FMS	102	109	NA
ISO15-0007-004	3M vijver; Sample	114	112	NA
ISO15-0007-005	3M vijver; Sample Dup	114	111	NA
ISO15-0007-006	3M vijver; FMS	113	112	NA
ISO15-0007-007	Blokkersdijkvijver std; Sample	112	116	NA
ISO15-0007-008	Blokkersdijkvijver std; Sample Dup	112	111	NA
ISO15-0007-009	L4; Sample	112	113	NA
ISO15-0007-010	L4; Sample Dup	113	114	NA
ISO15-0007-011	L4; FMS	116	116	NA
ISO15-0007-012	P114bis; Sample	115	116	NA
ISO15-0007-013	P114bis; Sample Dup	114	115	NA
ISO15-0007-014	P116; Sample	117	114	NA
ISO15-0007-015	P116; Sample Dup	116	116	NA
ISO15-0007-016	P116; FMS	115	113	NA
ISO15-0007-017	Effluent WWTP; Sample	89.4	115	NA
ISO15-0007-018	Effluent WWTP; Sample Dup	88.3	110	NA
ISO15-0007-019	Effluent WWTP; FMS	86.7	110	NA
ISO15-0007-020	PP01; Sample	112	107	114
ISO15-0007-021	PP01; Sample Dup	111	107	117
ISO15-0007-022	PP02; Sample	111	88.4	115
ISO15-0007-023	PP02; Sample Dup	112	84.8	110
ISO15-0007-024	PP04; Sample	109	105	NA
ISO15-0007-025	PP04; Sample Dup	110	106	NA
ISO15-0007-026	PP04; FMS	108	102	NA
ISO15-0007-027	PP05; Sample	113	112	124
ISO15-0007-028	PP05; Sample Dup	110	111	126
ISO15-0007-029	PP06; Sample	115	112	NA
ISO15-0007-030	PP06; Sample Dup	112	112	NA
ISO15-0007-031	PP06; FMS	108	108	NA
ISO15-0007-032	PP07; Sample	112	110	NA
ISO15-0007-033	PP07; Sample Dup	113	108	NA
ISO15-0007-034	PP08; Sample	112	106	NA
ISO15-0007-035	PP08; Sample Dup	114	105	NA
ISO15-0007-036	PP09; Sample	111	110	NA
ISO15-0007-037	PP09; Sample Dup	110	110	NA
ISO15-0007-038	PP09; FMS	107	109	NA
ISO15-0007-039	PP10; Sample	110	106	120
ISO15-0007-040	PP10; Sample Dup	111	97.7	119
ISO15-0007-041	12; Sample	112	110	NA
ISO15-0007-042	12; Sample Dup	111	110	NA
ISO15-0007-043	12; FMS	109	107	NA

NA = Not Applicable
(1) Surrogate recovery standards were added during sample preparation.

Table 22 continued. Surrogate Recovery (1)

3M LIMS ID	Description	$^{13}C_4$-PFOA %Recovery 5/8/15 Analysis	$^{13}C_4$-PFOS %Recovery 5/8/15 Analysis	$^{13}C_4$-PFOS %Recovery 5/11/15 Analysis
ISO15-0007-044	13; Sample	108	107	NA
ISO15-0007-045	13; Sample Dup	111	108	NA
ISO15-0007-046	13; FMS	106	108	NA
ISO15-0007-047	5; Sample	111	111	NA
ISO15-0007-048	5; Sample Dup	109	108	NA
ISO15-0007-049	Bemalingsstation; Sample	109	108	NA
ISO15-0007-050	Bemalingsstation; Sample Dup	110	110	NA
ISO15-0007-051	Collector put; Sample	109	110	NA
ISO15-0007-052	Collector put; Sample Dup	111	110	NA
ISO15-0007-053	Collector put; FMS	107	109	NA
ISO15-0007-054	Trip Blank; Sample	116	113	NA
ISO15-0007-055	Trip Blank; FMS Low	112	110	NA
ISO15-0007-056	Trip Blank; FMS High	110	110	NA

NA = Not Applicable
(1) Surrogate recovery standards were added during sample preparation.

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M Environmental Laboratory method ETS-8-044.2 "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 10-22 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 Susan T. Wolf
DN: c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory - authenticated by LRA, [redacted] o=3M, cn=Susan T. Wolf
Reason: I have reviewed this document
Date: 2015.05.29 09:55:54 -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: 2015.05.29 10:40:41 -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 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
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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 2015 Sampling

Laboratory Request Number: ISO15-0013

Report Date – Date of Last Signature

Testing Laboratory

3M EHS Operations
3M Environmental Laboratory
Building 260-5N-17
Maplewood, MN 55144-1000

Requester

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

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

3M Environmental Laboratory

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

Analytical Report ISO15-0013

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

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M Environmental Laboratory prepared and analyzed groundwater, surface water, and wastewater samples collected by Environmental Resources Management (ERM) personnel at the 3M Antwerp, Belgium facility. Samples were collected between July 16, 2015 and July 30, 2015. Samples were returned to the 3M Environmental Laboratory on August 5, 2015 and August 17, 2015 at ambient temperature for the analysis of Perfluorooctanoic acid (PFOA), Perfluorobutane sulfonate (PFBS), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO15-0013.

The 3M Environmental Laboratory prepared sample containers for sixty-five sampling locations. Each sample set consisted of a field sample and field sample duplicate. Twenty-six locations also included a target analyte field matrix spike. Each empty container was marked with a "fill to here" line that corresponded to a final volume of 200 mL. Containers reserved for field matrix spikes were fortified with an appropriate matrix spike solution containing the target analytes prior to being sent to the field for sample collection. All sample bottles were fortified with a mass-labeled internal standard mix and select sample bottles were fortified with surrogate recovery standards [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS prior to being sent to the field for sample collection. Sample locations B3 was not collected. Sample locations P56 and P262 were inadvertently added to the sample list twice, and therefore, two sets of samples were collected and analyzed.

Samples were prepared and analyzed using method ETS-8-044.3 "Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis". Select internal standards were used to aid in the data quality objectives.

Table 1 summarizes the sample results using the analytical method identified above. All results for quality control samples prepared and analyzed with the samples will be reported and discussed elsewhere in this report.

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

Table 1. Sample Results Summary [1]

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA [2]**
Zone: 2nd Aquifer					
ISO15-0013-001	BD24-3; Sample	146	95.6	1430	4.01
ISO15-0013-002	BD24-3; Sample Dup	148	99.9	1530	4.24
	Average	**147**	**97.8**	**1480**	**4.13**
	%RPD Sample/Sample Dup	**1.4**	**4.4**	**6.8**	**5.6**
ISO15-0013-004	BD24-4; Sample	10.5	4.12	7.44	NR [3]
ISO15-0013-005	BD24-4; Sample Dup	10.3	4.05	7.55	0.0862
	Average	**10.4**	**4.09**	**7.50**	**0.0862 [4]**
	%RPD Sample/Sample Dup	**1.9**	**1.7**	**1.5**	**NA**
ISO15-0013-006	D09; Sample	143	8370	1870	22.1
ISO15-0013-007	D09; Sample Dup	138	7910	1840	21.5
	Average	**141**	**8140**	**1860**	**21.8**
	%RPD Sample/Sample Dup	**3.6**	**5.7**	**1.6**	**2.8**
ISO15-0013-009	D10; Sample	534	202	30.5	0.278
ISO15-0013-010	D10; Sample Dup	534	201	30.6	0.314
	Average	**534**	**202**	**30.6**	**0.296 [4]**
	%RPD Sample/Sample Dup	**0.0**	**0.50**	**0.33**	**12**
ISO15-0013-011	D11; Sample	40.6	96.2	842	95.7
ISO15-0013-012	D11; Sample Dup	40.4	95.6	848	94.1
	Average	**40.5**	**95.9**	**845**	**94.9 [4]**
	%RPD Sample/Sample Dup	**0.49**	**0.63**	**0.71**	**1.7**
ISO15-0013-013	D14; Sample	2.24	1.42	1.30	0.247
ISO15-0013-014	D14; Sample Dup	2.05	1.26	1.60	0.203
	Average	**2.15**	**1.34**	**1.45**	**0.225 [4]**
	%RPD Sample/Sample Dup	**8.9**	**12**	**21 [5]**	**20**
ISO15-0013-015	P118A; Sample	1780	3120	1210	6.76
ISO15-0013-016	P118A; Sample Dup	1810	3260	1250	6.71
	Average	**1800**	**3190**	**1230**	**6.74**
	%RPD Sample/Sample Dup	**1.7**	**4.4**	**3.3**	**0.74**
ISO15-0013-018	P118B; Sample	2130	2760	10700	11.0
ISO15-0013-019	P118B; Sample Dup	2120	2780	10700	11.5
	Average	**2130**	**2770**	**10700**	**11.3 [4]**
	%RPD Sample/Sample Dup	**0.47**	**0.72**	**0.0**	**4.4**
ISO15-0013-020	P121; Sample	0.176	0.0694	2.74	0.238
ISO15-0013-021	P121; Sample Dup	0.160	0.0670	2.49	0.177
	Average	**0.168 [2]**	**0.0682 [2]**	**2.62 [2]**	**0.208 [4]**
	%RPD Sample/Sample Dup	**9.5**	**3.5**	**9.6**	**29 [5]**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 14%, PFBS ± 15%, PFHS ± 23%, and PFOS ± 15%.

(2) Sample set reported using internal standard calibration. The analytical method uncertainty associated with the reported results are as follows: PFOA ± 10%, PFHS ± 11%, PFOS ± 9.2%, and PFOSA ± 13%.

(3) NR = Not reportable; see section 4 for details.

(4) Sample results are qualitative due to the field matrix spike recovery being less than 50%. See section 4 for details.

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

(6) The analytical uncertainty has been expanded due to field matrix spike recovery. See section 4 for details.

Table 1 continued. Sample Results Summary [1]

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
Zone: 2nd Aquifer					
ISO15-0013-023	P321; Sample	1320	437	3980	15.9
ISO15-0013-024	P321; Sample Dup	1310	438	4100	15.2
	Average	**1320**	**438**	**4040**	**15.6 [4]**
	%RPD Sample/Sample Dup	**0.76**	**0.23**	**3.0**	**4.5**
Zone: Blokkersdijk nature reserve					
ISO15-0013-025	3M vijver; Sample	1.99	0.803	5.65	0.0949
ISO15-0013-026	3M vijver; Sample Dup	1.88	0.785	5.12	0.112
	Average	**1.94**	**0.794**	**5.39**	**0.103 [6]**
	%RPD Sample/Sample Dup	**5.7**	**2.3**	**9.8**	**17**
ISO15-0013-027	Blokkersdijkvijver std; Sample	1.19	0.662	2.49	0.226
ISO15-0013-028	Blokkersdijkvijver std;Sample Dup	1.24	0.679	2.59	0.166
	Average	**1.22**	**0.671**	**2.54**	**0.196 [6]**
	%RPD Sample/Sample Dup	**4.1**	**2.5**	**3.9**	**31 [5]**
ISO15-0013-029	L21; Sample	0.602	0.284	7.33	0.190
ISO15-0013-030	L21; Sample Dup	0.584	0.264	7.13	0.153
	Average	**0.593 [2]**	**0.274 [2]**	**7.23 [2]**	**0.172 [6]**
	%RPD Sample/Sample Dup	**3.0**	**7.3**	**2.8**	**22 [5]**
ISO15-0013-032	L22; Sample	1.46	0.503	14.6	0.675
ISO15-0013-033	L22; Sample Dup	3.33	1.17	47.1	2.95
	Average	**2.40 [2]**	**0.837 [2]**	**30.9 [2]**	**1.81 [6]**
	%RPD Sample/Sample Dup	**78 [5]**	**80 [5]**	**105 [5]**	**126 [5]**
ISO15-0013-034	L31; Sample	0.640	0.551	11.1	0.416
ISO15-0013-035	L31; Sample Dup	0.599	0.496	10.2	0.294
	Average	**0.620**	**0.524**	**10.7**	**0.355 [6]**
	%RPD Sample/Sample Dup	**6.6**	**11**	**8.5**	**34 [5]**
ISO15-0013-037	L4; Sample	2.33	2.58	29.7	0.931
ISO15-0013-038	L4; Sample Dup	2.39	2.64	30.2	0.934
	Average	**2.36**	**2.61**	**30.0**	**0.933 [6]**
	%RPD Sample/Sample Dup	**2.5**	**2.3**	**1.7**	**0.32**
ISO15-0013-039	P114bis; Sample	3.03	1.26	14.1	0.187
ISO15-0013-040	P114bis; Sample Dup	3.36	1.30	13.9	0.183
	Average	**3.20**	**1.28**	**14.0**	**0.185 [6]**
	%RPD Sample/Sample Dup	**10**	**3.1**	**1.4**	**2.2**
ISO15-0013-041	P115; Sample	4.02	2.00	0.264	0.155
ISO15-0013-042	P115; Sample Dup	4.07	1.99	0.212	0.0946
	Average	**4.05**	**2.00**	**0.238**	**0.125 [6]**
	%RPD Sample/Sample Dup	**1.2**	**0.50**	**22 [5]**	**48 [5]**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 14%, PFBS ± 15%, PFHS ± 23%, and PFOS ± 15%.

(2) Sample set reported using internal standard calibration. The analytical method uncertainty associated with the reported results are as follows: PFOA ± 10%, PFHS ± 11%, PFOS ± 9.2%, and PFOSA ± 13%.

(3) NR = Not reportable; see section 4 for details.

(4) Sample results are qualitative due to the field matrix spike recovery being less than 50%. See section 4 for details.

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

(6) The analytical uncertainty has been expanded due to field matrix spike recovery. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
Zone: Blokkersdijk nature reserve					
ISO15-0013-044	P116; Sample	1.71	0.689	9.11	0.326
ISO15-0013-045	P116; Sample Dup	1.73	0.722	9.74	0.367
	Average	**1.72**	**0.706**	**9.43**	**0.347 (6)**
	%RPD Sample/Sample Dup	**1.2**	**4.7**	**6.7**	**12**
ISO15-0013-046	P119C; Sample	1320	997	7430	122
ISO15-0013-047	P119C; Sample Dup	1310	995	7210	124
	Average	**1320**	**996**	**7320**	**123 (6)**
	%RPD Sample/Sample Dup	**0.76**	**0.20**	**3.0**	**1.6**
Zone: Effluent WWTP					
ISO15-0013-049	Effluent WWTP; Sample	10.8	11.8	2.39	<0.0750
ISO15-0013-050	Effluent WWTP; Sample Dup	11.4	11.7	2.53	<0.0750
	Average	**11.1**	**11.8**	**2.46**	**<0.0750**
	%RPD Sample/Sample Dup	**5.4**	**0.85**	**5.7**	**NA**
Zone: Palingbeek & Tophatgracht					
ISO15-0013-073	12; Sample	123	30.1	364	0.410
ISO15-0013-074	12; Sample Dup	122	29.3	363	0.448
	Average	**123**	**29.7**	**364**	**0.429 (6)**
	%RPD Sample/Sample Dup	**0.82**	**2.7**	**0.28**	**8.9**
ISO15-0013-075	13; Sample	107	29.2	281	0.372
ISO15-0013-076	13; Sample Dup	105	29.8	287	0.364
	Average	**106**	**29.5**	**284**	**0.368 (6)**
	%RPD Sample/Sample Dup	**1.9**	**2.0**	**2.1**	**2.2**
ISO15-0013-078	5; Sample	80.3	67.0	108	0.360
ISO15-0013-079	5; Sample Dup	80.5	66.3	112	0.377
	Average	**80.4**	**66.7**	**110**	**0.369 (6)**
	%RPD Sample/Sample Dup	**0.25**	**1.1**	**3.6**	**4.6**
ISO15-0013-080	Bemalingsstation; Sample	61.2	49.0	92.7	0.268
ISO15-0013-081	Bemalingsstation; Sample Dup	61.8	49.5	87.6	0.250
	Average	**61.5**	**49.3**	**90.2**	**0.259 (6)**
	%RPD Sample/Sample Dup	**0.98**	**1.0**	**5.7**	**6.9**
Zone: Sewer					
ISO15-0013-082	Collector put; Sample	92.9	30.1	315	22.7
ISO15-0013-083	Collector put; Sample Dup	91.5	27.6	321	22.0
	Average	**92.2**	**28.9**	**318**	**22.4 (6)**
	%RPD Sample/Sample Dup	**1.5**	**8.7**	**1.9**	**3.1**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 14%, PFBS ± 15%, PFHS ± 23%, and PFOS ± 15%.

(2) Sample set reported using internal standard calibration. The analytical method uncertainty associated with the reported results are as follows: PFOA ± 10%, PFHS ± 11%, PFOS ± 9.2%, and PFOSA ± 13%.

(3) NR = Not reportable; see section 4 for details.

(4) Sample results are qualitative due to the field matrix spike recovery being less than 50%. See section 4 for details.

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

(6) The analytical uncertainty has been expanded due to field matrix spike recovery. See section 4 for details.

Table 1 continued. Sample Results Summary [1]

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
Zone: Source area - WWTP					
ISO15-0013-101	L19; Sample	313	209	3720	143
ISO15-0013-102	L19; Sample Dup	290	210	3620	144
	Average	**302**	**210**	**3670**	**144 [4]**
	%RPD Sample/Sample Dup	**7.6**	**0.48**	**2.7**	**0.70**
ISO15-0013-103	M4; Sample	381	162	6300	265
ISO15-0013-104	M4; Sample Dup	414	168	7070	281
	Average	**398**	**165**	**6690**	**273 [4]**
	%RPD Sample/Sample Dup	**8.3**	**3.6**	**12**	**5.9**
ISO15-0013-106	P118C; Sample	278	101	1900	93.9
ISO15-0013-107	P118C; Sample Dup	271	98.7	1850	86.7
	Average	**275**	**99.9**	**1880**	**90.3 [4]**
	%RPD Sample/Sample Dup	**2.6**	**2.3**	**2.7**	**8.0**
ISO15-0013-108	P262; Sample (set 1)	369	162	2970	53.3
ISO15-0013-109	P262; Sample Dup (set 1)	376	169	3120	44.8
	Average	**373**	**166**	**3050**	**49.1 [4]**
	%RPD Sample/Sample Dup	**1.9**	**4.2**	**4.9**	**17**
ISO15-0013-159	P262; Sample (set 2)	379	166	3040	43.4
ISO15-0013-160	P262; Sample Dup (set 2)	384	168	3190	56.9
	Average	**382**	**167**	**3120**	**50.2 [4]**
	%RPD Sample/Sample Dup	**1.3**	**1.2**	**4.8**	**27 [5]**
ISO15-0013-110	P263; Sample	2270	727	11300	15.9
ISO15-0013-111	P263; Sample Dup	2440	731	11800	13.1
	Average	**2360**	**729**	**11600**	**14.5 [4]**
	%RPD Sample/Sample Dup	**7.2**	**0.55**	**4.3**	**19**
ISO15-0013-112	P264; Sample	299	168	1890	172
ISO15-0013-113	P264; Sample Dup	304	166	1870	173
	Average	**302**	**167**	**1880**	**173 [4]**
	%RPD Sample/Sample Dup	**1.7**	**1.2**	**1.1**	**0.58**
ISO15-0013-115	P265C; Sample	119	33.7	948	120
ISO15-0013-116	P265C; Sample Dup	113	32.3	930	123
	Average	**116**	**33.0**	**939**	**122 [4]**
	%RPD Sample/Sample Dup	**5.2**	**4.2**	**1.9**	**2.5**
ISO15-0013-117	P340; Sample	2630	583	16500	170
ISO15-0013-118	P340; Sample Dup	1670	483	6710	105
	Average	**2150**	**533**	**11600**	**138**
	%RPD Sample/Sample Dup	**45 [5]**	**19**	**84 [5]**	**47 [5]**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 14%, PFBS ± 15%, PFHS ± 23%, and PFOS ± 15%.

(2) Sample set reported using internal standard calibration. The analytical method uncertainty associated with the reported results are as follows: PFOA ± 10%, PFHS ± 11%, PFOS ± 9.2%, and PFOSA ± 13%.

(3) NR = Not reportable; see section 4 for details.

(4) Sample results are qualitative due to the field matrix spike recovery being less than 50%. See section 4 for details.

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

(6) The analytical uncertainty has been expanded due to field matrix spike recovery. See section 4 for details.

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

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
Zone: Source area - WWTP					
ISO15-0013-120	P341; Sample	1150	491	6090	57.9
ISO15-0013-121	P341; Sample Dup	1130	486	6040	59.5
	Average	**1140**	**489**	**6070**	**58.7 [(4)]**
	%RPD Sample/Sample Dup	**1.8**	**1.0**	**0.82**	**2.7**
ISO15-0013-122	P343; Sample	902	636	7040	16.3
ISO15-0013-123	P343; Sample Dup	907	612	6940	16.4
	Average	**905**	**624**	**6990**	**16.4 [(4)]**
	%RPD Sample/Sample Dup	**0.55**	**3.8**	**1.4**	**0.61**
ISO15-0013-125	P371; Sample	183	99.9	2210	87.3
ISO15-0013-126	P371; Sample Dup	183	108	2250	83.5
	Average	**183**	**104**	**2230**	**85.4 [(4)]**
	%RPD Sample/Sample Dup	**0.0**	**7.8**	**1.8**	**4.4**
ISO15-0013-127	P374; Sample	862	1060	3450	30.1
ISO15-0013-128	P374; Sample Dup	897	1110	3210	30.2
	Average	**880**	**1090**	**3330**	**30.2**
	%RPD Sample/Sample Dup	**4.0**	**4.6**	**7.2**	**0.33**
ISO15-0013-130	P379; Sample	102	26.8	922	72.3
ISO15-0013-131	P379; Sample Dup	107	26.2	931	70.9
	Average	**105**	**26.5**	**927**	**71.6 [(4)]**
	%RPD Sample/Sample Dup	**4.8**	**2.3**	**0.97**	**2.0**
ISO15-0013-132	P380; Sample	231	181	1110	140
ISO15-0013-133	P380; Sample Dup	218	164	1080	126
	Average	**225**	**173**	**1100**	**133 [(4)]**
	%RPD Sample/Sample Dup	**5.8**	**9.9**	**2.7**	**11**
ISO15-0013-134	P381; Sample	684	183	1480	18.4
ISO15-0013-135	P381; Sample Dup	666	186	1360	21.4
	Average	**675**	**185**	**1420**	**19.9 [(4)]**
	%RPD Sample/Sample Dup	**2.7**	**1.6**	**8.5**	**15**
ISO15-0013-136	P382; Sample	494	138	1070	104
ISO15-0013-137	P382; Sample Dup	497	142	1060	98.9
	Average	**496**	**140**	**1070**	**101**
	%RPD Sample/Sample Dup	**0.61**	**2.9**	**0.94**	**5.0**
Zone: Southern site boundary					
ISO15-0013-141	B7; Sample	29.8	17.6	395	2.43
ISO15-0013-142	B7; Sample Dup	30.1	17.2	389	2.26
	Average	**30.0**	**17.4**	**392**	**2.35**
	%RPD Sample/Sample Dup	**1.0**	**2.3**	**1.5**	**7.2**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 14%, PFBS ± 15%, PFHS ± 23%, and PFOS ± 15%.

(2) Sample set reported using internal standard calibration. The analytical method uncertainty associated with the reported results are as follows: PFOA ± 10%, PFHS ± 11%, PFOS ± 9.2%, and PFOSA ± 13%.

(3) NR = Not reportable; see section 4 for details.

(4) Sample results are qualitative due to the field matrix spike recovery being less than 50%. See section 4 for details.

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

(6) The analytical uncertainty has been expanded due to field matrix spike recovery. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
Zone: Southern site boundary					
ISO15-0013-144	P372; Sample	83.5	58.8	1520	43.3
ISO15-0013-145	P372; Sample Dup	81.4	60.9	1500	43.0
	Average	**82.5**	**59.9**	**1510**	**43.2**
	%RPD Sample/Sample Dup	**2.5**	**3.5**	**1.3**	**0.70**
ISO15-0013-146	P378; Sample	373	360	1290	1.85
ISO15-0013-147	P378; Sample Dup	375	368	1330	1.82
	Average	**374**	**364**	**1310**	**1.84**
	%RPD Sample/Sample Dup	**0.53**	**2.2**	**3.1**	**1.6**
ISO15-0013-149	PA109A; Sample	66.6	67.1	173	1.89
ISO15-0013-150	PA109A; Sample Dup	66.0	68.9	180	2.00
	Average	**66.3**	**68.0**	**177**	**1.95**
	%RPD Sample/Sample Dup	**0.90**	**2.6**	**4.0**	**5.7**
ISO15-0013-151	PA111A; Sample	945	1210	2640	12.4
ISO15-0013-152	PA111A; Sample Dup	932	1180	2550	12.2
	Average	**939**	**1200**	**2600**	**12.3**
	%RPD Sample/Sample Dup	**1.4**	**2.5**	**3.5**	**1.6**
ISO15-0013-154	PA112; Sample	151	31.6	422	64.2
ISO15-0013-155	PA112; Sample Dup	155	31.9	430	63.6
	Average	**153**	**31.8**	**426**	**63.9**
	%RPD Sample/Sample Dup	**2.6**	**0.94**	**1.9**	**0.94**

		Concentration (ng/mL)				
3M LIMS ID	**Sample Description**	**PFOA**	**PFBS**	**PFHS**	**PFOS**	**PFOSA**
Zone: Extractions wells P&T						
ISO15-0013-052	PP01; Sample	88.2	122	36700	7200	58.6
ISO15-0013-053	PP01; Sample Dup	81.8	122	38200	6880	60.4
	Average	**85.0**	**122**	**37500**	**7040**	**59.5**
	%RPD Sample/Sample Dup	**7.5**	**0.0**	**4.0**	**4.5**	**3.0**
ISO15-0013-055	PP02; Sample	532	15.8	1270	22200	37.8
ISO15-0013-056	PP02; Sample Dup	493	16.6	1210	20500	35.2
	Average	**513**	**16.2**	**1240**	**21400**	**36.5**
	%RPD Sample/Sample Dup	**7.6**	**4.9**	**4.8**	**8.0**	**7.1**
ISO15-0013-057	PP04; Sample	903	965	431	7120	99.6
ISO15-0013-058	PP04; Sample Dup	925	993	448	7970	110
	Average	**914**	**979**	**440**	**7550**	**105**
	%RPD Sample/Sample Dup	**2.4**	**2.9**	**3.9**	**11**	**9.9**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 14%, PFBS ± 15%, PFHS ± 23%, and PFOS ± 15%.

(2) Sample set reported using internal standard calibration. The analytical method uncertainty associated with the reported results are as follows: PFOA ± 10%, PFHS ± 11%, PFOS ± 9.2%, and PFOSA ± 13%.

(3) NR = Not reportable; see section 4 for details.

(4) Sample results are qualitative due to the field matrix spike recovery being less than 50%. See section 4 for details.

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

(6) The analytical uncertainty has been expanded due to field matrix spike recovery. See section 4 for details.

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

		Concentration (ng/mL)				
3M LIMS ID	**Sample Description**	**PFOA**	**PFBS**	**PFHS**	**PFOS**	**PFOSA**
Zone: Extractions wells P&T						
ISO15-0013-060	PP05; Sample	2550	27200	5950	168	10.7
ISO15-0013-061	PP05; Sample Dup	2510	27400	5900	208	12.0
	Average	**2530**	**27300**	**5930**	**188**	**11.4**
	%RPD Sample/Sample Dup	**1.6**	**0.73**	**0.84**	**21 [(5)]**	**11**
ISO15-0013-062	PP06; Sample	245	110	64.9	2150	66.2
ISO15-0013-063	PP06; Sample Dup	208	52.5	57.9	2020	71.2
	Average	**227**	**81.3**	**61.4**	**2090**	**68.7**
	%RPD Sample/Sample Dup	**16**	**71 [(5)]**	**11**	**6.2**	**7.3**
ISO15-0013-064	PP07; Sample	345	45.3	109	2910	107
ISO15-0013-065	PP07; Sample Dup	345	45.5	111	2930	86.2
	Average	**345**	**45.4**	**110**	**2920**	**96.6**
	%RPD Sample/Sample Dup	**0.0**	**0.44**	**1.8**	**0.68**	**22 [(5)]**
ISO15-0013-066	PP08; Sample	569	24.1	207	8730	73.3
ISO15-0013-067	PP08; Sample Dup	556	24.0	199	8530	68.1
	Average	**563**	**24.1**	**203**	**8630**	**70.7**
	%RPD Sample/Sample Dup	**2.3**	**0.42**	**3.9**	**2.3**	**7.4**
ISO15-0013-068	PP09; Sample	765	31.6	464	2470	12.3
ISO15-0013-069	PP09; Sample Dup	773	30.0	458	2410	12.2
	Average	**769**	**30.8**	**461**	**2440**	**12.3**
	%RPD Sample/Sample Dup	**1.0**	**5.2**	**1.3**	**2.5**	**0.82**
ISO15-0013-071	PP10; Sample	640	21.0	200	3910	16.1
ISO15-0013-072	PP10; Sample Dup	638	21.4	190	3700	14.2
	Average	**639**	**21.2**	**195**	**3810**	**15.2**
	%RPD Sample/Sample Dup	**0.31**	**1.9**	**5.1**	**5.5**	**13**
Zone: Source area – Building 16						
ISO15-0013-085	K3; Sample	99.2	1330	90000	3510	11.7
ISO15-0013-086	K3; Sample Dup	97.6	1320	91000	3570	13.1
	Average	**98.4**	**1325**	**90500**	**3540**	**12.4 [(6)]**
	%RPD Sample/Sample Dup	**1.6**	**0.75**	**1.1**	**1.7**	**11**
ISO15-0013-087	P27; Sample	190	419	24.2	1970	169
ISO15-0013-088	P27; Sample Dup	196	488	28.6	1980	161
	Average	**193**	**454**	**26.4**	**1980**	**165 [(6)]**
	%RPD Sample/Sample Dup	**3.1**	**15**	**17**	**0.51**	**4.8**
ISO15-0013-090	P21B; Sample	12300	6360	9400	46700	3.76
ISO15-0013-091	P21B; Sample Dup	12500	6120	8930	46500	3.80
	Average	**12400**	**6240**	**9170**	**46600**	**3.78 [(6)]**
	%RPD Sample/Sample Dup	**1.6**	**3.8**	**5.1**	**0.43**	**1.1**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 14%, PFBS ± 15%, PFHS ± 23%, and PFOS ± 15%.

