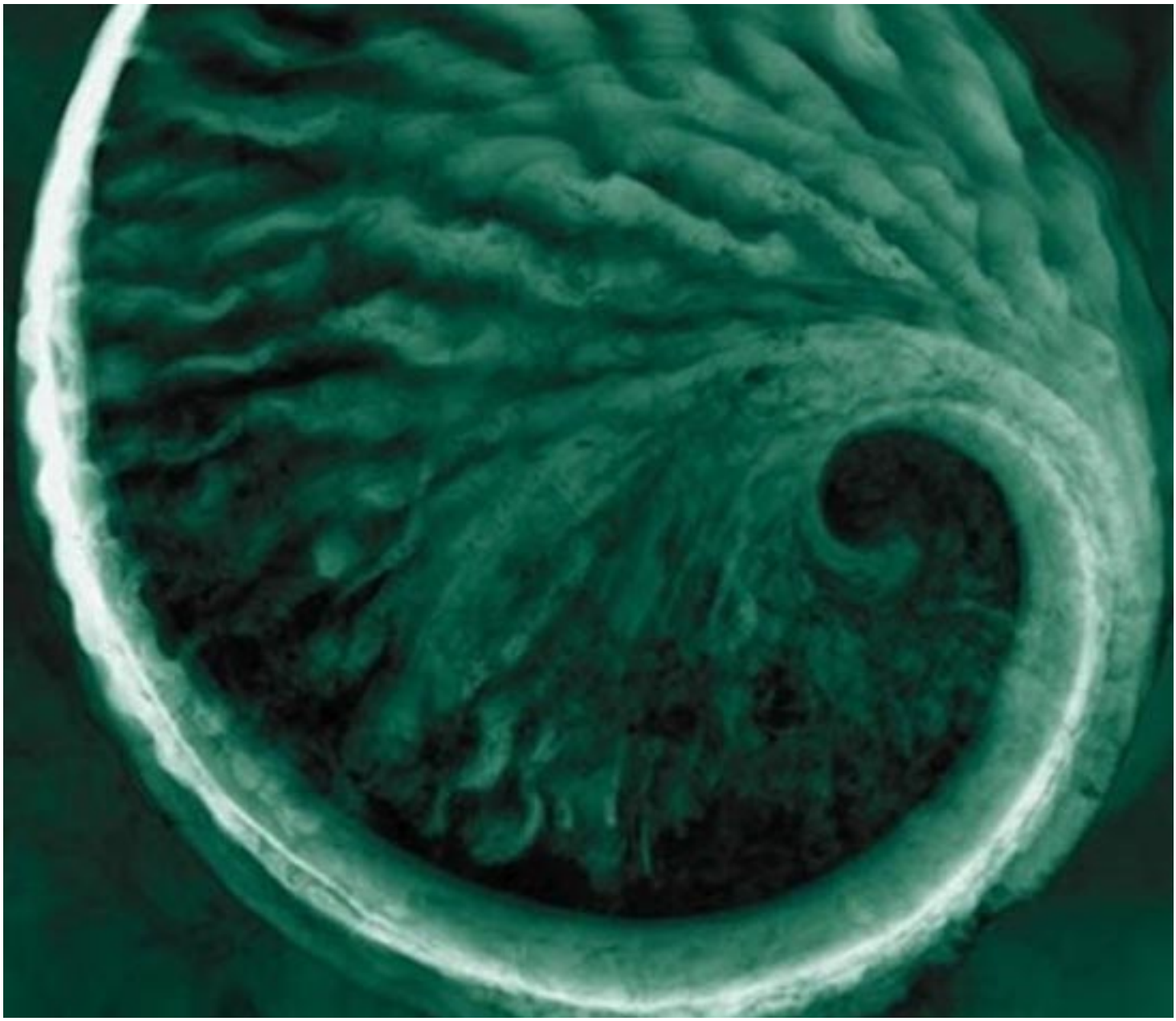

Eerste gefaseerd bodemsaneringsproject - Vijfde tussentijds verslag bodemsaneringswerken

Periode juli 2013 - juli 2014

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

2 februari 2015

www.erm.com

RAPPORT

Eerste gefaseerd bodemsaneringsproject - Vijfde tussentijds verslag bodemsaneringswerken

Periode juli 2013 – juli 2014

3M Belgium bvba
Canadastraat 11 te 2070 Zwijndrecht

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Voor en namens ERM nv

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Functie: Principal Partner

Handtekening:



Datum: 2 februari 2015

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

INHOUDSTAFEL

EXECUTIVE SUMMARY

ADMINISTRATIEVE GEGEVENS *I*

1 INLEIDING *1*

2 VOORSTUDIE *2*

2.1 OMGEVINGSKENMERKEN *2*
2.2 BEKNOPTE HISTORIEK EN BESCHRIJVING VAN HET TERREIN *2*
2.3 GEOLOGISCHE EN HYDROGEOLOGISCHE GEGEVENS *2*
2.4 RESULTATEN VOORMALIGE BODEMONDERZOEKEN EN/OF SANERINGEN *3*

3 ALGEMENE BESCHRIJVING SANERINGSWERKEN *5*

3.1 ALGEMEEN *5*
3.2 SANERINGSWERKEN ZOALS VOORZIEN IN HET EERSTE GEFASEERD BODEMSANERINGSPROJECT *5*
3.3 WIJZIGINGEN IN HET BODEMSANERINGSPROJECT VOLGENS HET EERSTE, TWEEDE, DERDE EN VIERDE TUSSENTIJDS VERSLAG *12*

4 UITGEVOERDE MONITORING EN RESULTATEN *19*

4.1 ALGEMEEN *19*
4.2 GRONDHOOP VERONTREINIGD MET KWIK EN FC VERBINDINGEN *20*
4.3 FC BRONZONES: GEBOUW 16 EN WWTP *20*
4.4 TWEEDE AQUIFER *27*
4.5 ZUIDELIJKE TERREINGRENS *30*
4.6 GEBIED TEN ZUIDEN VAN DE EXPRESWEG (Z-PEILBUIZEN) *33*
4.7 ZONE ONDERGRONDSE TANKEN *35*
4.8 NATUURGEBIED BLOKKERSDIJK *37*
4.9 PALINGBEEK EN TOPHAT GRACHT *41*
4.10 REGENWATERCOLLECTORPUT *43*
4.11 VUILVRACHT RICHTING SCHELDE *45*
4.12 VOORSTEL MONITORINGSPROGRAMMA 2014 *46*

5 GRONDWATERONTTREKKING BRONZONE *49*

5.1 OPVOLGING ONTTREKKINGSSYSTEEM *49*
5.2 MONITORING GRONDWATERSTANDEN *58*
5.3 VOORZIENE WERKEN VOOR VOLGEND JAAR *58*

6 VOORZIENE WERKEN IN VOLGENDE JAAR VAN DE SANERING *60*

6.1 GRONDWATERSANERING *60*

6.2 MONITORING GRONDWATER EN OPPERVLAKTEWATER 60
6.3 PLANNING 60

7 WIJZIGINGEN TEN OPZICHTE VAN BODEMSANERINGSPROJECT 61

8 CALAMITEITEN 62

8.1 PIPM INCIDENT TUSSEN GEBOUW 016 EN GEBOUW 003 62
8.2 20% HF INCIDENT TER HOOGTE VAN GEBOUW 017 62

9 TWEEDE FASE BODEMSANERINGSPROJECT 64

10 BESLUIT 65

11 VERKLARING EN ONDERTEKENING 67

BIJLAGEN

Bijlage 1 OVAM conformverklaring eerste gefaseerd bodemsaneringsproject
Bijlage 2 Situering onderzoekslocatie op topografische kaart
Bijlage 3 Overzichtsplan onderzoekslocatie
Bijlage 4 Detailplan ligging grondhopen
Bijlage 5 Detailplannen monsternamelocaties
Bijlage 6 Analysecertificaten
Bijlage 7 Overzicht veldmetingen en analyseresultaten
Bijlage 8 Statistische evaluatie analyseresultaten Blokkersdijk
Bijlage 9 Onttrekkingsfilters grondwateronttrekking
Bijlage 10 Schematische weergave pompput en inspectiekamer
Bijlage 11 Nota stabiliteitsdeskundige
Bijlage 12 Stijghoogtes en grondwaterisohypsen eerste en tweede aquifer

LIJST VAN AFKORTINGEN

ANB	Agentschap Natuur en Bos
BTEX	Benzeen, tolueen, ethylbenzeen en xylenen
ds	Droge stof
FC	Organofluoroverbindingen
IOA	Iso-octylacetaat
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
TFA	Trifluorazijnzuur
VMM	Vlaamse Milieumaatschappij
WWTP	Bedrijfswaterzuiveringsinstallatie 3M (“waste water treatment plant”)

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 fluorocarbons (FC) and mercury, and groundwater impacted with FC 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 FC 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 (road infrastructure) 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 2013 and July 2014. The remedial actions performed and monitoring results were discussed with OVAM during a meeting in November 2014.

In the period between July 2013 and July 2014 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;
   - Groundwater wells south of the express road E34 (Zwijndrecht Z-wells), final sampling;
   - Groundwater wells in the tank farm area (near Building 3), final sampling;
   - Blokkersdijk wells and pond, including 3M pond (groundwater and surface water);
   - Surface water in the Paling and Tophat ditches; and
   - 3M stormwater sewer and collector pit.

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

4. Environmental follow up of the following incidents at the site:
   - PIPM spill between Building 003 and Building 016 (October 2013), and
   - 20% HF spill outside Building 17 (September 2014).

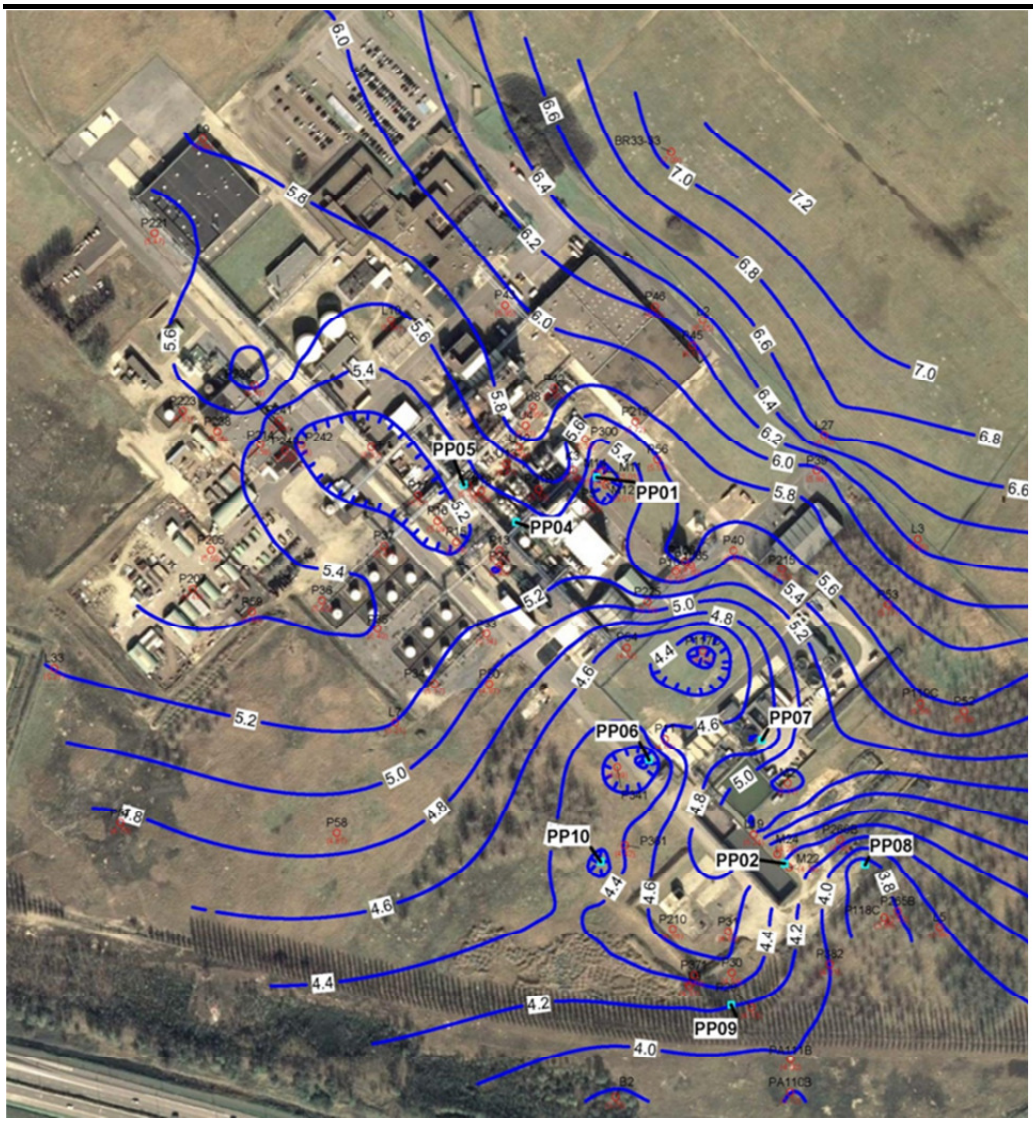
5. Evaluation of the analytical results of the July 2014 water samples by 3M Environmental Laboratory versus the analytical results of the duplicate samples by the laboratory of SGS.

Follow-up Monitoring of the P&T System

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

ERM inspects the P&T system regularly to verify 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 overall extraction system is functioning efficiently and effectively, but requires regular maintenance to remove sand and iron deposits accumulating in the wells. The system has a clear impact on the natural groundwater flow pattern of the first aquifer, as is shown in the figure below.

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

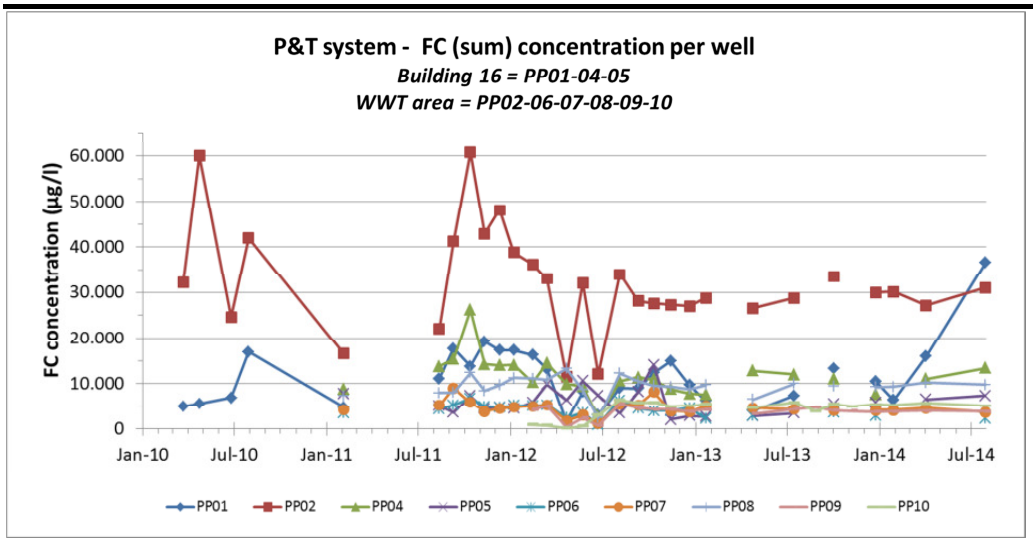


The stability of the buildings in the extraction areas is monitored on a six-monthly basis by an independent geotechnical expert to ensure no subsidence is occurring. The stability monitoring done in 2013 has indicated that the settlement caused by the extraction system stayed within the predefined limits. 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 can be ended.

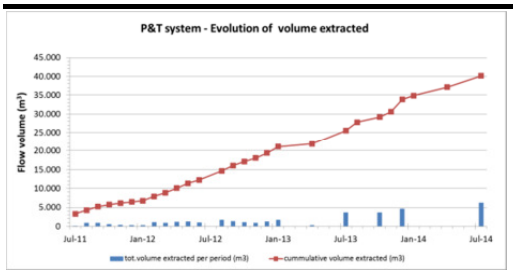
The average flow rate of the system (excluding well PP05 that operates intermittently) varies between minimum 0,9 m^3/day (well PP02) and maximum 9,1 m^3/day (well PP07). The average groundwater FC concentration ranges between 2,9 mg/L to 30,4 mg/L, depending on the extraction well, but the concentrations fluctuate significantly (see graph below). The long-term trend in FC concentrations in most of the extraction wells is stable, although fluctuations can occur.

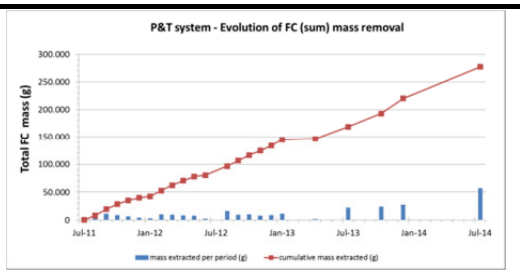
P&T system – Concentration Trends



Between July 2011 and June 2014 approximately 40.000 m^3 of contaminated groundwater was extracted and approximately 280 kg of FCs were removed, as shown in the graphs below.

P&T system – Efficiency Trends





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. Historical information and the available stability measurement data, as well as a review by an independent stability expert, seems to suggest that further stability monitoring is no longer necessary. The need for continuation of the settlement measurements will be assessed by an independent expert and discontinued if appropriate.

Monitoring of the Soil Pile Impacted with FCs and Mercury

A visual inspection by ERM in 2011 showed that the cover of the soil pile was damaged and needed to be replaced. In 2012 a new liner was place on top of the old damaged liner and in 2013 the annual visual inspection showed that the condition of the liner was intact. Historical data has indicated that the soil pile is placed on top of a liner.

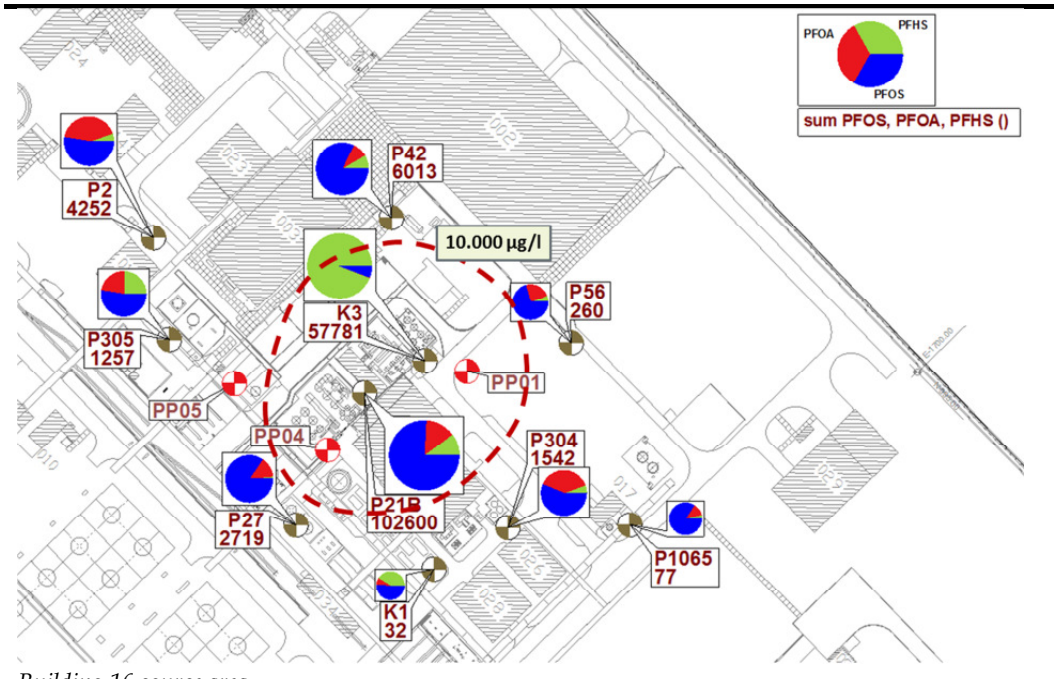
The new cover and the liner at the bottom prevent contaminant migration from the pile to the soil and first aquifer. Based on these observations and in consultation with OVAM (dd. Oct.2013) it was decided that the frequency of the visual inspection would be decreased from yearly to once every five years. Therefore the next visual inspection is scheduled for 2018 and no inspection was done this year (2014).