(2) Sample set reported using internal standard calibration. The analytical method uncertainty associated with the reported results are as follows: PFOA ± 10%, PFHS ± 11%, PFOS ± 9.2%, and PFOSA ± 13%.

(3) NR = Not reportable; see section 4 for details.

(4) Sample results are qualitative due to the field matrix spike recovery being less than 50%. See section 4 for details.

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

(6) The analytical uncertainty has been expanded due to field matrix spike recovery. See section 4 for details.

Table 1 continued. Sample Results Summary (1)

		Concentration (ng/mL)				
3M LIMS ID	**Sample Description**	**PFOA**	**PFBS**	**PFHS**	**PFOS**	**PFOSA**
Zone: Source area – Building 16						
ISO15-0013-092	P304; Sample	684	266	156	1070	25.8
ISO15-0013-093	P304; Sample Dup	665	253	143	1040	24.8
	Average	**675**	**260**	**150**	**1060**	**25.3 (6)**
	%RPD Sample/Sample Dup	**2.8**	**5.0**	**8.7**	**2.8**	**4.0**
ISO15-0013-095	P305; Sample	256	131	300	642	16.4
ISO15-0013-096	P305; Sample Dup	256	135	305	641	16.4
	Average	**256**	**133**	**303**	**642**	**16.4 (6)**
	%RPD Sample/Sample Dup	**0.0**	**3.0**	**1.7**	**0.16**	**0.0**
ISO15-0013-097	P42; Sample	440	67.0	587	3880	23.0
ISO15-0013-098	P42; Sample Dup	435	65.9	586	3930	21.9
	Average	**438**	**66.5**	**587**	**3910**	**22.5 (6)**
	%RPD Sample/Sample Dup	**1.1**	**1.7**	**0.17**	**1.3**	**4.9**
ISO15-0013-099	P56; Sample (set 1)	71.7	15.7	60.3	215	18.6
ISO15-0013-100	P56; Sample Dup (set 1)	72.8	16.1	62.1	215	17.7
	Average	**72.3**	**15.9**	**61.2**	**215**	**18.2 (6)**
	%RPD Sample/Sample Dup	**1.5**	**2.5**	**2.9**	**0.0**	**5.0**
ISO15-0013-156	P56; Sample (set 2)	72.7	15.7	60.3	222	18.4
ISO15-0013-157	P56; Sample Dup (set 2)	72.8	15.4	58.9	217	18.9
	Average	**72.8**	**15.6**	**59.6**	**220**	**18.7 (6)**
	%RPD Sample/Sample Dup	**0.14**	**1.9**	**2.3**	**2.3**	**2.7**

NA = Not Applicable

(1) Unless noted otherwise, samples were reported using external standard calibration. The analytical method uncertainties associated with the reported results are as follows: PFOA ± 14%, PFBS ± 15%, PFHS ± 23%, and PFOS ± 15%.

(2) Sample set reported using internal standard calibration. The analytical method uncertainty associated with the reported results are as follows: PFOA ± 10%, PFHS ± 11%, PFOS ± 9.2%, and PFOSA ± 13%.

(3) NR = Not reportable; see section 4 for details.

(4) Sample results are qualitative due to the field matrix spike recovery being less than 50%. See section 4 for details.

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

(6) The analytical uncertainty has been expanded due to field matrix spike recovery. See section 4 for details.

2 Methods - Analytical and Preparatory

2.1 Methods

Analysis was completed following 3M Environmental Laboratory method ETS-8-044.3 “Method of Analysis for the Determination of Perfluorinated Compounds in Water by LC/MS/MS; Direct Injection Analysis”.

Table 2. Target Analytes

Target Analytes	Acronym	Reference Material Structure
Perfluorooctanoic Acid (C8 Acid)	PFOA	Linear + Branched
Perfluorobutanesulfonate (C4 Sulfonate)	PFBS	Linear
Perfluorohexanesulfonate (C6 Sulfonate)	PFHS	Linear
Perfluorooctanesulfonate (C8 Sulfonate)	PFOS	Linear + Branched
Perfluorooctanesulfonamide	PFOSA	Linear + Branched

2.2 Sample Collection

Samples were collected between July 16, 2015 and July 30, 2015 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. Collected sample bottles were returned to the laboratory at ambient temperature on August 5, 2015 and August 17, 2015.

2.3 Sample Preparation

All samples were prepared for PFOA, PFBS, PFHS, and PFOS using a direct injection by methanol dilution method. Samples that required a 1:10 dilution were prepared by diluting 1 mL of a well-mixed sample with 9 mL of methanol. Samples that required a 1:100 dilution were prepared by diluting 0.1 mL of a well-mixed sample with 9.9 mL of methanol. Samples that required a 1:500 dilution were prepared by diluting 0.02 mL of a well-mixed sample with 9.9 mL of methanol. 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, and the Trip Blank sample 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.

Following the analysis of PFOA, PFBS, PFHS, and PFOS, all sample bottles were fortified with additional internal standard for a final concentration of 2.5 ng/mL (nominal) in order to achieve a consistent internal standard response for PFOSA. All samples were then prepared for PFOSA by removing a 0.16 mL aliquot of the well mixed sample and diluting it with 0.64 mL of methanol (dilution factor of 5). 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 2.5 ng/mL). The laboratory control samples were then diluted with methanol in the same manner. The calibration standards and method blanks were also prepared by removing a 0.4 mL aliquot of the well mixed sample and diluting it with 0.4 mL of methanol (dilution factor of 2).

2.4 Analysis

All samples and quality control samples were analyzed for five target analytes using high performance liquid chromatography/tandem mass spectrometry (HPLC/MS/MS). The rinseate blank was not fortified with SRSs prior to sample collection; however, SRSs were added and included in the analysis of these samples. Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables below.

Due to the nature of the sample, the wide range of concentrations found in the sample, and the environmental occurrence of multiple isomers of the laboratory's analytes of interest, the software used for processing the analytical results is not able to consistently integrate the analytical peak, manual integration of the analytical peak is necessary. All manual integrations are performed following the procedures outlined in method ETS-12-010.1. The consistency of the laboratory's integration is ensured through the training of laboratory personnel, the peer review process required for all manual integrations, the review of manual integrations by the QAU, and where necessary the review of manual integrations by laboratory management.

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

8/20/15 (ETS Kirk) External Standard Calibration Analysis for PFOA, PFBS, PFHS, PFOS, [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS for locations: BD24-3, BD24-4, D09, D10, D11, D14, P118A, P118B, P121, P321, 3M vijver, Blokkersdijkvijer std, L21, L22, L31, L4, P114bis, P115, P116, P119C, Effluent WWTP, PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09, PP10, 12, 13, 5, Bemalingsstation.

8/20/15 (ETS Buster) External Standard Calibration Analysis for PFOA, PFBS, PFHS, PFOS, [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS for locations: Collector put, K3, P27, P21B, P304, P305, P42, P56 (sets 1 and 2), L19, M4, P118C, P262 (sets 1 and 2), P263, P264, P265C, P340, P341, P343, P371, P374, P379, P380, P381, P383, B7, P372, P378, PA109A, PA111A, PA112, and Trip Blank set.

8/25/15 External Standard Calibration Analysis for locations: PP01 (PFHS and [$^{13}C_4$]-PFOS), PP02 (PFBS and [$^{13}C_4$]-PFOS), K3 (PFHS and [$^{13}C_4$]-PFOS), P21B (PFOS and [$^{13}C_4$]-PFOS), P340 (PFOA, PFOS, [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS).

8/26/15 Internal Standard Calibration Analysis for PFOA, PFHS, PFOS, [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS for locations: P121, L21, L22, and Trip Blank sample.

8/28/15 External Standard Calibration Analysis for PFBS for location PP02.

9/2/15 Internal Standard Calibration Analysis for PFOSA for locations: BD24-3, BD24-4, D09, D10, D11, D14, P118A, P118B, P121, P321, 3M vijver, Blokkersdijkvijer std, L21, L22, L31, L4, P114bis, P115, P116, P119C, Effluent WWTP, PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09, PP10, 12, 13, 5, Bemalingsstation.

9/8/15 Internal Standard Calibration Analysis for PFOSA for locations: Collector put, K3, P27, P21B, P304, P305, P42, P56 (sets 1 and 2), L19, M4, P118C, P262 (sets 1 and 2), P263, P264, P265C, P340, P341, P343, P371, P374, P379, P380, P381, P383, B7, P372, P378, PA109A, PA111A, PA112, and Trip Blank set.

Table 3. Instrument Parameters.

Instrument Name	**ETS Kirk**	**ETS Buster**
Liquid Chromatograph	Agilent 1260	Agilent 1100
Analysis Method	ETS-8-044.3	ETS-8-044.3
Analysis Date	8/20/15 & 8/26/15	8/20/15, 8/25/15, 8/28/15, 9/2/15, 9/8/15
Guard column	Betasil C18 (4.6 mm X 100 mm), 5 µ	Betasil C18 (4.6 mm X 100 mm), 5 µ
Analytical column	Betasil C18 (4.6 mm X 100 mm), 5 µ	Betasil C18 (4.6 mm X 100 mm), 5 µ
Injection Volume	5 µL	5 or 10 µL
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex API 4000
Ion Source	Turbo Spray	Turbo Spray
Polarity	Negative	Negative
Software	Analyst 1.6.2	Analyst 1.6.2

Table 4. Liquid Chromatography Gradient Program.

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

Table 5. Mass Transitions

Analyte	**Mass Transition Q1/Q3**	**Internal Standard (1)**	**Mass Transition Q1/Q3**
PFOA	413/369	$[^{13}C_8]$-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

Dwell time was 20, 50, or 100 msec for each transition. The individual transitions were summed to produce a "total ion chromatogram" (TIC), which was used for quantitation.

(1) Internal standards were used for samples analyzed on 8/26/15, 9/2/15, and 9/8/15.

3 Data Analysis

3.1 Calibration

8/20/15, 8/25/15, and 8/28/15 Analysis (External Standard Calibration): Samples were analyzed against an external standard calibration curve. Calibration standards were prepared by spiking known amounts of the stock solution containing the target analytes into 90:10 methanol: Milli-Q laboratory water. Calibration standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed on 8/20/15, 0.5 ng/mL to 100 ng/mL (nominal) were analyzed on 8/25/15, and 0.02 ng/mL to 2.5 ng/mL (nominal) were analyzed on 8/28/15. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL to 25 ng/mL (nominal) on 8/20/15, 0.5 ng/mL to 25 ng/mL (nominal) on 8/25/15, and 0.02 to 2.5 ng/mL (nominal) on 8/28/15. 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.

8/26/15 Analysis (Internal Standard Calibration): Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50 mL of 50:50 laboratory reagent water: methanol. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. A total of fourteen calibration standards ranging from 0.0125 ng/mL to 100 ng/mL (nominal) were analyzed. Of these calibration standards, ten contained the surrogates at concentrations ranging from 0.0125 ng/mL to 10 ng/mL (nominal). A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios or area counts and the resultant calibration curve confirmed accuracy of each curve point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

9/2/15 and 9/8/15 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 50 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 (nominal) 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 below.

Table 6. LOQ

Analyte	LOQ, ng/mL [1] 8/20/15 Analysis (ETS Buster)	LOQ, ng/mL [1] 8/20/15 Analysis (ETS Kirk)	LOQ, ng/mL [1] 8/25/15 Analysis	LOQ, ng/mL [2] 8/26/15 Analysis	LOQ, ng/mL [1] 8/28/15 Analysis	LOQ, ng/mL [3] 9/2/15 Analysis	LOQ, ng/mL [3] 9/8/15 Analysis
PFOA	0.0958	0.0479	0.479	0.0240	NA	NA	NA
PFBS	0.0200	0.0200	0.500	NA	0.0200	NA	NA
PFHS	0.0473	0.0189	0.473	0.0236	NA	NA	NA
PFOS	0.0464	0.0185	0.464	0.0232	NA	NA	NA
PFOSA	NA	NA	NA	NA	NA	0.0750	0.250

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 2.5 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. The reference standard used for the preparation of the mid and high level LCSs for PFHS on 8/19/15 and 8/21/15 and for all levels on 8/25/15, was not at a concentration sufficient for preparing at the desired concentration. In order to produce an appropriate on-column concentration, LCSs were spiked with PFHS post dilution. Surrogates [$^{13}C_4$]-PFOA and [$^{13}C_4$]-PFOS were also added post dilution when analyzed by external standard. The results in Table 7 for these LCSs are reported with the dilution factor applied.

Method ETS-8-044.3 states that the average recovery of LCSs at each spiking level must be within 80%-120% with a RSD ≤20%. All LCS samples met criteria with the following exceptions:

- LCSs prepared on 8/19/15 had multiple non-compliant recoveries for PFOA, PFBS, PFHS, and PFOS, indicating a preparation error. The LCSs were re-prepared on 8/21/15 for all analytes and added at the end of analytical batches k150820a and b150820b. All LCSs prepared on 8/21/15 met criteria except the high level LCSs had an average recovery of 123% for PFHS in batch b150820b. Only the LCSs prepared on 8/21/15 will be used to calculate analytical method uncertainty.
- For LCSs prepared on 8/25/15, the average recovery of the low and high level LCSs were 122% for PFHS.
- For LCSs prepared on 8/26/15, one low level LCS was calculated as an outlier for PFOS using the Q-test. The outlier is not included in the RPD or average LCS calculations nor is it used to calculate analytical method uncertainty.
- For LCSs prepared on 8/28/15, the low and mid-level LCSs had an average recovery of 121% and 127%, respectively for PFOS surrogate.

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 8/20/15 ETS Kirk	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-150821-1	95.8	83.0	86.7	100	96.1	96.1
LCS-150821-2	95.8	84.7	88.4	100	101	101
LCS-150821-3	95.8	90.9	94.9	100	101	101
Average ± %RSD	**90.0% ± 4.8%**			**99.4% ± 2.8%**		
LCS-150821-4	5000	4950	99.1	5000	5290	106
LCS-150821-5	5000	5000	100	5000	5280	106
LCS-150821-6	5000	4900	97.9	5000	5140	103
Average ± %RSD	**99.0% ± 1.1%**			**105% ± 1.6%**		
LCS-150821-7	35000	29800	85.2	35000	35800	102
LCS-150821-8	35000	29900	85.3	35000	35300	101
LCS-150821-9	35000	29900	85.3	35000	34800	99.3
Average ± %RSD	**85.3% ± 0.068%**			**101% ± 1.4%**		

ETS-8-044.3 External Calibration Analyzed 8/20/15 ETS Kirk	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-150821-1	94.6	93.6	99.0	92.7	86.8	93.6
LCS-150821-2	94.6	94.8	100	92.7	82.9	89.4
LCS-150821-3	94.6	97.5	103	92.7	91.5	98.7
Average ± %RSD	**101% ± 2.1%**			**93.9% ± 5.0%**		
LCS-150821-4	4730	5020	106	5000	4640	92.9
LCS-150821-5	4730	5030	107	5000	4640	92.9
LCS-150821-6	4730	4980	105	5000	4540	90.8
Average ± %RSD	**106% ± 0.94% (1)**			**92.2% ± 1.3%**		
LCS-150821-7	16600	17800	108	35000	33600	95.9
LCS-150821-8	16600	18100	109	35000	33600	95.9
LCS-150821-9	16600	17700	107	35000	33000	94.2
Average ± %RSD	**108% ± 0.93% (1)**			**95.3% ± 1.0%**		

(1) LCS spiked post dilution with dilution factor applied.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.
(3) LCS is an outlier and was not included in the accuracy or precision calculations.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 8/20/15 ETS Kirk	$[^{13}C_4]$-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-150821-1	99.8	88.8	89.0	95.6	85.5	89.4
LCS-150821-2	99.8	87.6	87.8	95.6	82.1	85.9
LCS-150821-3	99.8	90.9	91.1	95.6	85.6	89.6
Average ± %RSD	**89.3% ± 1.9% (1)**			**88.3% ± 2.4% (1)**		
LCS-150821-4	997	947	95.0	955	870	91.1
LCS-150821-5	997	974	97.7	955	885	92.7
LCS-150821-6	997	929	93.2	955	865	90.6
Average ± %RSD	**95.3% ± 2.4% (1)**			**91.5% ± 1.2% (1)**		

ETS-8-044.3 External Calibration Analyzed 8/20/15 ETS Buster	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-150821-1	95.8	84.5	88.2	100	106	106
LCS-150821-2	95.8	84.7	88.4	100	106	106
LCS-150821-3	95.8	90.6	94.5	100	111	111
Average ± %RSD	**90.4% ± 4.0%**			**108% ± 2.7%**		
LCS-150821-4	5000	5440	109	5000	5870	117
LCS-150821-5	5000	5310	106	5000	5830	117
LCS-150821-6	5000	5280	106	5000	5720	114
Average ± %RSD	**107% ± 1.6%**			**116% ± 1.5%**		
LCS-150821-7	35000	35600	102	35000	41000	117
LCS-150821-8	35000	35300	101	35000	39600	113
LCS-150821-9	35000	35200	101	35000	41200	118
Average ± %RSD	**101% ± 0.57%**			**116% ± 2.3%**		

(1) LCS spiked post dilution with dilution factor applied.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.
(3) LCS is an outlier and was not included in the accuracy or precision calculations.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 8/20/15 ETS Buster	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-150821-1	94.6	89.2	94.3	92.7	80.0	86.3
LCS-150821-2	94.6	92.2	97.4	92.7	66.4	71.7
LCS-150821-3	94.6	91.1	96.3	92.7	84.1	90.7
Average ± %RSD	**96.0% ± 1.6%**			**82.9% ± 12%**		
LCS-150821-4	4730	5380	114	5000	5360	107
LCS-150821-5	4730	5270	111	5000	5310	106
LCS-150821-6	4730	5320	113	5000	5390	108
Average ± %RSD	**113% ± 1.4% (1)**			**107% ± 0.93%**		
LCS-150821-7	16600	19800	120	35000	39500	113
LCS-150821-8	16600	20500	124	35000	39300	112
LCS-150821-9	16600	20800	126	35000	39200	112
Average ± %RSD	**123% ± 2.5% (1,2)**			**112% ± 0.51%**		

ETS-8-044.3 External Calibration Analyzed 8/20/15 ETS Buster	$[^{13}C_4]$-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-150821-1	99.8	104	104	95.6	91.5	95.7
LCS-150821-2	99.8	104	104	95.6	96.1	100
LCS-150821-3	99.8	103	103	95.6	97.2	102
Average ± %RSD	**104% ± 0.56% (1)**			**99.2% ± 3.2% (1)**		
LCS-150821-4	997	1070	107	955	1020	107
LCS-150821-5	997	1080	109	955	1040	108
LCS-150821-6	997	1040	105	955	1040	109
Average ± %RSD	**107% ± 1.9% (1)**			**108% ± 0.93% (1)**		

(1) LCS spiked post dilution with dilution factor applied.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.
(3) LCS is an outlier and was not included in the accuracy or precision calculations.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 8/25/15	PFOA (Linear + Branched)			PFBS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150825-1	5000	5630	113	5000	5980	120
LCS-150825-2	5000	5450	109	5000	5790	116
LCS-150825-3	5000	5550	111	5000	5900	118
Average ± %RSD	**111% ± 1.8%**			**118% ± 1.7%**		
LCS-150825-4	20000	21800	109	20000	24100	120
LCS-150825-5	20000	21900	109	20000	22900	114
LCS-150825-6	20000	21300	106	20000	23700	118
Average ± %RSD	**108% ± 1.6%**			**117% ± 2.6%**		
LCS-150825-7	70000	74000	106	70000	81900	117
LCS-150825-8	70000	73800	105	70000	83000	119
LCS-150825-9	70000	73600	105	70000	83000	119
Average ± %RSD	**105% ± 0.55%**			**118% ± 0.98%**		

ETS-8-044.3 External Calibration Analyzed 8/25/15	PFHS			PFOS (Linear + Branched)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150825-1	4720	5650	120	5000	5390	108
LCS-150825-2	4720	5750	122	5000	5440	109
LCS-150825-3	4720	5820	123	5000	5380	108
Average ± %RSD	**122% ± 1.3% (1,2)**			**108% ± 0.53%**		
LCS-150825-4	18900	22400	119	20000	22100	110
LCS-150825-5	18900	22400	119	20000	21700	108
LCS-150825-6	18900	22300	118	20000	21300	107
Average ± %RSD	**119% ± 0.49% (1)**			**108% ± 1.4%**		
LCS-150825-7	66000	80400	122	70000	78100	112
LCS-150825-8	66000	81300	123	70000	77400	111
LCS-150825-9	66000	80500	122	70000	77500	111
Average ± %RSD	**122% ± 0.47% (1,2)**			**111% ± 0.52%**		

(1) LCS spiked post dilution with dilution factor applied.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.
(3) LCS is an outlier and was not included in the accuracy or precision calculations.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 8/25/15	$[^{13}C_4]$-PFOA			$[^{13}C_4]$-PFOS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150825-1	1990	2320	117	1910	2120	111
LCS-150825-2	1990	2250	113	1910	2130	112
LCS-150825-3	1990	2270	114	1910	2160	113
Average ± %RSD	**115% ± 1.8% (1)**			**112% ± 0.89% (1)**		
LCS-150825-4	7970	8970	113	7630	8380	110
LCS-150825-5	7970	8660	109	7630	8150	107
LCS-150825-6	7970	8670	109	7630	8370	110
Average ± %RSD	**110% ± 2.1% (1)**			**109% ± 1.6% (1)**		

ETS-8-044.3 Internal Calibration Analyzed 8/26/15	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-150826-1	0.190	0.175	92.2	0.188	0.188	100
LCS-150826-2	0.190	0.181	95.4	0.188	0.192	102
LCS-150826-3	0.190	0.190	100	0.188	0.209	111
Average ± %RSD	**95.9% ± 4.1%**			**104% ± 5.6%**		
LCS-150826-4	24.0	23.4	97.7	23.7	23.2	97.9
LCS-150826-5	24.0	24.8	103	23.7	25.0	105
LCS-150826-6	24.0	23.9	99.4	23.7	24.8	105
Average ± %RSD	**100% ± 2.7%**			**103% ± 4.0%**		
LCS-150826-7	133	125	93.9	131	120	91.2
LCS-150826-8	133	128	96.1	131	121	92.2
LCS-150826-9	133	126	94.7	131	117	89.6
Average ± %RSD	**94.9% ± 1.2%**			**91.0% ± 1.4%**		

(1) LCS spiked post dilution with dilution factor applied.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.
(3) LCS is an outlier and was not included in the accuracy or precision calculations.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 8/26/15	PFOS (Linear + Branched)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150826-1	0.184	0.193	105
LCS-150826-2	0.184	0.189	103
LCS-150826-3	0.184	1.11	606 (3)
Average ± %RPD	**104% ± 1.9%**		
LCS-150826-4	23.2	24.1	104
LCS-150826-5	23.2	24.9	107
LCS-150826-6	23.2	24.2	104
Average ± %RSD	**105% ± 1.6%**		
LCS-150826-7	129	124	95.8
LCS-150826-8	129	120	93.0
LCS-150826-9	129	121	93.8
Average ± %RSD	**94.2% ± 1.5%**		

ETS-8-044.3 Internal Calibration Analyzed 8/26/15	$[^{13}C_4]$-PFOA			$[^{13}C_4]$-PFOS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150826-1	0.198	0.184	92.7	0.189	0.187	98.7
LCS-150826-2	0.198	0.189	95.3	0.189	0.184	97.5
LCS-150826-3	0.198	0.195	98.3	0.189	0.189	99.8
Average ± %RSD	**95.4% ± 2.9%**			**98.7% ± 1.2%**		
LCS-150826-4	2.00	2.14	107	1.91	2.12	111
LCS-150826-5	2.00	2.03	101	1.91	1.98	104
LCS-150826-6	2.00	2.00	99.9	1.91	1.98	104
Average ± %RSD	**103% ± 3.7%**			**106% ± 3.8%**		

(1) LCS spiked post dilution with dilution factor applied.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.
(3) LCS is an outlier and was not included in the accuracy or precision calculations.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 External Calibration Analyzed 8/28/15	PFBS			$[^{13}C_4]$-PFOS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150828-1	9.99	11.9	119	9.55	11.4	119
LCS-150828-2	9.99	11.0	110	9.55	11.6	121
LCS-150828-3	9.99	10.9	109	9.55	11.6	122
Average ± %RSD	**113% ± 4.9%**			**121% ± 1.3%** (1,2)		
LCS-150828-4	49.8	56.8	114	95.5	120	126
LCS-150828-5	49.8	54.9	110	95.5	123	129
LCS-150828-6	49.8	56.0	112	95.5	122	127
Average ± %RSD	**112% ± 1.8%**			**127% ± 1.2%** (1,2)		
LCS-150828-7	174	190	109			
LCS-150828-8	174	194	112			
LCS-150828-9	174	196	113			
Average ± %RSD	**111% ± 1.9%**					

ETS-8-044.3 Internal Calibration Analyzed 9/2/15	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150902-1	0.496	0.527	106
LCS-150902-2	0.496	0.511	103
LCS-150902-3	0.496	0.515	104
Average ± %RSD	**104% ± 1.5%**		
LCS-150902-4	49.6	49.8	100
LCS-150902-5	49.6	49.5	99.8
LCS-150902-6	49.6	50.7	102
Average ± %RSD	**101% ± 1.2%**		
LCS-150902-7	347	321	92.5
LCS-150902-8	347	323	93.1
LCS-150902-9	347	313	90.1
Average ± %RSD	**91.9% ± 1.7%**		

(1) LCS spiked post dilution with dilution factor applied.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.
(3) LCS is an outlier and was not included in the accuracy or precision calculations.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.3 Internal Calibration Analyzed 9/8/15	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-150908-1	0.496	0.481	96.9
LCS-150908-2	0.496	0.481	97.0
LCS-150908-3	0.496	0.489	98.6
Average ± %RSD	**97.5% ± 0.98%**		
LCS-150908-4	49.6	48.4	97.6
LCS-150908-5	49.6	47.9	96.5
LCS-150908-6	49.6	49.6	100
Average ± %RSD	**98.0% ± 1.8%**		
LCS-150908-7	347	318	91.6
LCS-150908-8	347	318	91.6
LCS-150908-9	347	316	91.0
Average ± %RSD	**91.4% ± 0.38%**		

(1) LCS spiked post dilution with dilution factor applied.
(2) LCSs do not meet acceptance criteria of 100 ± 20%.
(3) LCS is an outlier and was not included in the accuracy or precision calculations.

3.7 Analytical Method Uncertainty

Analytical uncertainty is based on historical QC data that is control charted and used to evaluate method accuracy and precision. The method uncertainty is calculated following ETS-12-012.3. The standard deviation is calculated for the set of accuracy results (in %) obtained for the QC samples. For method ETS-8-044.3, the most recent fifty QC samples were used. The expanded uncertainty is calculated by multiplying the standard deviation by a factor of 2, which corresponds to a confidence level of 95%.

The method uncertainty for PFHS using external calibration was calculated at ±16% following ETS-12-012.2; however, the method uncertainty was expanded to ±23% based on the recovery of the high level LCSs analyzed in batch b150820b.

Table 8. Analytical Method Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	7.17	±14%
PFBS	External	7.29	±15%
PFHS	External	NA	±23%
PFOS	External	7.33	±15%
PFOA	Internal	5.17	±10%
PFHS	Internal	5.65	±11%
PFOS	Internal	4.62	±9.2%
PFOSA	Internal	6.51	±13%

NA = Not Applicable

3.8 Field Matrix Spikes (FMS)

Target analyte field matrix spike samples were collected for select sampling points to verify that the analytical method is applicable for the collected matrix. Field matrix spikes are generated by adding a measured volume of field sample to a container spiked by the laboratory with the target analytes prior to shipping sample containers for sample collection. Field matrix spikes must be at least 50% of the analyte concentration to be considered an appropriate spike level. Field matrix spike recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The standards used for the preparation of the field matrix spiking solutions contained reference materials comprised of both linear and branched isomers for PFOS and only the linear isomer for PFOA. The stock solutions of PFOS and PFOA used to prepare the field matrix spikes were prepared from a different reference source than the one used to prepare the calibration standards. Field matrix spikes are presented in section 4 of this report.

In addition to target analyte field matrix spikes, select sample bottles contained stable isotope surrogate recovery spikes of $^{13}C_4$-PFOA and $^{13}C_4$-PFOS, which were added at a nominal concentration of 0.1 ng/mL to sample bottles prior to sample collection. The $^{13}C_4$- PFOA was selected to represent the C8 perfluorocarboxylic acid. The $^{13}C_4$-labeled PFOS was selected to represent the C4, C6 and C8 perfluorosulfonic acids and PFOSA. Surrogate matrix spike recoveries within method acceptance criteria of 100±30% confirm that "unknown" components in the sample matrix do not significantly interfere with the preparation and analysis of the analytes of interest. The surrogate spike recoveries are included in section 4 of this report.

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

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

Table 9. Field Matrix Spike Concentrations

Sampling Location	Spike Level	Final Concentration (ng/mL)				
		PFOA	PFBS	PFHS	PFOS	PFOSA
P121, L21, L31, P115, Effluent WWTP, B7	FMS	11.0	11.0	11.0	10.9	0.998
13, Collector pit, P56	FMS	100	100	100	100	10.0
BD24-3	FMS	1010	9.99	1010	1010	10.0
P119C, P27, P304, P264, P382	FMS	1000	99.9	1000	1000	100
P118A, P343, P378, PA111A	FMS	2010	9.99	2010	2010	10.0
D09, PP01, PP04, PP09, M4, P340, P374	FMS	2000	99.9	2000	2000	100
P343 (1)	FMS	1450	7.19	1450	1450	7.19
P382 (1)	FMS	875	87.2	873	873	87.3
Trip Blank	Low	11.0	11.0	11.0	10.9	0.998
	High	2000	99.9	2000	2000	100

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

4 Data Summary and Discussion

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

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 surrogate recovery standards met method acceptance criteria.

BD24-4: The initial sample (ISO15-0013-001) is not reportable for PFOSA. The area ratio (target analyte peak area counts over the internal standard area counts) for this sample was within the calibration range, however; the sample target analyte peak area counts for PFOSA were less than the peak area counts for the LOQ standard. Since the sample target analyte peak area counts were also less than twice the average area counts for the method blanks, the sample result is not reportable.

Sample locations L22, PP06, and P340: At least one analyte had a sample/sample duplicate RPD greater than 50%. The samples were re-analyzed and confirmed the high RPD values. Results from the initial analysis are reported.

PFOSA Analysis: All sample containers were pre-spiked with $^{13}C_8$-PFOSA and analyzed for PFOSA by internal standard calibration. During the review of the analysis it was noted that in most instances, sample sets that included a FMS sample had an area count response for $^{13}C_8$-PFOSA that was inconsistent with the area count response that was produced for the primary sample and sample duplicate, resulting in low FMS recoveries. To produce a more consistent internal standard response, additional internal standard was spiked into the sample bottle and the samples were diluted 1:5. However, several FMS recoveries were still below 70%; therefore, the method uncertainty was adjusted based on the FMS recovery. For the samples that did not have a FMS sample or the FMS was not calculated due to an inappropriate spike level, the lowest FMS recovery within each sampling zone was used to assess method uncertainty. The method uncertainty was not expanded for samples with FMS recoveries that met acceptance criteria.

- **Sample locations BD24-4, D10, D11, D14, P118B, P121, and P321 (2nd Aquifer zone)**: Locations from this zone that included FMS samples were: BD24-3, D09, P118A, and P121. The FMS recovery for locations BD24-3, D09, and P118A met method acceptance criteria, while sample location P121 had a FMS recovery of 44.6%. Since the recovery for location P121 was below 50%, this sample location and all other locations without a FMS sample in this zone have been flagged as qualitative. The results for these sampling locations were reviewed against historical results and found to be within the same order of magnitude, and had historical FMS recoveries within method acceptance criteria. While method ETS-8-044.3 indicates that results for sampling locations with FMS recoveries <50% should not be reported, the availability of historical data was used as justification for reporting the results, but flagging them as qualitative.

- **Sample locations 3M vijver, Blokkersdijkvijver – std, L21, L22, L31, L4, P114bis, P115, P116, and P119C (Blokkersdijk nature reserve zone)**: The FMS recovery was 62.4% for L21, 51.3% for L31, 56.2% for P115, and 56.0% for P119C. Since the lowest FMS recovery was 51.3%, the method uncertainty for all samples within this zone was expanded to ±49%.

- **Sample locations 12, 13, 5, and Bemalingsstation (Palingbeek & Tophatgracht zone)**: The FMS recovery was 61.9% for sample 13. Since this was the only FMS sample within the zone, the method uncertainty for all samples is expanded to ±38%.

- **Sample location Collector Put (Sewer zone)**: According to method recommendations, the FMS spike level must be at least 50% of the endogenous sample concentration and less than 10 times the endogenous sample concentration. For sampling location Collector Put, the spike level for PFOSA was just below the method recommendation at 45% of the endogenous level. To evaluate sample recovery for this zone, the FMS recovery was calculated for PFOSA and the FMS recovery is flagged as being below the recommended spike level. Since the FMS recovery was 57.5%, the method uncertainty is expanded to ±43%.

- **Sample locations K3, P27, P21B, P305, P42, and P56 (Source area – Building 16 zone)**: The FMS recovery was 54.0% for P27 and 57.5% for P56. Since the lowest FMS recovery was 54.0%, the method uncertainty for all samples within this zone is expanded to ±46%.

- **Sample locations L19, M4, P118C, P262, P263, P264, P265C, P341, P343, P371, P379, P380, and P381 (Source area – WWTP)**: Locations from this zone that included FMS samples were: M4, P264, P340, P343, P374, and P382. The FMS recovery for locations M4 and P343 could not be calculated as the spike level was not appropriate as compared to the endogenous sample concentration. The FMS recovery for locations P340, P374, and P382 met method acceptance criteria, while sample location P264 had a FMS recovery of 45.5%. Since the recovery is below 50%, this sample location and all other locations without a FMS sample in this zone have been flagged as qualitative. The results for these sampling locations were reviewed against historical results and found to be within the same order of magnitude, and had historical FMS recoveries within method acceptance criteria. While method ETS-8-044.3 indicates that results for sampling locations with FMS recoveries <50% should not be reported, the availability of historical data was used as justification for reporting the results, but flagging them as qualitative.

Table 10. Location ID: BD24-3

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-001	BD24-3; Sample	146	NA	95.6	NA
ISO15-0013-002	BD24-3; Sample Dup	148	NA	99.9	NA
ISO15-0013-003	BD24-3; FMS	1190	103	1130	102 (1)
Average Concentration (ng/mL) ± %RPD		**147 ng/mL ± 1.4%**		**97.8 ng/mL ± 4.4%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-001	BD24-3; Sample	1430	NA	4.01	NA
ISO15-0013-002	BD24-3; Sample Dup	1530	NA	4.24	NA
ISO15-0013-003	BD24-3; FMS	2520	103	12.6	84.8
Average Concentration (ng/mL) ± %RPD		**1480 ng/mL ± 6.8%**		**4.13 ng/mL ± 5.6%**	

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

Table 11. Location ID: D09

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-006	D09; Sample	143	NA	8370	NA
ISO15-0013-007	D09; Sample Dup	138	NA	7910	NA
ISO15-0013-008	D09; FMS	2260	106	10300	NC
Average Concentration (ng/mL) ± %RPD		**141 ng/mL ± 3.6%**		**8140 ng/mL ± 5.7%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-006	D09; Sample	1870	NA	22.1	NA
ISO15-0013-007	D09; Sample Dup	1840	NA	21.5	NA
ISO15-0013-008	D09; FMS	3790	96.8	113	91.2
Average Concentration (ng/mL) ± %RPD		**1860 ng/mL ± 1.6%**		**21.8 ng/mL ± 2.8%**	

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

Table 12. Location ID: P118A

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-015	P118A; Sample	1780	NA	3120	NA
ISO15-0013-016	P118A; Sample Dup	1810	NA	3260	NA
ISO15-0013-017	P118A; FMS	3930	106	5460	113
Average Concentration (ng/mL) ± %RPD		**1800 ng/mL ± 1.7%**		**3190 ng/mL ± 4.4%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-015	P118A; Sample	1210	NA	6.76	NA
ISO15-0013-016	P118A; Sample Dup	1250	NA	6.71	NA
ISO15-0013-017	P118A; FMS	3240	100	15.2	84.7
Average Concentration (ng/mL) ± %RPD		**1230 ng/mL ± 3.3%**		**6.74 ng/mL ± 0.74%**	

NA = Not Applicable

Table 13. Location ID: P121

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-020	P121; Sample	0.176	NA	0.0694	NA
ISO15-0013-021	P121; Sample Dup	0.160	NA	0.0670	NA
ISO15-0013-022	P121; FMS	11.9	107 (1)	12.4	112 (1)
Average Concentration (ng/mL) ± %RPD		**0.168 ng/mL ± 9.5%**		**0.0682 ng/mL ± 3.5%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-020	P121; Sample	2.74	NA	0.238	NA
ISO15-0013-021	P121; Sample Dup	2.49	NA	0.177	NA
ISO15-0013-022	P121; FMS	13.8	103	0.653	44.6 (2)
Average Concentration (ng/mL) ± %RPD		**2.62 ng/mL ± 9.6%**		**0.208 ng/mL ± 29% (3,4)**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) FMS concentration greater than 10 times the sample concentration.
(2) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%.
(3) Sample/sample duplicate RPD does not meet acceptance criteria of ≤20%.
(4) Sample results are considered qualitative due to field matrix spike recovery below 50%.

Table 14. Location ID: L21

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-029	L21; Sample	0.602	NA	0.284	NA
ISO15-0013-030	L21; Sample Dup	0.584	NA	0.264	NA
ISO15-0013-031	L21; FMS	12.8	111 (1)	12.5	111 (1)
Average Concentration (ng/mL) ± %RPD		**0.593 ng/mL ± 3.0%**		**0.274 ng/mL ± 7.3%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-029	L21; Sample	7.33	NA	0.190	NA
ISO15-0013-030	L21; Sample Dup	7.13	NA	0.153	NA
ISO15-0013-031	L21; FMS	19.7	114	0.794	62.4 (2)
Average Concentration (ng/mL) ± %RPD		**7.23 ng/mL ± 2.8%**		**0.172 ng/mL ± 22% (3,4)**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.
(2) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%.
(3) Sample/sample duplicate RPD does not meet acceptance criteria of ≤20%.
(4) Method uncertainty has been expanded to ±49%.

Table 15. Location ID: L31

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-034	L31; Sample	0.640	NA	0.551	NA
ISO15-0013-035	L31; Sample Dup	0.599	NA	0.496	NA
ISO15-0013-036	L31; FMS	9.44	80.5 (1)	11.0	95.2 (1)
Average Concentration (ng/mL) ± %RPD		**0.620 ng/mL ± 6.6%**		**0.524 ng/mL ± 11%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-034	L31; Sample	11.1	NA	0.416	NA
ISO15-0013-035	L31; Sample Dup	10.2	NA	0.294	NA
ISO15-0013-036	L31; FMS	19.4	80.1	0.867	51.3 (2)
Average Concentration (ng/mL) ± %RPD		**10.7 ng/mL ± 8.5%**		**0.355 ng/mL ± 34% (3,4)**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) FMS concentration greater than 10 times the sample concentration.
(2) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%.
(3) Sample/sample duplicate RPD does not meet acceptance criteria of ≤20%.
(4) Method uncertainty has been expanded to ±49%.

Table 16. Location ID: P115

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-041	P115; Sample	4.02	NA	2.00	NA
ISO15-0013-042	P115; Sample Dup	4.07	NA	1.99	NA
ISO15-0013-043	P115; FMS	13.9	89.9	12.2	92.8
Average Concentration (ng/mL) ± %RPD		**4.05 ng/mL ± 1.2%**		**2.00 ng/mL ± 0.50%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-041	P115; Sample	0.264	NA	0.155	NA
ISO15-0013-042	P115; Sample Dup	0.212	NA	0.0946	NA
ISO15-0013-043	P115; FMS	9.22	82.2 (1)	0.686	56.2 (3)
Average Concentration (ng/mL) ± %RPD		**0.238 ng/mL ± 22% (2)**		**0.125 ng/mL ± 48% (2,4)**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.
(2) Sample/sample duplicate RPD does not meet acceptance criteria of ≤20%.
(3) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%.
(4) Method uncertainty has been expanded to ±49%.

Table 17. Location ID: P119C

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-046	P119C; Sample	1320	NA	997	NA
ISO15-0013-047	P119C; Sample Dup	1310	NA	995	NA
ISO15-0013-048	P119C; FMS	2240	92.5	1980	98.4
Average Concentration (ng/mL) ± %RPD		**1320 ng/mL ± 0.76%**		**996 ng/mL ± 0.20%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-046	P119C; Sample	7430	NA	122	NA
ISO15-0013-047	P119C; Sample Dup	7210	NA	124	NA
ISO15-0013-048	P119C; FMS	7930	NC	179	56.0 (1)
Average Concentration (ng/mL) ± %RPD		**7320 ng/mL ± 3.0%**		**123 ng/mL ± 1.6% (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) Method uncertainty has been expanded to ±49%.

Table 18. Location ID: Effluent WWTP

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-049	Effluent WWTP; Sample	10.8	NA	11.8	NA
ISO15-0013-050	Effluent WWTP; Sample Dup	11.4	NA	11.7	NA
ISO15-0013-051	Effluent WWTP; FMS	21.6	95.8	21.8	91.4
Average Concentration (ng/mL) ± %RPD		**11.1 ng/mL ± 5.4%**		**11.8 ng/mL ± 0.85%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-049	Effluent WWTP; Sample	2.39	NA	<0.0750	NA
ISO15-0013-050	Effluent WWTP; Sample Dup	2.53	NA	<0.0750	NA
ISO15-0013-051	Effluent WWTP; FMS	11.8	85.5	0.951	95.3
Average Concentration (ng/mL) ± %RPD		**2.46 ng/mL ± 5.7%**		**<0.0750 ng/mL**	

NA = Not Applicable

Table 19. Location ID: PP01

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-052	PP01; Sample	88.2	NA	122	NA	36700	NA
ISO15-0013-053	PP01; Sample Dup	81.8	NA	122	NA	38200	NA
ISO15-0013-054	PP01; FMS	2080	99.8 (1)	236	114	NA (2)	NA (2)
Average Concentration (ng/mL) ± %RPD		**85.0 ng/mL ± 7.5%**		**122 ng/mL ± 0.0%**		**37500 ng/mL ± 4.0%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-052	PP01; Sample	7200	NA	58.6	NA
ISO15-0013-053	PP01; Sample Dup	6880	NA	60.4	NA
ISO15-0013-054	PP01; FMS	8470	NC	139	79.5
Average Concentration (ng/mL) ± %RPD		**7040 ng/mL ± 4.5%**		**59.5 ng/mL ± 3.0%**	

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

Table 20. Location ID: PP04

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-057	PP04; Sample	903	NA	965	NA	431	NA
ISO15-0013-058	PP04; Sample Dup	925	NA	993	NA	448	NA
ISO15-0013-059	PP04; FMS	2980	103	1100	NC	2580	107
Average Concentration (ng/mL) ± %RPD		**914 ng/mL ± 2.4%**		**979 ng/mL ± 2.9%**		**440 ng/mL ± 3.9%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-057	PP04; Sample	7120	NA	99.6	NA
ISO15-0013-058	PP04; Sample Dup	7970	NA	110	NA
ISO15-0013-059	PP04; FMS	10100	NC	178	73.2
Average Concentration (ng/mL) ± %RPD		**7550 ng/mL ± 11%**		**105 ng/mL ± 9.9%**	

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

Table 21. Location ID: PP09

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-068	PP09; Sample	765	NA	31.6	NA	464	NA
ISO15-0013-069	PP09; Sample Dup	773	NA	30.0	NA	458	NA
ISO15-0013-070	PP09; FMS	2670	95.1	132	101	2480	101
Average Concentration (ng/mL) ± %RPD		**769 ng/mL ± 1.0%**		**30.8 ng/mL ± 5.2%**		**461 ng/mL ± 1.3%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-068	PP09; Sample	2470	NA	12.3	NA
ISO15-0013-069	PP09; Sample Dup	2410	NA	12.2	NA
ISO15-0013-070	PP09; FMS	4270	91.5	105	92.8
Average Concentration (ng/mL) ± %RPD		**2440 ng/mL ± 2.5%**		**12.3 ng/mL ± 0.82%**	

NA = Not Applicable

Table 22. Location ID: 13

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-075	13; Sample	107	NA	29.2	NA
ISO15-0013-076	13; Sample Dup	105	NA	29.8	NA
ISO15-0013-077	13; FMS	198	92.0	127	97.5
Average Concentration (ng/mL) ± %RPD		**106 ng/mL ± 1.9%**		**29.5 ng/mL ± 2.0%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-075	13; Sample	281	NA	0.372	NA
ISO15-0013-076	13; Sample Dup	287	NA	0.364	NA
ISO15-0013-077	13; FMS	352	NC	6.56	61.9 (1)
Average Concentration (ng/mL) ± %RPD		**284 ng/mL ± 2.1%**		**0.368 ng/mL ± 2.2%** (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) Method uncertainty has been expanded to ±38%.

Table 23. Location ID: Collector put

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-082	Collector put; Sample	92.9	NA	30.1	NA
ISO15-0013-083	Collector put; Sample Dup	91.5	NA	27.6	NA
ISO15-0013-084	Collector put; FMS	195	103	136	107
Average Concentration (ng/mL) ± %RPD		**92.2 ng/mL ± 1.5%**		**28.9 ng/mL ± 8.7%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-082	Collector put; Sample	315	NA	22.7	NA
ISO15-0013-083	Collector put; Sample Dup	321	NA	22.0	NA
ISO15-0013-084	Collector put; FMS	435	NC	28.1	57.5 (1,2)
Average Concentration (ng/mL) ± %RPD		**318 ng/mL ± 1.9%**		**22.4 ng/mL ± 3.1%** (3)	

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) Although the FMS spike level was less than 50% of the endogenous level (45%), the FMS spike recovery was calculated was used to assess accuracy.
(3) Method uncertainty has been expanded to ±43%.

Table 24. Location ID: P27

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-087	P27; Sample	190	NA	419	NA	24.2	NA
ISO15-0013-088	P27; Sample Dup	196	NA	488	NA	28.6	NA
ISO15-0013-089	P27; FMS	1240	105	512	NC	1130	110 (1)
Average Concentration (ng/mL) ± %RPD		**193 ng/mL ± 3.1%**		**454 ng/mL ± 15%**		**26.4 ng/mL ± 17%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-087	P27; Sample	1970	NA	169	NA
ISO15-0013-088	P27; Sample Dup	1980	NA	161	NA
ISO15-0013-089	P27; FMS	2900	92.5	219	54.0 (2)
Average Concentration (ng/mL) ± %RPD		**1980 ng/mL ± 0.51%**		**165 ng/mL ± 4.8% (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.
(2) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%.
(3) Method uncertainty has been expanded to ±46%.

Table 25. Location ID: P304

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-092	P304; Sample	684	NA	266	NA	156	NA
ISO15-0013-093	P304; Sample Dup	665	NA	253	NA	143	NA
ISO15-0013-094	P304; FMS	1650	97.6	357	NC	1180	103
Average Concentration (ng/mL) ± %RPD		**675 ng/mL ± 2.8%**		**260 ng/mL ± 5.0%**		**150 ng/mL ± 8.7%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-092	P304; Sample	1070	NA	25.8	NA
ISO15-0013-093	P304; Sample Dup	1040	NA	24.8	NA
ISO15-0013-094	P304; FMS	2090	104	109	83.7
Average Concentration (ng/mL) ± %RPD		**1060 ng/mL ± 2.8%**		**25.3 ng/mL ± 4.0%**	

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

Table 26. Location ID: P56 (set 2)

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-156	P56; Sample	72.7	NA	15.7	NA	60.3	NA
ISO15-0013-157	P56; Sample Dup	72.8	NA	15.4	NA	58.9	NA
ISO15-0013-158	P56; FMS	181	108	134	118	171	111
Average Concentration (ng/mL) ± %RPD		**72.8 ng/mL ± 0.14%**		**15.6 ng/mL ± 1.9%**		**59.6 ng/mL ± 2.3%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-156	P56; Sample	222	NA	18.4	NA
ISO15-0013-157	P56; Sample Dup	217	NA	18.9	NA
ISO15-0013-158	P56; FMS	335	NC	24.4	57.5 (1)
Average Concentration (ng/mL) ± %RPD		**220 ng/mL ± 2.3%**		**18.7 ng/mL ± 2.7% (2)**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%.
(2) Method uncertainty has been expanded to ±46%.

Table 27. Location ID: M4

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-103	M4; Sample	381	NA	162	NA
ISO15-0013-104	M4; Sample Dup	414	NA	168	NA
ISO15-0013-105	M4; FMS	2540	107	2350	109 (1)
Average Concentration (ng/mL) ± %RPD		**398 ng/mL ± 8.3%**		**165 ng/mL ± 3.6%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-103	M4; Sample	6300	NA	265	NA
ISO15-0013-104	M4; Sample Dup	7070	NA	281	NA
ISO15-0013-105	M4; FMS	8980	NC	297	NC
Average Concentration (ng/mL) ± %RPD		**6690 ng/mL ± 12%**		**273 ng/mL ± 5.9% (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 results are considered qualitative based on the lowest field matrix spike recovery within the sampling zone being below 50%.

Table 28. Location ID: P264

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-112	P264; Sample	299	NA	168	NA
ISO15-0013-113	P264; Sample Dup	304	NA	166	NA
ISO15-0013-114	P264; FMS	1290	98.9	1190	102
Average Concentration (ng/mL) ± %RPD		**302 ng/mL ± 1.7%**		**167 ng/mL ± 1.2%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-112	P264; Sample	1890	NA	172	NA
ISO15-0013-113	P264; Sample Dup	1870	NA	173	NA
ISO15-0013-114	P264; FMS	2800	92.0	218	45.5 (1)
Average Concentration (ng/mL) ± %RPD		**1880 ng/mL ± 1.1%**		**173 ng/mL ± 0.58% (2)**	

NA = Not Applicable
(1) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%.
(2) Sample results are considered qualitative due to field matrix spike recovery below 50%.

Table 29. Location ID: P340

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-117	P340; Sample	2630	NA	583	NA
ISO15-0013-118	P340; Sample Dup	1670	NA	483	NA
ISO15-0013-119	P340; FMS	4300	108	2700	108
Average Concentration (ng/mL) ± %RPD		**2150 ng/mL ± 45% (1)**		**533 ng/mL ± 19%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-117	P340; Sample	16500	NA	170	NA
ISO15-0013-118	P340; Sample Dup	6710	NA	105	NA
ISO15-0013-119	P340; FMS	17300	NC	247	110
Average Concentration (ng/mL) ± %RPD		**11600 ng/mL ± 84% (1)**		**138 ng/mL ± 47% (1)**	

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

Table 30. Location ID: P343

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-122	P343; Sample	902	NA	636	NA
ISO15-0013-123	P343; Sample Dup	907	NA	612	NA
ISO15-0013-124	P343; FMS	2480	109	2200	109
Average Concentration (ng/mL) ± %RPD		**905 ng/mL ± 0.55%**		**624 ng/mL ± 3.8%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-122	P343; Sample	7040	NA	16.3	NA
ISO15-0013-123	P343; Sample Dup	6940	NA	16.4	NA
ISO15-0013-124	P343; FMS	9870	NC	19.4	NC
Average Concentration (ng/mL) ± %RPD		**6990 ng/mL ± 1.4%**		**16.4 ng/mL ± 0.61% (1)**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Sample results are considered qualitative based on the lowest field matrix spike recovery within the sampling zone being below 50%.

Table 31. Location ID: P374

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-127	P374; Sample	862	NA	1060	NA
ISO15-0013-128	P374; Sample Dup	897	NA	1110	NA
ISO15-0013-129	P374; FMS	2940	103	3610	126
Average Concentration (ng/mL) ± %RPD		**880 ng/mL ± 4.0%**		**1090 ng/mL ± 4.6%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-127	P374; Sample	3450	NA	30.1	NA
ISO15-0013-128	P374; Sample Dup	3210	NA	30.2	NA
ISO15-0013-129	P374; FMS	5400	104	113	82.9
Average Concentration (ng/mL) ± %RPD		**3330 ng/mL ± 7.2%**		**30.2 ng/mL ± 0.33%**	

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

Table 32. Location ID: P382

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-136	P382; Sample	494	NA	138	NA
ISO15-0013-137	P382; Sample Dup	497	NA	142	NA
ISO15-0013-138	P382; FMS	1480	113	1160	117
Average Concentration (ng/mL) ± %RPD		**496 ng/mL ± 0.61%**		**140 ng/mL ± 2.9%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-136	P382; Sample	1070	NA	104	NA
ISO15-0013-137	P382; Sample Dup	1060	NA	98.9	NA
ISO15-0013-138	P382; FMS	2030	110	164	71.6
Average Concentration (ng/mL) ± %RPD		**1070 ng/mL ± 0.94%**		**101 ng/mL ± 5.0%**	

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

Table 33. Location ID: B7

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-141	B7; Sample	29.8	NA	17.6	NA
ISO15-0013-142	B7; Sample Dup	30.1	NA	17.2	NA
ISO15-0013-143	B7; FMS	38.7	NC	29.0	105
Average Concentration (ng/mL) ± %RPD		**30.0 ng/mL ± 1.0%**		**17.4 ng/mL ± 2.3%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-141	B7; Sample	395	NA	2.43	NA
ISO15-0013-142	B7; Sample Dup	389	NA	2.26	NA
ISO15-0013-143	B7; FMS	391	NC	2.61	NC
Average Concentration (ng/mL) ± %RPD		**392 ng/mL ± 1.5%**		**2.35 ng/mL ± 7.2%**	

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

Table 34. Location ID: P378

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-146	P378; Sample	373	NA	360	NA
ISO15-0013-147	P378; Sample Dup	375	NA	368	NA
ISO15-0013-148	P378; FMS	2430	102	2700	116
Average Concentration (ng/mL) ± %RPD		**374 ng/mL ± 0.53%**		**364 ng/mL ± 2.2%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-146	P378; Sample	1290	NA	1.85	NA
ISO15-0013-147	P378; Sample Dup	1330	NA	1.82	NA
ISO15-0013-148	P378; FMS	3520	110	11.2	93.7
Average Concentration (ng/mL) ± %RPD		**1310 ng/mL ± 3.1%**		**1.84 ng/mL ± 1.6%**	

NA = Not Applicable

Table 35. Location ID: PA111A

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-151	PA111A; Sample	945	NA	1210	NA
ISO15-0013-152	PA111A; Sample Dup	932	NA	1180	NA
ISO15-0013-153	PA111A; FMS	2870	96.1	3610	120
Average Concentration (ng/mL) ± %RPD		**939 ng/mL ± 1.4%**		**1200 ng/mL ± 2.5%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-151	PA111A; Sample	2640	NA	12.4	NA
ISO15-0013-152	PA111A; Sample Dup	2550	NA	12.2	NA
ISO15-0013-153	PA111A; FMS	4670	103	20.1	78.0
Average Concentration (ng/mL) ± %RPD		**2600 ng/mL ± 3.5%**		**12.3 ng/mL ± 1.6%**	

NA = Not Applicable

Table 36. Trip Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-161	Trip Blank; Sample	<0.0240	NA	<0.200	NA	<0.0236	NA
ISO15-0013-162	Trip Blank; FMS Low	11.3	103	12.5	114	10.8	98.2
ISO15-0013-163	Trip Blank; FMS High	2110	106	114.0	114	2250	113

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO15-0013-161	Trip Blank; Sample	<0.0232	NA	<0.250	NA
ISO15-0013-162	Trip Blank; FMS Low	11.4	104	0.995	99.7
ISO15-0013-163	Trip Blank; FMS High	2190	110	93.1	93.1

NA = Not Applicable

Table 37. Surrogate Recovery Standard Results [1]

		Percent Recovery (%)	
3M LIMS ID	**Sample Description**	**[$^{13}C_4$]-PFOA**	**[$^{13}C_4$]-PFOS**
ISO15-0013-001	BD24-3; Sample	103	101
ISO15-0013-002	BD24-3; Sample Dup	103	101
ISO15-0013-003	BD24-3; FMS	98.2	97.2
ISO15-0013-004	BD24-4; Sample	98.3	95.6
ISO15-0013-005	BD24-4; Sample Dup	99.6	100
ISO15-0013-006	D09; Sample	103	100
ISO15-0013-007	D09; Sample Dup	100	99.0
ISO15-0013-008	D09; FMS	95.7	92.8
ISO15-0013-009	D10; Sample	95.5	99.1
ISO15-0013-010	D10; Sample Dup	93.7	96.9
ISO15-0013-011	D11; Sample	102	89.8
ISO15-0013-012	D11; Sample Dup	98.0	84.4
ISO15-0013-013	D14; Sample	98.7	98.5
ISO15-0013-014	D14; Sample Dup	91.8	91.7
ISO15-0013-015	P118A; Sample	99.7	99.3
ISO15-0013-016	P118A; Sample Dup	96.5	97.6
ISO15-0013-017	P118A; FMS	100	99.5
ISO15-0013-018	P118B; Sample	98.5	93.9
ISO15-0013-019	P118B; Sample Dup	99.5	98.0
ISO15-0013-020	P121; Sample	89.8 [2]	90.1 [2]
ISO15-0013-021	P121; Sample Dup	91.2 [2]	86.2 [2]
ISO15-0013-022	P121; FMS	102 [2]	84.8 [2]
ISO15-0013-023	P321; Sample	92.5	88.1
ISO15-0013-024	P321; Sample Dup	93.3	89.8
ISO15-0013-025	3M vijver; Sample	98.7	98.1
ISO15-0013-026	3M vijver; Sample Dup	101	97.8
ISO15-0013-027	Blokkersdijkvijver std; Sample	97.2	94.3
ISO15-0013-028	Blokkersdijkvijver std; Sample Dup	94.2	94.3
ISO15-0013-029	L21; Sample	90.2 [2]	87.5 [2]
ISO15-0013-030	L21; Sample Dup	90.2 [2]	79.8 [2]
ISO15-0013-031	L21; FMS	94.2 [2]	87.9 [2]
ISO15-0013-032	L22; Sample	98.8 [2]	91.7 [2]
ISO15-0013-033	L22; Sample Dup	98.3 [2]	86.6 [2]
ISO15-0013-034	L31; Sample	100	98.7
ISO15-0013-035	L31; Sample Dup	102	100
ISO15-0013-036	L31; FMS	98.5	97.6
ISO15-0013-037	L4; Sample	96.0	92.8
ISO15-0013-038	L4; Sample Dup	100	97.4
ISO15-0013-039	P114bis; Sample	93.7	92.4
ISO15-0013-040	P114bis; Sample Dup	95.6	92.0
ISO15-0013-041	P115; Sample	99.0	97.9
ISO15-0013-042	P115; Sample Dup	98.3	95.1
ISO15-0013-043	P115; FMS	97.9	94.2