Monitoring Groundwater, Surface Water and Storm Water Quality

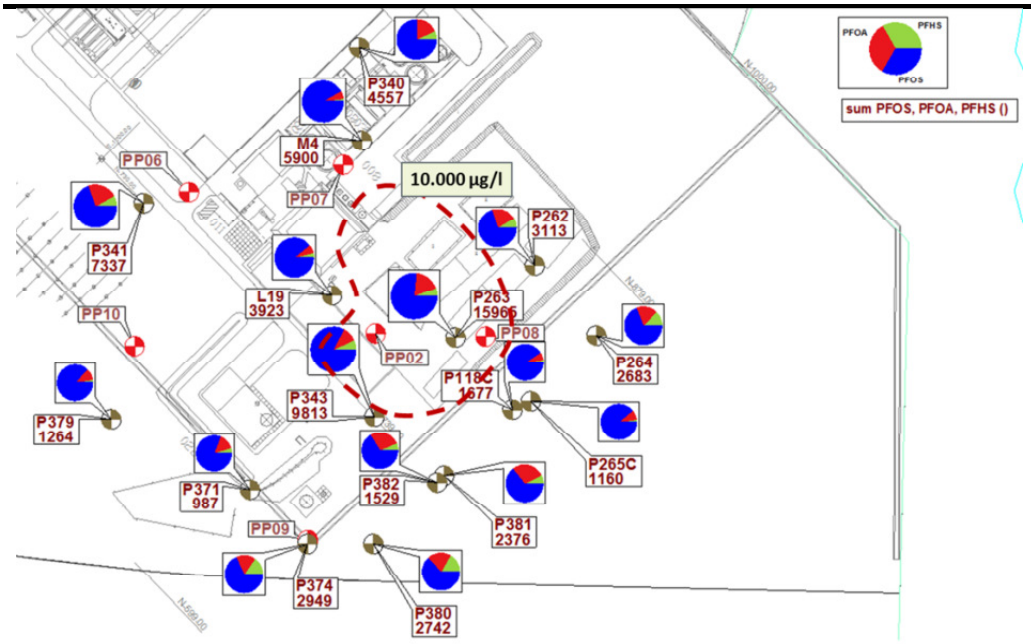
The remedial objectives of the monitoring program are to verify if the P&T system is mitigating the horizontal migration in the first aquifer to the areas of concern (Blokkersdijk nature reserve and the Paling ditch at the southern site boundary) as well as the vertical migration of contaminants from 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** seven groundwater wells are sampled annually in the Building 16 area and in the WWTP area nine wells are sampled annually, and seven wells sampled semi-annually. The results of the July 2014 monitoring are shown in the figures below together with the sum FC-concentration contour (10.000 µg/l).

FC Source Areas - Results July 2014 Monitoring



Building 16 source area

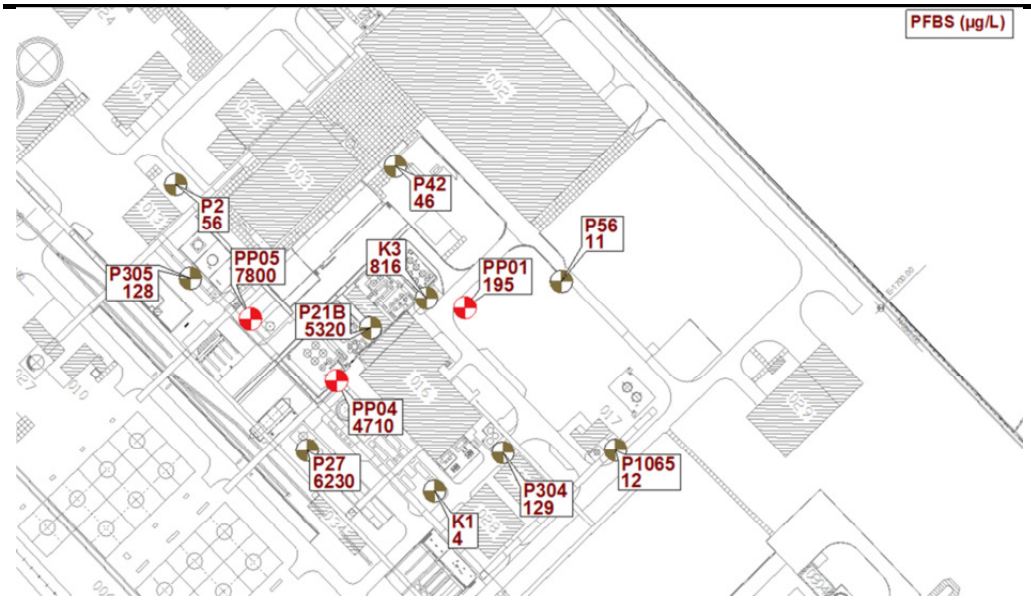


WWTP source area

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 and P42. The extent of the sum FC concentration contour appears to be slightly increased compared to the contour based on the results of the previous monitoring round (2012-13). In the WWTP area, groundwater concentrations in most of the wells are stable or decreasing, except for well P263 in the center of the plume. The extent of the sum FC concentration contour appears to be similar to the contour based on the previous monitoring results (2012-13).
The general stable/decreasing trend in FC concentrations suggest that the shallow P&T system installed in the two source areas is effectively reducing the concentrations of FCs in groundwater in the Building 16 and WWTP area.

The use of PFOS on-site was eliminated in 2002 and it was replaced with PFBS. 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. In addition, PFBS has a significantly lower toxicity than PFOS, which is why PFBS was selected to replace PFOS. Recent groundwater analytical data indicates the presence, and increase in concentrations, of PFBS in the area of Building 16.

During the July 2014 monitoring, additional wells were sampled and analyzed for PFBS to assess the PFBS impact. The results of the monitoring is shown in the following figure. None of the measured concentrations exceeds the monitoring value for sum FC's (10.000 µg/L).



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.

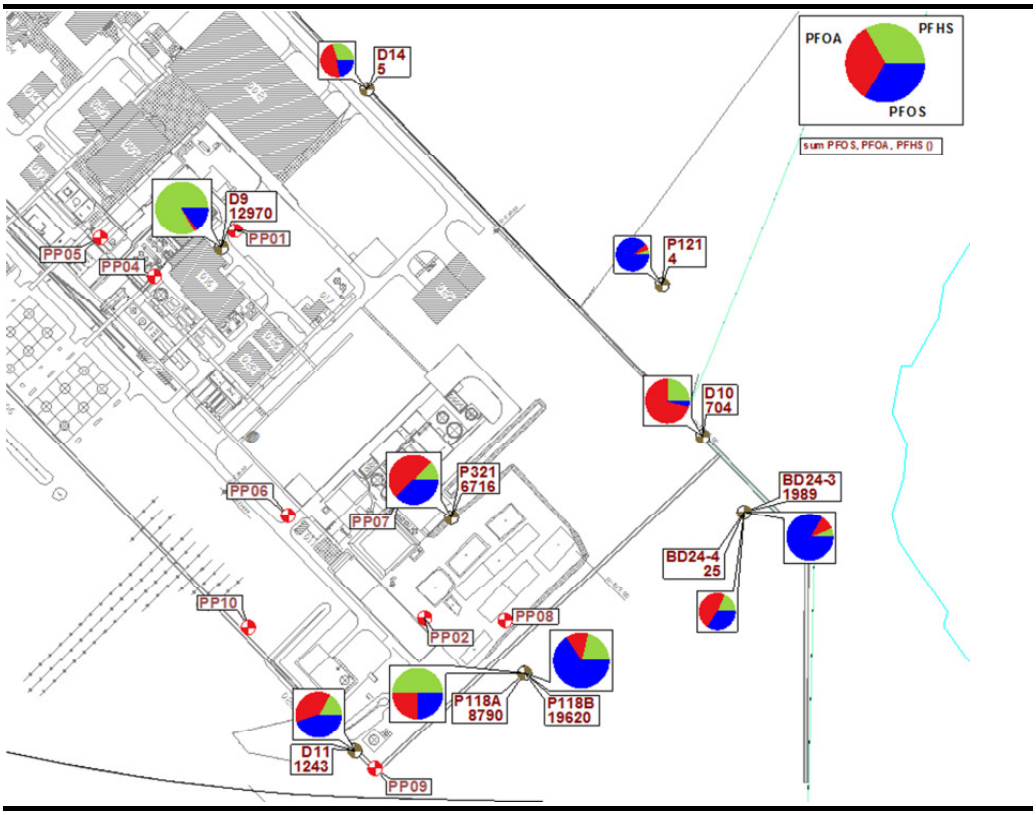
ERM recommends to continue the current groundwater monitoring program for wells completed in the shallow (1st) aquifer, but to increase the monitoring frequency of wells P21B, P42, P304 en P305 (Building 16 area) and M4, L19 en P263 (WWTP area) to semi-annually to allow a more frequent (semi-annual) evaluation of the evolution of the sum FC-contour. For the Building 18 source area it is further more recommended to replace the monitoring of well K1 with well P27, as the concentrations in well K1 have been stable and low since the start of the monitoring in 2009 (the current sum FC concentrations is about 30 µg/l). Well P27 would be monitored semi-annually.

To monitor the groundwater quality of the **second aquifer**, five wells are sampled annually, four 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 2014 monitoring are shown in the following figure.

The evaluation of the monitoring results shows that the groundwater concentrations in the second aquifer fluctuate but in general are decreasing or stable, suggesting that the shallow P&T system in the first aquifer is achieving capture and mitigating downward migration of impacted groundwater. Near Building 16, the PFOS concentration in well D09 appears to be increasing, but more data are needed to confirm any concentration trends in this well. In the WWTP area, most FC concentrations show a stable to declining trend in the most recent data. FC concentrations in wells P321 and BD24-3 have fluctuated

and continued monitoring of these wells will be performed to assess the long-term trend.

Second Aquifer - Results July 2014 Monitoring



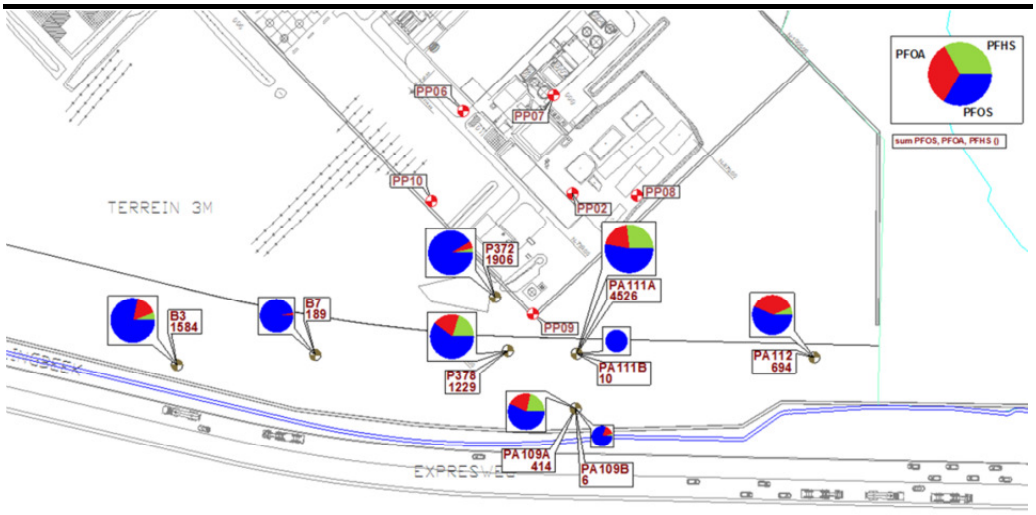
The FC concentrations are below the five time limit value based on the concentrations measured during startup of the remediation (mid 2011). At the northeast property boundary (downgradient from the source areas in the first aquifer) concentrations are below 10% of the solubility. Therefore, remedial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

Based on the results of the monitoring ERM recommends that the impact of the P&T system on the FC concentrations in the second aquifer be assessed through continued monitoring. In addition, ERM recommends to keep the current monitoring frequency for wells P321 (quarterly) and BD 24-3 (semi-annually). Furthermore, ERM recommends increasing the monitoring frequency from annually to semi-annually for well D9.

The groundwater quality along the **southern site boundary** is currently being monitored semi-annually in seven wells. The results of the July 2014 monitoring are shown in the following figure.

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. The concentrations in well B3 appear to be increasing, but more data are needed to confirm any concentration trends in this well.

Southern Site Boundary - Results July 2014 Monitoring

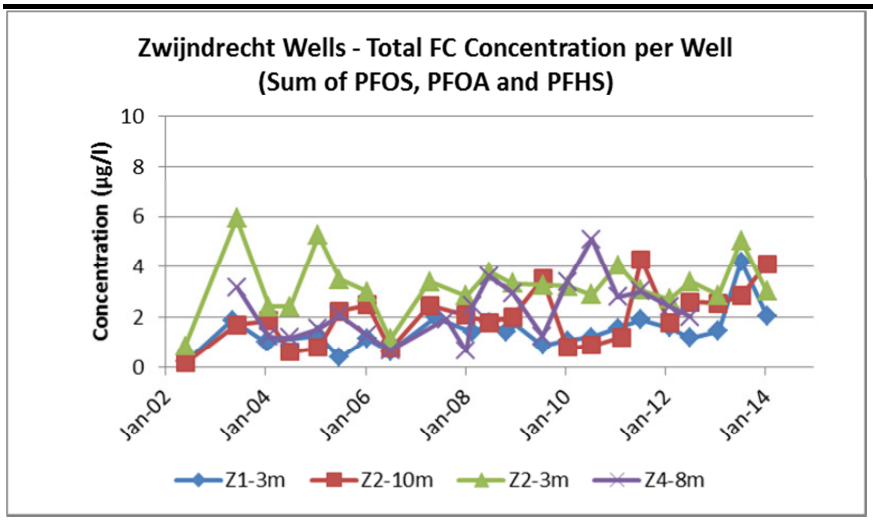


ERM recommends continuing the current groundwater monitoring program (semi-annual sampling).

The four groundwater monitoring wells south of the express road (highway E34), the **Zwijndrecht Z-wells**, have been periodically monitored since 2002 (and semi-annually since 2009). The purpose of the monitoring is to evaluate if the FC concentrations in this area are increasing compared to the results prior to the start of the remedial monitoring.

A review of the historic data (prior to 2009) shows that FC concentrations lie around the detection limit, see graph below.

Zwijndrecht Z-wells – Monitoring Results since 2002



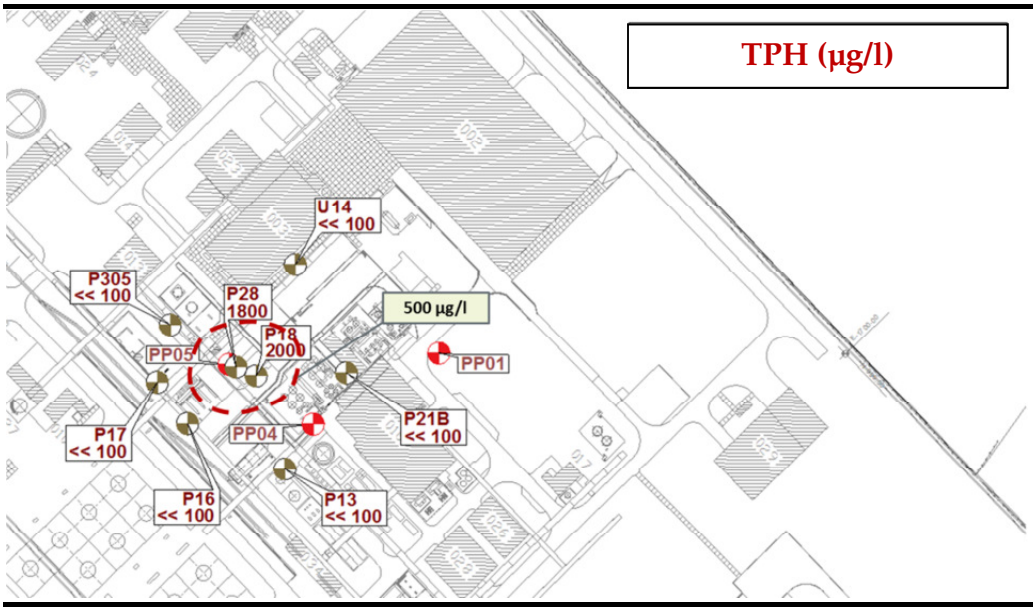
The graph of the sum of the FC compounds shows that the concentrations fluctuate, but remain in the low µg/L range since 2009. No significant concentration increase is observed since 2009, and concentrations measured between 2009-2014 are comparable to the concentrations measured between 2002-2006. Therefore it can be concluded that the remedial goal included in the approved RAP for the contamination with FC's in groundwater in the "Zwijndrecht Z-well" area has been met.

During the meeting with OVAM in October 2013, OVAM requested that a partial end evaluation investigation be performed for the area of the Zwijndrecht wells. The purpose of such an investigation is to remove the area of concern from the remedial action plan. The partial end evaluation report was submitted to OVAM for review on 24/Oct./2014.

ERM recommends abandoning all Zwijndrecht wells after OVAM concurrence of the end evaluation investigation. Well abandonment should be done in accordance with the relevant regulations.

The groundwater quality in the **tank farm area** (near Building 3) is monitored annually for pH, conductivity and TPH in two wells (P18 and P28) as in both wells increased levels of these parameters are measured. In 2014 a total of eight wells were sampled and analyzed to delineate the pH, conductivity and TPH in wells P18 and P28. The results of the July 2014 monitoring are shown in the figure below.

Wells near tank farm area – Results July 2014 Monitoring



The delineation investigation confirms that only in wells P18 and P28 elevated levels of pH, conductivity and TPH are measured. The highest TPH concentrations is about twice the remedial norm (500 µg/l).

The source of the TPH contamination is unknown but maybe 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 of TPH.

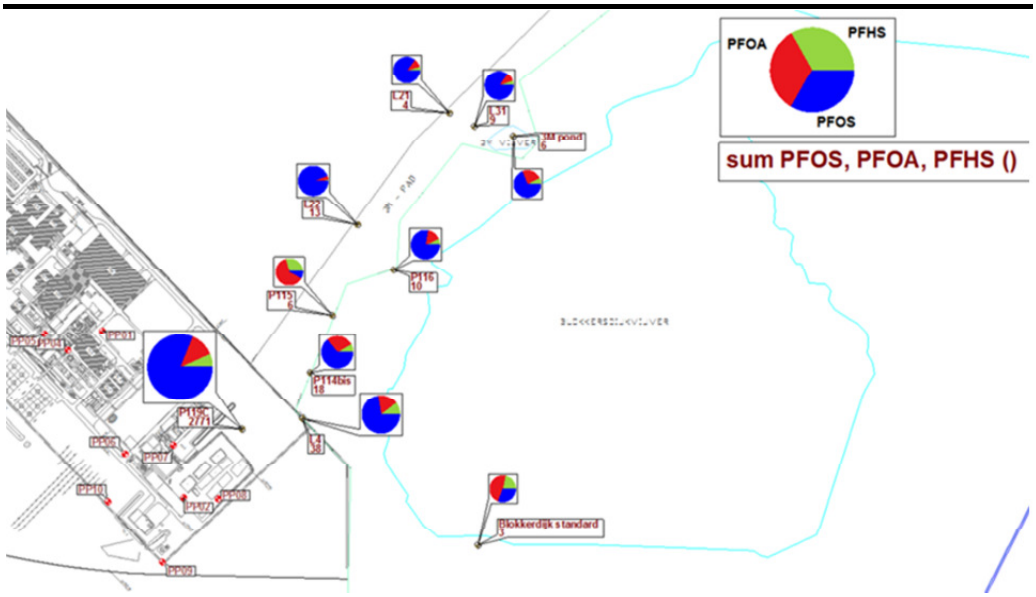
The groundwater monitoring for BTEX was discontinued last year as the concentrations were stable and below the remedial value. During the meeting with OVAM in October 2013, OVAM requested an end evaluation investigation for the (former) BTEX contamination in groundwater at this

zone. The partial end evaluation report was submitted to OVAM for review on 24/Oct./2014.