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 37 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
ISO15-0013-044	P116; Sample	97.1	92.7	NA
ISO15-0013-045	P116; Sample Dup	96.6	95.4	NA
ISO15-0013-046	P119C; Sample	96.8	89.0	NA
ISO15-0013-047	P119C; Sample Dup	94.7	86.5	NA
ISO15-0013-048	P119C; FMS	96.4	86.0	NA
ISO15-0013-049	Effluent WWTP; Sample	99.4	97.6	NA
ISO15-0013-050	Effluent WWTP; Sample Dup	101	97.4	NA
ISO15-0013-051	Effluent WWTP; FMS	100	100	NA
ISO15-0013-052	PP01; Sample	99.1	94.3	114
ISO15-0013-053	PP01; Sample Dup	94.9	92.0	119
ISO15-0013-054	PP01; FMS	98.5	96.6	NA
ISO15-0013-055	PP02; Sample	91.7	84.4	NA
ISO15-0013-056	PP02; Sample Dup	94.5	84.9	NA
ISO15-0013-057	PP04; Sample	94.5	91.6	NA
ISO15-0013-058	PP04; Sample Dup	95.1	90.9	NA
ISO15-0013-059	PP04; FMS	90.9	84.7	NA
ISO15-0013-060	PP05; Sample	92.9	89.7	NA
ISO15-0013-061	PP05; Sample Dup	88.9	87.8	NA
ISO15-0013-062	PP06; Sample	96.5	90.3	NA
ISO15-0013-063	PP06; Sample Dup	95.0	90.3	NA
ISO15-0013-064	PP07; Sample	83.6	79.2	NA
ISO15-0013-065	PP07; Sample Dup	98.0	90.9	NA
ISO15-0013-066	PP08; Sample	97.6	87.2	NA
ISO15-0013-067	PP08; Sample Dup	94.6	85.9	NA
ISO15-0013-068	PP09; Sample	96.1	91.4	NA
ISO15-0013-069	PP09; Sample Dup	95.5	90.5	NA
ISO15-0013-070	PP09; FMS	93.5	87.3	NA
ISO15-0013-071	PP10; Sample	94.3	89.0	NA
ISO15-0013-072	PP10; Sample Dup	96.3	88.9	NA
ISO15-0013-073	12; Sample	93.3	88.9	NA
ISO15-0013-074	12; Sample Dup	91.9	86.6	NA
ISO15-0013-075	13; Sample	92.1	87.9	NA
ISO15-0013-076	13; Sample Dup	93.7	90.8	NA
ISO15-0013-077	13; FMS	90.9	86.0	NA
ISO15-0013-078	5; Sample	89.3	88.2	NA
ISO15-0013-079	5; Sample Dup	93.4	89.2	NA
ISO15-0013-080	Bemalingsstation; Sample	91.4	87.4	NA
ISO15-0013-081	Bemalingsstation; Sample Dup	86.2	86.6	NA
ISO15-0013-082	Collector put; Sample	101	96.0	NA
ISO15-0013-083	Collector put; Sample Dup	107	103	NA
ISO15-0013-084	Collector put; FMS	104	100	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 37 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
ISO15-0013-085	K3; Sample	110	103	119
ISO15-0013-086	K3; Sample Dup	106	102	115
ISO15-0013-087	P27; Sample	113	104	NA
ISO15-0013-088	P27; Sample Dup	110	103	NA
ISO15-0013-089	P27; FMS	107	101	NA
ISO15-0013-090	P21B; Sample	103	99.5	116
ISO15-0013-091	P21B; Sample Dup	97.1	99.1	126
ISO15-0013-092	P304; Sample	108	105	NA
ISO15-0013-093	P304; Sample Dup	109	103	NA
ISO15-0013-094	P304; FMS	103	103	NA
ISO15-0013-095	P305; Sample	108	105	NA
ISO15-0013-096	P305; Sample Dup	110	111	NA
ISO15-0013-097	P42; Sample	110	101	NA
ISO15-0013-098	P42; Sample Dup	113	108	NA
ISO15-0013-099	P56; Sample	112	113	NA
ISO15-0013-100	P56; Sample Dup	106	102	NA
ISO15-0013-101	L19; Sample	111	99.7	NA
ISO15-0013-102	L19; Sample Dup	108	101	NA
ISO15-0013-103	M4; Sample	113	107	NA
ISO15-0013-104	M4; Sample Dup	109	106	NA
ISO15-0013-105	M4; FMS	111	110	NA
ISO15-0013-106	P118C; Sample	115	109	NA
ISO15-0013-107	P118C; Sample Dup	111	103	NA
ISO15-0013-108	P262; Sample	112	102	NA
ISO15-0013-109	P262; Sample Dup	107	103	NA
ISO15-0013-110	P263; Sample	112	111	NA
ISO15-0013-111	P263; Sample Dup	105	102	NA
ISO15-0013-112	P264; Sample	103	101	NA
ISO15-0013-113	P264; Sample Dup	114	108	NA
ISO15-0013-114	P264; FMS	104	104	NA
ISO15-0013-115	P265C; Sample	110	106	NA
ISO15-0013-116	P265C; Sample Dup	110	106	NA
ISO15-0013-117	P340; Sample	102	86.0	122
ISO15-0013-118	P340; Sample Dup	107	96.0	117
ISO15-0013-119	P340; FMS	100	85.9	118
ISO15-0013-120	P341; Sample	102	101	NA
ISO15-0013-121	P341; Sample Dup	106	97.7	NA
ISO15-0013-122	P343; Sample	113	108	NA
ISO15-0013-123	P343; Sample Dup	106	101	NA
ISO15-0013-124	P343; FMS	110	109	NA
ISO15-0013-125	P371; Sample	110	102	NA
ISO15-0013-126	P371; Sample Dup	113	106	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 37 continued. Surrogate Recovery Standard Results (1)

		Percent Recovery (%)			
3M LIMS ID	Sample Description	[$^{13}C_4$]-PFOA	[$^{13}C_4$]-PFOS	[$^{13}C_4$]-PFOA Re-analysis	[$^{13}C_4$]-PFOS Re-analysis
ISO15-0013-127	P374; Sample	110	102	NA	NA
ISO15-0013-128	P374; Sample Dup	108	99.1	NA	NA
ISO15-0013-129	P374; FMS	98.8	102	NA	NA
ISO15-0013-130	P379; Sample	111	108	NA	NA
ISO15-0013-131	P379; Sample Dup	116	110	NA	NA
ISO15-0013-132	P380; Sample	107	106	NA	NA
ISO15-0013-133	P380; Sample Dup	113	112	NA	NA
ISO15-0013-134	P381; Sample	108	107	NA	NA
ISO15-0013-135	P381; Sample Dup	112	114	NA	NA
ISO15-0013-136	P382; Sample	106	107	NA	NA
ISO15-0013-137	P382; Sample Dup	109	108	NA	NA
ISO15-0013-138	P382; FMS	107	107	NA	NA
ISO15-0013-141	B7; Sample	107	95.6	NA	NA
ISO15-0013-142	B7; Sample Dup	110	107	NA	NA
ISO15-0013-143	B7; FMS	106	97.4	NA	NA
ISO15-0013-144	P372; Sample	111	103	NA	NA
ISO15-0013-145	P372; Sample Dup	106	106	NA	NA
ISO15-0013-146	P378; Sample	109	107	NA	NA
ISO15-0013-147	P378; Sample Dup	107	108	NA	NA
ISO15-0013-148	P378; FMS	101	103	NA	NA
ISO15-0013-149	PA109A; Sample	110	109	NA	NA
ISO15-0013-150	PA109A; Sample Dup	114	110	NA	NA
ISO15-0013-151	PA111A; Sample	112	109	NA	NA
ISO15-0013-152	PA111A; Sample Dup	109	104	NA	NA
ISO15-0013-153	PA111A; FMS	100	104	NA	NA
ISO15-0013-154	PA112; Sample	93.7	90.2	NA	NA
ISO15-0013-155	PA112; Sample Dup	102	97.0	NA	NA
ISO15-0013-156	P56; Sample	103	99.1	NA	NA
ISO15-0013-157	P56; Sample Dup	104	103	NA	NA
ISO15-0013-158	P56; FMS	103	104	NA	NA
ISO15-0013-159	P262; Sample	113	104	NA	NA
ISO15-0013-160	P262; Sample Dup	104	101	NA	NA
ISO15-0013-161	Trip Blank; Sample	106	106	96.8 (2)	85.0 (2)
ISO15-0013-162	Trip Blank; FMS Low	105	106	NA	NA
ISO15-0013-163	Trip Blank; FMS High	107	103	NA	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.

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 10-37 of this report.

6 Data / Sample Retention

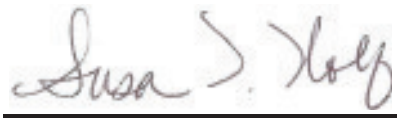
All remaining sample and associated project data (hardcopy and electronic) will be archived according to 3M Environmental Laboratory standard operating procedures.

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
Reason: I am the author of this document
Date: 2015.10.02 09:50:58 -05'00'

Chelsie Grochow, 3M Report Author



Digitally signed by Susan T. Wolf
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Reason: I have reviewed this document
Date: 2015.10.02 09:12:42 -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: 2015.10.05 09:16:57 -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 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
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Quality Assurance Representative

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SGS

3M Corporate Environmental Programs
Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC14-05296

3M Lab Request Number: E14-2082

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 4th, 2014

Requester

Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
Tel: [redacted]

Registered office: Noorderlaan 87 B-2030 Antwerpen H.R. Antwerpen 141.810 BTW BE 404.882.750 Belfius 550-3560000-93
All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction - Summary.

At the request (Lab Request Number: E14-2082) of Robert A. Paschke, 3M Manager Corporate Environmental Programs, water samples were collected on October 20th 2014 from 2 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
Collector Put	39.2	20.8	86.8	249	12.5
Effluent WWTP	2339	5.88	3.72	<0.364	<0.311

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)
Collector Put	100
Effluent WWTP	100
Field Trip Blank	100

2.2. Direct injection.

All samples and associated quality control samples were prepared using a modified procedure of ECO/AV/IAC/064 "Bepaling door directe injectie van pergefluoreerde componenten in water en sediment 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
Collector Put	1.0	0.100	Solution A	5.0	-
Effluent WWTP	1.0	0.100	Solution A	5.0	-
Effluent WWTP (PFBS)	0.5	0.100	Solution A	10.0	-
Field Trip Blank	1.0	0.100	Solution A	10.0	-

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

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ^{13}C-PFOA, PFOS, FOSA and ^{13}C-PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetrie C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
^{13}C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
PFBS	2.08 - 2.50	298.93/79.82	40	28	45
		298.93/98.78	40	29	45
PFHS	2.45 - 3.05	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.80 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
^{13}C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ^{13}C-PFOA, PFOS, FOSA and ^{13}C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of ± 25% (± 30% for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.0510 to 0.136 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.311 to 0.828 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 blank was 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 Spike (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
22 Oct 2014	LCS1	80	79.8	86.7	90.6	84.9	93.4	87.7	87.4

3.6. QC Lab Standards (QCs)

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

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C-PFOA	PFOS	FOSA	^{13}C-PFOS
16 July 2014	QC Lab Low inj1	10	103	91.9	121	102	100	103	101
	QC Lab High inj1	100	99.0	88.0	121	92.9	108	93.5	102
	QC Lab Low inj2	10	111	101	124	98.8	111	110	114
	QC Lab High inj2	100	108	97.0	120	100	119	104	115

3.7. Equation.

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
Collector Put	39.2	20.8	86.8	249	12.5
Effluent WWTP	2339	5.88	3.72	<0.364	<0.311
Field Trip Blank	<1.51	<0.810	<0.797	<0.663	<0.566

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

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
Collector Put	112	93.6	124	101
Effluent WWTP	123	103	134	109
Field Trip Blank	107	89.6	119	96.9

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 Marleen Coetsiers (ERM Belgium).

8. Signatures.



Geert De Smet, Date November 5th, 2014
Lab Operations Manager



Marc Van Ryckeghem, Date November 5th, 2014
Business Unit Manager Environmental Laboratories

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

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

3M Corporate Environmental Programs.
Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC15-00181-001

3M Lab Request Number: E15-0044

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: February 27th, 2015

Requester

Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
Tel: ++1 651 737 3634

Registered office: Noorderlaan 87 B-2030 Antwerpen H.R. Antwerpen 141.810 BTW BE 404.882.750 Dexia 550-3560000-93
All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction - Summary.

At the request (Lab Request Number: E15-0044) of Robert A. Paschke, 3M Manager Corporate Environmental Programs, water samples were collected between January 26th and 28th, and February 5th, 2015 from 22 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C-labeled analogue of PFOA ($C_6F_{13}{}^{13}CF_2{}^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C-labeled analogue of PFOS ($C_7F_{15}{}^{13}CF_2SO3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}\ SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Geert De Smet. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles.
Table 1 summarizes the sample results. All PFBS, PFHS, PFOA, PFOS and FOSA results have an accuracy of ± 30%.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
BD24-3		72.4	126	912	<1.32
D09		5913	135	1114	<14.5
P121		<0.877	<0.826	5.60	<1.32
P119C		80.0	193	1194	<14.5
Effluent WWTP		6.97	17.0	9.5	<1.32
Bemalingsstation		6.17	14.1	23.8	<1.32
Collector Put		11.2	51.3	91.8	3.96
P27	1608	16.0	176	734	93.9
P304	103	74.6	560	549	<14.5
P305	70.6	179	224	375	<14.5
P42	23.4	359	443	2468	<14.5
M4		212	1725	9018	138.1
P118C		71.7	228	1028	<14.5
P264		238	437	1048	<14.5
P265C		<9.65	86.9	508	15.2
P371		27.0	159	481	<14.5
P374		411	613	1252	<14.5
P381		117	610	697	<14.5
P382		61.4	423	532	<14.5
B7		<0.877	0.977	7.20	<1.32
P378		234	320	444	<14.5
PA112		62.0	277	212	21.5

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)
BD24-3	100
D09	100
P121	100
P119C	100
Effluent WWTP	100
Bemalingsstation	100
Collector Put	100
P27	100
P304	100
P305	100
P42	100
M4	100
P118C	100
P264	100
P265C	100
P371	100
P374	100
P381	100
P382	100
B7	100
P378	100
PA112	100
Field Trip Blank	100

2.2. Direct injection.

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

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
BD24-3	1.0	0.100	Solution A	10.0	-
D09	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P121	1.0	0.100	Solution A	10.0	-
P119C	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
Effluent WWTP	1.0	0.100	Solution A	10.0	-
Bemalingsstation	1.0	0.100	Solution A	10.0	-
Collector Put	1.0	0.100	Solution A	10.0	-
P27	1.0	0.100	Solution A	10.0	-
P304	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P305	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P42	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
M4	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P118C	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P264	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P265C	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P371	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P374	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P381	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P382	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
B7	1.0	0.100	Solution A	10.0	-
P378	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PA112	1.0	0.100	Solution A	10.0	-
Field Trip Blank	1.0	0.100	Solution A	10.0	-

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

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ^{13}C-PFOA, PFOS, FOSA and ^{13}C-PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetrie C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
^{13}C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
PFBS	2.08 - 2.50	298.93/79.82	40	28	45
		298.93/98.78	40	29	45
PFHS	2.45 - 3.05	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.80 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
^{13}C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ^{13}C-PFOA, PFOS, FOSA and ^{13}C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of ± 25% (± 30% for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.0608 to 0.119 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.775 to 14.5 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within ± 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
25 February 2015	LCS1	80	75.4	83.1	99.9	91.6	79.5	75.6	82.9

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
16 July 2014	QC Lab Low inj1	10	90.0	96.7	122	98.1	81.3	99.2	96.6
	QC Lab High inj1	100	87.0	81.8	120	87.9	88.2	82.9	86.8
	QC Lab Low inj2	10	102	111	115	87.4	105	125	89.3
	QC Lab High inj2	100	79.2	76.6	111	83.0	85.1	85.6	78.5

3.7. Equations.

Recovery.

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

4. Data Summary.

The following tables summarize the results for the sample locations.

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

	PFBS	PFHS	PFOA	PFOS	FOSA
	ng/ml	ng/ml	ng/ml	ng/ml	ng/ml
BD24-3		72.4	126	912	<1.32
D09		5913	135	1114	<14.5
P121		<0.877	<0.826	5.60	<1.32
P119C		80.0	193	1194	<14.5
Effluent WWTP		6.97	17.0	9.5	<1.32
Bemalingsstation		6.17	14.1	23.8	<1.32
Collector Put		11.2	51.3	91.8	3.96
P27	1608	16.0	176	734	93.9
P304	103	74.6	560	549	<14.5
P305	70.6	179	224	375	<14.5
P42	23.4	359	443	2468	<14.5
M4		212	1725	9018	138.1
P118C		71.7	228	1028	<14.5
P264		238	437	1048	<14.5
P265C		<9.65	86.9	508	15.2
P371		27.0	159	481	<14.5
P374		411	613	1252	<14.5
P381		117	610	697	<14.5
P382		61.4	423	532	<14.5
B7		<0.877	0.977	7.20	<1.32
P378		234	320	444	<14.5
PA112		62.0	277	212	21.5
Field Trip Blank	<0.675	<0.877	<0.826	<0.775	<1.32

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

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
BD24-3	122	102	102	82.5
D09	124	104	119	96.5
P121	122	102	119	96.4
P119C	104	87.0	103	83.7
Effluent WWTP	110	91.8	114	92.2
Bemalingsstation	109	91.5	119	96.5
Collector Put	117	97.9	111	90.5
P27	115	96.4	101	82.3
P304	113	94.4	106	85.8
P305	122	102	108	87.8
P42	90.5	75.6	99.1	80.5
M4	119	99.8	98.1	79.7
P118C	114	95.2	104	84.7
P264	109	91.4	105	85.4
P265C	115	95.7	108	87.7
P371	103	86.3	104	84.8
P374	117	97.8	108	88.0
P381	101	84.6	115	93.3
P382	93.7	78.3	101	82.1
B7	115	96.5	116	94.3
P378	110	92.0	110	89.3
PA112	113	94.1	112	91.1
Field Trip Blank	121	101	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 Ruben Maes (ERM Belgium).

8. Signatures.



Geert De Smet,
Lab Operations Manager Date March 2nd, 2015



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories Date March 2nd, 2015

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

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

3M Corporate Environmental Programs.
Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC15-00181-002

3M Lab Request Number: E15-0044

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: February 27th, 2015

Requester

Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
Tel: [redacted]

Registered office: Noorderlaan 87 B-2030 Antwerpen H.R. Antwerpen 141.810 BTW BE 404.882.750 Dexia 550-3560000-93
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1. Introduction - Summary.

At the request (Lab Request Number: E15-0044) of Robert A. Paschke, 3M Manager Corporate Environmental Programs, water samples were collected on January 27th 2015 from 4 locations and analyzed for the following perfluorinated compounds:

- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$) (and its ^{13}C-labeled analogues)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$) (and its ^{13}C-labeled analogues)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$) (and its ^{13}C-labeled analogues)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}\ SO_2NH_2$) (and its ^{13}C-labeled analogue)
- ^{13}C-labeled analogue of PFUdA ($C_4F_{21}{}^{13}C_6F_2{}^{13}COO^-$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared one sample container for each sampling location under the direction of Geert De Smet. Each empty container was marked with a "fill to here" line and was fortified with a surrogate recovery spike and an internal standard spike, prior to being sent to the field for sample collection.

Table 1 summarizes the sample results with their uncertainty. All PFHS, PFOA, PFOS and FOSA results have an accuracy of ± 30% unless noted otherwise. See Section 4 of the report for additional information on the method QC used to assess method uncertainty.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)			
	PFHS	PFOA	PFOS	FOSA
L21	0.120	0.433	3.98	0.0336 (b)
L22	0.284	0.607	13.5	0.0349 (c)
L31	0.519	0.584	6.09	<0.025 (b)
P115	1.89	4.53	0.542	<0.025 (a)

(a) The accuracy for FOSA for P115 is ± 55%.
(b) The accuracy for FOSA for L21 and L31 is ± 60%.
(c) The accuracy for FOSA for L22 is ± 65%.

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory. Based on the concentrations as reported in previous reports, all bottles were 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 samples collected and spikes added to each bottle.

Table 2. Sample Collection and Spike Information.

Sample Identification	Nominal Final Volume Collected (ml)	Volume of Spike Solution (mL)	Spike Solution Description	Final Nominal Spike Concentration (ng/mL)	
				^{13}C-PFC-SS	^{13}C-PFC-IS
L21	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
L22	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
L31	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
P115	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0

Solution A = 1000 ng/mL (nominal) ^{13}C-PFC Surrogate Recovery Standards
Solution B = 1500 ng/mL (nominal) ^{13}C-PFC Internal Standards

2.2. Extraction.

All samples, calibration standards, and associated quality control samples were extracted using a modified procedure of ECO/AV/IAC/064 “Bepaling van pergefluoreerde componenten in water met hoge druk vloeistof-chromatografie electrospray ionisatie tandem massaspectrometrie (LC-ESI-MS-MS)”. Briefly, an amount of sample (see table 3) was loaded, onto a pre-conditioned Waters tC18 solid phase extraction (SPE) cartridge (Sep-Pak, 1g, 6cc) using a vacuum manifold. The loaded SPE cartridges were then eluted with 5 mL of methanol using vacuum.

Table 3. Sample amount used.

Sample	Amount of Sample used (mL)	Concentration Factor
L21	40	8
L22	40	8
L31	40	8
P115	40	8
Field Trip Blank	40	8

2.3. Analysis.

All solutions and extracts were analyzed for the PFCs (PFHS, PFOA, PFOS, FOSA) and the surrogate recovery standards ($^{13}C_2$-PFHS, $^{13}C_4$-PFOA, $^{13}C_4$-PFOS, $^{13}C_7$-PFUdA) and internal standards ($^{13}C_3$-PFHS, $^{13}C_8$-PFOA, $^{13}C_8$-PFOS, $^{13}C_8$-FOSA) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetrie C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
$^{13}C_3$-PFHS	2.00 - 3.00	402.0 /80.0	60	38	51
$^{13}C_2$-PFHS	2.30 - 3.30	403.0/84.0	60	38	51
$^{13}C_8$-PFOS	3.00 - 4.00	507.0/80.0	60	48	56
$^{13}C_4$-PFOA	2.30 - 3.50	417.0 /372.0	100	11	14
$^{13}C_8$PFOA	2.30 - 3.50	421.0 /376.0	60	11	14
PFHS	2.00 - 3.00	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.30 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
$^{13}C_4$-PFOS	3.00 - 4.00	503.0 /80.0	60	48	56
$^{13}C_7$PFUdA	4.30 - 5.60	570.0 /525.0	60	12	17
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
$^{13}C_8$FOSA	4.30 - 5.70	506.0/78.0	60	34	44

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Extracted Calibration Standard.

Extracted calibration standards were prepared by spiking known amounts of stock solutions containing PFHS, PFOA, PFOS, FOSA and ^{13}C-labeled analogues into 40 mL of HPLC water. Each spiked water standard was then extracted in the same manner as the collected samples. A total of 12 spiked standards ranging from 0.005 ng/mL to 100 ng/mL (nominal) were prepared. Each curve point contains the mixture of internal standards at a nominal concentration of 1 ng/mL. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. The calibration curve will be generated by taking the ratio of the standard peak area counts over the internal standard peak area counts to fit the data for each analyte. Each extracted calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of ± 25% (± 30% for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The 0.025ng/mL (nominal) reporting limit is a practical quantitation limit (PQL) required by the requester and it is possible that the samples contain target analytes at quantifiable concentrations below the PQL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within ± 25%, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by loading 40 mL of HPLC water onto a Waters tC18 solid phase extraction (SPE) cartridge (SEP-Pak, 1g, 6cc) and eluting with 5 mL of methanol using the same extraction procedure as the samples. Method blanks were prepared to evaluate the levels of background contamination in the overall extraction process (glassware, SPE cartridges, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carryover.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of HPLC water, spiked with the surrogate recovery standards and internal standards, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCSs)

Low (0.125 ng/mL nominal concentration) and high (2.5 ng/mL nominal concentration) lab control spikes were prepared and analyzed in duplicate. LCSs were prepared by spiking known amounts of the analytes and surrogates into 40 mL of HPLC water to produce the desired concentration. The spiked water samples were extracted and analyzed in the same manner as the samples.
All LCSs produced recoveries within the method acceptance criteria of ± 25% for accuracy.
All LCSs produced recoveries within the method acceptance criteria of ± 15% RPD for precision, except for *$^{13}C_4$-PFOA* LCSH (16%) and $^{18}C_2$-PFHS LCSL (22%).

Table 7 summarize the LCS recovery results.

Table 7. Lab Control Spike Results.

Extraction date	Description	Nominal Spike Level (ng/mL)	Percent Recovery							
			$^{13}C_4$-PFOA	$^{13}C_4$-PFOS	$^{13}C_2$-PFHS	$^{13}C_7$-PFuDA	PFHS	PFOA	PFOS	FOSA
04 February 2015	LCSL01	0.125	98.9	92.9	99.8	85.6	77.1	125	80.2	90.2
	LCSL02	0.125	88.0	101	80.1	81.1	83.8	108	87.4	104
	Average		**93.5**	**96.8**	**90.0**	**83.4**	**80.5**	**117**	**83.8**	**97.0**
	%RPD		**12**	**8.0**	**22 (a)**	**5.4**	**8.3**	**14**	**8.6**	**14**
	LCSH 01	2.5	92.6	93.8	113	109	78.8	100	81.9	82.5
	LCSH 02	2.5	108	102	106	102	75.0	106	83.7	84.8
	Average		**100**	**98.1**	**109**	**105**	**76.9**	**103**	**82.8**	**83.7**
	%RPD		**16 (a)**	**8.7**	**6.2**	**6.7**	**4.9**	**5.7**	**2.2**	**2.7**

(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 8 and 9 summarizes the results for the 4 sample locations.

Table 8. Sample Results PFHS and PFOA.

	PFHS	$^{13}C_2$-PFHS	PFOA	$^{13}C_4$-PFOA
	ng/ml	%Rec	ng/ml	%Rec
Field Trip Blank	<0.025	105	<0.025	97.0
L21	0.120	79.5	0.433	80.0
L22	0.284	83.1	0.607	84.8
L31	0.519	90.1	0.584	89.1
P115	1.89	90.6	4.53	89.8

Table 9. Sample Results PFOS and FOSA.

	PFOS	$^{13}C_4$-PFOS	FOSA	$^{13}C_7$-PFuDA
	ng/ml	%Rec	ng/ml	%Rec
Field Trip Blank	<0.025	112	<0.025	113
L21	3.98	80.0	0.0336	43.0 (a)
L22	13.5	91.5	0.0349	38.1 (a)
L31	6.09	87.8	<0.025	41.9 (a)
P115	0.542	90.8	<0.025	45.2 (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 P115 is ± 55%.
- The accuracy for FOSA for L21 and L31 is ± 60%.
- The accuracy for FOSA for L22 is ± 65%.

5. Conclusion.

- The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130% for four 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 Ruben Maes (ERM Belgium).

8. Signatures.



Geert De Smet,
Lab Operations Manager Date March 2nd, 2015



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories Date March 2nd, 2015

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

3M Corporate Environmental Programs.
Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC15-02056-001

3M Lab Request Number: E15-0427

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

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: June 1st, 2015

Requester

Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
Tel: [redacted]

Registered office: Noorderlaan 87 B-2030 Antwerpen H.R. Antwerpen 141.810 BTW BE 404.882.750 Dexia 550-3560000-93
All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction - Summary.

At the request (Lab Request Number: E15-00) of Robert A. Paschke, 3M Manager Corporate Environmental Programs, water samples were collected between April 21st and 28th, 2015 from 10 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C-labeled analogue of PFOA ($C_6F_{13}{}^{13}CF_2{}^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C-labeled analogue of PFOS ($C_7F_{15}{}^{13}CF_2SO3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}\ SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Geert De Smet. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles.
Table 1 summarizes the sample results. All PFBS, PFHS, PFOA, PFOS and FOSA results have an accuracy of ± 30%.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
P321		958	4003	4178	<12.6
Effluent WWTP		14.2	15.7	10.0	<1.15
Bemalingsstation		19.0	27.9	42.3	<1.15
Collector Put		19.4	54.8	186	<1.15
PP02	22.3	2131	565	18979	<12.6
PP05	10359	3645	1153	268	<12.6
PP07	43.1	162	360	3719	<12.6
PP09	39.3	443	688	2158	<12.6
12		16.6	87.8	255	<1.15
5		17.0	27.3	56.3	<1.15

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

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

Table 2 below details the samples collected.

Table 2. Sample Collection.

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

2.2. Direct injection.

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

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
P321	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
Effluent WWTP	1.0	0.100	Solution A	10.0	-
Bemalingsstation	1.0	0.100	Solution A	10.0	-
Collector Put	1.0	0.100	Solution A	10.0	-
PP02	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP05	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP07	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP09	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
12	1.0	0.100	Solution A	10.0	-
5	1.0	0.100	Solution A	10.0	-
Field Trip Blank	1.0	0.100	Solution A	10.0	-

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

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ^{13}C-PFOA, PFOS, FOSA and ^{13}C-PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetrie C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
^{13}C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
PFBS	2.08 - 2.50	298.93/79.82	40	28	45
		298.93/98.78	40	29	45
PFHS	2.45 - 3.05	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.80 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
^{13}C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ^{13}C-PFOA, PFOS, FOSA and ^{13}C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of ± 25% (± 30% for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.103 to 0.181 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 1.15 to 22.1 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within ± 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
13 May 2015	LCS1	80	102	101	97.9	99.7	102	92.9	98.8

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing PFBS, PFHS, PFOA, ^{13}C-PFOA, PFOS, FOSA and ^{13}C-PFOS (QC Lab Low and QC Lab High).
The QCs produced recoveries within the method acceptance criteria of ± 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
16 July 2014	QC Lab Low inj1	10	121	113	124	119	110	119	122
	QC Lab High inj1	100	107	94.3	123	102	116	103	99.0
	QC Lab Low inj2	10	87.7	94.4	120	109	106	116	106
	QC Lab High inj2	100	86.7	75.4	122	97.8	97.8	86.6	89.0

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		958	4003	4178	<12.6
Effluent WWTP		14.2	15.7	10.0	<1.15
Bemalingsstation		19.0	27.9	42.3	<1.15
Collector Put		19.4	54.8	186	<1.15
PP02	22.3	2131	565	18979	<12.6
PP05	10359	3645	1153	268	<12.6
PP07	43.1	162	360	3719	<12.6
PP09	39.3	443	688	2158	<12.6
12		16.6	87.8	255	<1.15
5		17.0	27.3	56.3	<1.15
Field Trip Blank	<2.00	<1.49	<1.62	<1.60	<1.15

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

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
P321	96.8	80.9	125	102
Effluent WWTP	123	103	121	97.9
Bemalingsstation	127	106	126	102
Collector Put	128	107	125	101
PP02	94.0	78.6	98.5	80.0
PP05	91.5	76.5	112	91.1
PP07	85.8	71.7	103	84.0
PP09	86.9	72.6	109	88.4
12	125	104	115	93.8
5	123	103	114	92.3
Field Trip Blank	132	110	135	110

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 June 1st, 2015



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories Date June 1st, 2015

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3M Corporate Environmental Programs.
Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC15-02056-002

3M Lab Request Number: E15-0427

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

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: June 1st, 2015

Requester

Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
Tel: [redacted]

Registered office: Noorderlaan 87 B-2030 Antwerpen H.R. Antwerpen 141.810 BTW BE 404.882.750 Dexia 550-3560000-93
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1. Introduction - Summary.

At the request (Lab Request Number: E15-00) of Robert A. Paschke, 3M Manager Corporate Environmental Programs, water samples were collected between April 20th and 28th, 2015 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 (filter)	0.175	1.25	0.0294	0.0461 (a)
Blokkersdijkvijver (nt filter)	0.955	1.30	7.06	0.0377
P114 bis	3.75	4.50	37.4	<0.025 (b)

(a) The accuracy for FOSA for Blokkersdijkvijver (filter) is ± 76.5%.
(b) The accuracy for FOSA for P114bis is ± 68.5%.

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory. Based on the concentrations as reported in previous reports, all bottles were 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 samples collected and spikes added to each 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 (filter)	400	0.1	Solution A	0.25	-
		0.25	Solution B	-	1.0
Blokkersdijkvijver (nt filter)	400	0.1	Solution A	0.25	-
		0.25	Solution B	-	1.0
P114 bis	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 (filter)	40	8
Blokkersdijkvijver (nt filter)	40	8
P114 bis	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)	0.003

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 5 to 7.

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 carryover.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of HPLC water, spiked with the surrogate recovery standards and internal standards, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCSs)

Low (0.125 ng/mL nominal concentration) and high (2.5 ng/mL nominal concentration) lab control spikes were prepared and analyzed in duplicate. LCSs were prepared by spiking known amounts of the analytes and surrogates into 40 mL of HPLC water to produce the desired concentration. The spiked water samples were extracted and analyzed in the same manner as the samples.
All LCSs produced recoveries within the method acceptance criteria of ± 25% for accuracy.
All LCSs produced recoveries within the method acceptance criteria of ± 15% RPD for precision, except for PFOS LCSH (23%).
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
13 May 2015	LCSL01	0.125	113	108	101	118	102	119	111	88.3
	LCSL02	0.125	114	100	102	104	95.7	121	110	79.1
	Average		**113**	**104**	**101**	**111**	**99.1**	**120**	**111**	**83.7**
	%RPD		**0.5**	**7.5**	**1.1**	**13**	**6.8**	**1.8**	**0.4**	**11**
	LCSH 01	2.5	90.1	85.2	95.9	81.2	97.0	110	80.6	96.2
	LCSH 02	2.5	104	95.3	105	89.7	102	112	102	95.9
	Average		**96.9**	**90.3**	**100**	**85.3**	**99.6**	**111**	**91.2**	**96.1**
	%RPD		**14**	**11**	**9.1**	**10**	**5.1**	**1.9**	**23(a)**	**0.3**

(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 4 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	94.4	<0.025	95.4
Blokkersdijkvijver (filter)	0.175	110	1.25	109
Blokkersdijkvijver (nt filter)	0.955	98.6	1.30	105
P114 bis	3.75	96.6	4.50	102

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	88.3	<0.025	94.5
Blokkersdijkvijver (filter)	0.0294	98.5	0.0461	23.9(a)
Blokkersdijkvijver (nt filter)	7.06	103	0.0377	72.8
P114 bis	37.4	101	<0.025	31.5(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 Blokkersdijkvijver (filter) is ± 76.5%.
- The accuracy for FOSA for P114bis is ± 68.5%.

5. Conclusion.

- The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130% for two sample locations.
- Lab control spike recoveries fell within the method acceptance criteria of 25%.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Ruben Maes (ERM Belgium).

8. Signatures.



Geert De Smet,
Lab Operations Manager Date June 1st, 2015



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories Date June 1st, 2015

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

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

ANALYTICAL REPORT

IAC15-03861

3M Lab Request Number: E15-0533

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

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: August 13th, 2015

Requester

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

Registered office: Noorderlaan 87 B-2030 Antwerpen H.R. Antwerpen 141.810 BTW BE 404.882.750 Dexia 550-3560000-93
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1. Introduction - Summary.

At the request (Lab Request Number: E15-0533) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected on July 16th, 17th, 22nd, 23rd, 24th, 27th, 28th and 30th, 2015 from 24 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C-labeled analogue of PFOA ($C_6F_{13}{}^{13}CF_2{}^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C-labeled analogue of PFOS ($C_7F_{15}{}^{13}CF_2SO3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}\ SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Geert De Smet. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles.
Table 1 summarizes the sample results. All PFBS, PFHS, PFOA, PFOS and FOSA results have an accuracy of ± 30%.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
BD24-3		65.3	142	1177	<10.0
BD24-4		2.27	10.1	3.54	<0.315
D10		174	518	<28.0	<9.99
D11		46.7	35.0	718	<9.99
D14		<0.928	2.04	<0.884	<0.315
P118A		2948	1848	979	<18.3
P118B		2805	2344	11355	<18.3
Effluent WWTP		13.6	12.8	<0.884	<0.315
Bemalingsstation		58.6	67.2	96.2	<0.465
Collector Put		30.3	87.4	431	16.3
K3	1671	79341	129	4177	<18.3
P21B	8893	12817	13575	46411	<18.3
P56	20.1	80.3	70.3	276	12.9
L19		268	256	4700	97.4
P262		213	390	3962	<18.3
P263		948	2444	13056	<18.3
P340		550	841	769	<18.3
P341		581	1123	7190	<18.3
P343		980	1039	9830	<18.3
P379		36.9	100	1426	49.0
P380		379	276	1461	76.8
P372		87.5	86.9	2378	<9.99
PA109A		123	49.4	175	<3.33
PA111A		2162	1007	3454	<9.99

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

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

Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
BD24-3	100
BD24-4	100
D10	100
D11	100
D14	100
P118A	100
P118B	100
Effluent WWTP	100
Bemalingsstation	100
Collector Put	100
K3	100
P21B	100
P56	100
L19	100
P262	100
P263	100
P340	100
P341	100
P343	100
P379	100
P380	100
P372	100
PA109A	100
PA111A	100
Field Trip Blank	100

2.2. Direct injection.

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

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
BD24-3	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
BD24-4	1.0	0.100	Solution A	1.0	-
D10	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
D11	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
D14	1.0	0.100	Solution A	1.0	-
P118A	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P118B	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
Effluent WWTP	1.0	0.100	Solution A	1.0	-
Bemalingsstation	1.0	0.100	Solution A	2.0	-
Collector Put	1.0	0.100	Solution A	10.0	-
K3	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P21B	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P56	1.0	0.100	Solution A	2.0	-
L19	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P262	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P263	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P340	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P341	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P343	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P379	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P380	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P372	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
PA109A	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
PA111A	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
Field Trip Blank	1.0	0.100	Solution A	10.0	-

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

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ^{13}C-PFOA, PFOS, FOSA and ^{13}C-PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetrie C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
^{13}C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
PFBS	2.08 - 2.50	298.93/79.82	40	28	45
		298.93/98.78	40	29	45
PFHS	2.45 - 3.05	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.80 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
^{13}C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ^{13}C-PFOA, PFOS, FOSA and ^{13}C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of ± 25% (± 30% for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.0610 to 0.442 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.128 to 54.0 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
07 August 2015	LCS1	80	76.7	83.1	119	80.0	103	117	79.4

3.6. QC Lab Standards (QCs)

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

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C-PFOA	PFOS	FOSA	^{13}C-PFOS
02 July 2015	QC Lab Low inj1	10	92.0	84.9	123	109	112	95.5	93.9
	QC Lab High inj1	100	77.5	78.5	123	89.5	114	90.0	85.2
	QC Lab Low inj2	10	124	124	101	78.7	119	106	91.2
	QC Lab High inj2	100	120	124	97.6	76.0	120	121	91.6

3.7. Equations.

Recovery.

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

4. Data Summary.

The following tables summarize the results for the sample locations.

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

	PFBS	PFHS	PFOA	PFOS	FOSA
	ng/ml	ng/ml	ng/ml	ng/ml	ng/ml
BD24-3		65.3	142	1177	<10.0
BD24-4		2.27	10.1	3.54	<0.315
D10		174	518	<28.0	<9.99
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P379		36.9	100	1426	49.0
P380		379	276	1461	76.8
P372		87.5	86.9	2378	<9.99
PA109A		123	49.4	175	<3.33
PA111A		2162	1007	3454	<9.99
Field Trip Blank	<1.60	<4.91	<0.680	<4.67	<1.15

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

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
BD24-3	115	96.0	107	87.3
BD24-4	121	101	98.5	80.0
D10	113	94.8	125	102
D11	103	86.1	126	102
D14	121	101	98.2	79.8
P118A	91.8	76.8	122	99.4
P118B	84.2	70.4	111	90.0
Effluent WWTP	126	105	116	94.2
Bemalingsstation	125	104	116	94.2
Collector Put	131	110	129	105
K3	114	94.9	148	120
P21B	85.1	71.2	126	103
P56	125	105	125	101
L19	124	104	135	110
P262	100	83.7	132	107
P263	93.5	78.2	130	106
P340	121	101	130	105
P341	112	93.4	141	114
P343	108	90.5	139	113
P379	110	92.0	138	112
P380	131	110	147	119
P372	128	107	148	120
PA109A	108	90.3	132	108
PA111A	131	109	145	118
Field Trip Blank	107	89.4	142	115

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 August 13th, 2015



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories Date August 13th, 2015

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

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

ANALYTICAL REPORT

IAC15-03864

3M Lab Request Number: E15-0533

Analysis of Total Petroleum Hydrocarbons in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
Haven 407
B-2030 Antwerpen

Author: Sandra Graré
Date issued: September 02nd, 2015

Requester

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

Registered office: Noorderlaan 87 B-2030 Antwerpen H.R. Antwerpen 141.810 BTW BE 404.882.750 Belfius 550-3560000-93
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1. Introduction.

At the request (Lab Request Number: E15-0533) of Jim Kotsmith, 3M Manager Corporate Environmental Programs, water samples were collected on July 30th 2015 from 2 locations and analyzed for:

- Total Petroleum Hydrocarbons (TPH)

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

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

2.2. Analysis.

2.2.1. Mineral oil.

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

2.2.2. Volatile compounds.

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

3. Results.

Table 1-2 summarizes the sample results.

Table 1. Mineral oil results.

Sample identification	Concentration (in µg/L)				
	Fraction C-10 -C-12	Fraction C-12 -C-20	Fraction C-20 -C-30	Fraction C-30 -C-40	Mineral oil
P18	180	1000	850	93	2100
P28	*	*	*	*	*

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

Table 2. Volatile compounds.

Sample identification	Concentration (in µg/L)					
	Benzene	Ethylbenzene	m- + p-xylene	o-xylene	Sum xylene	Toluene
P18	< 5.0	< 5.0	< 3.0	< 1.5	< 4.5	< 5.0
P28	< 5.0	< 5.0	< 3.0	< 1.5	< 4.5	13

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 September 2nd, 2015



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories Date September 2nd, 2015

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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.

servaco
laboratorium voor industriële analyses

Servaco n.v.
Vlamingstraat 19
8560 Wevelgem
Tel. 056 43 27 30
Fax 056 40 41 85
e-mail: info@servaco.be

Pullaar 207
2870 Puurs
Tel. 03 860 04 90
Fax 03 860 04 99

BELAC 052-TEST

ANALYSERAPPORT

Rapportnummer : 1429116

Opdrachtomschrijving : 0243999 3M

Project :
Projectomschrijving :

ERM
Marleen Coetsiers
Posthoflei 5 6
2600 Antwerpen-Berchem

Monsternamerapport : niet beschikbaar

Wevelgem, 28-10-2014

Geachte

Gelieve hierbij het rapport van bovenvermeld onderzoek te vinden.
Dit rapport omvat 3 Pagina(s)



Dr. N.E. Van Landuyt
Algemeen Directeur

Monsteromschrijving

1429116-01	bodem	Bunker 1
	Verpakking Agl	Staal bekomen via ophaling
1429116-02	bodem	Bunker 2
	Verpakking Agl	Staal bekomen via ophaling
1429116-03	bodem	Bunker 3
	Verpakking Agl	Staal bekomen via ophaling

Resultaten

Code Servaco		1429116-01	1429116-02	1429116-03
Monsternamedatum		21-10-2014	21-10-2014	21-10-2014
Ontvangstdatum :		22-10-2014	22-10-2014	22-10-2014
Omschrijving component				
droge stof	% (g/100g)	58.8	48.8	33.6
bads : !	Startdatum analyse	23-10-2014 *	23-10-2014 *	23-10-2014 *
pH in 1M KCl		8.59	8.21	8.17
baph_KCl : !	Startdatum analyse	27-10-2014	27-10-2014	27-10-2014
organische stof	% DS	4.1	5.6	5.7
baos : !	Startdatum analyse	27-10-2014	27-10-2014	27-10-2014
lutum gehalte (fraktie <2µm)	% DS	2.9	6.6	19.1
balut : !	Startdatum analyse	24-10-2014	24-10-2014	24-10-2014

Opmerkingen

1429116-01

bads : geur (AT)

1429116-02

bads : geur (AT)

1429116-03

bads : geur (AT)

Bijlagen:

Methode-omschrijving:

bads !	Droge stof op bodem, afval, bodemslib en waterzuiveringsslib - gravimetrie 105°C (CMA/2/II/A.1)
balut !	lutumbepaling op bodem en pasteus afval - bezinkingstechniek (CMA/2/II/A.6)
baos !	organisch koolstofgehalte op bodem en bodemslib - spectrofotometrisch (CMA/2/II/A.10)
baph_KCl !	zuurtegraad (pH) (gravimetrisch 1 + 5 - 1M KCl) op bodem - electrometrie (CMA/2/II/A.20)

!:geaccrediteerde methode voor de monstertypes terug te vinden in de bijlage van het BELAC-certificaat nr. 052-TEST.De beproevingsresultaten hebben enkel betrekking op de beproefde objecten. Dit verslag mag alleen gedeeltelijk worden gereproduceerd met schriftelijke toestemming van Servaco.De meetonzekerheid en omschrijving van de vermelde onderzoeksmethoden zijn op aanvraag ter beschikking evenals eventueel aangewende verpakkingscode's

Opmerkingen i.v.m de houdbaarheid :

-

Bijlage 3

Overzicht veldmetingen en analyseresultaten

Overzicht veldmetingen

Project	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933
Location	12	12	12	13	13	13	3M vijver	3M vijver	3M vijver	5	5	5	B3
Filter top													
Filter bottom													
Date	20-10-2015	20-10-2014	21-04-2015	20-10-2015	20-10-2014	21-04-2015	20-10-2015	20-10-2014	21-04-2015	20-10-2015	20-10-2014	21-04-2015	01-08-2014
EC (µs/cm)	903	863	711	895	835	720	741	543	509	1242	1127	905	655
pH	7,58	7,97	7,17	7,71	7,69	6,95	7,95	7,47	7,55	7,58	7,86	7,12	6,84
ORP	120,1	82,9	0	112,8	102,7	0	110,3	154,3	0	107,4	89,3	0	-45,2
Temperature	10,54	13,31	13,97	10,58	13,01	13,29	10,64	13,66	13,62	9,51	13,01	13,06	16,63
Oxigen (mg/l)	0	2,24	0	0	2,84	0	0,17	1,1	0	0	0,98	0	0

Overzicht veldmetingen

Project	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933
Location	B3-bis	B7	B7	B7	BD24-3	BD24-3	BD24-3	BD24-4	BD24-4	Bemalingssta	Bemalingssta	Bemalingssta	Blokkersdijk
Filter top	250	180	180	180	1700	1700	1700	2200	2200				
Filter bottom	350	380	380	380	1800	1800	1800	2400	2400				
Date	22-10-2015	28-07-2015	01-08-2014	27-01-2015	28-07-2015	30-07-2014	27-01-2015	28-07-2015	30-07-2014	20-10-2015	20-10-2014	21-04-2015	20-10-2015
EC (µs/cm)	419	422	293	281	702	757	665	1157	12,91	1363	1223	982	570
pH	7,36	7,47	7,33	7,75	7,43	7,26	6,65	7,14	6,96	7,58	7,89	7,18	7,11
ORP	-70,1	0	81,3	0	-93,8	-354,1	-180,9	-124,6	-335,4	120,1	97,7	0	99,6
Temperature	13,29	14,2	17,21	7,8	13,31	12,56	9,36	13,46	13,51	10,54	13,22	14,2	10,22
Oxigen (mg/l)	0	1,7	1,1	6,99	0	0	0	0	0	0	0,51	0	0,14

Overzicht veldmetingen

Project	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933
Location	Blokkersdijk	Blokkersdijk	Collector pit	Collector pit	D11	D11	D14	D14	D9	D9	D9	Effluent WW	Effluent WW
Filter top					1490	1490	1550	1550	1300	1300	1300		
Filter bottom					1590	1590	1650	1650	1500	1500	1500		
Date	20-10-2014	21-04-2015	22-10-2015	21-04-2015	28-07-2015	31-07-2014	27-07-2015	30-07-2014	19-10-2015	31-07-2014	26-01-2015	22-10-2015	21-04-2015
EC (µs/cm)	424	458	500	619	376	738	2877	3450	973	1167	853	5276	3821
pH	8,17	7,92	7,68	7,76	7,37	6,97	6,91	6,89	7,02	7,14	6,57	7,57	7,84
ORP	120	0	-51	66	137,1	-270,6	-94,9	0	-113,2	0	-119,6	-93,4	60,5
Temperature	13,91	13,32	15,15	14,88	13,98	14,87	14,5	17,1	16,3	19	13,64	15,26	16,07
Oxigen (mg/l)	4,84	0	0	0	0	0	0	0,42	0	0,34	0	0,17	0

Overzicht veldmetingen

Project	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933
Location	K1	K3	L19	L19	L21	L21	L21	L22	L31	L31	L4	L4	L4
Filter top			280	280	300	300	300	200	120	120	200	200	200
Filter bottom			480	480	500	500	500	400	320	320	400	400	400
Date	31-07-2014	31-07-2014	22-07-2015	26-01-2015	28-07-2015	30-07-2014	27-01-2015	21-10-2015	28-07-2015	27-01-2015	21-10-2015	20-10-2014	27-01-2015
EC (µs/cm)	1860	634	848	393	363	382	388	385	506	497	699	696	216
pH	7,4	7,31	7,18	6,62	7,47	7,5	7,66	7,43	7,11	7,43	7,33	6,64	6,97
ORP	0	0	0	-102,9	153,8	0	0	-54,7	-58,4	0	-148,9	-176,4	-139,2
Temperature	19,8	19,8	25,1	9,58	15,92	16,6	8,5	13,5	15,59	7,5	13,36	15,12	8,3
Oxigen (mg/l)	0,54	0,61	2,1	7,02	0	0,24	0,28	0	0	7,66	0	0	1,44

Overzicht veldmetingen

Project	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933
Location	L4	M4	M4	P1065	P114bis	P114bis	P114bis	P114bis	P114bis	P114bis	P115	P115	P116
Filter top	200	100	100	0	360	360	360	360	360	360			250
Filter bottom	400	300	300	0	460	460	460	460	460	460			350
Date	21-04-2015	27-07-2015	26-01-2015	30-07-2014	21-10-2015	23-07-2015	30-07-2014	20-10-2014	27-01-2015	21-04-2015	30-07-2014	27-01-2015	21-10-2015
EC (µs/cm)	326	949	772	469	2628	1507	1825	2113	1515	1665	3440	3260	612
pH	6,16	1,06	6,73	7,02	6,97	7,05	7,04	6,5	7,18	7,17	6,96	6,98	7,4
ORP	-108,4	0	-101,2	-169,5	-117	0	0	-306,8	0	-154,2	0	0	-77,9
Temperature	12,7	18	9,64	22,53	13,82	16,6	14,1	16,02	10,7	12,69	14,6	10,5	13,54
Oxigen (mg/l)	3,03	7,2	8,35	0,86	0	0	0,15	0	0,23	0	0,18	0,19	0

Overzicht veldmetingen

Project	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933
Location	P116	P116	P116	P116	P118A	P118A	P118B	P118C	P118C	P119C	P119C	P119C	P119C
Filter top	250	250	250	250	2250	2250	1300			250	250	250	250
Filter bottom	350	350	350	350	2350	2350	1400			450	450	450	450
Date	30-07-2014	20-10-2014	27-01-2015	21-04-2015	17-07-2015	29-07-2014	29-07-2014	29-07-2014	26-01-2015	29-10-2015	30-07-2015	30-07-2014	26-01-2015
EC (µs/cm)	657	610	695	1169	1928	2320	1806	608	986	592	519	700	490
pH	7,59	7,19	7,61	7,6	6,95	6,87	7,04	7,19	7,18	7,19	7,52	7,24	6,86
ORP	0	-418,5	0	-177	-79,9	0	0	0	0	9	-127,7	-379,6	-177,8
Temperature	14,7	15,67	10,4	13,33	15,04	14,5	14,3	16,3	9,5	12,36	12,45	13,76	9,52
Oxigen (mg/l)	0,23	0	0,29	0	0	0,34	0,36	0,61	0,69	0	0	0	5,01

Overzicht veldmetingen

Project	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933
Location	P121	P121	P13	P16	P17	P18	P21B	P21B	P21B	P222	P222	P231	P262
Filter top	2350	2350				140	350	350	350				620
Filter bottom	2450	2450				340	550	550	550				720
Date	30-07-2015	27-01-2015	01-08-2014	01-08-2014	01-08-2014	21-10-2015	21-10-2015	28-07-2015	31-07-2014	30-07-2014	27-01-2015	30-07-2014	23-07-2015
EC (µs/cm)	2442	2580	5,95	721	616	0	2064	1397	2095	356	603	582	627
pH	6,71	6,43	7,33	7,38	7,41	0	7,31	7,46	7,36	7,4	7,47	7,19	7,22
ORP	-59,7	0	0	0	0	0	-122,8	-176,9	0	0	0	0	0
Temperature	13,9	11,5	17,3	17,1	16,1	0	12,38	16,23	18,6	7,4	8,3	16,1	13,9
Oxigen (mg/l)	0	0,17	0,2	0,24	0,19	0	0	0	0,14	0,34	0,21	0,73	0,65

Overzicht veldmetingen

Project	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933
Location	P263	P263	P264	P264	P264	P265C	P265C	P265C	P27	P27	P28	P28	P283
Filter top	740	740	500	500	500	450	450	450			110	110	
Filter bottom	840	840	550	550	550	550	550	550			310	310	
Date	22-07-2015	27-01-2015	17-07-2015	29-07-2014	27-01-2015	22-07-2015	29-07-2014	26-01-2015	01-08-2014	26-01-2015	29-10-2015	31-07-2014	29-07-2014
EC (µs/cm)	1871	1243	610	821	732	597	531	596	824	541	0	0	667
pH	7,46	6,53	7,28	7,28	6,62	7,54	7,39	7,49	7,37	6,97	0	0	7,05
ORP	0	-151,6	-161,2	0	-136	0	0	0	0	-72,5	0	0	0
Temperature	17,3	11,75	18,08	16,8	9,56	16,2	18,1	9,8	22,1	8,24	0	0	14,8
Oxigen (mg/l)	0,51	1,14	0	0,4	0	0,73	0,77	1,04	0,44	0	0	0	0,41

Overzicht veldmetingen

Project	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933
Location	P304	P304	P305	P305	P305	P321	P321	P321	P321	P321	P340	P340	P341
Filter top			350	350	350	1550	1550	1550	1550	1550	400	400	360
Filter bottom			550	550	550	1650	1650	1650	1650	1650	600	600	560
Date	31-07-2014	26-01-2015	28-07-2015	31-07-2014	26-01-2015	22-10-2015	28-07-2015	20-10-2014	26-01-2015	21-04-2015	20-10-2015	27-07-2015	27-07-2015
EC (µs/cm)	1075	797	778	965	732	1068	1039	1920	1170	1363	1626	1396	853
pH	7,1	6,5	6,98	6,99	6,46	7,11	7,16	6,64	6,53	6,38	6,74	6,83	7,12
ORP	0	-131,8	-137	0	-144,7	-73,4	-127,3	-364,2	-230,5	-184,3	-74,4	0	0
Temperature	17,4	12,19	17,01	18,8	11,64	13,5	15,28	14,83	12,11	14,41	16,21	11	12,9
Oxigen (mg/l)	0,35	0	0	0,43	0	0	0	0	0	0	0	0,54	0,37

Overzicht veldmetingen

Project	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933
Location	P341	P343	P371	P371	P371	P372	P372	P372	P374	P374	P374	P378	P378
Filter top	360		150	150	150	480	480	480	500	500	500	350	350
Filter bottom	560		350	350	350	580	580	580	600	600	600	550	550
Date	30-07-2014	29-07-2014	27-07-2015	29-07-2014	26-01-2015	30-07-2015	29-07-2014	26-01-2015	27-07-2015	29-07-2014	26-01-2015	28-07-2015	01-08-2014
EC (µs/cm)	656	1152	546	539	517	354	491	466	1111	883	1047	864	798
pH	6,84	6,91	7,28	7,03	7,34	7,13	6,88	7,09	6,07	6,54	6,79	6,84	6,66
ORP	-311,2	-363,2	0	-381,1	0	-119,6	-266,2	0	0	-353,2	0	0	-202,1
Temperature	16,78	16,84	14,7	15,97	8,7	12,73	18,28	10,4	14,7	16,72	9,7	14,3	16,63
Oxigen (mg/l)	0	0	1,46	0,45	0,39	0	0	0,37	0,49	0	0,57	0,26	0

Overzicht veldmetingen

Project	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933
Location	P378	P379	P379	P380	P380	P381	P381	P381	P382	P382	P42	P42	P42
Filter top	350	350	350	350	350	500	500	500	250	250	300	300	300
Filter bottom	550	550	550	550	550	600	600	600	350	350	500	500	500
Date	28-01-2015	27-07-2015	30-07-2014	22-07-2015	29-07-2014	22-07-2015	29-07-2014	26-01-2015	22-07-2015	26-01-2015	27-10-2015	01-08-2014	02-11-2015
EC (µs/cm)	1039	550	532	1009	749	862	852	877	846	679	1136	6,94	975
pH	6,87	7,17	6,95	6,63	6,63	7,29	6,82	6,87	7,17	7,15	6,86	6,94	6,56
ORP	0	0	-233,3	0	-354,4	0	0	0	0	0	-127,4	0	-166,7
Temperature	10,7	15,4	18,91	19,7	18,16	18,1	17,5	9,5	13,2	9,6	17,9	18,5	10,56
Oxigen (mg/l)	0,26	0,38	0	0,64	0	2,18	0,61	0,91	0,26	0,3	0	0,23	0

Overzicht veldmetingen

Project	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933
Location	P56	PA109A	PA109A	PA109A	PA109B	PA111A	PA111A	PA111A	PA111B	PA112	PA112	PA112	PP01
Filter top	0	500	500	500	0	400	400	400	0	345	345	345	260
Filter bottom	0	600	600	600	0	600	600	600	0	545	545	545	560
Date	01-08-2014	28-07-2015	01-08-2014	28-01-2015	01-08-2014	28-07-2015	01-08-2014	28-01-2015	01-08-2014	28-07-2015	01-08-2014	28-01-2015	22-10-2015
EC (µs/cm)	500	466	548	1468	331	1245	1313	1470	323	782	676	1179	674
pH	7,52	7,75	6,9	7,03	7,56	7,06	6,48	6,58	7,14	7,34	7,06	7,43	7,27
ORP	0	0	-208,9	0	-163,8	0	-257,8	0	-230,2	0	-60,4	0	-29
Temperature	18,7	14,1	17,5	10,5	22,38	15,7	20,39	10,7	21,41	13,6	14,84	9,5	13,81
Oxigen (mg/l)	1,79	0,37	0	0,42	0,47	1,2	0	0,61	0	0,35	0	0,54	0

Project	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933
Location	PP01	PP01	PP01	PP02	PP02	PP02	PP04	PP04	PP04	PP05	PP05	PP05	PP06
Filter top	260	260	260	320	320	320	200	200	200	160	160	160	130
Filter bottom	560	560	560	620	620	620	600	600	600	560	560	560	530
Date	27-07-2015	20-10-2014	21-04-2015	22-10-2015	20-10-2014	21-04-2015	27-07-2015	20-10-2014	21-04-2015	20-10-2015	27-07-2015	21-04-2015	22-10-2015
EC (µs/cm)	521	751	565	781	899	1051	611	1004	898	0	0	0	763
pH	7,29	7,23	7,52	7,13	6,93	7,29	7,18	685	7,12	0	0	0	8,04
ORP	104,2	8,2	-141	-52,8	23,5	29,7	-78,2	41	-117,2	0	0	0	-83,1
Temperature	17,27	18,5	11,35	14,61	16,9	13,51	16,68	18,8	12,41	0	0	0	15,83
Oxigen (mg/l)	8,52	0	0	0	0	0	2,9	0	0	0	0	0	0