ERM recommends abandoning all out of use monitoring wells in this area after OVAM conformity of the end evaluation investigation. Well abandonment should be done in accordance with the relevant regulations.

Groundwater and surface water quality in, and close to, the **Blokkersdijk nature reserve** is monitored via eight wells and the 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 2014 monitoring are shown in the figure below.

Blokkersdijk Nature Reserve - Results July 2014 Monitoring



The statistical evaluation of the PFOS results in the surface water show a stable or no trend for the Blokkersdijk pond and a significant increasing trend(1) for the 3M pond. The significantly increasing trend in well L4 is not confirmed and for the replacement well of well P114 (which was irreparably damaged) not enough data is yet available to allow a statistical evaluationof the concentration. Groundwater concentrations at L21 shows a potential increasing trend(2).

There is no need for additional remedial actions in this area as there is no significant increasing trend observed in the Blokkersdijk pond. However, it is recommended to investigate the Blokkersdijk area for potential sources, as the monitoring wells and 3M pond are located upgradient from the known nearby FC source area (WWTP).

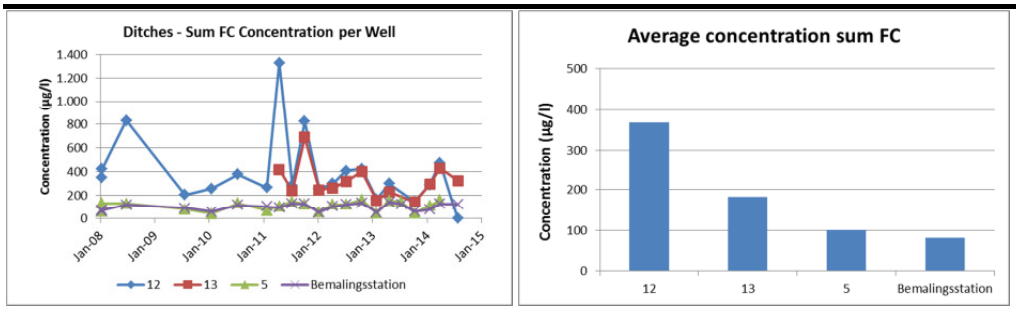
Based on the results of the monitoring, ERM recommends to continue the assessment of the FC concentrations in and close to the Blokkersdijk nature

(1) A significant increasing trend indicates that an increasing trend is calculated with two different statistical test methods
(2) A potential increasing trend indicates that an increasing trend is calculated with one of two statistical test methods

reserve through monitoring. To verify the increasing trend for the 3M pond and the three groundwater wells (L4, P114bis and P116), ERM recommends to monitor these three wells and the surface water of the Blokkersdijk and 3M pond on a quarterly basis. Wells L21, L22, L31 and P115 will be monitored semi-annually. It is also recommended that well P119C is moved to the monitoring program of the WWTP source area, as this well is located downgradient from the Blokkerdijk area but upgradient from the WWTP source area.

The quality of the **surface water in the Paling and Tophat ditches** is monitored quarterly for FC compounds at four different locations. The evaluation of the monitoring results shows that FC concentrations in groundwater are stable at present, see graph below.

Paling and Tophat Ditch - Monitoring Results since 2008

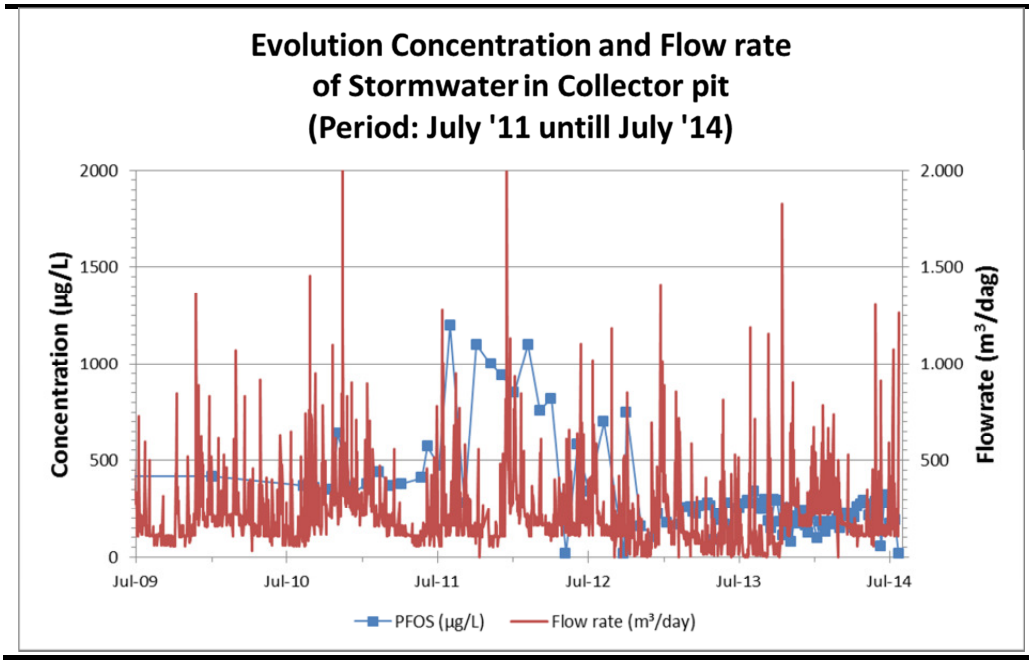


An inspection of the sampling results for the Paling and Tophat ditches shows less variability in 2012 and 2013. FC concentrations in the Tophat ditch are lower than those measured in the Paling ditch.

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.

To monitor the PFOS mass flux leaving the 3M site due to infiltration of contaminated groundwater into the storm sewer system, monthly samples are taken from the **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 sewer system.

Since November 2012 PFOS concentrations in the collector pit show a stable trend (see following figure). Concentrations can fluctuate. The higher PFOS concentrations in the collector pit measured during the 2011-2012 monitoring period were 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 and the FC concentrations decreased.



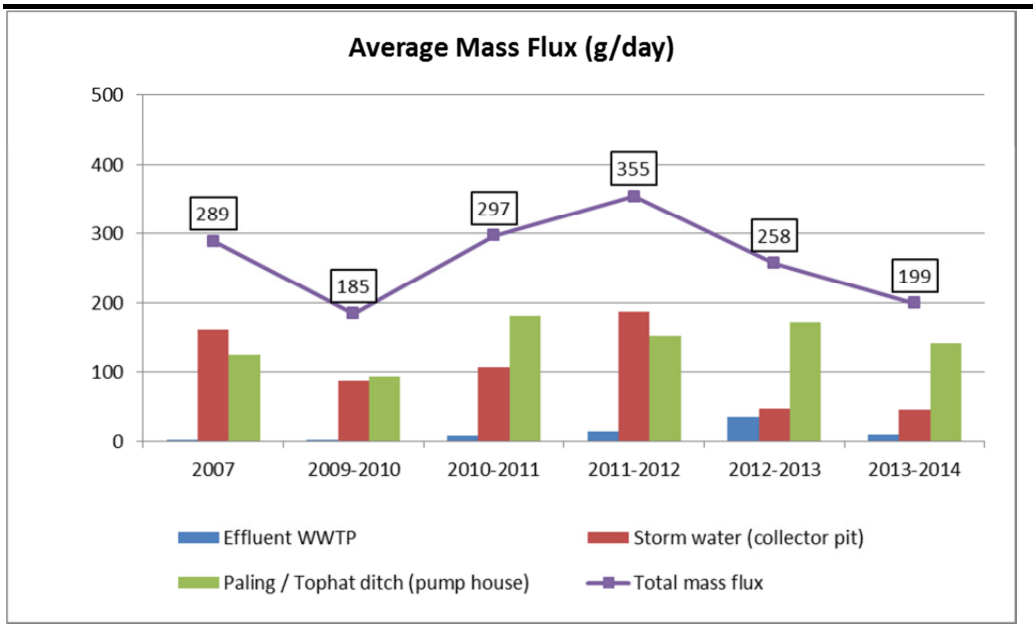
ERM recommends continuation of the monthly monitoring of the stormwater sewer system via the collector pit. The collector pit results are less sensitive to fluctuations caused by rainwater and therefore more reliable.

Mass Flux to the Schelde River

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 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 (PNEC: 370 g/day). The results since 2007 are summarized in the following graph.

As discussed in previous reports, the higher mass flux calculated for the previous 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 decreased, and also the average PFOS mass flux to the Schelde River decreased significantly. The calculated mass flux value has never exceeded the PNEC value (370 g/dag).

Summary of the Average PFOS Mass Flux to the Schelde River



* PNEC value: 370 g/dag

The monitoring of the yearly mass flux will be continued.

Environmental Follow-up of Incidents

At the request of 3M, ERM conducted the environmental follow-up of the following spill incidents that occurred at the 3M site:

- On October 15th, 2013, approximately 1900 kg PIPM was discharged as a spray from an overhead transfer line running between building 03 and 16. The transfer was stopped and the following day the impacted area (gravel and soil) was excavated and seven soil samples were taken for analysis on PIPM. The incident was reported to OVAM in accordance with local regulations.

  The analyses indicated that the soil was not impacted and it was concluded that no further actions were needed. The excavated soil was returned to the excavation pit after approval from OVAM.
  The results and conclusions were reported to OVAM. In their conformity letter OVAM required that the area would be included in the next periodic soil investigation of the site (to be conducted in 2017).

- On August 11th, 2014, a minor leak of a 20% HF Solutia was noticed in Building 17. 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 an undisturbed zone. The analyses

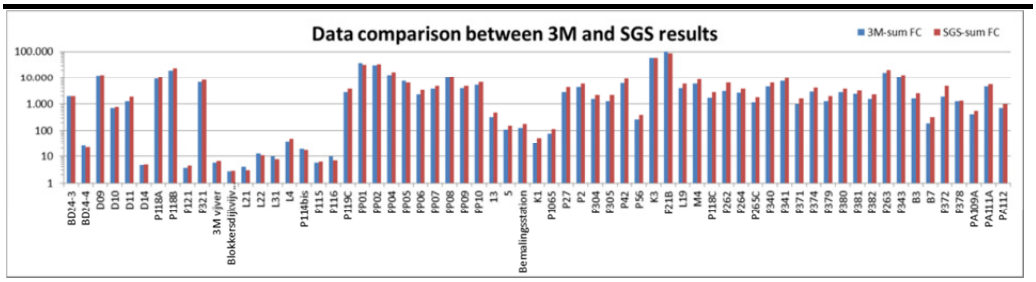
showed an elevated fluoride concentration in the impacted area. The concentration is below the EPA screening level for industrial use. The pH levels are normal.

Because it was a minor impact and the concentration of fluoride lies below the relevant screening level, ERM concludes that no further measures 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 will be reported to OVAM for approval.

Evaluation analytical results water samples SGS vs 3M Environmental Laboratory

In agreement with OVAM (meeting dd. 22 October 2013) all water samples taken in 2014 are analyzed by the 3M Environmental Laboratory in the USA. As verification and to comply with local legislation the duplicate water samples taken during the July 2014 monitoring event were analyzed by SGS nv (an OVAM certified lab) as well as the 3M Environmental Laboratory. For the sake of consistency in the report at hand only the 3M results are shown. An evaluation of both data sets (see graph below) shows that the results between both labs are comparable, but in general the 3M results (primarily PFOS) tend to be lower than the SGS results. This is likely due to different sample preparation techniques and analytical methods (instrument parameters and calibration). The difference between the results is within the acceptable range of variation.

Lab results comparison (SGS vs 3M Environmental Laboratory)



In consultation with OVAM (dd. 12/Nov./2014) it has been decided that in 2015 all water samples will again be analyzed by the 3M Environmental Laboratory, but the duplicate sampling and evaluation will be spread out over the four monitoring events. All water samples will be analyzed once by the SGS laboratory during the monitoring period (January, April, July and October 2015).

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.

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. ERM recommends continuing the groundwater monitoring program to assess the long-term FC concentration trends. If an increase is observed, the need for optimization of the P&T system will be evaluated. The (potential) increasing trend in some of the Blokkersdijk monitoring wells and the 3M pond needs to be verified via monitoring and the cause of the concentration increase needs to be further investigated. At present, there is no need for additional remedial actions in this area as there is no increasing trend observed in the Blokkersdijk pond.

ERM recommends that the remedial actions and regular monitoring continue at the 3M site as summarized in the following table.

The yearly average mass flux of PFOS towards the Schelde River lies well below the relevant PNEC value and has decreased further, after having increased during the 2011-12 monitoring period. This continued decrease confirms that the mitigation measure taken mid 2012 (the sludge removal from the collector pit) was effective.

Two minor incidents occurred at the 3M site between July 2013 - July 2014. The sampling results identified no impact to soil and/or groundwater, therefore, no further action is required.

The next interim report will be submitted to OVAM before the end of 2015, describing the remedial actions and monitoring results taken from July 2014 through July 2015.

■

Proposed Remedial Monitoring Program for 2015

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; 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
	5	Semi-annual	P21B, P27, P42, P304, P305	Increased frequency to allow semi-annual evaluation of the sum FC-contour	K1 is replaced by P27, as the concentrations in K1 have been low and stable since the start of the monitoring in 2008
WWTP	6	Annual	P262, P340, P341, P343, P379, P380	-	-
	7	Semi-annual	P118C, P264, P265C, P371, P374, P381, P382	-	-
	4	Semi-annual	L19, M4, P119C, P263	Increased frequency to allow semi-annual evaluation of the sum FC-contour	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)
Second Aquifer	4	Annual	BP24-4, D11, D14, P118B	-	-
		Annual	D10, P118A	Increasing trend not confirmed	
	1	Semi-annual	D9	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
	4	Semi-annual	BD24-3, P121	-	-
	1	Quarterly	P321	-	-
Southern Site Boundary	7	Semi-annual	B3, B7, PA111A, PA112, P378, PA109A, P372	-	-

Location	Number of Wells	Frequency	Well ID	Rationale	Remarks
South of the Express Road (Zwijndrecht or Z-wells)	-	-	-	Monitoring was ended, as the concentrations are stable and low	An end evaluation investigation for this zone was performed. After OVAM concurrence of the end evaluation investigation, all Zwijndrecht wells will be abandoned in accordance with relevant regulations
Tank Farm Area					
BTEX	-	-		BTEX monitoring was discontinued early 2013 as the remedial goal was met	An end evaluation investigation for this zone was performed. After OVAM conformity of the end evaluation investigation all out of use monitoring wells will be abandoned in accordance with relevant legal guidelines
TPH	2	Annual	P18, P28	TPH monitoring at request of OVAM	Source investigation ongoing
Blokkersdijk Nature Reserve					
Wells	4	Semi-annual	L21, L22, P115, L31	-	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

DEEL 1: ADMINISTRATIEVE GEGEVENS

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 - Vijfde tussentijds verslag bodemsaneringswerken: Periode juli 2012 – juli 2013 3M Belgium bvba Canadastraat 11 te 2070 Zwijndrecht**
Referentie EBSD:	**R03 0243999 TTV5 v1.docx**
Rapportdatum:	22 januari 2015
Onderzoekslocatie:	3M Belgium bvba
- Straat + nummer of omschrijving:	Canadastraat 11
- Postcode:	2070
- Fusiegemeente:	Zwijndrecht
- Deelgemeente:	Zwijndrecht
Aanleiding:	☐ Overdracht grond ☐ Sluiting bedrijf ☐ Stopzetting activiteit ☐ Overdracht grond + sluiting bedrijf ☐ Periodieke verplichting ☒ Decretaal verplicht ☐ Decretaal vrijwillig ☐ Vrijwillig door derden ☐ Ambtshalve[1] ☐ Verspreiding ☐ Onteigening ☐ Onbekend
Naam opdrachtgever:	3M Belgium bvba
- Straat + nummer:	Hermeslaan 7
- Postcode:	1831
- Fusiegemeente:	Machelen
- Land:	België
- Telefoon:	-
- Fax:	-
- E-mail:	-
Hoedanigheid:	☒ Eigenaar ☐ Gebruiker ☒ Exploitant ☐ Optredend in opdracht van de eigenaar/gebruiker/exploitant ☐ Andere: ……

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

Titel:	**Eerste gefaseerd bodemsaneringsproject - Vijfde tussentijds verslag bodemsaneringswerken: Periode juli 2012 – juli 2013 3M Belgium bvba Canadastraat 11 te 2070 Zwijndrecht**
Referentie EBSD:	**R03 0243999 TTV5 v1.docx**
Rapportdatum:	22 januari 2015

Naam contactpersoon:	Kristof Verstraeten
- Telefoon:	[redacted]
- Fax:	[redacted]
- E-mail:	[redacted]

Naam contactpersoon ter plaatse:	Kristof Verstraeten
- Telefoon:	[redacted]
- Fax:	[redacted]
- E-mail:	[redacted]

Naam bodemsaneringsdeskundige:	Environmental Resources Management - ERM nv
Naam contactpersoon:	Nanda Hermes
- Telefoon:	[redacted]
- Fax:	-
- E-mail:	[redacted]

Dossiernummer OVAM:	732

Tabel 0.2 Identificatie van de betrokken kadastrale percelen

Gemeentenummer	Sectie	Perceelnummer	Adres	Gemeente	Oppervlakte perceel (m^2)	Periode		Persoon (Eigenaar / gebruiker / exploitant)				Huidig bestemmingstype (1)	Toekomstig bestemmingstype (1)	Grondwaterkwetsbaarheid (2)
						Van	Tot	Type (3)	Naam	Adres	Letter (4)			
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.

DEEL 2: RAPPORT

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 vijfde tussentijds verslag (TTV5) van de saneringswerken, zoals beschreven in het door de OVAM conform verklaarde bodemsaneringsproject (1e fase) (1) en de vier voorgaande tussentijdse verslagen (2), (3) (4) (5). Het betreft de saneringswerken op het deel productiezone, voormalige slibbekkens/waterzuiveringsinstallatie, natuurgebied Blokkersdijk en de 2e aquifer. De beschouwde saneringsperiode loopt van juli 2013 tot en met juli 2014.

De organofluor (FC) impact ter hoogte van de zuidelijke perceelsgrens en de Palingbeek wordt momenteel door middel van een grondwatermonitoring opgevolgd. Wanneer er meer duidelijkheid is omtrent 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.

Op 12 november 2014 heeft het project team (3M en ERM) een onderhoud gehad met OVAM om de stand van zaken van de bodemsanering te bespreken.

(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

2 VOORSTUDIE

2.1 OMGEVINGSKENMERKEN

De ligging van de onderzoekslocatie is weergegeven op een kopie van topografische kaart van België de opgenomen in Bijlage **2Error! Reference source not found.**. 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 Bijlage ***3***.
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 2014) 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.

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. 2013)

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**

* 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 en de schommelende vuilvrachten in de regenwaterriolering.

De verhoogde vuilvrachtwaarde berekend voor de periode juli 2011 – juni 2012 ten opzichte van de waarden berekend in de voorgaande periodes 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 regenwater collectorput terug gedaald (zie paragraaf 4.10).

3 ALGEMENE BESCHRIJVING SANERINGSWERKEN

3.1 ALGEMEEN

In het eerste gefaseerd bodemsaneringsproject [1] zijn de voorgestelde bodemsaneringswerken beschreven. Het beschrijft de selectie en uitwerking van de saneringsaanpak voor de verontreinigingen met FC verbindingen en BTEX waarvoor een saneringsnoodzaak bestaat. De saneringswerken uitgevoerd tussen juli 2009 en juni 2013 zijn beschreven in de vier voorgaande tussentijdse verslagen [2], [3], [4], [5].

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

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.

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.

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

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.

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.

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

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	-

Gefaseerde opbouw grondwaterextractiesysteem

Volgende fases zijn gedefinieerd in het bodemsaneringsproject:

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

- Fase 3: Evaluatie van de efficiëntie van het onttrekkingssysteem gedurende vijf jaar; en
- Fase 4: eventuele uitbreiding van het onttrekkingssysteem.