Overzicht veldmetingen

Project	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933	0287933
Location	PP06	PP06	PP06	PP07	PP07	PP07	PP07	PP08	PP08	PP08	PP08	PP09	PP09
Filter top	130	130	130	200	200	200	200	320	320	320	320	300	300
Filter bottom	530	530	530	600	600	600	600	720	720	720	720	600	600
Date	27-07-2015	20-10-2014	21-04-2015	29-10-2015	27-07-2015	20-10-2014	21-04-2015	22-10-2015	27-07-2015	20-10-2014	21-04-2015	22-10-2015	20-10-2014
EC (µs/cm)	335	636	755	717	738	849	913	908	521	1223	1309	605	916
pH	7,28	7,68	7,13	7,24	6,81	6,84	7,28	6,93	7,22	6,72	7	7,19	6,61
ORP	-90,5	42,5	-30,5	-41,3	17,9	55,2	-47,3	-26,2	92,4	14,6	-91,5	-24,1	73,4
Temperature	16,89	17,5	13,3	16,57	17,79	17,9	14,27	13,47	15,57	16,9	10,72	12,67	16,1
Oxigen (mg/l)	5,81	0	0	0	0	0	0	0	8,31	0	0	0	0

Project	0287933	0287933	0287933	0287933	0287933
Location	PP09	PP10	PP10	PP10	U14
Filter top	300	300	300	300	
Filter bottom	600	600	600	600	
Date	21-04-2015	22-10-2015	20-10-2014	21-04-2015	01-08-2014
EC (µs/cm)	990	361	911	953	6,85
pH	6,97	7,36	6,67	7,38	6,85
ORP	29,9	-51,6	44	56,9	0
Temperature	16,72	13,57	15,3	17,72	21
Oxigen (mg/l)	0	0	0	0	0,57

Locatie	**L4**	**P114bis**	**P116**	**P321**	**D9**	**L19**	**M4**	**P118C**	**P119C**	**P21B**	**P263**	**P265C**
Bemonsteringsdatum	*20-10-14*	*20-10-14*	*20-10-14*	*20-10-14*	*26-01-15*	*26-01-15*	*26-01-15*	*26-01-15*	*26-01-15*	*26-01-15*	*26-01-15*	*26-01-15*
Benzeen												
Ethylbenzeen												
Tolueen												
Xyleen												
Orthoxyleen												
Meta- en paraxyleen												
Minerale olie C10 - C12												
Minerale olie C10 - C40												
Minerale olie C12 - C20												
Minerale olie C20 - C30												
Minerale olie C30 - C40												
PFHS	2,39	1,63	0,619	892	7161,5	102	264	95,85	91	10000	770	17,975
PFOA	3,14	4,53	1,09	3140	130,5	237	1652,5	217	181	12900	1990	79,35
PFOS	29,9	13,4	8,77	3210	1402	2900	13409	1314	1462	79300	9820	660
PFOSA	1,05	0,0284	0,0673	0,438	17,7	188	341	60,3	62,9	27,3	21,5	118
FOSA					14,5		138,1	14,5	14,5			15,2
PFBS										4740		

Locatie	**P27**	**P304**	**P305**	**P321**	**P371**	**P372**	**P374**	**P381**	**P382**	**P42**	**BD24-3**	**L21**
Bemonsteringsdatum	*26-01-15*	*26-01-15*	*26-01-15*	*26-01-15*	*26-01-15*	*26-01-15*	*26-01-15*	*26-01-15*	*26-01-15*	*26-01-15*	*27-01-15*	*27-01-15*
Benzeen												
Ethylbenzeen												
Tolueen												
Xyleen												
Orthoxyleen												
Meta- en paraxyleen												
Minerale olie C10 - C12												
Minerale olie C10 - C40												
Minerale olie C12 - C20												
Minerale olie C20 - C30												
Minerale olie C30 - C40												
PFHS	17,95	89,3	215	915	40,35	66,4	478	147	79,85	425	83,1	0,16
PFOA	167,5	526,5	205,5	3570	143,5	80,6	534,5	584	387,5	406	124,5	0,448
PFOS	872	669,5	456,5	4120	558	1380	1841	933,5	712,5	2874	996	4,17
PFOSA	153	25	15,3	5,84	48,3	38,5	31,4	12,5	86	29	2,82	0,0398
FOSA	93,9	14,5	14,5		14,5		14,5	14,5	14,5	14,5	1,32	0,0336
PFBS	2004	133,5	86,8							32,55		

Locatie	**L22**	**L31**	**L4**	**P114bis**	**P115**	**P116**	**P121**	**P264**	**B7**	**P378**	**PA109A**	**PA111A**
Bemonsteringsdatum	*27-01-15*	*27-01-15*	*27-01-15*	*27-01-15*	*27-01-15*	*27-01-15*	*27-01-15*	*27-01-15*	*28-01-15*	*28-01-15*	*28-01-15*	*28-01-15*
Benzeen												
Ethylbenzeen												
Tolueen												
Xyleen												
Orthoxyleen												
Meta- en paraxyleen												
Minerale olie C10 - C12												
Minerale olie C10 - C40												
Minerale olie C12 - C20												
Minerale olie C20 - C30												
Minerale olie C30 - C40												
PFHS	0,3245	0,5985	0,681	1,47	1,94	0,735	0,5385	274	0,762	285,5	330	1250
PFOA	0,6345	0,5925	1,02	3,43	4,105	1,74	0,537	413	1,2335	300	291	834
PFOS	12,45	5,905	38,6	14,2	0,5045	6,87	4,895	1324	9,05	546	429	1960
PFOSA	0,0445	0,025	13	0,0409	0,025	0,0557	0,258	84,7	0,0839	0,943	1,26	14,7
FOSA	0,0349	0,025			0,025		1,32	14,5	1,32	14,5		
PFBS												

Locatie	**PA112**	**L4**	**P114bis**	**P116**	**P321**	**P118A**	**P118B**	**P118C**	**P264**	**L19**	**P263**	**P265C**
Bemonsteringsdatum	*28-01-15*	*21-04-15*	*21-04-15*	*21-04-15*	*21-04-15*	*17-07-15*	*17-07-15*	*17-07-15*	*17-07-15*	*17-07-15*	*17-07-15*	*17-07-15*
Benzeen												
Ethylbenzeen												
Tolueen												
Xyleen												
Orthoxyleen												
Meta- en paraxyleen												
Minerale olie C10 - C12												
Minerale olie C10 - C40												
Minerale olie C12 - C20												
Minerale olie C20 - C30												
Minerale olie C30 - C40												
PFHS	73	0,659	1,85	0,722	1094,150925	3069	2787,5	99,9	167	239	838,5	33
PFOA	264,5	0,84	3,48	1,85	4216,408255	1824	2237	275	302	279	2402	116
PFOS	254,5	19	16,2	7,71	4908,82575	1104,5	11027,5	1880	1880	4185	12328	939
PFOSA	44,3	10,6	0,061	0,0617	0,457	6,74	11,3	90,3	173	144	14,5	122
FOSA	21,5				12,6	18,3	181,3			97,4	18,3	
PFBS					331,79454							

Locatie	**P343**	**P380**	**P381**	**P382**	**D10**	**L22**	**L4**	**P114bis**	**P115**	**P116**	**P262**	**D9**
Bemonsteringsdatum	*17-07-15*	*17-07-15*	*17-07-15*	*17-07-15*	*23-07-15*	*23-07-15*	*23-07-15*	*23-07-15*	*23-07-15*	*23-07-15*	*23-07-15*	*24-07-15*
Benzeen												
Ethylbenzeen												
Tolueen												
Xyleen												
Orthoxyleen												
Meta- en paraxyleen												
Minerale olie C10 - C12												
Minerale olie C10 - C40												
Minerale olie C12 - C20												
Minerale olie C20 - C30												
Minerale olie C30 - C40												
PFHS	802	276	185	140	188	0,837	2,61	1,28	2	0,706	189,75	8140
PFOA	972	250,5	675	496	526	2,4	2,36	3,2	4,05	1,72	383,75	141
PFOS	8410	1280,5	1420	1070	29,3	30,9	30	14	0,238	9,43	3522,25	1860
PFOSA	16,4	133	19,9	101	0,296	1,81	0,933	0,185	0,125	0,347	49,65	21,8
FOSA	18,3	76,8			9,99						18,3	
PFBS												

Locatie	**K3**	**P27**	**P304**	**P56**	**D14**	**M4**	**P340**	**P341**	**P371**	**P374**	**P379**	**P42**
Bemonsteringsdatum	*24-07-15*	*24-07-15*	*24-07-15*	*24-07-15*	*27-07-15*	*27-07-15*	*27-07-15*	*27-07-15*	*27-07-15*	*27-07-15*	*27-07-15*	*27-07-15*
Benzeen												
Ethylbenzeen												
Tolueen												
Xyleen												
Orthoxyleen												
Meta- en paraxyleen												
Minerale olie C10 - C12												
Minerale olie C10 - C40												
Minerale olie C12 - C20												
Minerale olie C20 - C30												
Minerale olie C30 - C40												
PFHS	84920,5	26,4	150	70,35	1,134	165	541,5	535	104	1090	31,7	587
PFOA	113,7	193	675	71,425	2,095	398	1495,5	1131,5	183	880	102,5	438
PFOS	3858,5	1980	1060	246,75	1,167	6690	6184,5	6630	2230	3330	1176,5	3910
PFOSA	12,4	165	25,3	18,45	0,225	273	138	58,7	85,4	30,2	71,6	22,5
FOSA	18,3			12,9	0,315		18,3	18,3			49	
PFBS	1498	454	260	17,925								66,5

Locatie	**B7**	**BD24-3**	**BD24-4**	**D11**	**L21**	**L31**	**P21B**	**P305**	**P321**	**P378**	**PA109A**	**PA111A**
Bemonsteringsdatum	*28-07-15*	*28-07-15*	*28-07-15*	*28-07-15*	*28-07-15*	*28-07-15*	*28-07-15*	*28-07-15*	*28-07-15*	*28-07-15*	*28-07-15*	*28-07-15*
Benzeen												
Ethylbenzeen												
Tolueen												
Xyleen												
Orthoxyleen												
Meta- en paraxyleen												
Minerale olie C10 - C12												
Minerale olie C10 - C40												
Minerale olie C12 - C20												
Minerale olie C20 - C30												
Minerale olie C30 - C40												
PFHS	17,4	81,55	3,18	71,3	0,274	0,524	10993,5	303	438	364	95,5	1681
PFOA	30	144,5	10,25	37,75	0,593	0,62	12987,5	256	1320	374	57,85	973
PFOS	392	1328,5	5,52	781,5	7,23	10,7	46505,5	642	4040	1310	176	3027
PFOSA	2,35	4,13	0,0862	94,9	0,172	0,355	3,78	16,4	15,6	1,84	1,95	12,3
FOSA		10	0,315	9,99			18,3				3,33	9,99
PFBS							7566,5	133				

Locatie	**PA112**	**P119C**	**P121**	**P18**	**P28**	**P372**
Bemonsteringsdatum	*28-07-15*	*30-07-15*	*30-07-15*	*30-07-15*	*30-07-15*	*30-07-15*
Benzeen				5	5	
Ethylbenzeen				5	5	
Tolueen				5	13	
Xyleen				4,5	4,5	
Orthoxyleen				1,5	1,5	
Meta- en paraxyleen				3	3	
Minerale olie C10 - C12				180		
Minerale olie C10 - C40				2100		
Minerale olie C12 - C20				1000		
Minerale olie C20 - C30				850		
Minerale olie C30 - C40				93		
PFHS	31,8	996	0,0682			73,7
PFOA	153	1320	0,168			84,7
PFOS	426	7320	2,62			1944
PFOSA	63,9	123	0,208			43,2
FOSA						9,99
PFBS						

Bijlage 4

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	0341743
Subject	Re-evaluation of the statistical trend analysis of the ground- and surface water concentrations collected in the Blokkersdijk nature reserve
Date	31-March-2016

Environmental Resources Management

ERM Belgium

Posthoflei 3
2600 Antwerp
Tel [redacted]

Email: [redacted]

ERM

Introduction

In this memo the results of a revised evaluation of the statistical trend analysis of the perfluorooctane sulfonate (PFOS) concentrations in the Blokkerdijk nature reserve are described. 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 in St. Paul, Minnesota, it has been determined that the analytical data generated by the 3M Environmental Laboratory 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, a revised evaluation of the PFOS trend analysis has been performed by comparing the total data set (which includes the pre-3M lab analysis data) with only the 3M lab data set.

The results of the revised evaluation will be included in the sixth annual interim report ("*Eerste gefaseerd bodemsaneringsproject - Zesde tussentijds verslag bodemsaneringswerken, Periode juli 2014 - juli 2015*", dd. 31 maart 2016) that was submitted to the Flemish environmental agency OVAM.

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

Background

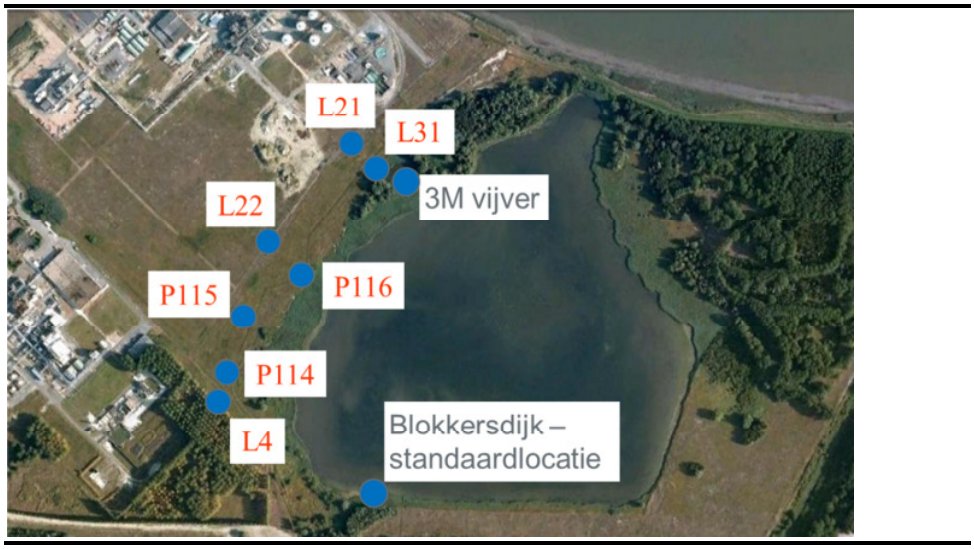
The Blokkerdijk nature reserve lies east and adjacent to the 3M site (see Figure 1). During the evaluation of remedial options in 2008, monitoring of the water quality in the nature reserve was selected as the best remedial technique and 'a statistically increasing trend' was defined as trigger value. 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

As noted earlier, multiple analytical laboratories were used to determine PFOS concentrations at the above sampling locations. Questions have been raised as to the accuracy of the data collected from some of these labs, and as discussed earlier, the lab currently used (3M Environmental Lab) has provided the most accurate data to date. The use of multiple laboratories over the period of data collection could confound the results of a PFOS trend analysis. In order to determine if this might be occurring, the PFOS trend analyses were conducted on two sets of samples: 1) the entire dataset for each location, and 2) only the 3M lab data for each location. The 3M lab data analysis started in January 2014 and continued through January 2016.

The trend analysis was conducted as follows:

1. Data distribution was determined using the Shapiro-Wilks and Lillifors tests for the entire data set and only the 3M lab data for each location;
2. Q-Q plots were developed for each location using the entire data set and the 3M data set, to evaluate if data outliers were present in the data;
3. Time series figures were created for each location showing the pre-3M and the 3M PFOS concentrations over time; and
4. Trend analyses were conducted using ordinary least-square (OLS) regression on both the original and log-transformed (natural log) data sets, as well as the non-parametric Mann-Kendall trend test as defined in the remedial action plan of 2008 [(1)]. The appropriate trend analysis used for each location and data set was based on the distribution of the data determined in step 1.

Table 1 (included in Annex 1) shows the data used in the trend analysis. PFOS was detected in all samples, except for the July 30, 2014 sampling in well P115, which was non-detect.

All statistical analyses were conducted using the U.S. EPA statistical software program ProUCL version 5.0.00.

Results

Figure 1 (included as Annex 2) shows time series graphs of PFOS concentrations for samples collected from each of the sampling locations, broken down into pre-3M analysis and 3M analysis samples. In some of the locations the apparent trend (or lack thereof) can be observed between the pre-3M and 3M lab data. 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 for the entire dataset shows that wells L21 and P114, and the 3M vijver had significant increasing trends of PFOS over time. However, these locations did not show a significant trend when only the 3M lab data was utilized. The following subsections discuss the trend analysis results for each sampling location.

Well L21

Figure 1 (see Annex 2) shows well L21 with apparent trend in the pre-3M lab data set, but an apparent upward trend in the 3M lab data. The Q-Q plots for the entire data set show that the highest three values might be from a different population (i.e., are potential outliers (Figure 2); however, the Q-Q plot for the 3M lab data set (Figure 3) does not show any potential outliers. The entire data set is log-normally distributed, and the OLS regression for the log-transformed data showed a significant upward trend in PFOS (Table 2; see Annex 3). However, there was no significant trend in the 3M lab data for this location. There is much variability in the PFOS concentrations in this well. The lack of a significant trend in the 3M lab data set might be due to this variability. For example, the last two sampling periods showed a decrease in PFOS concentrations.

Well L22

Figure 1 (see Annex 2) shows well L22 with no apparent upward trend in either the pre-3M or 3M lab data sets. However, two intermediate values are substantially higher than the norm for this well. The Q-Q plots for the entire data set and the 3M lab data set show that these two values might be from a different population (i.e., are potential outliers) (Figures 2 and 3; see Annex 4). The entire data set is log-normally distributed, and the OLS regression for the log-transformed data showed no significant trend in PFOS, nor was there any significant trend in the 3M lab data set (Table 2; see Annex 3).

Well L31

Figure 1 (see Annex 2) shows well L31 with no apparent upward trend in either the pre-3M or 3M lab data sets. Neither the entire data set nor the 3M lab data set showed a significant trend (Table 2; see Annex 3).

Well L4

Figure 1 (see Annex 2) shows well L4 with an apparent upward trend in the pre-3M lab data set, and a potential upward trend in the 3M lab data set. However, the apparent trends are not consistent between the pre-3M and the 3M sampling periods (i.e., the pre-3M trend does not coincide with the 3M trend). The entire data set is normally distributed, and the OLS regression for the original data showed no significant trend in PFOS, nor was there any significant trend in the 3M lab data set (Table 2; see Annex 3).

Well P114

Figure 1 (see Annex 2) shows well P114 with an apparent upward trend in the pre-3M lab data, but no apparent trend in the 3M lab data. The entire data set is normally distributed, and the OLS regression for the original data showed a significant upward trend in PFOS. However, the 3M lab data did not show any significant trend (Table 2; see Annex 3).

Well P115

Figure 1 (see Annex 2) shows well P115 with no apparent upward trend in either the pre-3M or 3M lab data sets, although there is much variability in PFOS concentrations in the two data sets. Neither the entire data set the 3M lab data set showed any significant trend in PFOS (Table 2; see Annex 3).

Well P116

Figure 1 (see Annex 2) shows well P116 with no apparent upward trend in either the pre-3M or 3M lab data sets. However, there appears to be a data outlier in the 3M lab data set, which is also suggested in the Q-Q plots (Figures 2 and 3; see Annex 4). The entire data set is non-parametric, and the Mann-Kendall trend showed no significant trend in PFOS, nor was there any significant trend in the 3M lab data set (Table 2; see Annex 3).

3M vijver

Figure 1 (see Annex 2) shows the 3M vijver PFOS concentrations with no apparent upward trend in either the pre-3M or the 3M lab data sets, although there is much variability in PFOS concentrations in the two data sets. The entire data set is log-normally distributed, and the OLS regression for the log-transformed data showed an upward trend in PFOS; however, there was no significant trend in the 3M PFOS concentration data (Table 2; see Annex 3). These results are consistent with the graphic analysis presented in Figure 1 (see Annex 2).

Blokkersdijk vijver standard

Figure 1 (see Annex 2) shows the Blokkersdijk vijver standard PFOS concentrations with no apparent upward trend in either the pre-3M or 3M lab data sets, but one intermediate value in the 3M lab data set is substantially higher than the norm for this location. The Q-Q plots for the entire data set and the 3M lab data set show that this value might be from a different population (i.e., is a potential outlier) (Figures 2 and 3; see Annex 4). The entire data set is non-parametric, and the Mann-Kendall test showed no significant trend in PFOS, nor was there any significant trend in the 3M lab data set (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 entire data set for each location, and a trend analysis for only the data which was analyzed by 3M Environmental Lab for each location. Three of the locations showed statistically significant upward trends when the entire data set was utilized in the trend analysis: L21, P114, and 3M vijver. However, none of these three locations showed a significant trend in PFOS when only the 3M lab data was utilized in the analysis. In the case of well L21, any apparent trend is masked by the variability in PFOS data during the period of sampling.

Significant upward trends in three locations (L21, P114, and 3M vijver) appear to be due to changes in analytical laboratories. When the PFOS concentration trends were evaluated for only the data analyzed by 3M Environmental Lab, no significant trends were observed in those three locations. For this reason it is suggested that only data analyzed by 3M be utilized for future trend analysis. Only five to ten sampling periods are included in the 3M lab data evaluation. Those locations with a smaller number of samples might contain too much variability in PFOS concentrations, or not have enough statistical power to adequately evaluate a statistical trend analysis. Therefore, it is suggested that in all the sample locations, quarterly samples be taken to populate the data set and that a graphical and statistical trend analysis be continued.

■

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 (all data)
Figure 3:	Q-Q plots (3M lab data)

Annex 1

Table 1: Data used in the trend analysis

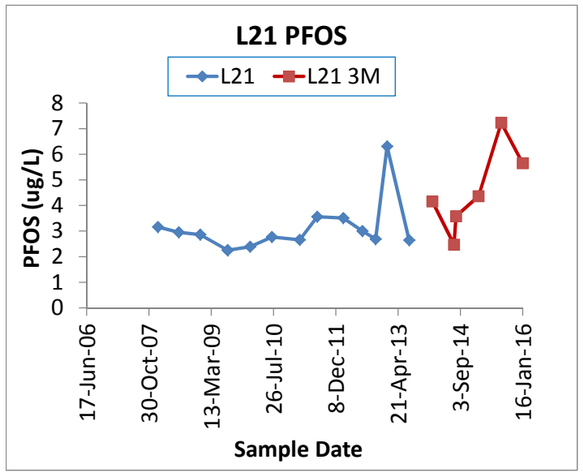
Table 1. PFOS Data from Selected Site Wells
Highlighted dates (red) are analyses conducted by 3M
P114 highlighted wells (red) are labelled P114bis

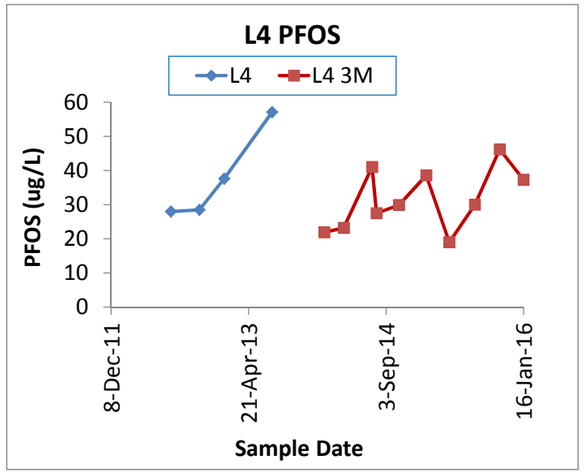
L21		L22		L31		L4		P114		P115		P116		3M vijver		BD standaard	
Date	PFOS (ug/L)	Date	PFOS (ug/L)	Date	PFOS (ug/L)	Date	PFOS (ug/L)	Date	PFOS (ug/L)	Date	PFOS (ug/L)	Date	PFOS (ug/L)	Date	PFOS (ug/L)	Date	PFOS (ug/L)
10-Jan-08	3,16	10-Jan-08	9,2	10-Jan-08	8,2	12-Jul-12	28	10-Jan-08	2,75	10-Jan-08	0,56	10-Jan-08	9,28	26-Apr-05	3,1	10-Aug-05	1,1
26-Jun-08	2,95	26-Jun-08	14,2	26-Jun-08	8,13	24-Oct-12	28,5	26-Jun-08	4,84	26-Jun-08	0,6	26-Jun-08	9,12	10-Aug-05	2,7	1-Mar-06	1,26
15-Dec-08	2,86	24-Jul-09	12,2	15-Dec-08	8,88	22-Jan-13	37,6	15-Dec-08	4,54	15-Dec-08	0,24	15-Dec-08	7,8	1-Mar-06	2,05	18-Oct-06	1,07
23-Jul-09	2,25	20-Jan-10	16,2	24-Jul-09	10,03	15-Jul-13	57,1	24-Jul-09	7,71	24-Jul-09	0,246	24-Jul-09	8,41	18-Oct-06	2,76	5-Apr-07	6,33
24-Jul-09	2,25	14-Jul-10	13,3	20-Jan-10	6,21	21-jan-14	21,9	20-Jan-10	5,45	20-Jan-10	0,213	20-Jan-10	8,35	5-Apr-07	4,13	20-Aug-07	0,62
20-Jan-10	2,39	26-Jan-11	7,73	14-Jul-10	7,56	02-apr-14	23,2	14-Jul-10	7,33	14-Jul-10	0,164	14-Jul-10	5,86	20-Aug-07	6,62	10-Jan-08	0,67
14-Jul-10	2,77	6-Feb-12	8,22	22-Feb-11	8,14	14-jul-14	41,04	22-Feb-11	7,5	22-Feb-11	0,083	26-Jan-11	7,38	10-Jan-08	2,81	26-Jun-08	6,23
22-Feb-11	2,66	9-Jul-12	12,7	6-Feb-12	7,39	30-jul-14	27,5	12-Jul-11	12	12-Jul-11	0,661	12-Jul-11	9,42	26-Jun-08	4,1	28-Jul-08	2,83
12-Jul-11	3,56	25-Oct-12	10	9-Jul-12	12,4	20-okt-14	29,9	6-Feb-12	11,5	6-Feb-12	0,409	6-Feb-12	9,39	15-Dec-08	3,05	2-Oct-08	0,82
6-Feb-12	3,51	23-Jan-13	10	23-Oct-12	8,6	27-jan-15	38,6	9-Jul-12	17,8	9-Jul-12	0,313	9-Jul-12	12,8	7-Apr-09	3,82	15-Dec-08	5,9
9-Jul-12	3	19-Jul-13	10,3	23-Jan-13	7,39	21-apr-15	19	24-Oct-12	13	24-Oct-12	0,126	24-Oct-12	9,48	24-Jul-09	5,21	7-Apr-09	0,67
23-Oct-12	2,69	21-Jan-14	12,1	24-Apr-13	9,57	23-jul-15	30	25-Jan-13	16,4	25-Jan-13	0,564	23-Jan-13	9,73	20-Jan-10	4,14	23-Jul-09	0,799
23-Jan-13	6,31	30-Jul-14	11,9	19-Jul-13	10,9	21-okt-15	46,2	24-Apr-13	21,9	15-Jul-13	0,436	24-Apr-13	15,3	14-Jul-10	4,04	14-Oct-09	0,62
19-Jul-13	2,64	27-Jan-15	11,4	14-Oct-13	11,6	16-jan-16	37,3	15-Jul-13	18,5	21-jan-14	0,518	15-Jul-13	10,1	18-Feb-11	3,55	27-Oct-09	0,62
21-Jan-14	4,16	23-Jul-15	30,9	21-jan-14	8,07			02-apr-14	16,3	14-jul-14	0,911	14-Oct-13	11	12-Jul-11	6,16	20-Jan-10	2,65
30-Jul-14	3,58	21-Oct-15	38,6	14-jul-14	6,3			14-jul-14	11,05	30-jul-14	<0.464	21-jan-14	9,17	28-Feb-12	4,38	15-Apr-10	4,6
27-Jan-15	4,36	16-jan-16	5,39	30-jul-14	8,03			30-jul-14	11,5	27-jan-15	0,467	02-apr-14	7,46	10-Jul-12	3,84	14-Jul-10	1,08
28-Jul-15	7,23			27-jan-15	5,72			20-okt-14	13,4	23-jul-15	0,238	14-jul-14	5,25	23-Oct-12	3,83	7-Oct-10	0,65
16-jan-16	5,65			28-jul-15	10,7			27-jan-15	14,2	16-jan-16	0,594	30-jul-14	7,42	31-Jan-13	4,93	18-Feb-11	4,34
				16-jan-16	7,61			21-apr-15	16,2			20-okt-14	8,77	25-Apr-13	5,91	19-Apr-11	1,3
								23-jul-15	14			27-jan-15	6,87	19-Jul-13	6,97	12-Jul-11	3,51
								21-okt-15	14			21-apr-15	7,71	14-Oct-13	3,59	4-Oct-11	0,74
								16-jan-16	4,29			23-jul-15	9,43	23-jan-14	4,13	28-Feb-12	0,813
												21-okt-15	34,6	02-apr-14	4	10-Apr-12	0,652
												16-jan-16	8,51	31-jul-14	3,85	10-Jul-12	0,516
														21-okt-14	3,18	23-Oct-12	1,27
														27-jan-15	5,27	31-Jan-13	0,617
														21-apr-15	4,3	25-Apr-13	0,533
														27-jul-15	5,39	19-Jul-13	4,35
														20-okt-15	4,78	14-Oct-13	0,198
														16-jan-16	3,29	23-jan-14	1,06
																02-apr-14	1,17
																31-jul-14	0,86
																21-okt-14	26,9
																27-jan-15	0,792
																21-apr-15	5,01
																27-jul-15	2,54
																20-okt-15	7,18
																16-jan-16	1,23

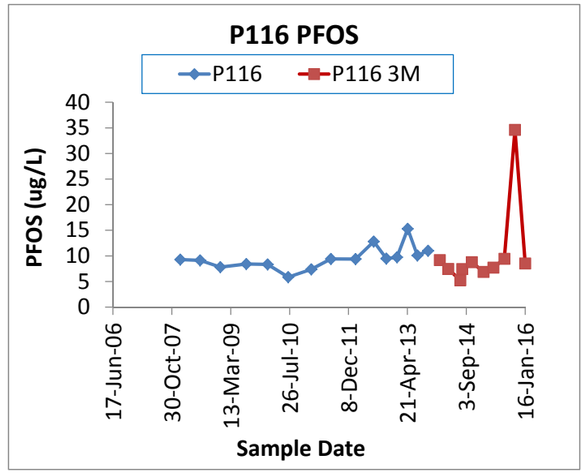
Annex 2

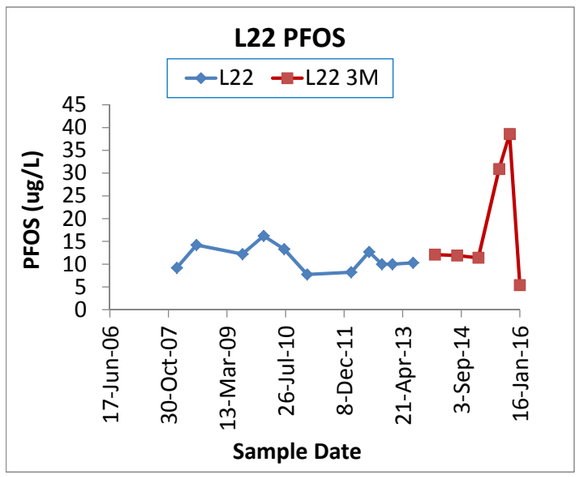
Figure 1: Time series graphs of PFOS

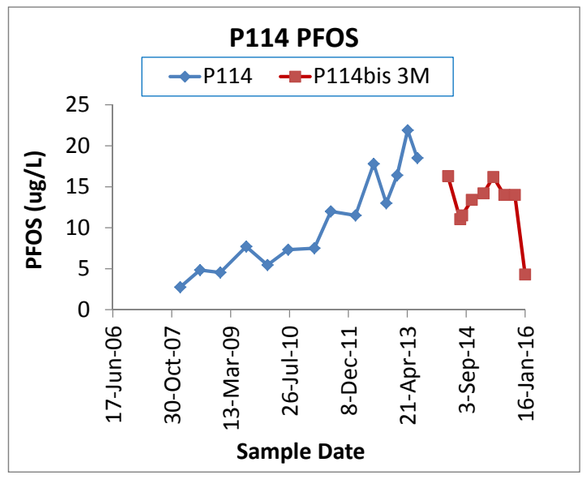
Figure 1. Time series plots of PFOS data with 3M-analyzed data demarcated (in red).