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

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

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 16, 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

Doelstelling

Vanuit risicogebaseerd oogpunt is het niet noodzakelijk de vuilvracht welke dagelijks via de regenwaterriolering geloosd wordt te reduceren. De dagelijkse PFOS-vuilvracht naar de Schelde overschrijdt immers de ecotoxicologisch veilige waarde niet (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.

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.

Monitoring

Om de vuilvracht die het 3M terrein verlaat als gevolg van het infiltreren van 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 regenwater collectorput net vóór het punt waar het regenwater wordt samengevoegd met het gezuiverde afvalwater dat in de Schelde wordt geloosd.

3.2.8 Natuurgebied Blokkersdijk

Doelstelling

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

Monitoring

Ter hoogte van het natuurreservaat 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.

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 perceelsgrens, 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 HET EERSTE, TWEEDE, DERDE EN VIERDE TUSSENTIJDS VERSLAG

Ten opzichte van de saneringsaanpak zoals voorzien in het eerste gefaseerd bodemsaneringsproject (1) zijn in de voorgaande tussentijdse verslagen (2), (3), (4), (5) een aantal wijzigingen voorgesteld. Deze wijzigingen zijn toegelicht in onderstaande paragrafen. In paragraaf 3.3.11 wordt een overzicht gegeven van het monitoringsprogramma voor 2014.

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

is 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.

Intussen is deze daling bevestigd en is ook de saneringsdoelstelling behaald (zie TTV4 [(5)]). Daarom is in het vierde tussentijds verslag voorgesteld om de monitoring van de BTEX concentraties in het grondwater stop te zetten. 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. Na conformiteit van het verslag door OVAM zullen de alle peilbuizen in de zone van de ondergrondse tanks, die niet meer opgevolgd worden, definitief buiten gebruik gesteld worden conform de wettelijke voorschriften.

Ter hoogte van twee peilbuizen (P18 en P28) zijn verhoogde waarden voor pH, geleidbaarheid en minerale olie in grondwater gemeten waarvoor geen verklaring gegeven kon worden. De mogelijkheid van interferentie door een andere verontreiniging kon niet uitgesloten worden. Bijkomend historisch en analytisch brononderzoek is in het derde tussentijds verslag aanbevolen. 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.

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).

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

Grondwatermonitoring

De grondwatermonitoring ter hoogte van zone Gebouw 16 loopt intussen vijf 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 vijf 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.

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 op te volgen.

3.3.5 Reductie van de FC impact in de regenwaterriolering

Omdat de regenwaterriolering zeer gevoelig is aan fluctuaties als gevolg van neerslag is 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. 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.

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, ondanks het feit dat er in deze zone geen toename van verontreiniging verwacht wordt, de grondwaterkwaliteit ten zuiden van de Expresweg verder op te volgen. 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 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 het gebied ten zuiden van de Expressweg (Z-peilbuizen). Na conformiteit van het rapport door OVAM zullen alle peilbuizen in deze zone definitief buiten gebruik gesteld worden conform de wettelijke voorschriften.

3.3.10 Vuilvracht richting de Schelde

Geen wijzigingen.

3.3.11 Monitoringsprogramma 2014

Op basis van de resultaten uit de periode juli 2011 tot en met juli 2013 is het monitoringsprogramma zoals opgenomen in het conformverklaarde BSP fase 1 waar nodig of wenselijk aangepast. In de volgende tabel is een overzicht gegeven van het gewijzigde monitoringsprogramma voor de periode juni 2013 – juli 2014, met verantwoording. Voor meer details wordt verwezen naar het vierde TTV[(5)].

Tabel 3.4 Monitoringsprogramma 2014

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
P&T systeem	7	Tweemaandelijks	PP01, PP02,PP04, PP05, PP06, PP07, PP08	-	Minstens maandelijkse inspectie van de installatie en de onttrekkingsputten
	2	Maandelijks	PP09, PP10		
Stabiliteit	-	Halfjaarlijks (stopzetting indien akkoord stabiliteitsdeskundige)		Geen onaanvaardbare zettingen en een technische evaluatie door 3M geeft aan dat er geen verder noodzaak is voor monitoring. Te bevestigen door onafhankelijke stabiliteitsdeskundige.	-
Grondhoop	-	Vijfjaarlijks	Visuele inspectie van grondhoop	Bovenafdek vervangen in 2012	Vanaf 2014, vijfjaarlijkse monitoring, aangezien bovenafdek recentelijk vervangen is
FC Bronzones					
Gebouw 16	8	Jaarlijks	K1, K3, P21B, P304, P305 P42, P56	-	**PFBS** zal worden toegevoegd aan het analysepakket om de concentraties verder op te volgen
WWTP	9	Jaarlijks	L19, M4, P262, P263, P340, P341, P343, P379, P380	-	-
	7	Halfjaarlijks	P118C, P264, P265C, **P371**, **P374**, P381, P382	**P371** en **P374** zullen eveneens 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 halfjaarlijks
Tweede aquifer	5	Jaarlijks	BP24-4, D9, D11, D14, P118B	-	-
	2	Halfjaarlijks	**P118A, P121**	**P118A** en **P121** zullen 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
	2	Halfjaarlijks	**BD24-3, D10**	**BD24-3** en **D10** zullen halfjaarlijks bemonsterd worden aangezien de stijgende trend niet bevestigd is	
	1	Driemaandelijks	P321	-	-
Zuidelijke terreingrens	7	Halfjaarlijks	B3, B7, PA111A, PA112, P378, PA109A, P372	-	-
Ten zuiden van expresweg (Z-peilbuizen)	4	-	Z1-3m, Z4-8m, Z2-3m, Z2-10m	**Z4-8m** is beschadigd en niet meer bruikbaar. Z1-3m, Z2-3m en Z2-10m worden nog éénmaal bemonsterd, nadien wordt monitoring stopgezet	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

Locatie	Aantal peilbuizen	Frequentie	Peilbuis ID	Verantwoording	Opmerkingen
				aangezien concentraties relatief laag zijn.	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	5	Halfjaarlijks	L21, L22, P115, **L31**, P119C	Verlaagde frequentie voor **L31** aangezien stijgende trend niet is bevestigd	-
	3	Driemaandelijks	**L4**, P114, P116	Verhoogde frequentie voor **L4** om potentiële stijgende trend te verifiëren	Indien de stijgende trend niet wordt bevestigd bij twee opeenvolgende monitoringsrondes, zal de monitoringsfrequentie terug worden verlaagd naar jaarlijks
3M vijver	1	Driemaandelijks	3M vijver	-	Indien de stijgende trend niet wordt bevestigd bij twee opeenvolgende monitoringsrondes, zal de monitoringsfrequentie terug worden verlaagd naar halfjaarlijks
Blokkersdijk vijver	1	Driemaandelijks	Blokkersdijk standaard	-	-
	1	-	Blokkersdijk oost	**Blokkersdijk oost** wordt geschrapt aangezien dit staalnamepunt niet meer bereikbaar is	
Palingbeek en Tophat gracht	4	Driemaandelijks	12, 13, 5, Bemalingsstation	-	-
Regenwaterriolering					
Regenwaterriolering	4	-	RW3, RW4, RW12, RW13	Monitoring wordt vervangen door maandelijkse monitoring van de collector put	-
Collector put	1	Maandelijks	Collector put	Maandelijkse stalen geanalyseerd door 3M labo te Zwijndrecht	Jaarlijkse verificatie van de stalen door een extern labo
Effluent WWTP	1	Driemaandelijks	Lozingspijp	Maandelijkse stalen geanalyseerd door 3M labo te Zwijndrecht	Driemaandelijkse verificatie van de stalen door een extern labo

Legende

Verhoogde monitoringsfrequentie

Verlaagde monitoringsfrequentie

Geen wijzigingen

4 UITGEVOERDE MONITORING EN RESULTATEN

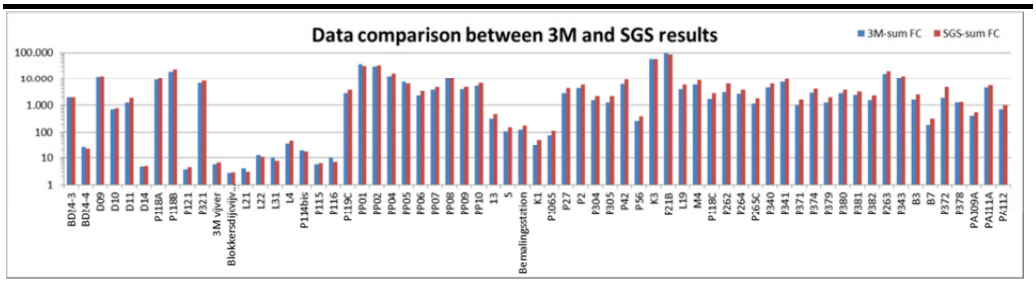
4.1 ALGEMEEN

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 22 oktober 2013 en beschreven in het vierde tussentijds verslag zijn de waterstalen door het 3M Environmental Laboratory in de Verenigde Staten geanalyseerd op FC verbindingen. Tijdens de volledige monitoringronde van juli 2014 zijn – ter controle – duplo stalen genomen, die geanalyseerd zijn door het door OVAM erkende laboratorium SGS. 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 twee datasets (SGS vs. 3M labo) volgt dat de resultaten van beide labo's vergelijkbaar zijn (zie Grafiek 4.1), maar dat de waarden (met name voor PFOS) gemeten door het 3M Environmental Laboratory over het algemeen lager liggen dan de concentraties gemeten door SGS. Het verschil wordt waarschijnlijk veroorzaakt door een verschil in de voorbereiding van de waterstalen en de analytische methode (de gebruikte apparatuur en de calibratie ervan). Gemiddeld ligt het verschil tussen de resultaten van beide labo's binnen de normale analysevariatie.

Grafiek 4.1 Evaluatie som FC meetresultaten SGS vs 3M Environmental Laboratory



In overleg met OVAM (dd. 12 november 2014) is besloten om in 2015 de waterstaalanalyses van alle monitoringsrondes opnieuw te laten analyseren door het 3M Environmental Laboratory, maar de controle staalname en analyse te spreiden over de vier monitoringsperiodes (in plaats van één controle tijdens de monitoring van juli). Er dient wel voor worden gezorgd dat alle waterstalen éénmaal door het SGS labo geanalyseerd zullen worden tijdens de monitoringsperiode (januari, april, juli en oktober 2015).

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 in Bijlage ***4*** 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 16 en zone WWTP) elk jaar 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 Bijlage ***5***.

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

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum januari 2014	Bemonsteringsdatum juli 2014	Analyse
Zone Gebouw 16				
K1	2,3-4,3		X	FC inclusief PFBS
K3	3,6-5,6		X	FC inclusief PFBS
P21B	3,5-5,5		X	FC inclusief PFBS
P304	4,0-6,0		X	FC inclusief PFBS
P305	3,5-5,5		X	FC inclusief PFBS
P42	3,0-5,0		X	FC inclusief PFBS
P56	1,2-3,2		X	FC inclusief PFBS
P1065 *	?-2,0		X	FC inclusief PFBS
P27 *	?-2,9		X	FC inclusief PFBS
P2 *	?-?		X	FC inclusief PFBS
Zone WWTP				
P118C	2,5-4,5	X	X	
L19	2,8-4,8		X	FC
M4	1,0-3,0		X	FC
P262	6,2-7,2		X	FC
P263	7,4-8,4		X	FC
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

* *Eenmalige staalname voor PFBS onderzoek*

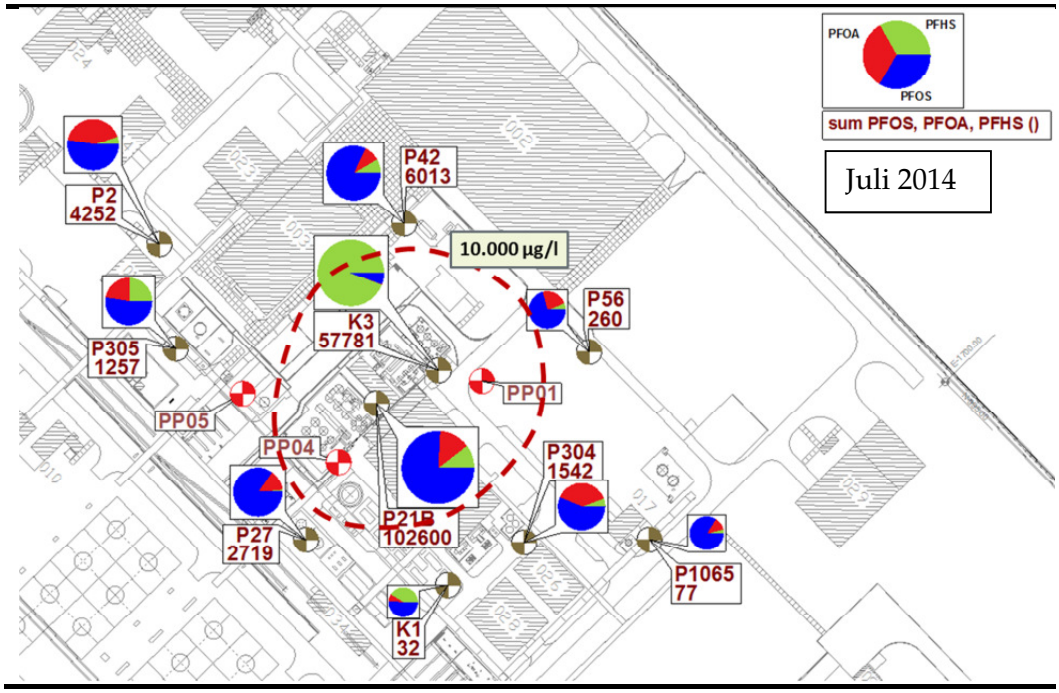
4.3.2 Bespreking resultaten, conclusies en aanbevelingen

De analyseverslagen zijn toegevoegd in Bijlage ***6***. Een toetsingstabel met deze resultaten is opgenomen in Bijlage ***7*** samen met de resultaten van de veldmetingen.

Gebouw 16

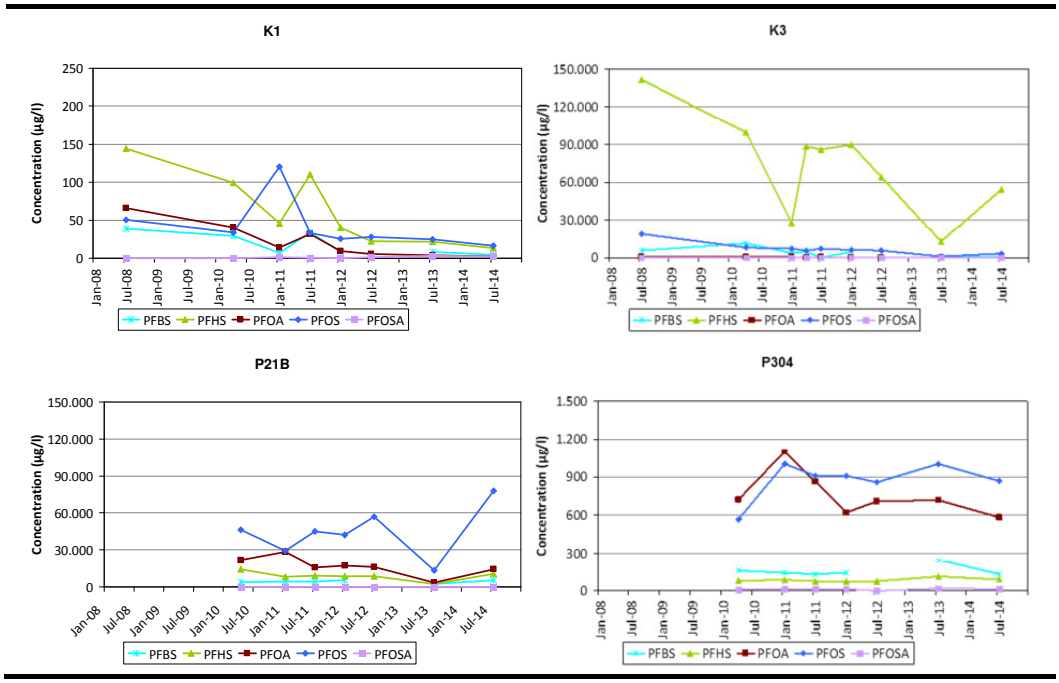
De grondwateranalyseresultaten voor de som van PFOS, PFHS en PFOA van juli 2014 ter hoogte van gebouw 16 zijn op de volgende figuur voorgesteld. De rode lijn stelt de contour van 10.000 µg/l voor de som van PFOS, PFHS en PFOA voor.

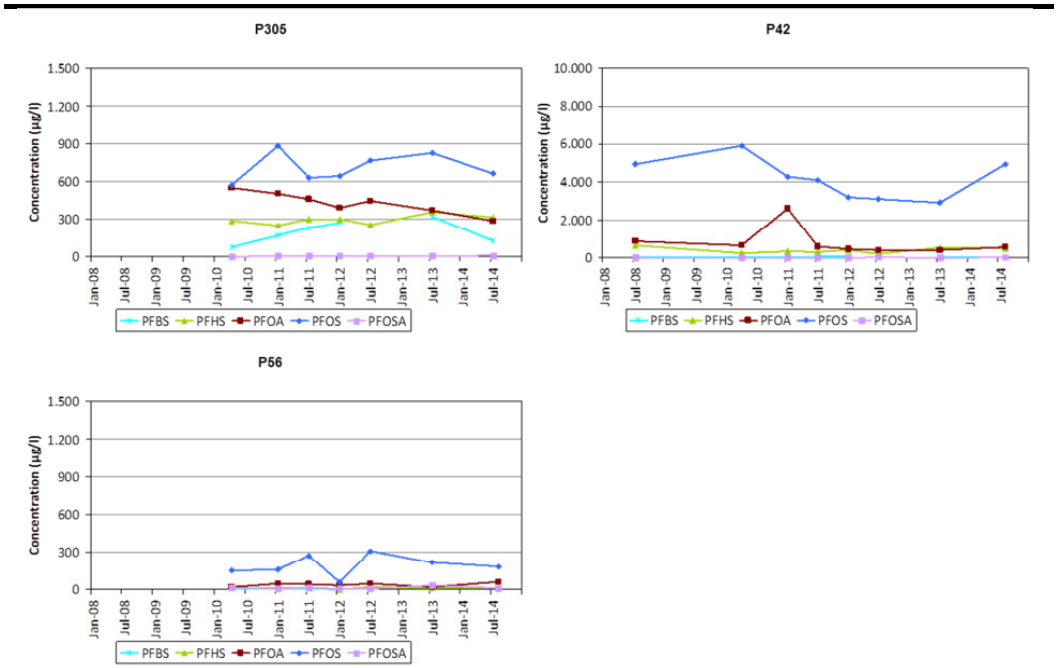
Figuur 4.1 Schematische voorstelling FC grondwateranalyseresultaten thv. Gebouw 16



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

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



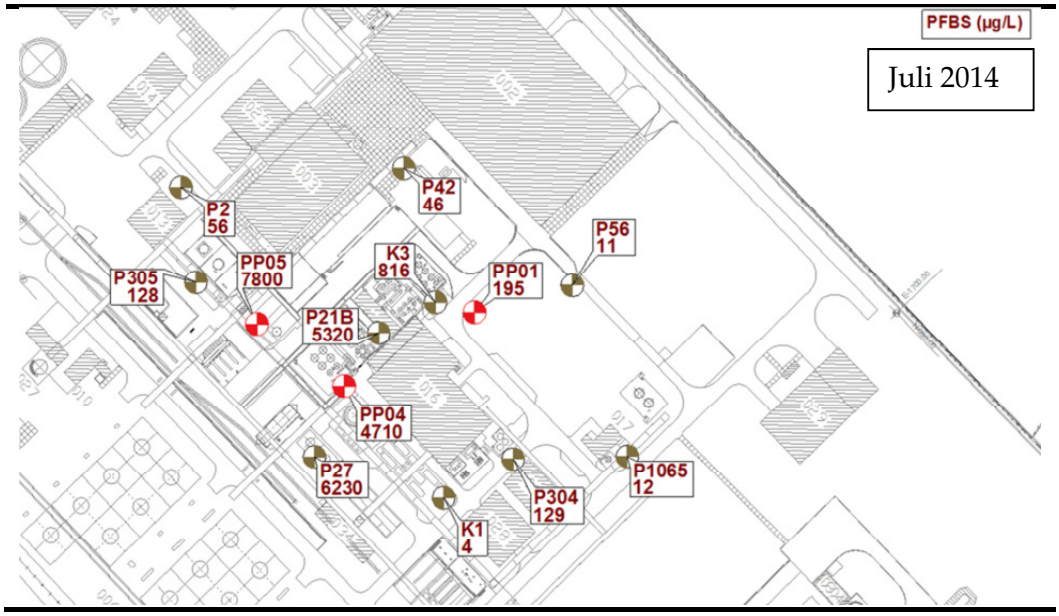


In het algemeen kan gesteld worden dat de FC concentraties in het grondwater ter hoogte van gebouw 16 dalen of stabiel zijn in de tijd, met uitzondering van de peilbuizen P21B en P42 waar de resultaten in juli 2014 een stijging vertonen ten opzichte van eerdere metingen. Deze stijging dient verder opgevolgd te worden. De omvang van de som FC concentratiecontour lijkt licht groter geworden te zijn ten opzichte van de contour van vorige monitoringsperiode (2012-13).
Echter, 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 16 effectief capteert.

PFBS is de vervanger van PFOS en wordt sinds 2002 op de site geproduceerd. 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. Bovendien heeft PFBS een significant lagere toxiciteit dan PFOS en is daarom als vervanger van PFOS geselecteerd.

De resultaten voor PFBS van juli 2014 in het grondwater ter hoogte van gebouw 16 zijn op de volgende figuur voorgesteld.

Figuur 4.2 Schematische voorstelling PFBS grondwateranalyseresultaten thv. gebouw 16



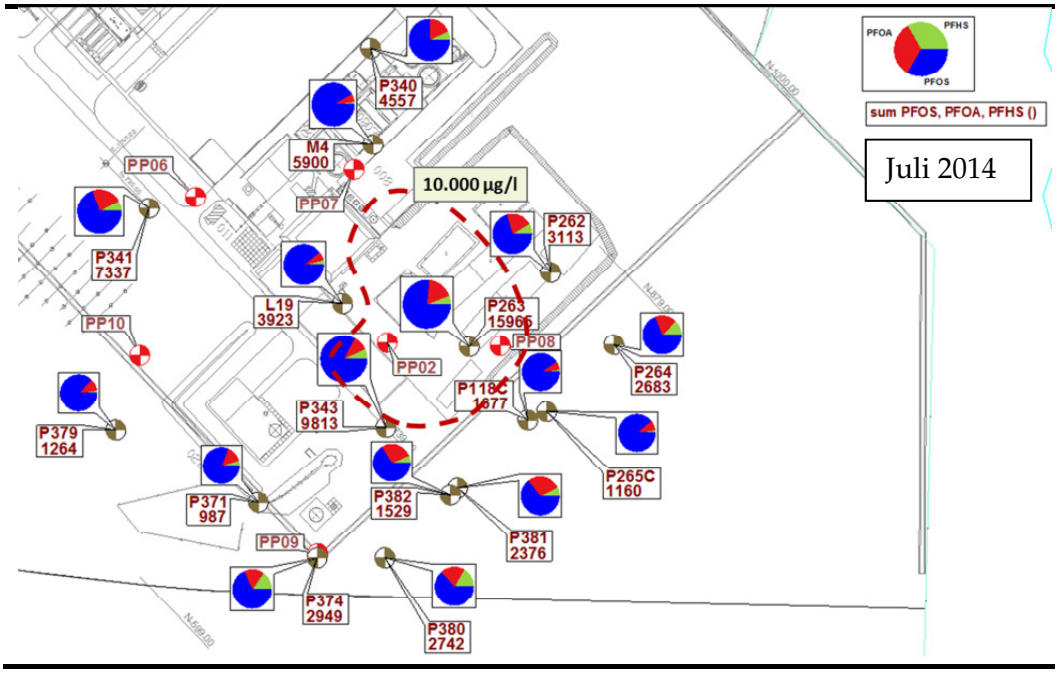
Geen van de gemeten concentraties overschrijdt de monitoringswaarde voor de som FC's (10.000 µg/l). De eerder vastgestelde stijging van PFBS concentraties ter hoogte van peilbuis P305 is niet bevestigd. De oorzaak van de PFBS detectie is momenteel onvoldoende gekend, en wordt verder onderzocht.

De jaarlijkse monitoring in de kernzone van gebouw 16 blijft behouden en de volgende volledige monitoring zal plaatsvinden **in juli 2015**.
Echter, om de evolutie van de som FC concentratiecontour (10.000 µg/l) beter te kunnen opvolgen (halfjaarlijks, ipv. jaarlijks) wordt aanbevolen om de monitoringsfrequentie in de volgende vier peilbuizen te verhogen van jaarlijks naar halfjaarlijks: P21B, P42, P304 en P305. Verder wordt aanbevolen om de monitoring van de concentraties in peilbuis K1 stop te zetten en te vervangen door peilbuis P27, omdat de concentraties in peilbuis K1 sinds de start van de monitoring in 2008 stabiel en laag liggen (huidige som FC concentratie ca. 30 µg/l). Deze peilbuis zou eveneens halfjaarlijks worden gemonitoord.
Tot slot wordt aanbevolen om de PFBS concentraties ter hoogte van bronzone Gebouw 16 verder af te perken en de oorzaak van de verontreiniging verder te onderzoeken en jaarlijks te monitoren.

Zone WWTP

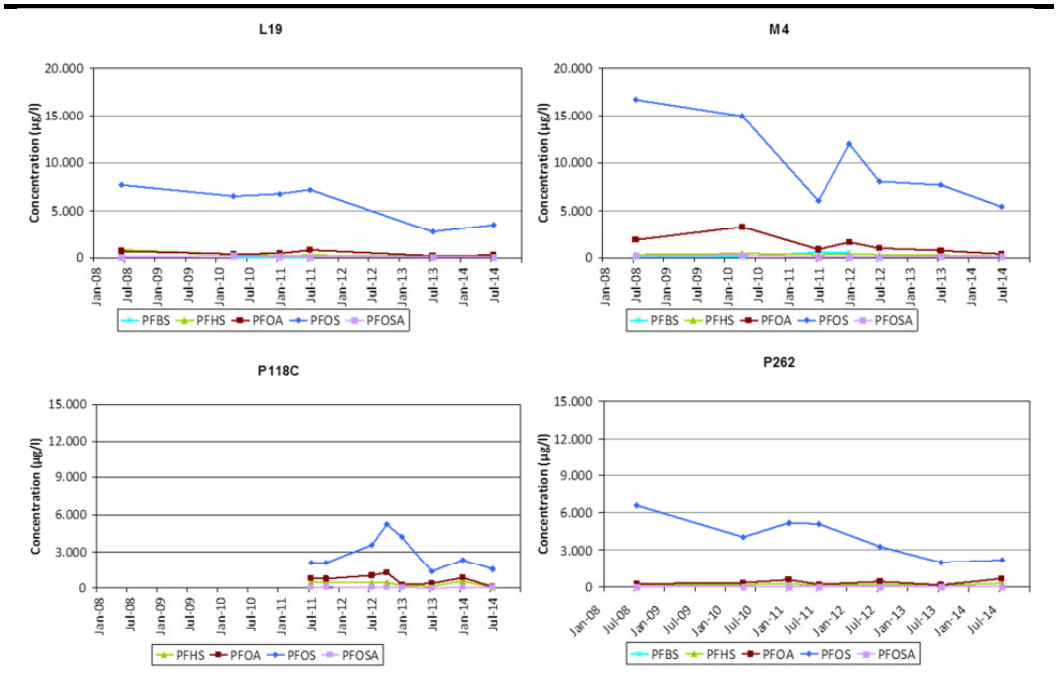
De grondwateranalyseresultaten voor de som van PFOS, PFHS en PFOA van juli 2014 ter hoogte van de WWTP zijn op de volgende figuur voorgesteld. De rode lijn stelt de concentratiecontour van 10.000 µg/l voor de som van PFOS, PFHS en PFOA voor.

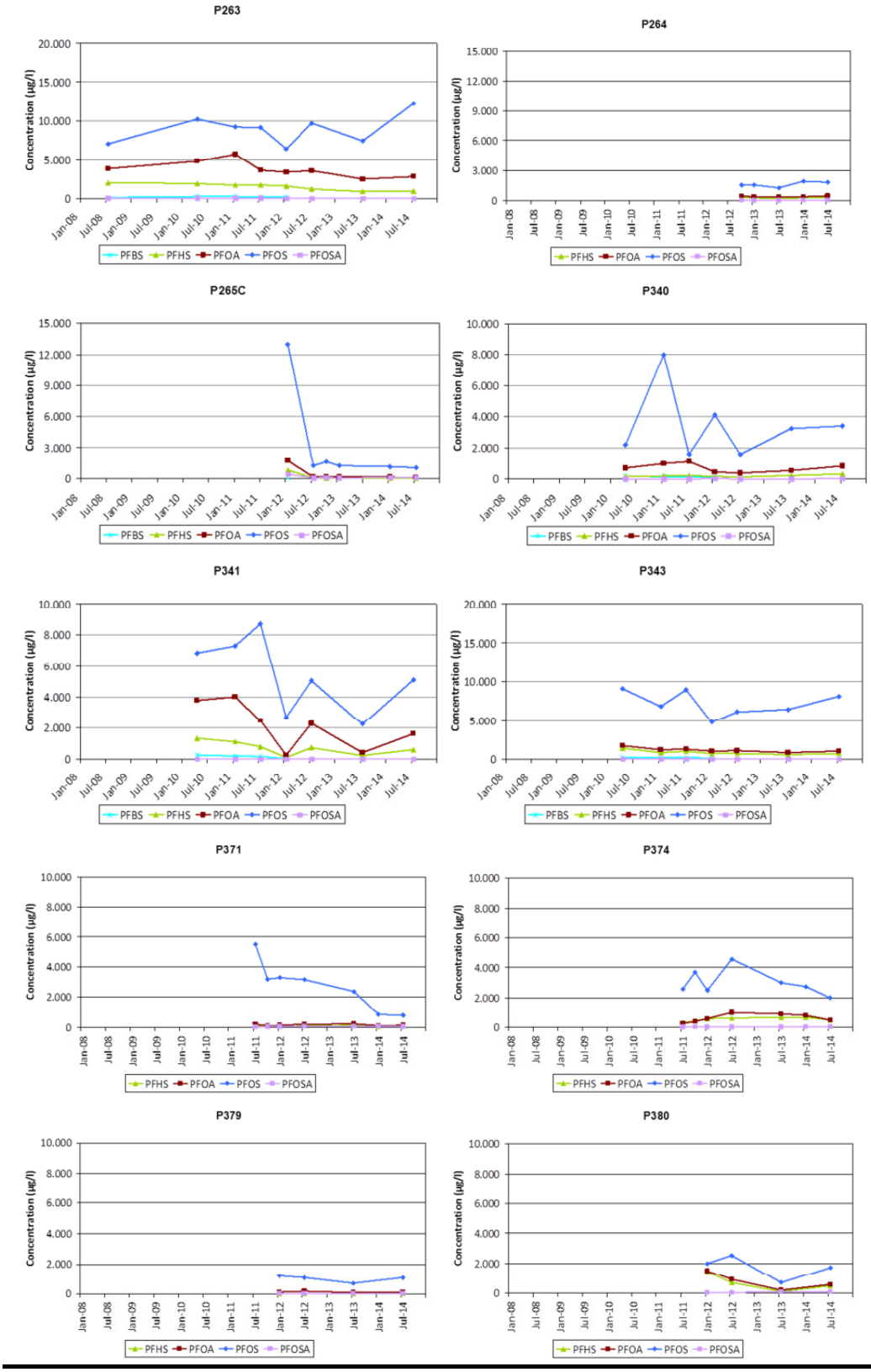
Figuur 4.3 Schematische voorstelling FC grondwateranalyseresultaten thv. WWTP

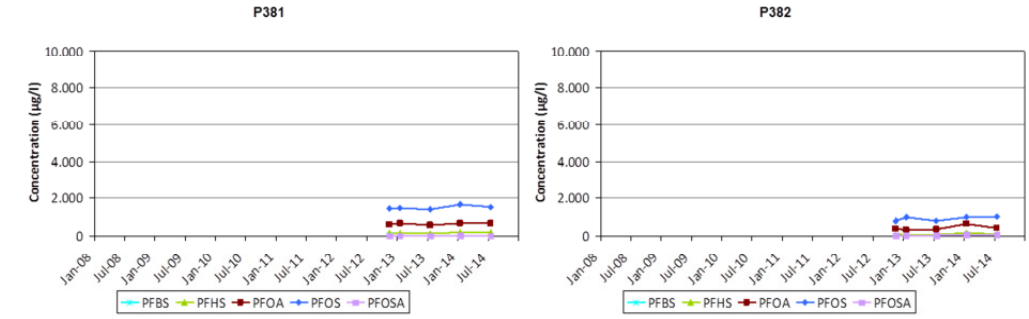


De evolutie van de concentraties aan PFOS, PFHS, PFOA en PFOSA in het grondwater ter hoogte van zone WWTP is voorgesteld in de volgende grafieken.

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




P263
P264
P265C
P340
P341
P343
P371
P374
P379
P380
Concentration (µg/l)
PFBS
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 in peilbuis P263 waar tijdens de meting van juli 2014 een stijging in de PFOS concentratie is vastgesteld. Deze stijging dient verder opgevolgd te worden. De eerder vastgestelde stijgingen in peilbuis P374 is niet bevestigd door de recente metingen. De meest recente concentraties in deze peilbuis tonen een dalende trend.
De omvang van de som FC concentratiecontour is vergelijkbaar met de contour van vorige monitoringsperiode (2013-13).
De algemene stabiele/dalende trend in FC-concentraties lijkt erop te duiden dat het onttrekkingssysteem de FC-concentraties in het grondwater in de WWTP bronzone effectief capteert.

De jaarlijkse monitoring in de kernzone WWTP blijft behouden en de volgende volledige monitoring zal plaatsvinden **in juli 2015**. Aanbevolen wordt om voor de peilbuizen M4, L19 en P263 de monitoringsfrequentie te verlagen naar halfjaarlijks en om voor peilbuizen P118C, P263, P264, P265C, P371, P374, P381 en P382 de halfjaarlijkse monitoringsfrequentie te behouden om de evolutie van de som FC concentratiecontour (10.000 µg/l) beter te kunnen opvolgen (halfjaarlijks, ipv. jaarlijks).

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 plan in Bijlage 5. 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. 2013	Jan. 2014	Apr. 2014	Jul. 2014	
Zone Gebouw 16						
D09	13,0-15,0				X	FC
D14	15,5-16,5				X	FC
Zone WWTP						
D10	15,0-16,0	X	X		X	FC
D11	14,9-15,9				X	FC

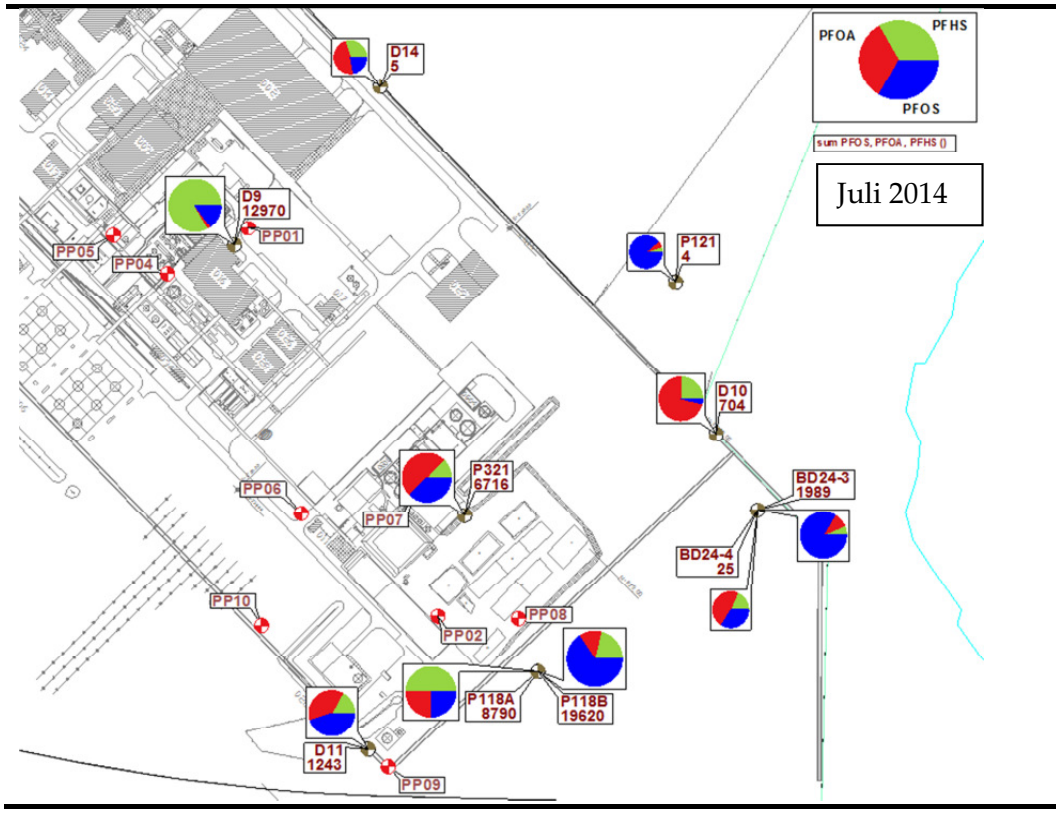
Peilbuis	Filterdiepte	Bemonsteringsdatum				Analyse
	(m-mv)	Okt. 2013	Jan. 2014	Apr. 2014	Jul. 2014	
BD24-3	17,0-18,0	X	X		X	FC
BD24-4	22,0-24,0				X	FC
P118A	22,5-23,5		X		X	FC
P118B	13,0-14,0				X	FC
P121	23,5-24,5		X		X	FC
P321	15,5-15,6	X	X	X	X	FC

4.4.2 *Bespreking resultaten*

De analyseverslagen zijn toegevoegd in Bijlage ***6***. Een toetsingstabel met deze resultaten is opgenomen in Bijlage **7** samen met de resultaten van de veldmetingen.

De grondwateranalyseresultaten voor de som van PFOS, PFHS en PFOA van juli 2014 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 baseline voor de verdere opvolging van de evolutie van de concentraties in de tweede aquifer.