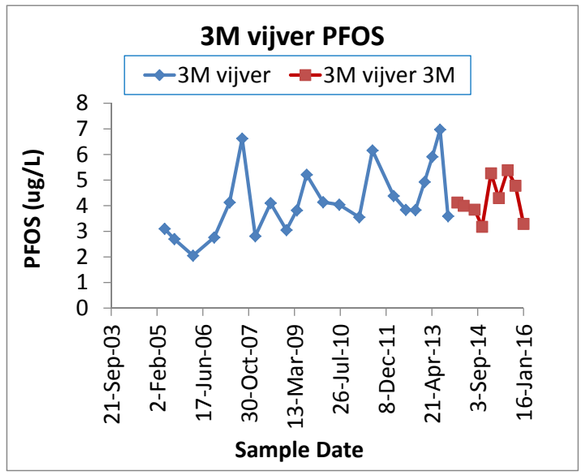


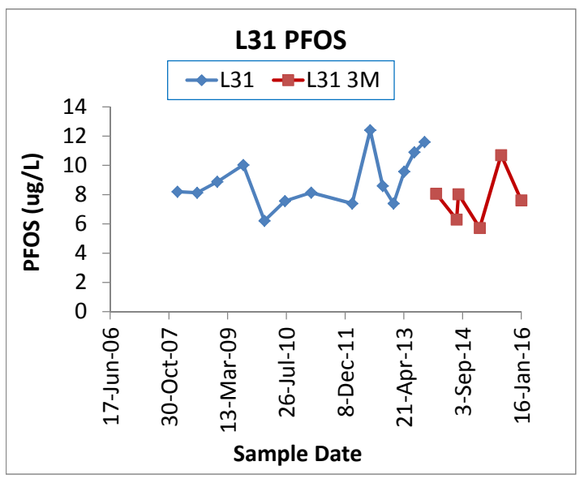


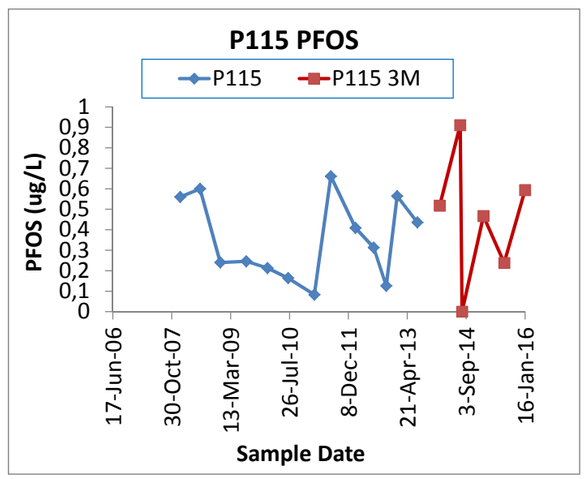


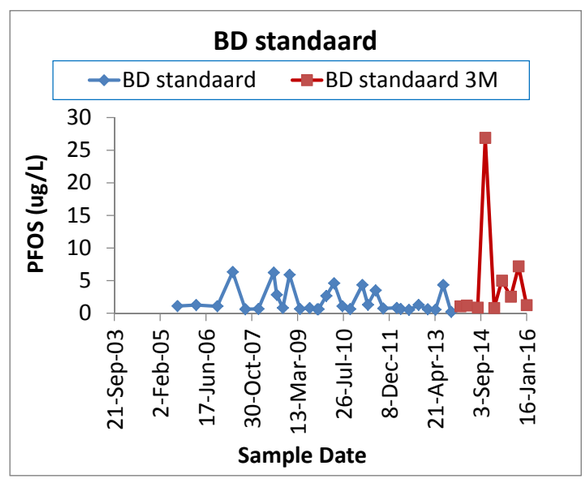












Annex 3

Table 2: Results of the trend analyses

Table 2. PFOS Trend Analysis p-values.

Well	L21	L22	L31	L4	P114	P115	P116	3M vijver	Blokkersdijkvijver standaard
All PFOS data									
Data Distribution	LN	LN	N	N	N	N	NP	LN	NP
OLS trend	0,0047	0,192	0,891	0,878	0,0025	0,429	0,239	0,046	0,266
Ln-OLS trend	0,0037	0,451	0,987	0,863	0,0009	0,446	0,359	0,016	0,625
Mann-Kendall Trend	0,0097	0,435	0,348	0,256	0,0056	0,288	0,399	0,018	0,49
3M PFOS data									
Data Distribution	N	N	N	N	NP	N	NP	N	LN
OLS trend	0,105	0,489	0,621	0,15	0,267	0,764	0,19	0,615	0,999
Ln-OLS trend	0,122	0,79	0,663	0,168	0,196	0,516	0,163	0,688	0,524
Mann-Kendall Trend	0,066	0,5	0,5	0,076	0,417	0,5	0,142	0,377	0,233

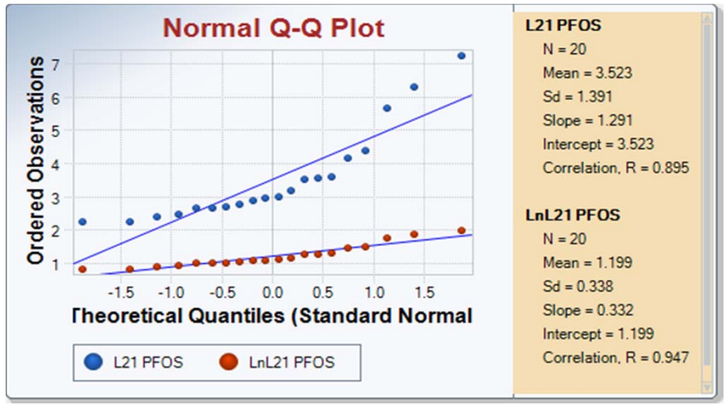
N = normal
LN = lognormal
NP = non-parametric

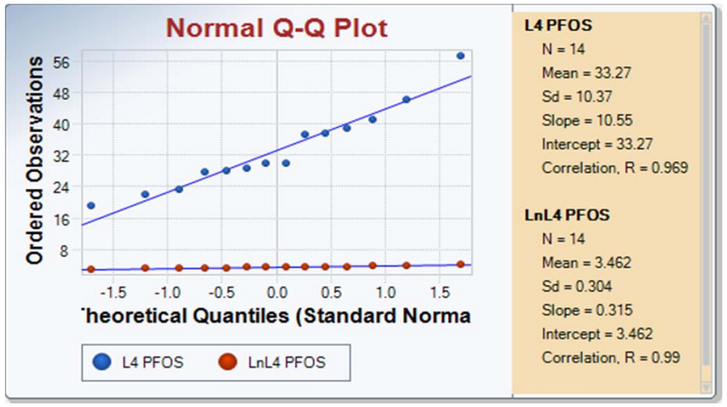
indicates significant trend

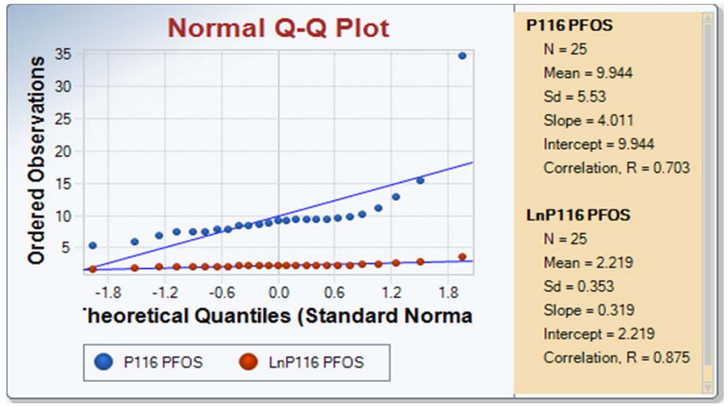
Annex 4

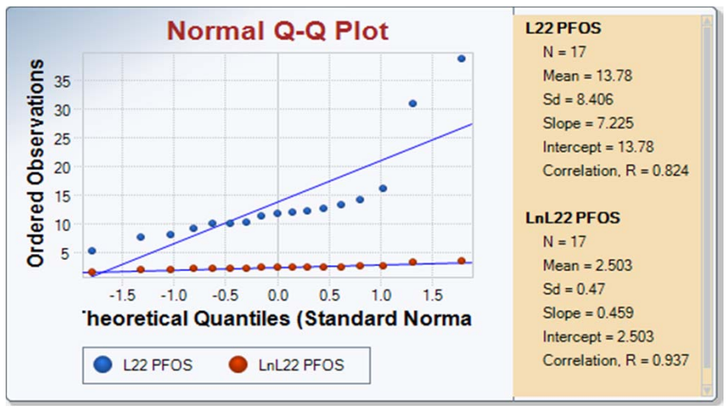
Figure 2: Q-Q plots (all data)
Figure 3: Q-Q plots (3M lab data)

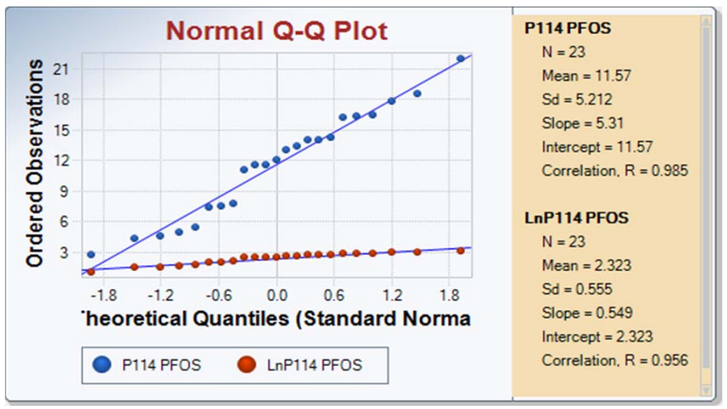
Figure 2. Q-Q plots for the entire data set.

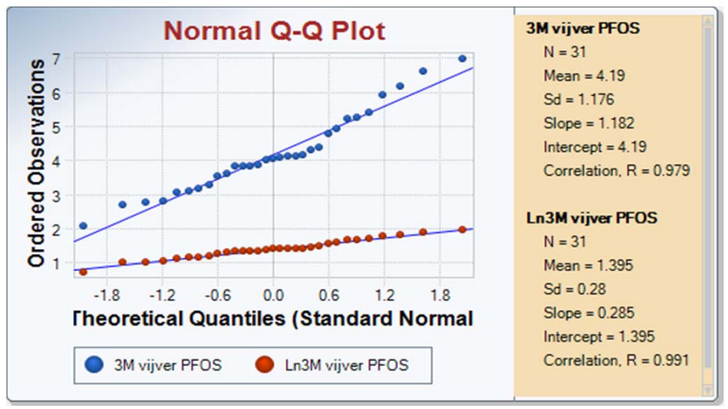


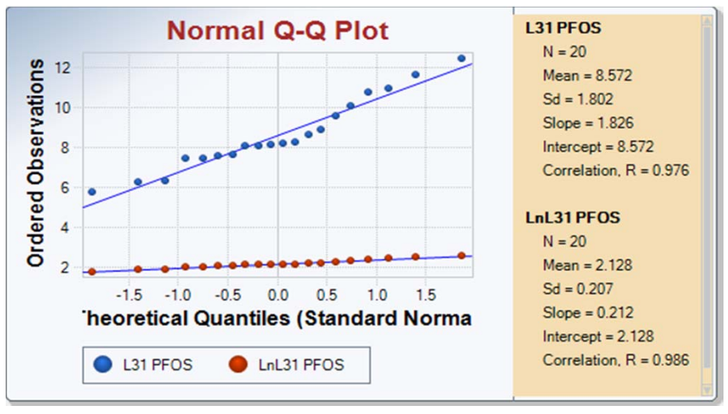


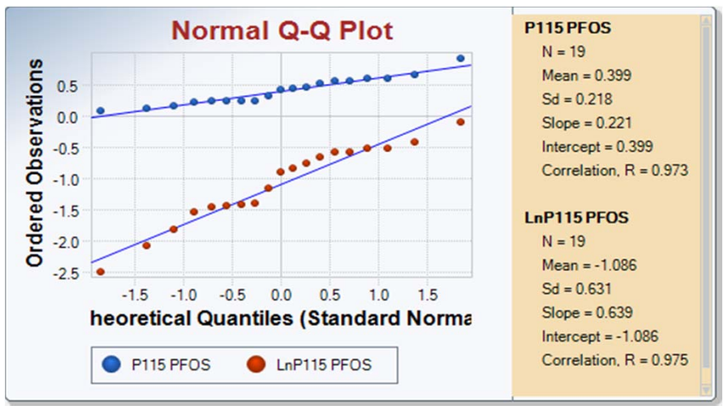












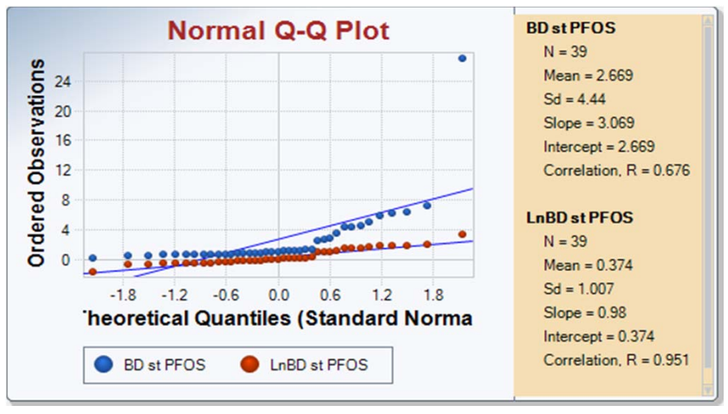
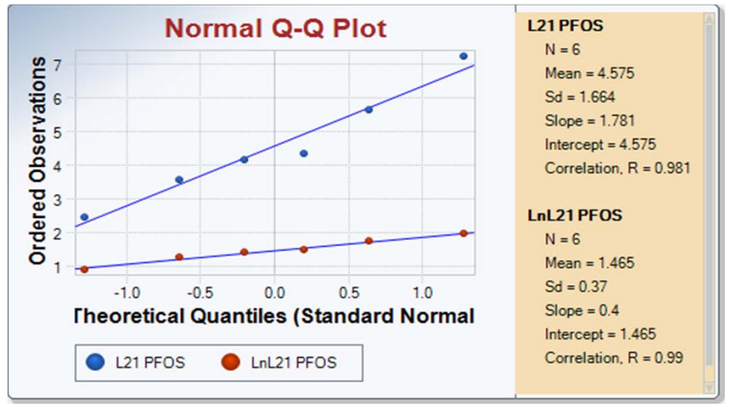
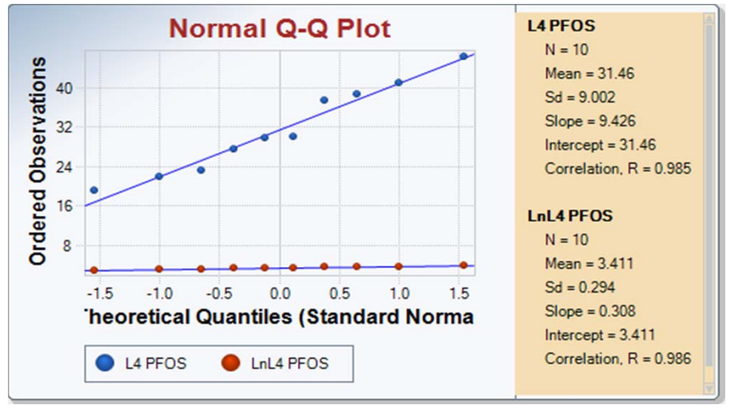
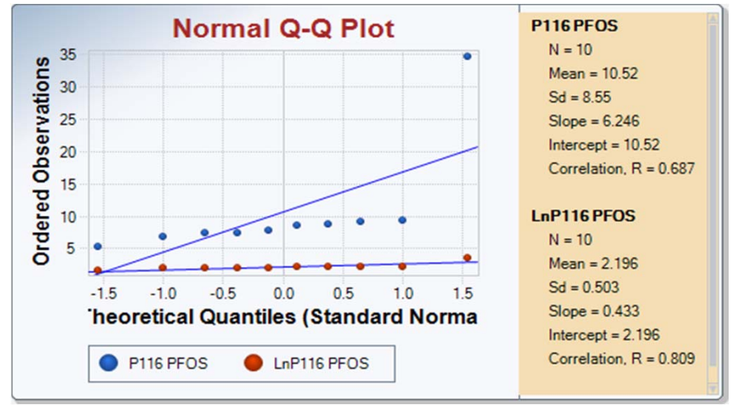
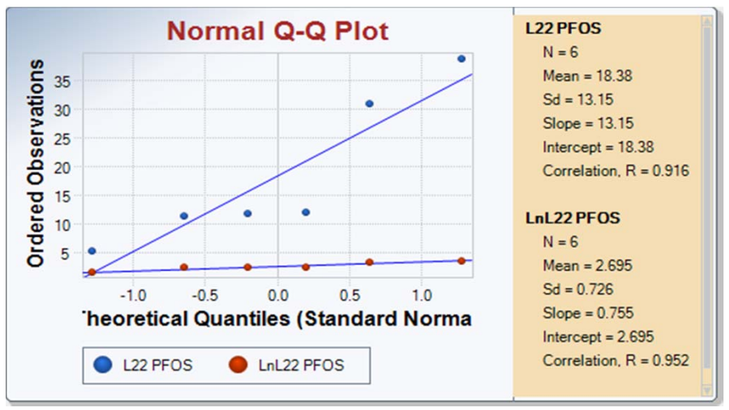


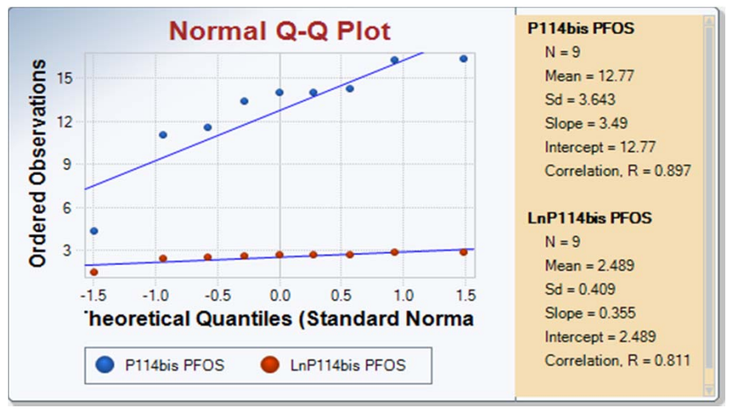
Figure 3. Q-Q plots for the 3M data sets.

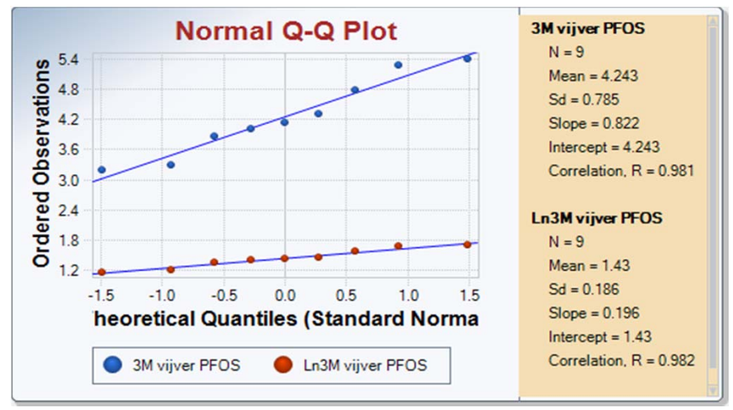


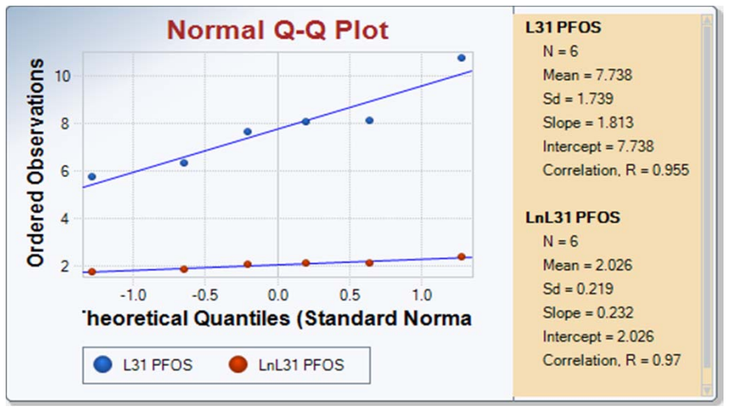


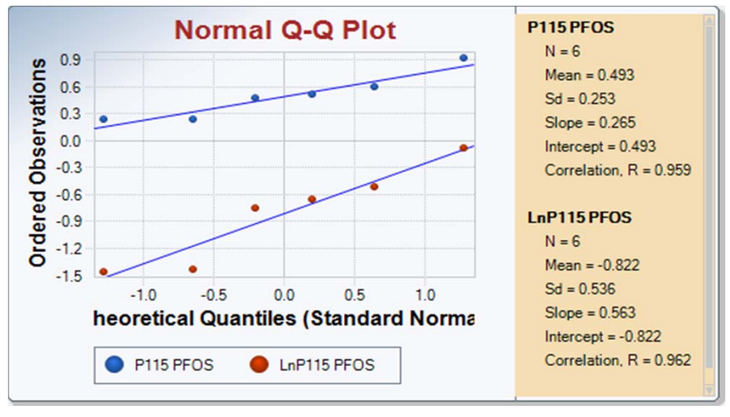


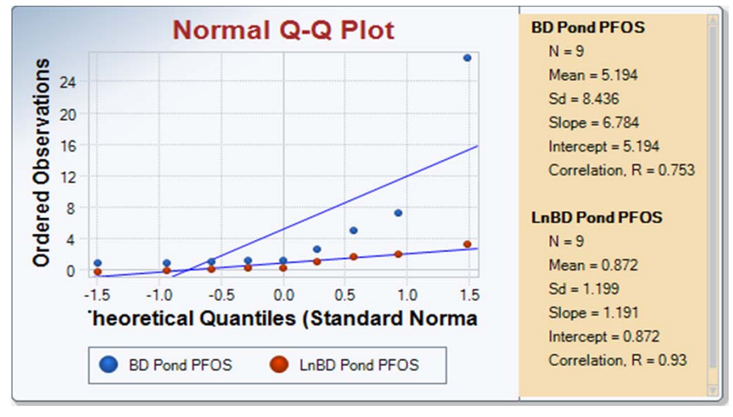






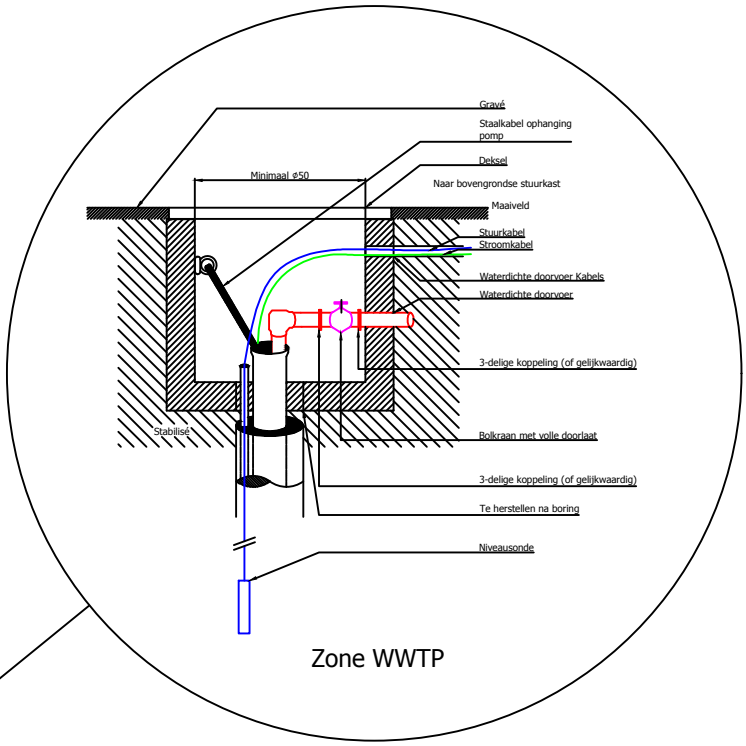




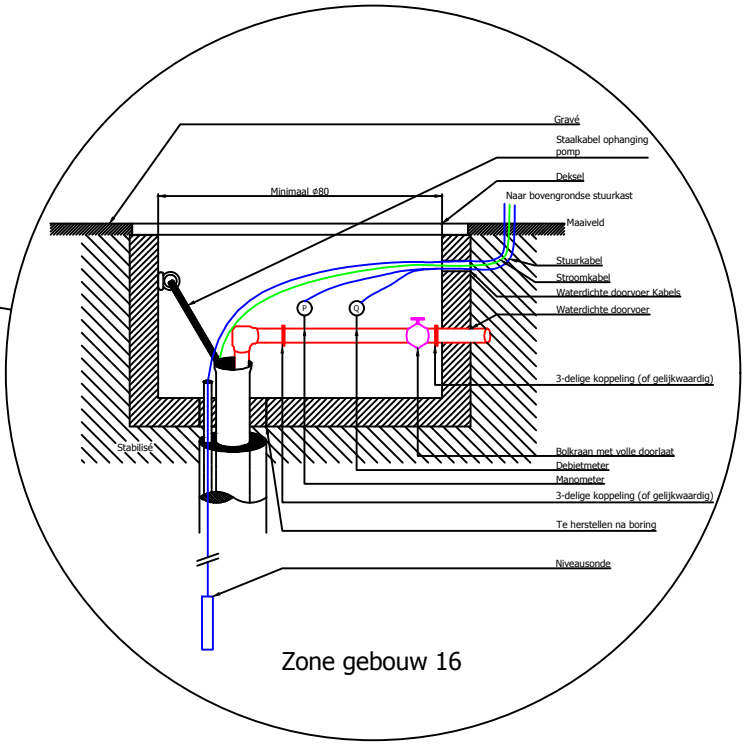


Bijlage 5

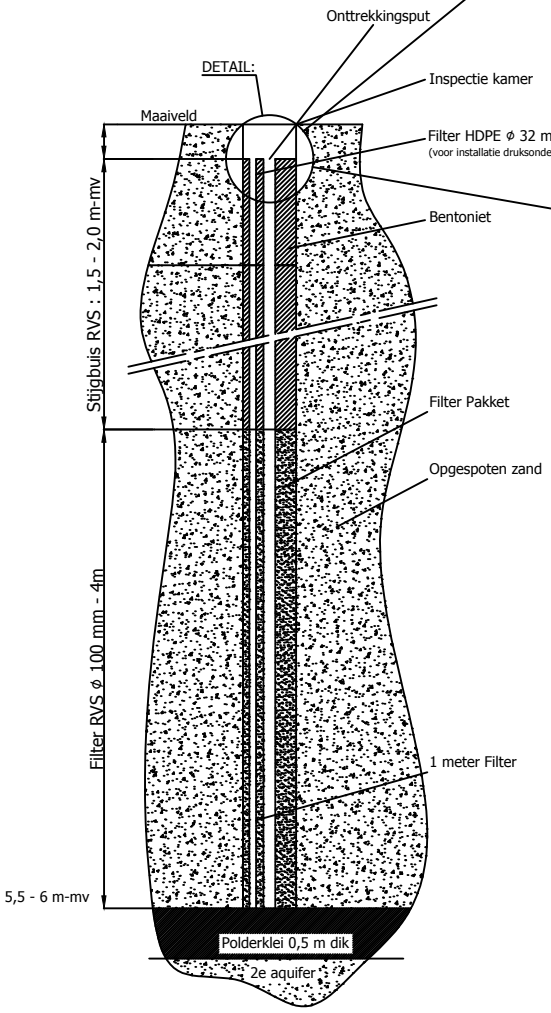
Schematische weergave pompput en inspectiekamer



Zone WWTP



Zone gebouw 16



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NVE	RMA	NHE	v0.1	☐ FINAL ☒ DRAFT

Environmental Resources Management n.v.