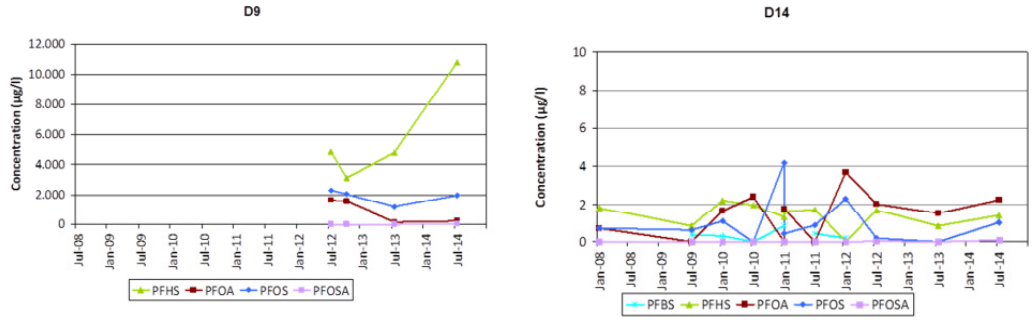
Figuur 4.4 ***Schematische voorstelling FC grondwateranalyseresultaten in de 2de aquifer***



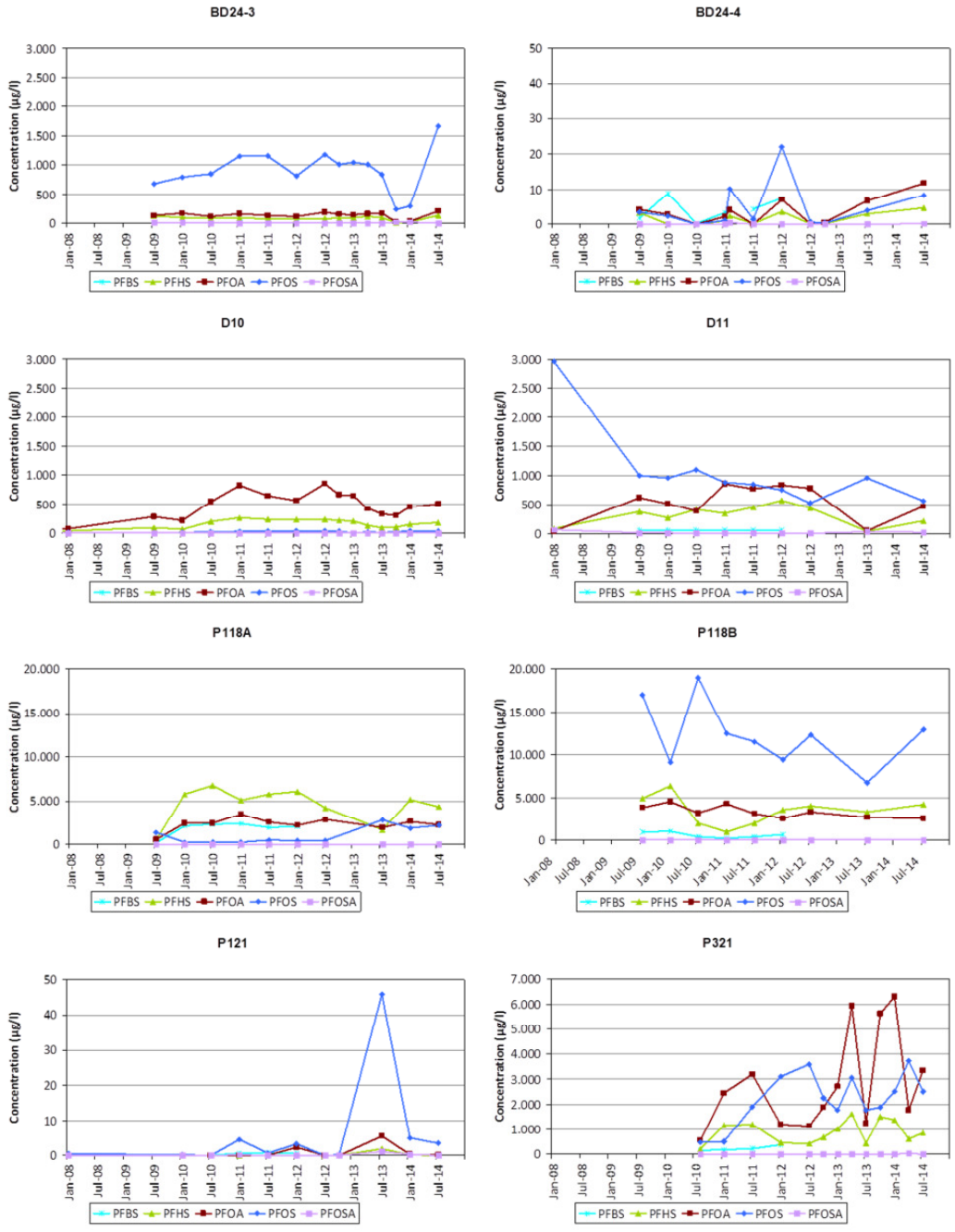
De evolutie van de concentraties aan PFOS, PFHS, PFOA en PFOSA in het grondwater van de tweede aquifer is voorgesteld in de volgende grafieken.

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

Bronzone Gebouw 16



Bronzone WWTP



Ter hoogte van gebouw 16 is er een stijging in de PFHS concentraties van peilbuis D09 vastgesteld, terwijl de PFOS en PFOA concentraties stabiel of dalend lijken. Er is echter meer data nodig om een duidelijke uitspraak te doen voor peilbuis D09. 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, met uitzondering van peilbuis P321 waar de fluctuerende concentraties een stijgende trend lijken te tonen. Ter hoogte van peilbuis BD24-3 is tijdens de laatste monitoring een verhoogde PFOS concentratie gemeten, maar blijven de concentraties algemeen gezien stabiel in de tijd. De recente stijging zal verder opgevolgd worden.

In het algemeen kan gesteld worden dat de concentraties aan FC verbindingen in het grondwater in de tweede aquifer stabiel zijn (P121, BD24-3, BD24-4, D10, D11, D14, P118A) of dalen (P118B) in de tijd, maar fluctuaties zijn mogelijk. De resultaten lijken erop te duiden dat het onttrekkingssysteem geïnstalleerd ter hoogte van de bronzones in de eerste aquifer de verontreiniging capteert en neerwaartse verspreiding van het verontreinigd grondwater mitigeert.

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 perceelsgrens (stroomafwaarts van de bronzones in de eerste aquifer) zijn de concentraties aan FC verbindingen lager dan 10% van de oplosbaarheid. Hieruit volgt dat momenteel wordt voldaan aan de saneringsdoelstellingen voor de tweede aquifer. De noodzaak om deze doelstellingen op te volgen (door monitoring) blijft behouden.

Om het effect van het onttrekkingssysteem op de FC concentraties in de tweede aquifer te evalueren wordt aanbevolen om de jaarlijkse monitoring van peilbuizen in de tweede aquifer te behouden. De eerst volgende volledige monitoring zal plaatsvinden **in juli 2015**. Voor peilbuis P321 blijft de driemaandelijkse monitoring behouden en voor peilbuizen BD24-3 en P121 een halfjaarlijkse monitoring. Aanbevolen wordt om de monitoringsfrequentie van peilbuis D09 te verhogen van jaarlijks naar halfjaarlijks. Voor peilbuizen D10 en P118A wordt voorgesteld de frequentie te verlagen van halfjaarlijks naar jaarlijks.

4.5 ZUIDELIJKE TERREINGRENS

4.5.1 Uitgevoerd veldwerk

Een overzicht van de bemonsterde peilbuizen langs de zuidelijke 3M perceelsgrens is weergegeven in onderstaande tabel. De locaties van deze

peilbuizen zijn aangeduid op plan in Bijlage **5**. De stalen zijn door 3M en SGS geanalyseerd op FC verbindingen.

Peilbuis B3 is niet bemonsterd in januari 2014 omdat de omgeving van de peilbuis onder water stond. Tijdens de monitoring van juli 2014 zijn bijkomend eenmalig de peilbuizen PA109B en PA111B bemonsterd.

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

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum januari 2014	Bemonsteringsdatum juli 2014	Analyse
B3	0,4-1,4	-	X	FC
B7	1,8-3,8	X	X	FC
P372	4,8-5,8	X	X	FC
P378	3,5-5,5	X	X	FC
PA109A	5,0-6,0	X	X	FC
PA109B *	2,0-3,0		X	FC
PA111A	4,0-6,0	X	X	FC
PA111B *	1,5-3,5		X	FC
PA112	3,5-5,5	X	X	FC

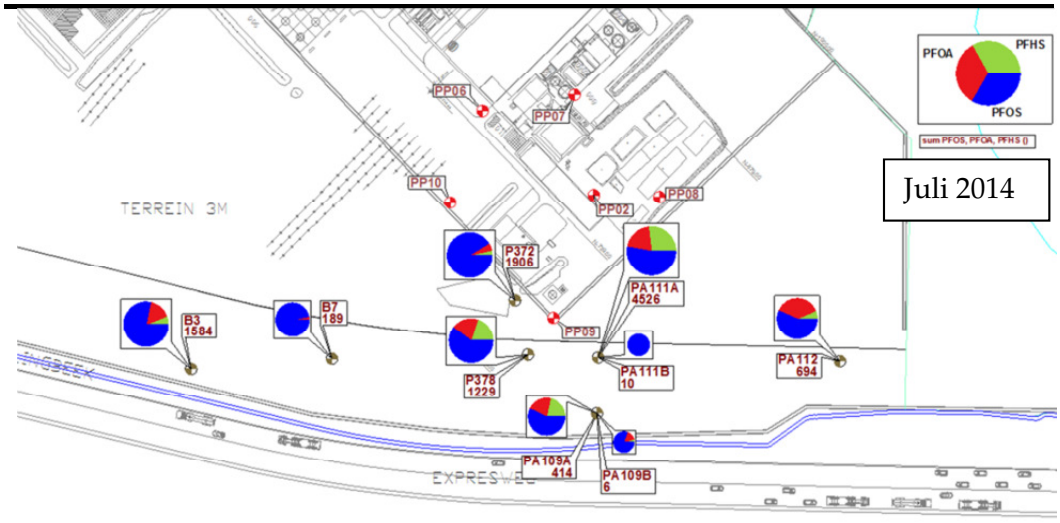
* *Eenmalige staalname*

4.5.2 *Bespreking resultaten*

De analyseverslagen zijn toegevoegd in Bijlage **6**. Een toetsingstabel met deze resultaten is opgenomen in Bijlage **7** samen met de resultaten van de veldmetingen.

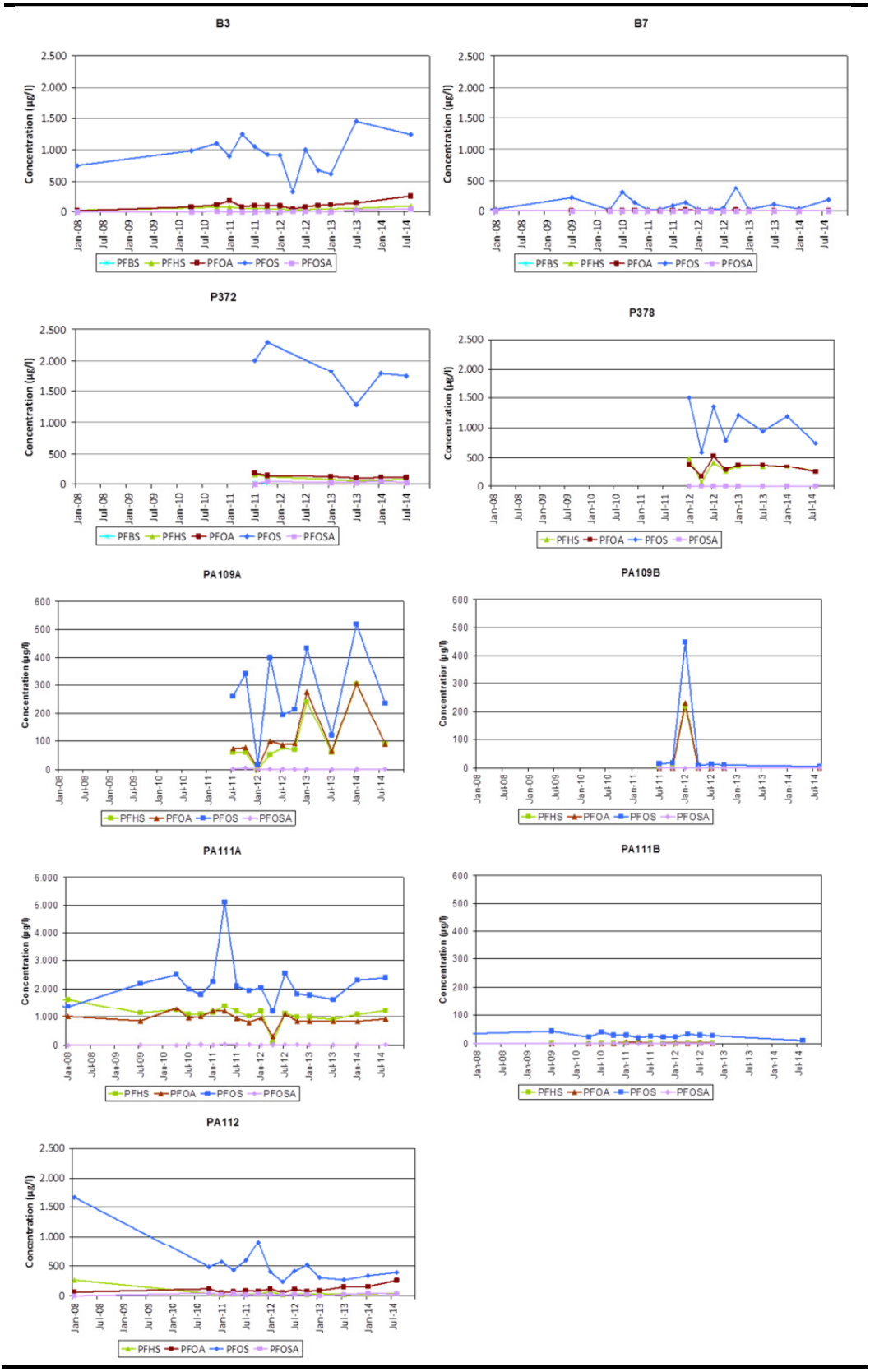
De grondwateranalyseresultaten voor de som van PFOS, PFHS en PFOA van juli 2014 ter hoogte van de zuidelijke perceelsgrens zijn op de onderstaande figuur voorgesteld.

Figuur 4.5 ***Schematische voorstelling FC grondwateranalyseresultaten thv. zuidelijke perceelsgrens***



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

Grafiek 4.5 Evolutie FC concentraties t.h.v. zuidelijke perceelsgrens



In het algemeen vertonen de FC-concentraties in het grondwater ter hoogte van de zuidelijke perceelsgrens een stabiele of dalende trend met fluctuaties in de tijd, met uitzondering van peilbuis B3 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 perceelsgrens 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 perceelsgrens beperkt.

De halfjaarlijkse monitoring blijft behouden en de volgende monitoring zal plaatsvinden **in januari 2015**.

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

4.6.1 Uitgevoerd veldwerk

Een overzicht van de bemonsterde peilbuizen ten zuiden van de Expresweg (Z-peilbuizen) is weergegeven in onderstaande tabel. De locaties van deze peilbuizen zijn aangeduid op plan in Bijlage **5**. De stalen zijn door 3M en SGS geanalyseerd op FC verbindingen.

Tabel 4.4 Overzicht bemonsterde peilbuizen ten zuiden van de Expresweg (Z-peilbuizen)

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum jan 2014	Analyse
Z1-3m	2,0-4,0	X	FC
Z2-3m	3,5-5,5	X	FC
Z2-10m	8,0-10,0	X	FC
Z4-8m	6,0-8,0	-	FC

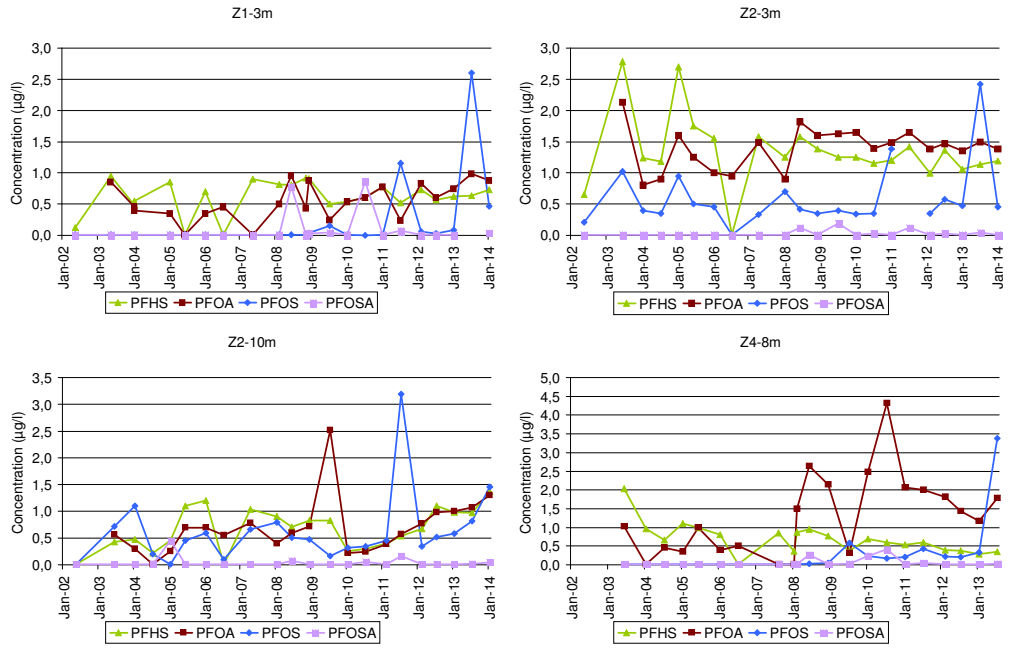
Peilbuis Z4-8m kon niet meer bemonsterd worden omdat deze beschadigd is en niet langer bruikbaar.

4.6.2 Bespreking resultaten

De analyseverslagen zijn toegevoegd in Bijlage ***6***. Een toetsingstabel met deze resultaten is opgenomen in Bijlage ***7*** samen met de resultaten van de veldmetingen.

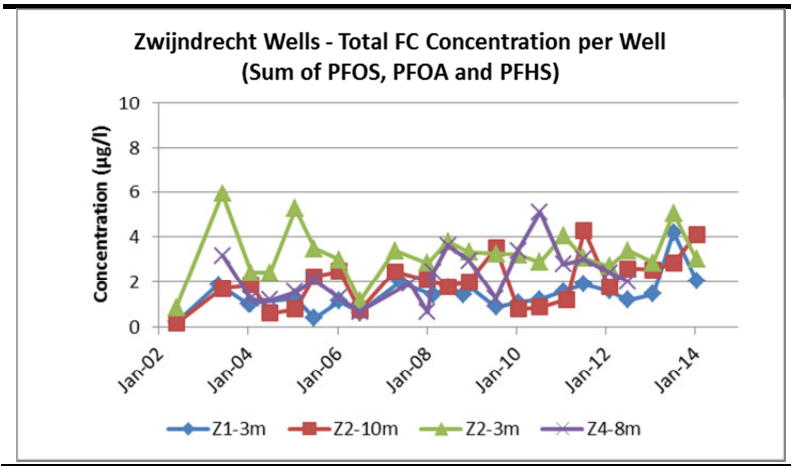
De evolutie van de FC concentraties in het grondwater ten zuiden van de expresweg is voorgesteld in onderstaande grafieken.

Grafiek 4.6 Evolutie FC concentraties t.h.v. Zwijndrecht Z-peilbuizen



De evolutie van de som FC concentraties (som PFOS, PFHS en PFOA) in het grondwater ten zuiden van de expresweg is voorgesteld in onderstaande grafiek.

Grafiek 4.7 Evolutie som FC concentraties t.h.v. Zwijndrecht Z-peilbuizen



De gemeten concentraties fluctueren, maar zijn laag (< 5 µg/L).

4.6.3 *Conclusies en aanbevelingen*

Algemeen kan gesteld worden dat de gemeten concentraties FC verbindingen in grondwater ter hoogte van de Zwijndrecht peilbuizen in dezelfde lijn liggen als de concentraties gemeten tijdens voorgaande bemonsteringen. De gemeten concentraties zijn relatief laag.

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 organofluoconcentraties 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 eindevaluatieonderzoek opgelegd voor de zone ten zuiden van de Expresweg (Z-peilbuizen). Dit eindevaluatieonderzoek met referentie R002-0243999 is op 24/10/2014 aan OVAM overgemaakt. Aanbevolen wordt om na conformiteit door OVAM van het eindevaluatie onderzoek alle peilbuizen in deze zone definitief buitengebruik te stellen conform de wettelijke voorschriften.

4.7 ZONE ONDERGRONDSE TANKEN

4.7.1 Uitgevoerd veldwerk

In juli 2014 zijn naast peilbuis P18 en P28 een aantal bijkomende peilbuizen bemonsterd voor de afperking van de verontreiniging met minerale olie. In onderstaande tabel is een overzicht gegeven van de bemonsterde peilbuizen. De locaties van deze peilbuizen zijn aangeduid op plan in Bijlage ***5***. De stalen zijn door SGS geanalyseerd op minerale olie, pH en EC.

Tabel 4.5 Overzicht bemonsterde peilbuizen zone ondergrondse tanks

Peilbuis	Filterdiepte (m-mv)	Bemonsteringsdatum Juli 2014	Analyses
P13	onbekend	X	MO
P16	4,0-6,0	X	MO
P17	4,0-6,0	X	MO
P21B	3,5-5,5	X	MO
P305	3,5-5,5	X	MO
U14	Onbekend	X	MO
P18	1,4-3,4	X	MO
P28	1,1-3,1	X	MO

4.7.2 Bespreking resultaten

De analyseverslagen zijn toegevoegd in Bijlage ***6***. Een toetsingstabel met deze resultaten is opgenomen in Bijlage ***7*** samen met de resultaten van de veldmetingen.

Veldparameters

De pH en geleidbaarheid van peilbuizen P18 en P28 zijn niet tijdens de bemonstering gemeten omdat de sterk verhoogde waarden de veldmeetapparatuur beschadigt. In het grondwater ter hoogte van de peilbuizen P18 en P28 zijn in het labo een verhoogde conductiviteit (> 58 000 µS/cm) en een verhoogde pH (> 12,4) gemeten. In de afperkende peilbuizen

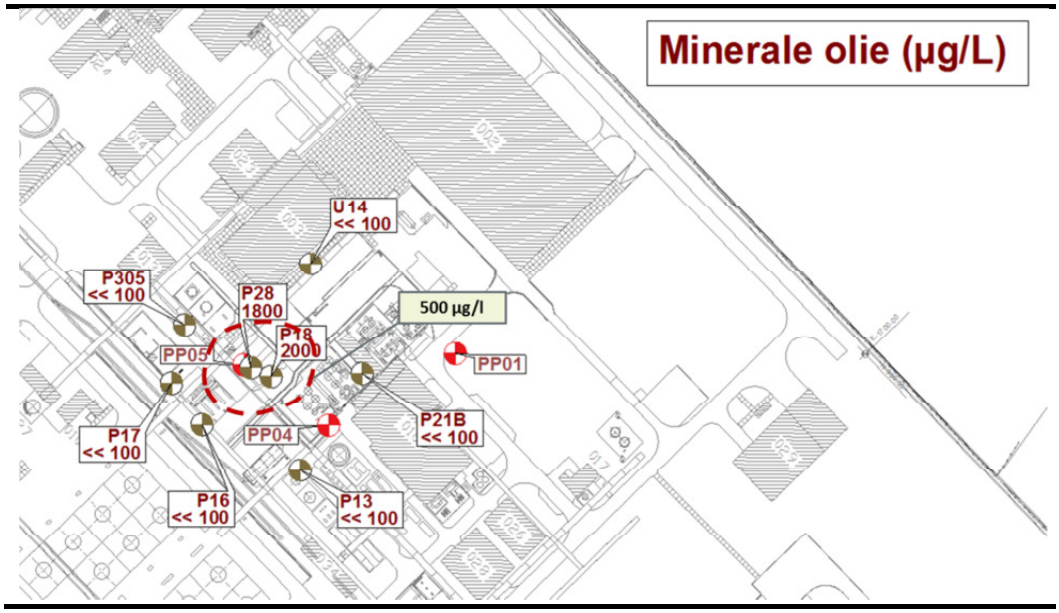
zijn normale waarden voor pH en geleidbaarheid (EC) gemeten, met uitzondering van P21B waar een licht verhoogde EC waarde van 2400 µS/cm is gemeten.

Minerale olie

In het grondwater ter hoogte van de peilbuizen P18 en P28 is een verhoogde concentratie aan minerale olie gemeten. De gemeten concentraties zijn vergelijkbaar met de concentraties gemeten in juli 2012 en juli 2013. De gemeten minerale olie 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.

Ter hoogte van de afperkende peilbuizen zijn in het grondwater geen minerale olie concentraties boven de detectielimiet vastgesteld.

Figuur 4.6 ***Schematische voorstelling minerale olie grondwateranalyseresultaten thv. tankenpark***



4.7.3 Conclusies en aanbevelingen

De verontreiniging met minerale olie, pH en geleidbaarheid is afgeperkt en is gelokaliseerd rond peilbuizen P18 en P28. De hoogst gemeten concentratie minerale olie bedraagt ca. 2 keer 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 2015**.

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. Aanbevolen wordt om na conformiteit door OVAM van het eindevaluatie onderzoek alle peilbuizen in de zone van de ondergrondse tanks, die niet meer opgevolgd zullen worden, definitief buitengebruik te stellen conform de wettelijke voorschriften.

4.8 NATUURGEBIED BLOKKERSDIJK

4.8.1 Uitgevoerd veldwerk

De peilbuizen langs het 3M pad ten westen van het natuurgebied Blokkersdijk en het oppervlaktewater van de 3M-vijver zijn halfjaarlijks bemonsterd en het oppervlaktewater van de Blokkersdijkvijver is driemaandelijks bemonsterd om de evolutie in FC concentraties in deze zones na te gaan. Sinds januari 2014 worden de peilbuizen L4, P114 en P116 driemaandelijks opgevolgd, wordt P119C bijkomend halfjaarlijks bemonsterd en wordt L31 opnieuw halfjaarlijks bemonsterd. Peilbuis P114 was niet meer aanwezig en is op 6 maart 2014 vervangen door peilbuis P114bis met een identieke filterstelling.

In onderstaande tabel is een overzicht gegeven van de grondwater- en oppervlaktewater monitoring ter hoogte van het 3M pad ten westen van het natuurreservaat Blokkersdijk. De locaties van de peilbuizen en de monsternamepunten voor het oppervlaktewater zijn aangeduid op plan in Bijlage **5**. De grondwater- en oppervlaktewaterstalen zijn geanalyseerd op FC verbindingen door 3M en SGS.

Tabel 4.6 Overzicht staalnamelocaties natuurgebied Blokkersdijk

Locatie	Filterdiepte	Bemonsteringsdatum				Analyse
	(m-mv)	okt 2013	jan 2014	apr 2014	jul 2014	
L21	3,0-5,0		X		X	FC
L22	2,0-4,0		X		X	FC
L31	1,2-3,2	X	X		X	FC
L4	2,0-4,0		X	X	X	FC
P114	3,6-4,6	-	-			FC
P114bis	3,6-4,6			X	X	
P115	3,3-4,3		X		X	FC
P116	2,5-3,5	X	X	X	X	FC
P119C	2,5-4,5		X		X	FC
3M vijver	-	X	X	X	X	FC
Blokkersdijk standaard	-	X	X	X	X	FC
Blokkersdijk oost	-	X	X			FC

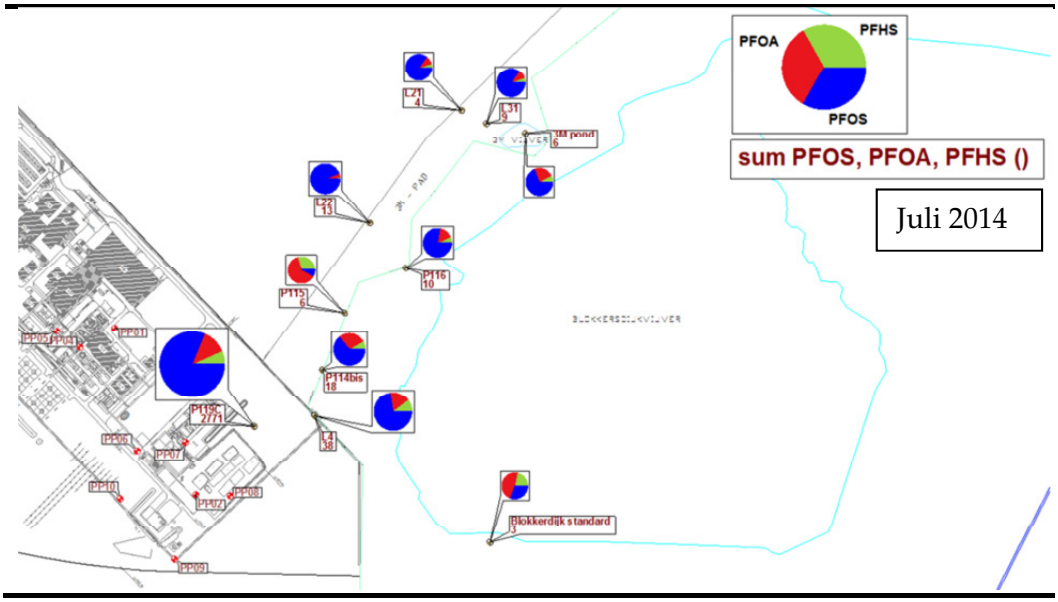
* Staalnamepunt "Blokkersdijk oost" is sinds april 2014 geschrapt omwille van de slechte toegankelijkheid

4.8.2 *Bespreking resultaten*

De analyseverslagen zijn toegevoegd in Bijlage ***6***. Een toetsingstabel met deze resultaten is opgenomen in Bijlage **7** samen met de resultaten van de veldmetingen.

Op onderstaande figuren zijn de meest recente (juli 2014) 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 4.7 ***Schematische voorstelling FC grondwateranalyseresultaten thv. natuurgebied Blokkersdijk***

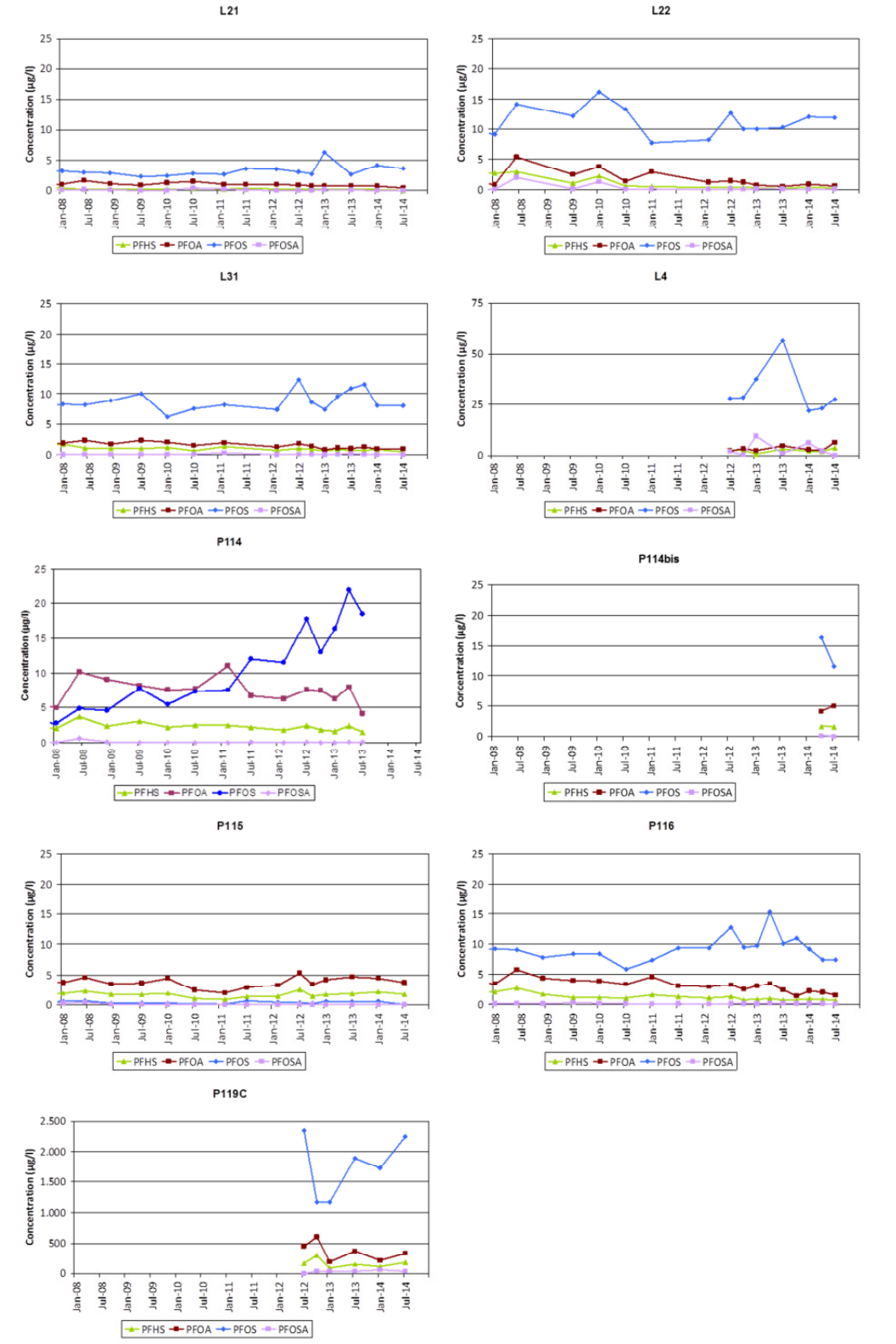


Algemeen kan gesteld worden dat de concentraties in grond- en oppervlaktewater relatief laag zijn, met uitzondering van peilbuis 119C. Hierbij dient te worden opgemerkt dat peilbuis P119C is dichtbij de WWTP bronzone bevindt en stroomopwaarts gelegen is van het 3M pad dat langs westzijde van het natuurreservaat loopt.

Grondwater: 3M pad

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

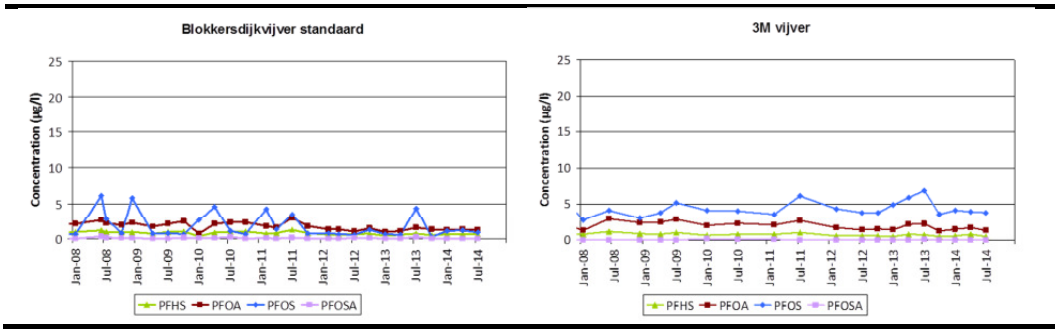
Grafiek 4.8 Evolutie FC concentraties in grondwater t.h.v. 3M pad (westelijk van Blokkersdijk natuurreservaat)



Oppervlaktewater: Blokkersdijkvijver en 3M-vijver

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

Grafiek 4.9 Evolutie FC concentraties in oppervlaktewater Blokkersdijk natuurreservaat



Statistiek

Er zijn voldoende data verzameld voor een statistische analyse voor de PFOS concentraties in het oppervlaktewater van Blokkersdijksvijver, de 3M vijver en de stroomopwaartse peilbuizen conform de bepalingen van het eerste gefaseerd bodemsaneringsproject.

De rapportage van deze trendanalyse, waarbij gebruik gemaakt is van zowel de "t-test" als de "Mann-Kendall analyse", is opgenomen in Bijlage *8*. Enkel indien beide statische analyses een stijgende trend als resultaat geven, wordt gesproken van een significante stijging.

In onderstaande tabel is een overzicht gegeven van de resultaten van de uitgevoerde statistische analyses.

Tabel 4.7 Overzicht resultaten statistische analyse PFOS concentraties

Locatie	Mann-Kendall	t-Test
L21	waarschijnlijk stijgend	geen trend
L22	stabiel	geen trend
L31	stabiel	geen trend
L4	stabiel	geen trend
P115	stabiel	geen trend
P116	stabiel	geen trend
P119C	Stabiel	geen trend
Blokkersdijk vijver (standaard locatie)	waarschijnlijk dalend	geen trend
3M vijver	stijgend	significante trend

Voor peilbuis L4 is de in het vorige tussentijds rapport bepaalde stijgende trend niet bevestigd. Het huidige groter aantal analyseresultaten geeft een meer betrouwbaar resultaat.
Ter hoogte van peilbuis P114 is voorheen een significante stijging (beide testen) van de PFOS concentraties waargenomen. Deze peilbuis is beschadigd en niet meer bruikbaar; de peilbuis is vervangen door peilbuis P114bis. Van peilbuis P114bis zijn er nog onvoldoende analyseresultaten beschikbaar om een statistische analyse uit te voeren.
Ter hoogte van peilbuis L21 is een potentiële stijgende trend (één test) van de PFOS concentraties waargenomen. Hierbij dient benadrukt te worden dat het om lage concentraties (< 10 µg/l) gaat.

In het oppervlaktewater van de 3M vijver is een significante stijging (beide testen) van de PFOS concentraties waargenomen. Hierbij dient eveneens benadrukt te worden dat het om lage concentraties (< 10 µg/l) gaat en dat de concentratietrends voor PFOA, PFHS en som FC stabiel of dalend is.

4.8.3 Conclusies en aanbevelingen

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

Er wordt echter aanbevolen om nader onderzoek uit te voeren naar mogelijk bronzones in deze zone aangezien de peilbuizen en de 3M vijver stroomopwaarts liggen van de nabij gelegen gekende bronzone (WWTP). Verder wordt aanbevolen om de FC concentraties in de 3M vijver, de Blokkersdijkvijver en de peilbuisP114bis, L4 en P116 (driemaandelijks), en L21, L22, L31 en P115 (halfjaarlijks) verder op te volgen. Tot slot wordt aanbevolen om peilbuis P119C toe te voegen aan de monitoring van de WWTP bronzone, aangezien deze peilbuis stroomafwaarts ligt van het natuurreservaat maar stroomopwaarts van de WWTP bronzone. De volgende volledige monitoring zal plaats vinden **in januari 2015**.

4.9 PALINGBEEK EN TOPHAT GRACHT

4.9.1 Uitgevoerd veldwerk

Om de FC vuilvracht naar de Schelde op te volgen worden het oppervlaktewater van de Palingbeek en de Tophatgracht en het oppervlaktewater ter hoogte van het bemalingsstation driemaandelijks bemonsterd. De staalnamelocaties zijn aangeduid op plan in Bijlage ***5***. De stalen zijn door 3M en SGS geanalyseerd op FC verbindingen.

Tabel 4.8 Overzicht staalnamelocaties Palingbeek en Tophatgracht

Locatie	Bemonsteringsdatum				Analyse
	Okt. 2013	Jan. 2014	Apr. 2014	Jul. 2014	
12	X	X	X	*	FC
13	X	X	X	X	FC
5	X	X	X	X	FC
Bemalingsstation	X	X	X	X	FC

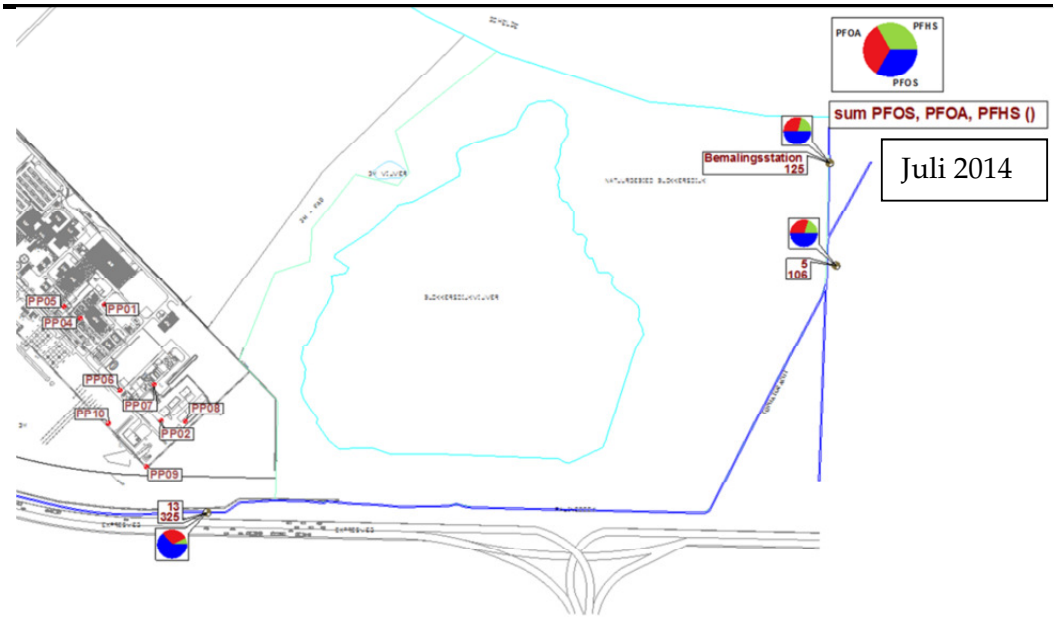
* In juli 2014 was geen water aanwezig ter hoogte van locatie 12

4.9.2 Bespreking resultaten

De analyseverslagen zijn toegevoegd in ***Bijlage 6***. In de toetsingstabellen in Bijlage 7 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 2014 zijn op de onderstaande figuur voorgesteld.