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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

Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.:	Titel:
0287933	6de Tussentijds verslag Bodemsaneringswerken

Bijlage:	Beschrijving:
10	Doorsnede Pompput en inspectiekamer

Fase:
TTV-6

Schaal:	Formaat:	Bestandsnaam:	Datum:
-/-	A4	3M-Doorsnedes.dwg	07/10/2015

Deel 3: Kaartmateriaal

Eerste gefaseerd bodemsaneringsproject -
Zesde tussentijds verslag bodemsaneringswerken
Periode juli 2014 - juli 2015

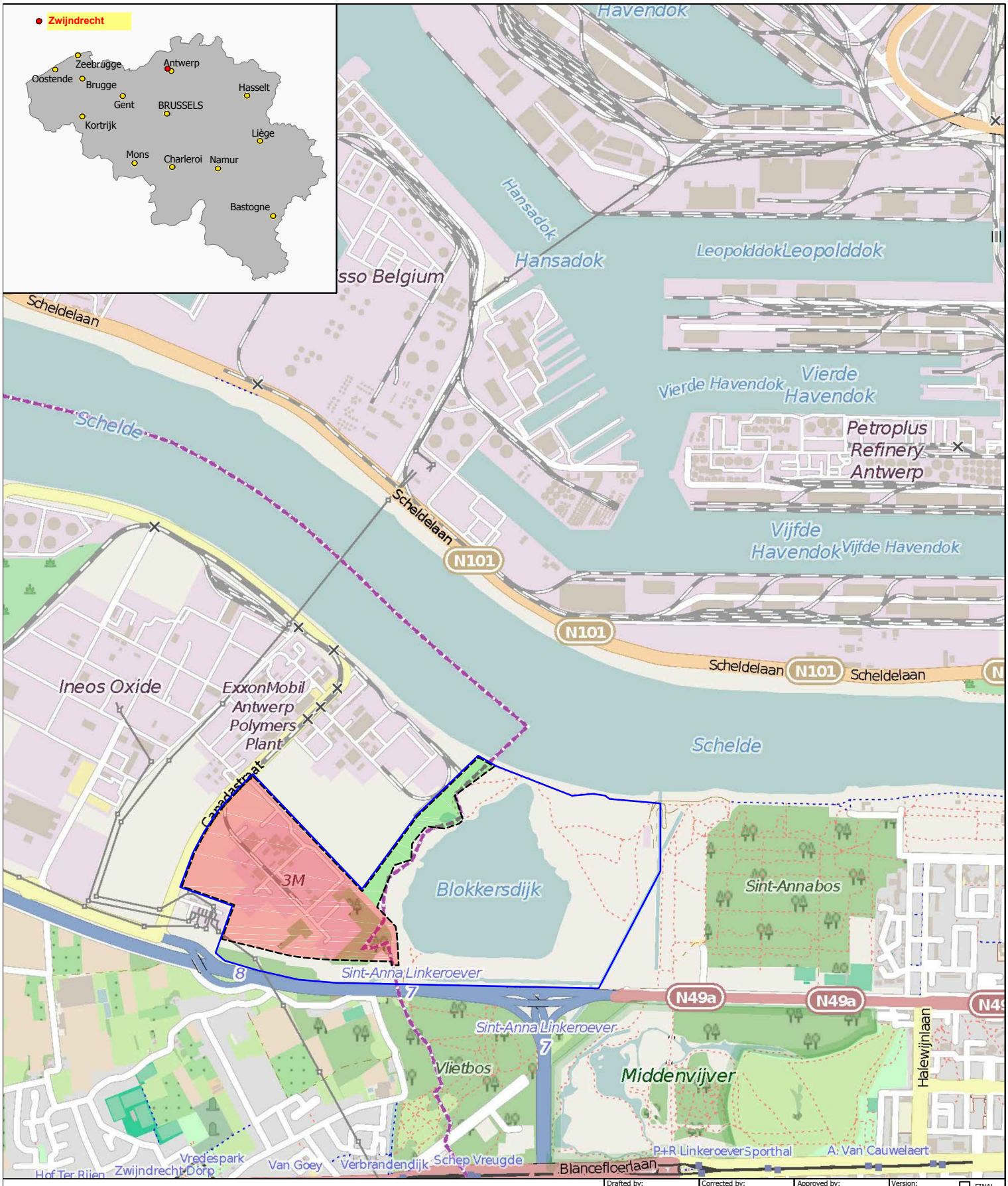
3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

5 april 2016

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)

Drafted by:	Corrected by:	Approved by:	Version:
NVE	RMA	NHE	v0.1

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1348 Louvain-la-Neuve
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Fax.: 010/48.35.36

Client / Project Name:	Location:
3M Belgium bvba	Zwijndrecht

Project Number:	Title:
0287933	6de Tussentijds Verslag Bodemsaneringswerken

Bijlage:	Description:
2	Topografische situering
Phase: TTV-6	© OpenStreetMap-contributors

Scale:	Paper Size:	File Name:	Date:
1:25 000	A4	3M Zwijndrecht_Topo.dwg	07/10/2015

Kaart 2

Overzichtsplan onderzoekslocatie

SCHELDE

NATUURGEBIED BLOKKERSDIJK

3M VIJVER

3M - PAD

BLOKKERSDIJKVIJVER

TOPHATGRACHT

CANADASTRAAT

TERREIN 3M

PALINGBEEK

EXPRESWEG - A11

CHARLES DE COSTERLAAN - N49A

RING - A14

Legende:

- 3M terreingrens
- Kadastrale grens
- Kadastrale nummer
- Gebouw
- Nummer grondhoop
- Ondergrondse dijk
- Hek
- Stelcon plaat
- Peilbuis
- Peilbuis verdwenen
- Onttrekkingsput
- Oppervlaktewaterstaalnamepunt
- Regenwaterrioleringsput

0 35.0 70.0 105.0 140.0 175.0 (m)

Getekend: NVE	Gecontroleerd: RMA	Goedgekeurd: NHE	Versie: v0.1	FINAL / DRAFT

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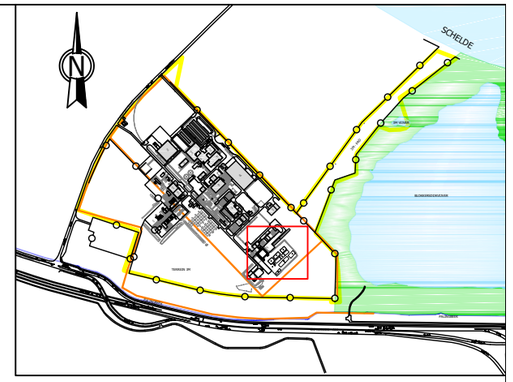
Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht
Projectnr.: 0287933	Titel: 6de Tussentijds Verslag Bodemsaneringswerken
Bijlage: 3	Beschrijving: Overzicht van de onderzoekslocatie met aanduiding van Peilbuizen
Fase: TTV-6	
Schaal: 1:3500	Formaat: A3
Bestandsnaam: 3M Zwijndrecht.dwg	Datum: 07/10/2015

Kaart 3

Detailplan ligging grondhopen



0804
0803
0801
008
011
①
②
③
④
⑤
⑥
⑦
⑧
⑨
⑩

Legende:

- 3M terreingrens
- Kadastrale grens
- 267v2 Kadastrale nummer
- 029 Gebouw
- ③ Nummer grondhoop
- Ondergrondse dijk
- Hek
- Stelcon plaat

0 7.5 15.0 22.5 30.0 37.5 (m)

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
NVE	RMA	NHE	v0.1	☐ FINAL ☒ DRAFT

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Place de l'Université 16, 1348 Louvain-la-Neuve, Tel.: 010/48.35.39, Fax.: 010/48.35.36

Klant / Projectnaam:	3M Belgium bvba
Locatie:	Zwijndrecht
Projectnr.:	0287933
Titel:	6de Tussentijds Verslag Bodemsaneringswerken
Bijlage:	4
Beschrijving:	Overzicht Grondhopen
Fase:	TTV-6
Schaal:	1:750
Formaat:	A3
Bestandsnaam:	3M Zwijndrecht.dwg
Datum:	07/10/2015

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 Palingbeek
G. Monsternamelocaties Regenwaterriolering



Legende:

- 3M terreingrens
- Kadastrale grens
- 267v2 — Kadastrale nummer
- 029 — Gebouw
- ③ — Nummer grondhoop
- Ondergrondse dijk
- Hek
- Stelcon plaat
- Peilbuis
- Peilbuis verdwenen
- Onttrekkingsput
- Oppervlaktewaterstaalnamepunt
- Regenwaterrioleringsput

0 7.5 15.0 22.5 30.0 37.5 (m)

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
NVE	RMA	NHE	v0.1	☐ FINAL ☒ DRAFT

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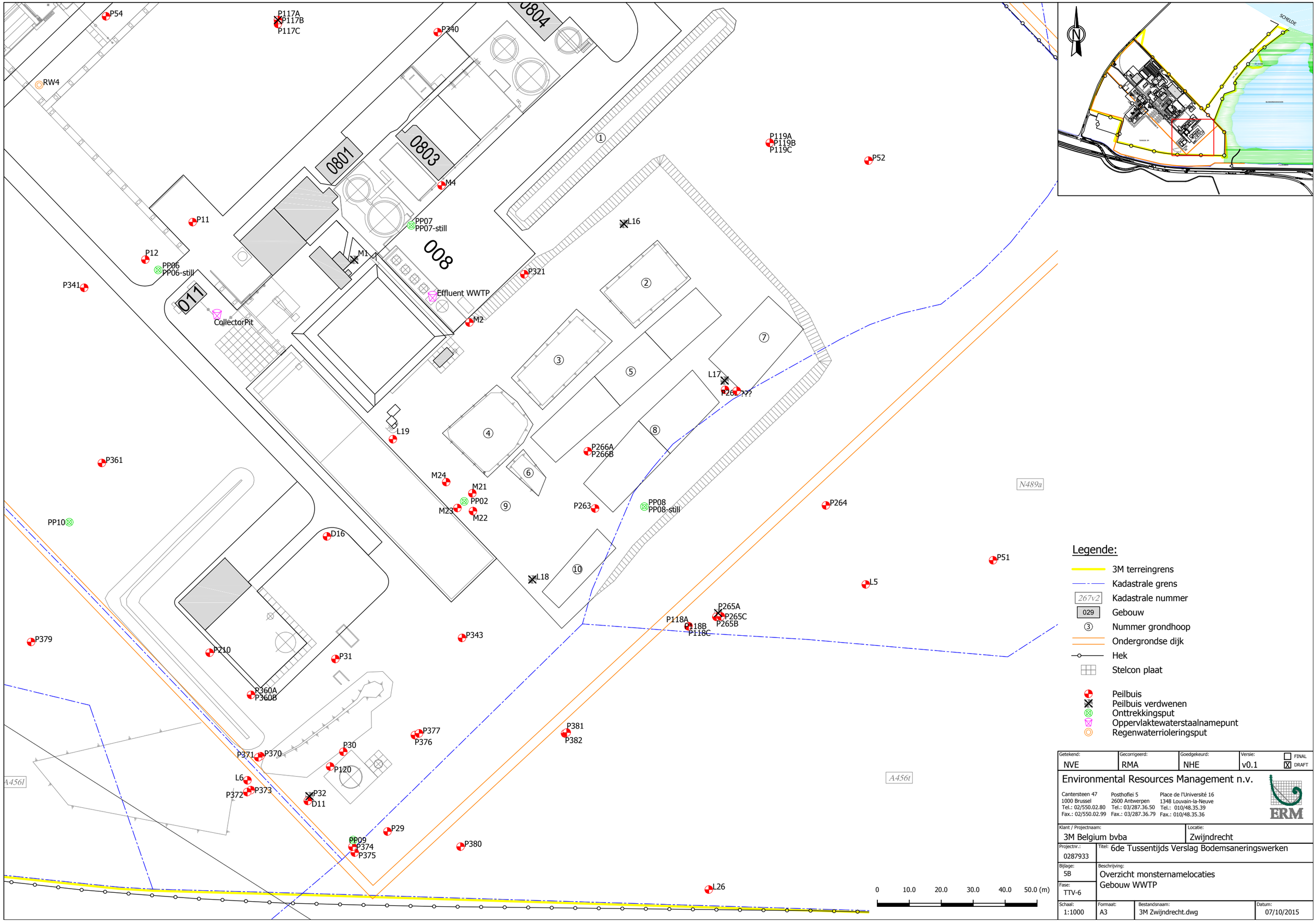
Place de l'Université 16, 1348 Louvain-la-Neuve, Tel.: 010/48.35.39, Fax.: 010/48.35.36

Klant / Projectnaam:	3M Belgium bvba
Locatie:	Zwijndrecht
Projectnr.:	0287933
Titel:	6de Tussentijds Verslag Bodemsaneringswerken
Bijlage:	5A
Beschrijving:	Overzicht monsternamelocaties Bronzone Gebouw 16 en zone ondergrondse tanken
Fase:	TTV-6
Schaal:	1:750
Formaat:	A3
Bestandsnaam:	3M Zwijndrecht.dwg
Datum:	07/10/2015



Legende:

- 3M terreingrens
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- 267v2 Kadastrale nummer
- 029 Gebouw
- ③ Nummer grondhoop
- Ondergrondse dijk
- Hek
- Stelcon plaat
- Peilbuis
- Peilbuis verdwenen
- Onttrekkingsput
- Oppervlaktewaterstaalnamepunt
- Regenwaterrioleringsput

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
NVE	RMA	NHE	v0.1	☐ FINAL ☒ DRAFT

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Place de l'Université 16, 1348 Louvain-la-Neuve, Tel.: 010/48.35.39, Fax.: 010/48.35.36

Klant / Projectnaam:	3M Belgium bvba
Locatie:	Zwijndrecht
Projectnr.:	0287933
Titel:	6de Tussentijds Verslag Bodemsaneringswerken
Bijlage:	5B
Beschrijving:	Overzicht monsternamelocaties Gebouw WWTP
Fase:	TTV-6
Schaal:	1:1000
Formaat:	A3
Bestandsnaam:	3M Zwijndrecht.dwg
Datum:	07/10/2015



Legende:

- 3M terreingrens
- Kadastrale grens
- 267v2 Kadastrale nummer
- 029 Gebouw
- ③ Nummer grondhoop
- Ondergrondse dijk
- Hek
- Stelcon plaat

- Peilbuis
- Peilbuis verdwenen
- Onttrekkingsput
- Oppervlaktewaterstaalnamepunt
- Regenwaterrioleringsput

0 15.0 30.0 45.0 60.0 75.0 (m)

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
NVE	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

Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.:	Titel:
0287933	6de Tussentijds Verslag Bodemsaneringswerken

Bijlage:	Beschrijving:
5C	Overzicht monsternamelocaties 2de Aquifer
Fase: TTV-6	

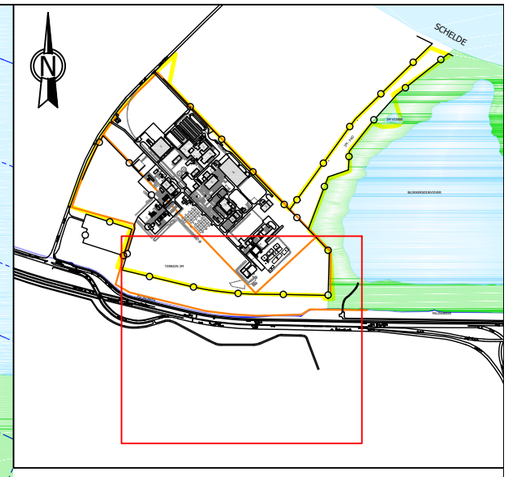
Schaal:	Formaat:	Bestandsnaam:	Datum:
1:1500	A3	3M Zwijndrecht.dwg	07/10/2015

TERREIN 3M

PALINGBEEK

EXPRESWEG - A11

SCHELDE



Legende:

- 3M terreingrens
- Kadastrale grens
- 267v2 Kadastrale nummer
- 029 Gebouw
- ③ Nummer grondhoop
- Ondergrondse dijk
- Hek
- Stelcon plaat

- Peilbuis
- Peilbuis verdwenen
- Onttrekkingsput
- Oppervlaktewaterstaalnamepunt
- Regenwaterrioleringsput

0 30.0 60.0 90.0 120.0 150.0 (m)

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
NVE	RMA	NHE	v0.1	☐ FINAL ☒ DRAFT

Environmental Resources Management n.v.

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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

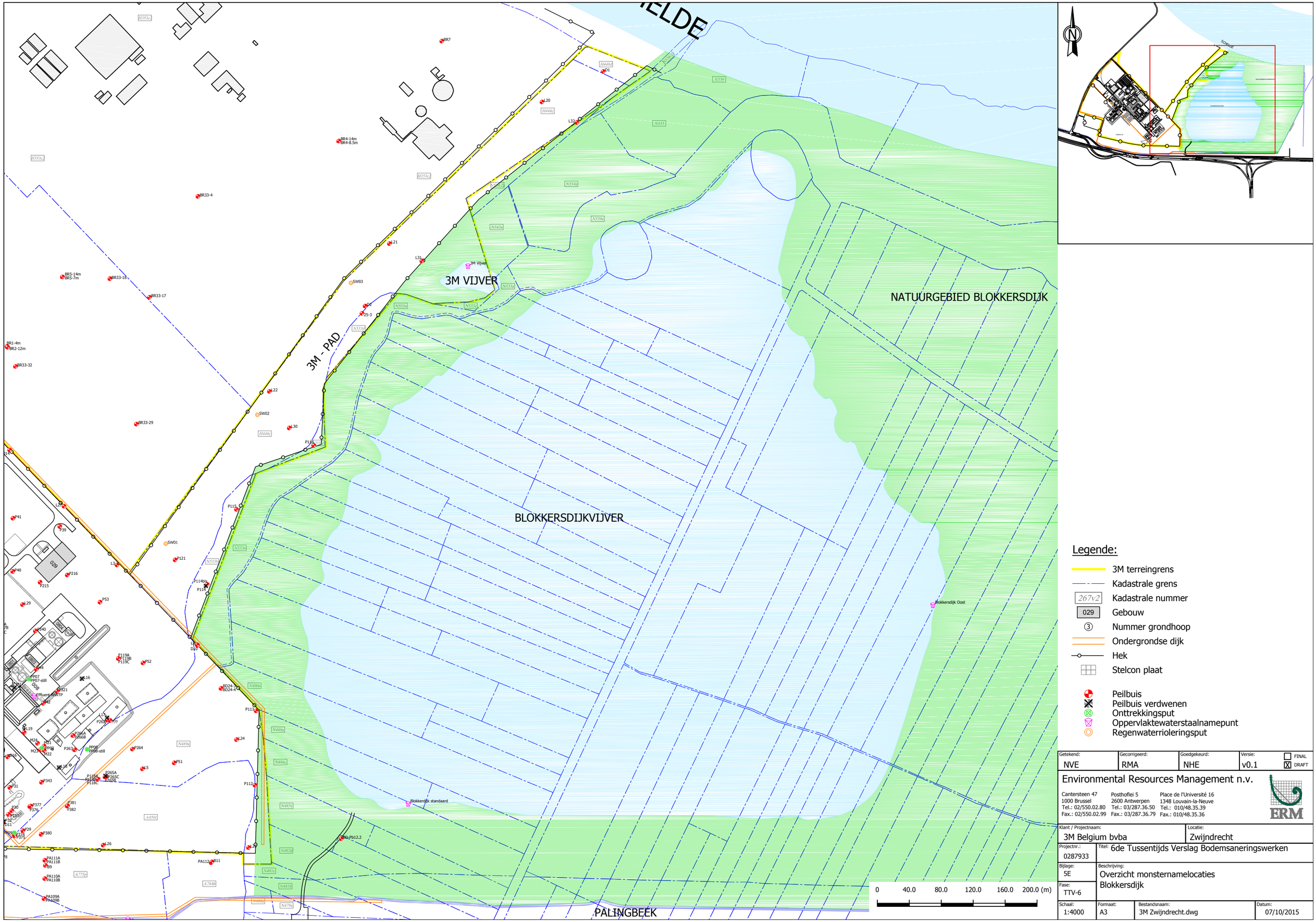
Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.:	Titel:
0287933	6de Tussentijds Verslag Bodemsaneringswerken

Bijlage:	Beschrijving:
5D	Overzicht monsternamelocaties Zuidelijke grens en Zwijndrecht wells

Fase:
TTV-6

Schaal:	Formaat:	Bestandsnaam:	Datum:
1:3000	A3	3M Zwijndrecht.dwg	07/10/2015


3M VIJVER
3M - PAD
NATUURGEBIED BLOKKERSDIJK
BLOKKERSDIJKVIJVER
PALINGBEEK
3M Vijver
Blokkersdijk standaard
Blokkersdijk Oost
SW01
SW02
SW03
BR1-4m
BR2-12m
BR33-32
BR5-14m
BR5-7m
BR33-18
BR33-17
BR33-29
BR33-4
BR4-14m
BR4-8.5m
BR7
P41
P39
P40
P216
P215
P53
P52
P51
P121
P115
P113
P112
P264
P343
P380
P29
P30
P377
P378
P381
P382
P373
P375
P374
P119A
P119B
P119C
P265A
P265B
P265C
P265D
P118A
P118B
P118C
P263
P266A
P266B
P268
P268-still
P267
P269
P270
P271
P272
P273
P274
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P276
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P278
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P304
P305
P306
P307
P308

SCHELDE

3M VIJVER

3M - PAD

NATUURGEBIED BLOKKERSDIJK

BLOKKERSDIJKVIJVER

PALINGBEEK

EXPRESWEG - A11

CHARLES DE COSTERLAAN - N49A

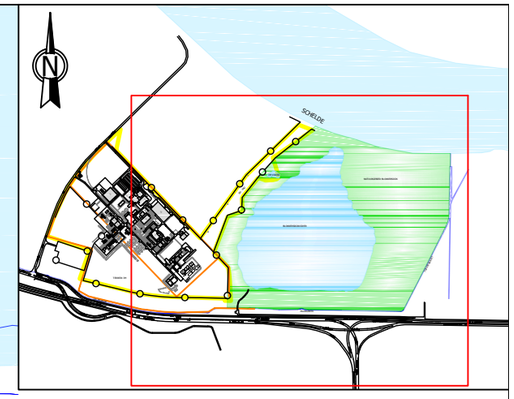
TOPHATGRACHT

RING 1 - A12

Remaingestation

Blokkersdijk Oost

Blokkersdijk standaard



Legende:

- 3M terreingrens
- Kadastrale grens
- 267v2 Kadastrale nummer
- 029 Gebouw
- ③ Nummer grondhoop
- Ondergrondse dijk
- Hek
- Stelcon plaat
- Peilbuis
- Peilbuis verdwenen
- Onttrekkingsput
- Oppervlaktewaterstaalnamepunt
- Regenwaterrioleringsput

0 60.0 120.0 180.0 240.0 300.0 (m)

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	☐ FINAL ☒ DRAFT
NVE	RMA	NHE	v0.1	

Environmental Resources Management n.v.

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Tel.: 02/550.02.80
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Posthoflei 5
2600 Antwerpen
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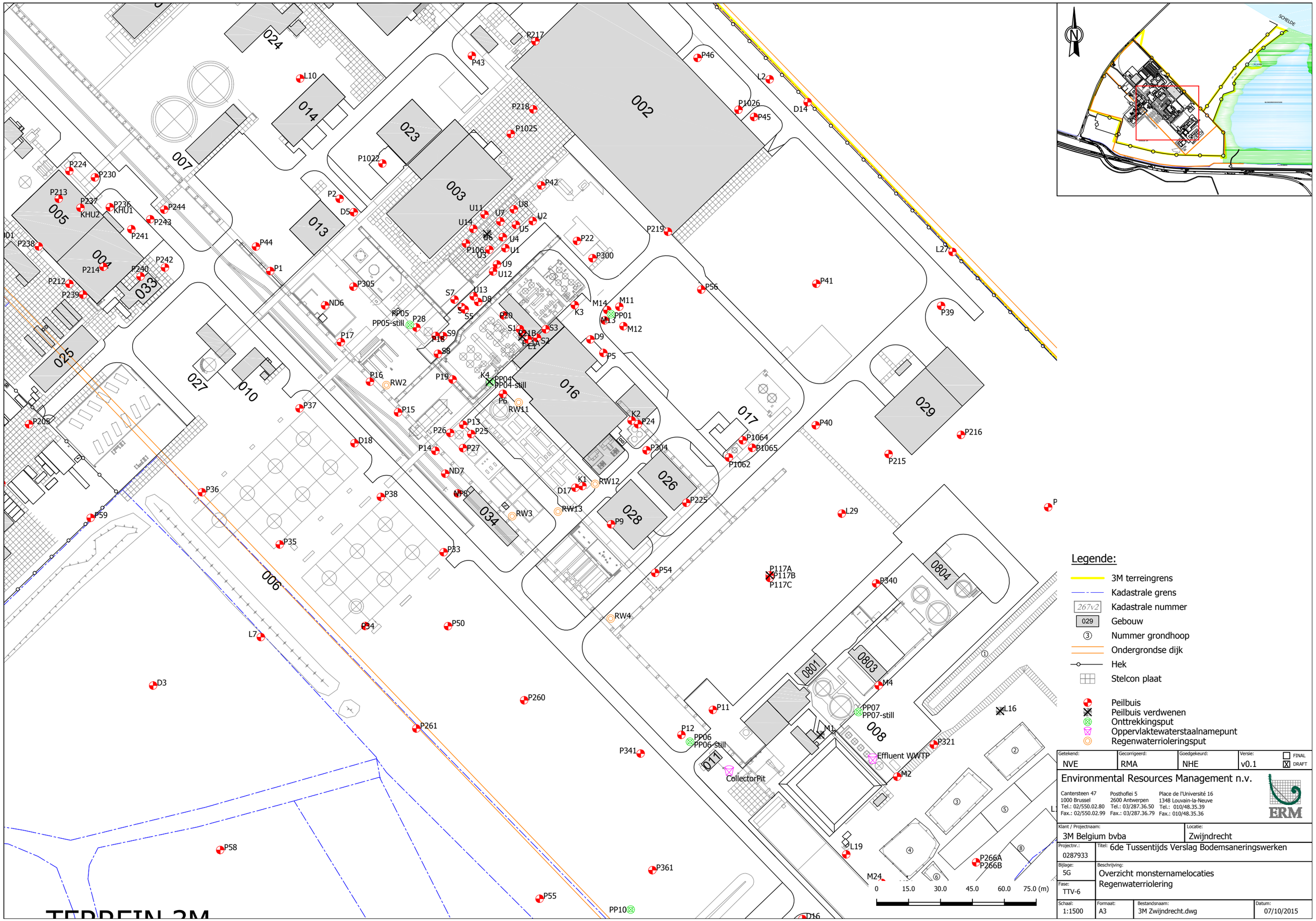
Place de l'Université 16
1348 Louvain-la-Neuve
Tel.: 010/48.35.39
Fax.: 010/48.35.36

Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.:	Titel:
0287933	6de Tussentijds Verslag Bodemsaneringswerken

Bijlage:	Beschrijving:
5F	Overzicht monsternamelocaties Palingbeek
Fase: TTV-6	

Schaal:	Formaat:	Bestandsnaam:	Datum:
1:6000	A3	3M Zwijndrecht.dwg	07/10/2015



Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
NVE	RMA	NHE	v0.1	FINAL / DRAFT

Environmental Resources Management n.v.

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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.:	Titel:
0287933	6de Tussentijds Verslag Bodemsaneringswerken

Bijlage:	Beschrijving:
5G	Overzicht monsternamelocaties Regenwaterriolering

Fase:
TTV-6

Schaal:	Formaat:	Bestandsnaam:	Datum:
1:1500	A3	3M Zwijndrecht.dwg	07/10/2015

Kaart 5

Onttrekkingsfilters grondwateronttrekking

004
033
027
010
006
PP05
PP05-still
PP04
PP04-still
016
PP01
017
029
026
028
034
0804
0801
0803
PP07
PP07-still
008
PP06
PP06-still
011
PP10
PP02
PP08
PP08-still
PP09
RREIN 3M



Legende:

- 3M terreingrens
- Kadastrale grens
- 267v2 — Kadastrale nummer
- 029 — Gebouw
- ③ — Nummer grondhoop
- Ondergrondse dijk
- Hek
- Stelcon plaat
- Onttrekkingsput

0 15.0 30.0 45.0 60.0 75.0 (m)

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	FINAL / DRAFT
NVE	RMA	NHE	v0.1	☐ FINAL ☒ DRAFT

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1000 Brussel
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2600 Antwerpen
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Klant / Projectnaam:	3M Belgium bvba
Locatie:	Zwijndrecht
Projectnr.:	0287933
Titel:	6de Tussentijds Verslag Bodemsaneringswerken
Bijlage:	9
Beschrijving:	Onttrekkingsfilters grondwateronttrekking
Fase:	TTV-6
Schaal:	1:1500
Formaat:	A3
Bestandsnaam:	3M Zwijndrecht.dwg
Datum:	07/10/2015