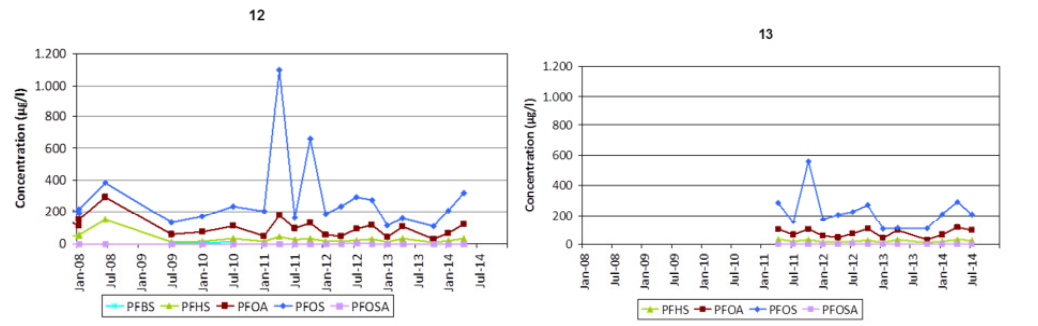
Figuur 4.8 ***Schematische voorstelling FC grondwateranalyseresultaten thv. Palingbeek en de Tophatgracht***



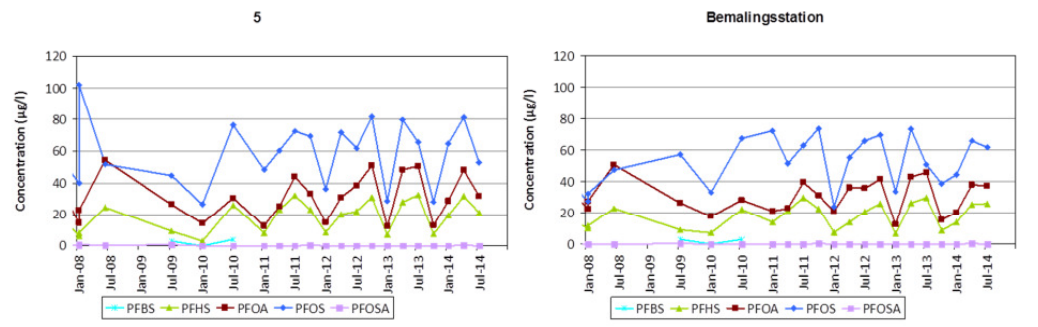
De evolutie van de FC concentraties in het oppervlaktewater van de Palingbeek en de Tophatgracht is voorgesteld in onderstaande grafieken.

Grafiek 4.10 ***Evolutie FC concentraties in oppervlaktewater Palingbeek en de Tophatgracht***

Staalnamepunten Palingbeek



Staalnamepunten Tophatgracht



De FC-concentraties ter hoogte van de Palingbeek en Tophat gracht vertonen een vrij stabiel verloop sinds 2012. De concentraties in de Tophat gracht zijn lager dan de concentraties gemeten 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 143 g voor de periode 1 juli 2013 tot 30 juni 2014. In onderstaande tabel is een overzicht gegeven van de berekende vuilvracht per jaar.

Tabel 4.9 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

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 (Tophatgracht). Dit is vermoedelijk het gevolg van verdunning door het instromen van minder of niet-verontreinigd grond- of oppervlaktewater. De concentraties in het oppervlaktewater van de Tophatgracht (locatie 5) en het bemalingsstation zijn vergelijkbaar.

Aanbevolen wordt om de driemaandelijkse bemonsteringsfrequentie te behouden. De volgende monitoring zal plaats vinden **in oktober 2014**.

4.10 REGENWATERCOLLECTORPUT

4.10.1 Uitgevoerd veldwerk

Om de invloed van de lopende grondwatersanering op de concentraties en debieten in de regenwaterriolering na te gaan, zijn maandelijks stalen genomen van de regenwater collectorput. Het debiet van de regenwater collectorput wordt dagelijks opgevolgd.

De locatie van de collectorput is aangeduid op plan in Bijlage **5**.

De waterstalen zijn geanalyseerd op de FC verbindingen PFOS en PFOA in het laboratorium van 3M en SGS.

4.10.2 Bespreking resultaten, conclusies en aanbevelingen

De analyseverslagen zijn toegevoegd in Bijlage ***6***. Een toetsingstabel met deze resultaten is opgenomen in Bijlage **7**.

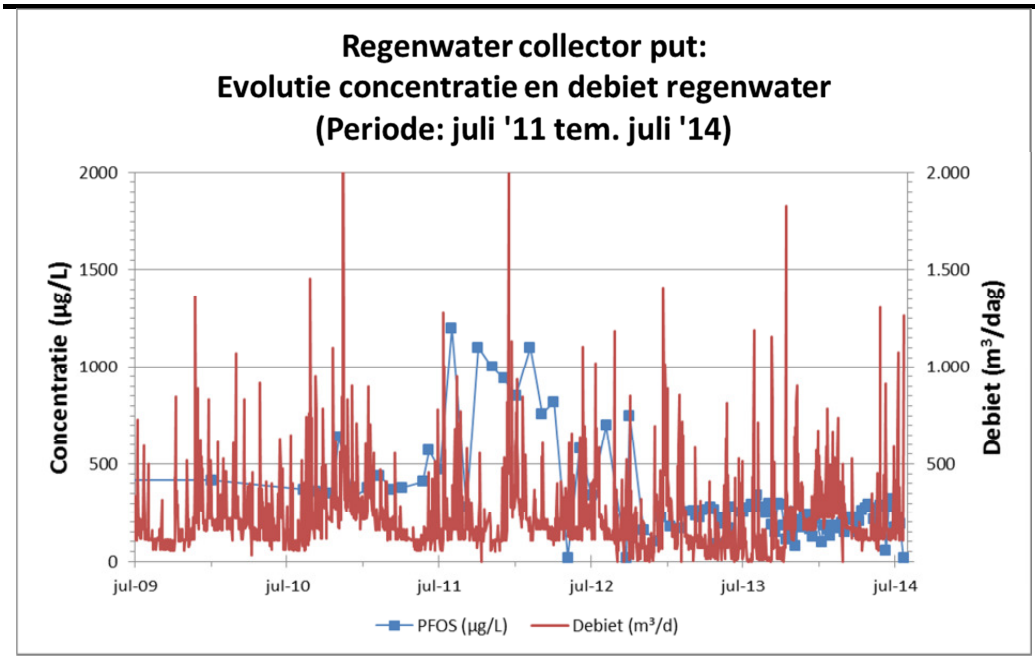
Regenwater collectorput

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 maandelijkse 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.

Regenwater collectorput

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

Grafiek 4.11 Evolutie PFOS concentratie in collector put



Sinds november 2012 vertonen de PFOS concentraties in de collectorput een stabiel verloop. De concentraties kunnen wel fluctueren. De gemiddelde concentratie PFOS in de periode juli 2013 – juni 2014 bedraagt 217 µg/l.

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

Tabel 4.10 Vuilvrachtberekening regenwater collectorput voor PFOS

Periode	Vuilvracht per jaar (kg)	Vuilvracht per dag (g)
2007	42	115
2008	32	89
juli 2009 tem. juni 2010	32	88
juli 2010 tem. juni 2011	39	107
juli 2011 tem. juni 2012	68	187
juli 2012 tem. juni 2013	18	48
juli 2013 tem. juni 2014	17	47

Deze gegevens maken deel uit van de totale vuilvrachtberekening naar de Schelde (zie paragraaf 4.11). De hogere vuilvracht 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 redelijk stabiel.

Aanbevolen wordt om de dagelijkse opvolging van het debiet en de maandelijkse monitoring van het hemelwater in de collectorput te blijven behouden. De resultaten van de collectorput zijn minder gevoelig voor fluctuaties veroorzaakt door neerslag en daardoor meer betrouwbaar.

4.11 VUILVRACHT RICHTING SCHELDE

Op basis van de gegevens bekomen uit de monitoringscampagne van 1 juli 2013 tot 30 juni 2014 is de vuilvracht welke dagelijks naar de Schelde stroomt opnieuw berekend worden (zie Tabel 4.11). De vermoedelijke PFOS vuilvracht is in het eerste gefaseerd bodemsaneringsproject geraamd op 290 g/dag. De veilige vuilvracht voor PFOS (PNEC waarde) is vastgelegd op 370 g/dag.

Tabel 4.11 Gemiddelde PFOS vuilvracht richting de Schelde (juli 2013 tem. juni 2014)

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)	692	14,0	9,7
Regenwaterriolering	216	217	47
Palingbeek/Tophatgracht***	2.865	49,8	143
Totale vuilvracht			**199**

* 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 tabel en grafiek zijn de gemiddelde berekende vuilvrachten van de voorbije jaren en het huidige jaar samengevat.

Tabel 4.12 Overzicht PFOS vuilvracht richting de Schelde

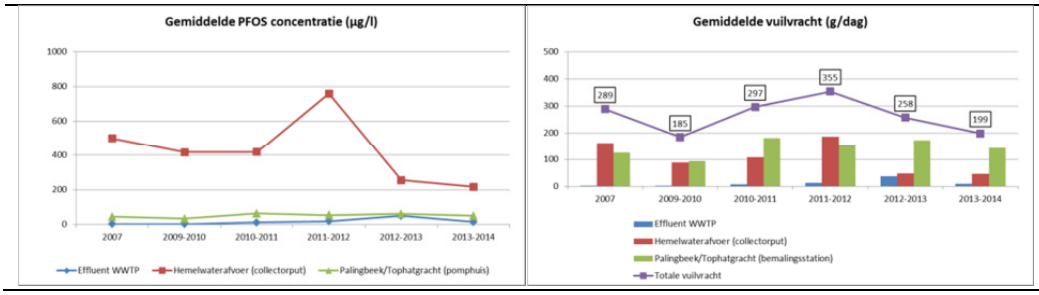
Periode	Gemiddelde vuilvracht (g/dag)
2007	289
2009-2010	185
2010-2011	297
2011-2012	355
2012-2013	258
2013-2014	199

* 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

Grafiek 4.12 Evolutie gemiddelde PFOS concentratie en vuilvracht naar de Schelde



Voor de periode juli 2013 tot en met juni 2014 bedraagt de berekende totale PFOS vuilvracht naar de Schelde 199 g/dag. Zoals aangegeven in de voorgaande rapporten, was de hogere berekende vuilvracht voor de vorige periode (2011-2012) waarschijnlijk het gevolg van het incident in 2011 waarbij slib in de collectorput van de regenwaterriolering terecht is gekomen. In september 2012 is dit slib geruimd en sindsdien zijn de PFOS concentraties in de regenwaterriolering en de berekende gemiddelde vuilvracht opnieuw gedaald.

De berekende vuilvracht aan PFOS naar de Schelde heeft nooit de PNEC waarde (370 g/dag) overschreden.

4.12 VOORSTEL MONITORINGSPROGRAMMA 2014

In de volgende tabel is een overzicht gegeven van het voorgestelde monitoringsprogramma voor de periode oktober 2014 – juli 2015.

Tabel 4.13 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 analysepakket 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 stopgegezet 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

5 GRONDWATERONTTREKKING BRONZONE

5.1 OPVOLGING ONTTREKKINGSSYSTEEM

De hieronder besproken resultaten hebben betrekking op de periode van juli 2013 tot juli 2014.

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 volledig operationeel.

In totaal zijn er negen pompputten in bedrijf (zie plan in Bijlage ***9***). Er zijn drie pompputten in de "zone gebouw 16" en zes pompputten in de "zone WWTP". In de regel is het filterend gedeelte van de pompputten geplaatst tot net boven de polderklei, behalve bij pompputten PP09 en PP10, die in een zone staan waar geen polderklei is aangetroffen. De polderklei op de site bevindt zich op een variabele diepte, maar algemeen geldt: hoe dichter bij de Schelde, hoe dieper de polderklei ligt ten opzichte van het maaiveld. De pompputten hebben bijgevolg allemaal een verschillende diepte t.o.v. het maaiveld. In onderstaande tabel is een overzicht gegeven.

Tabel 5.1 Technisch overzicht pompputten

Pompput	Locatie	Diepte (m-mv)	Gemiddeld jaardebiet (l/min)
PP01	Gebouw 16	2,0-5,5	3,1
PP04	Gebouw 16	2,0-6,0	1,8
PP05	Gebouw 16	1,7-5,7	*
PP02	WWTP	3,2-6,2	0,9
PP06	WWTP	1,3-5,3	3,5
PP07	WWTP	2,0-6,0	9,1
PP08	WWTP	3,2-7,2	5,8
PP09	WWTP	3,0-6,0	2,5
PP10	WWTP	3,0-6,0	8,6

*geen constant debiet door softwareproblemen

Elke pompput beschikt over een niveausensor (zie schematisch overzicht in ***Bijlage 10***). 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

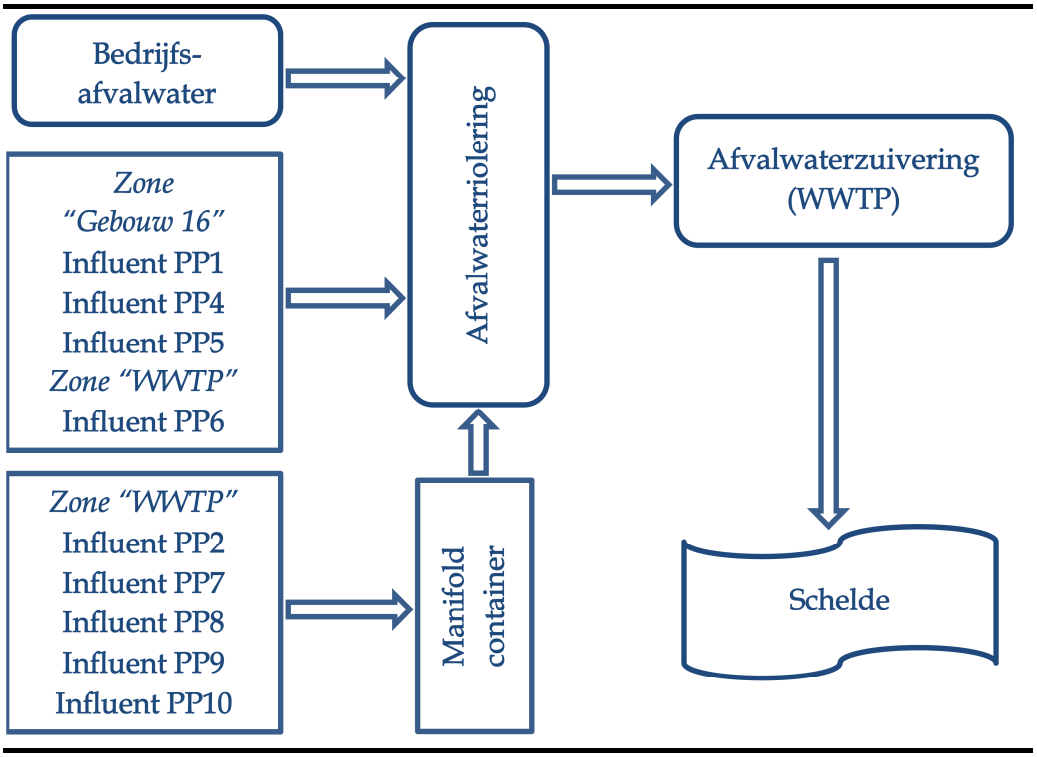
pompputten die veel water geven, wordt het niveau waarop de pompen terug aangeschakeld worden iets hoger ingesteld. In onderstaande tabel worden deze schakelniveaus weergegeven.

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 16 zijn rechtstreeks aangesloten op de chemische riolering van 3M. In de zone WWTP zijn de pompputten, met uitzondering van PP06, eerst aangesloten op een manifold 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.

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 2013 tot 1 juli 2014 heeft ERM bezoeken uitgevoerd op de data vermeld in onderstaande tabel. ERM komt ook steeds ter plaatse tijdens of kort na interventies door de aannemer, deze data zijn eveneens mee opgenomen in onderstaande tabel.

Tabel 5.3 Data controlebezoeken

Controlebezoeken ERM	Controlebezoeken ERM	Controlebezoeken ERM
02-07-13	17-10-13	11-02-14
03-07-13	25-10-12	18-02-14
17-07-13	29-10-13	25-02-14
24-07-13	07-11-13	03-03-14
30-07-13	14-11-13	11-03-14
01-08-13	10-12-13	21-03-14
09-08-13	18-12-14	26-03-14
12-08-13	23-12-13	02-04-14
14-08-13	30-12-13	08-04-14
27-08-13	07-01-14	16-04-14
06-09-13	13-01-14	29-04-14
17-09-13	16-01-14	14-05-14
24-09-13	21-01-14	17-06-14
01-10-13	28-01-14	01-07-14
07-10-13	05-02-14	

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
17/07/2013	PP01, PP02, PP04, PP05, PP07, PP08, PP09 & PP10
28/08/2013	PP09 & PP10
01/10/2013	PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 & PP10
07/11/2013	PP09 & PP10
23/12/2013	PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 & PP10
28/01/2014	PP01, PP02, PP07, PP08 & PP10
25/02/2014	PP09 & PP10
02-04-2014	PP01, PP02, PP04, PP05, PP07, PP08, PP09 & PP10
29-07-2014	PP01, PP02, PP04, PP05, PP06, PP07, PP08, PP09 & PP10

Opvolging zettingen

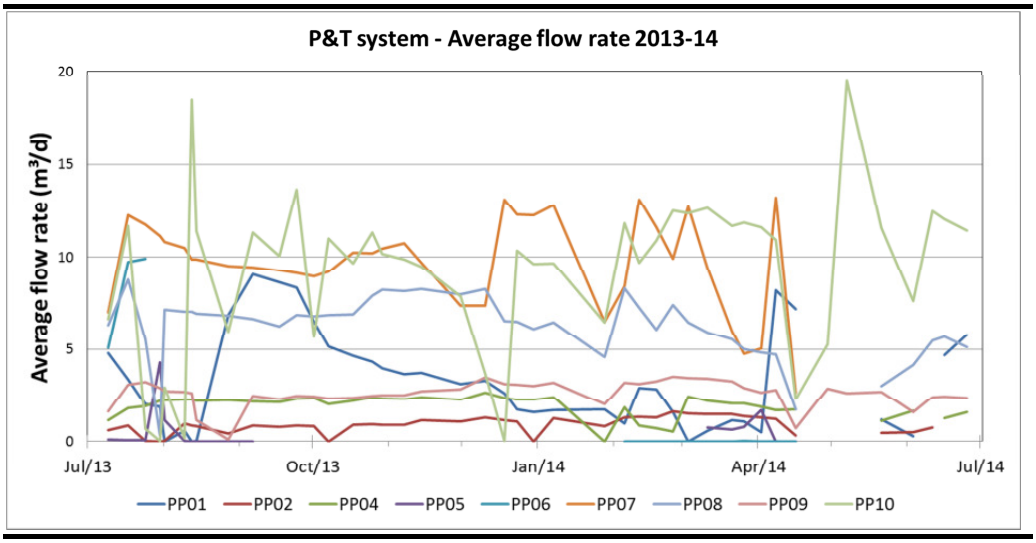
Er zijn verspreid over het terrein 20 zettingsbouten geplaatst. Deze zettingsbouten zijn in de periode 2009-2013 geregeld ingemeten. Eind 2013 is door de onafhankelijk stabiliteitsexpert Grontmij een evaluatie van de data uitgevoerd om na te gaan of de stabiliteitsmetingen kunnen worden stopgezet.

5.1.3 *Resultaten*

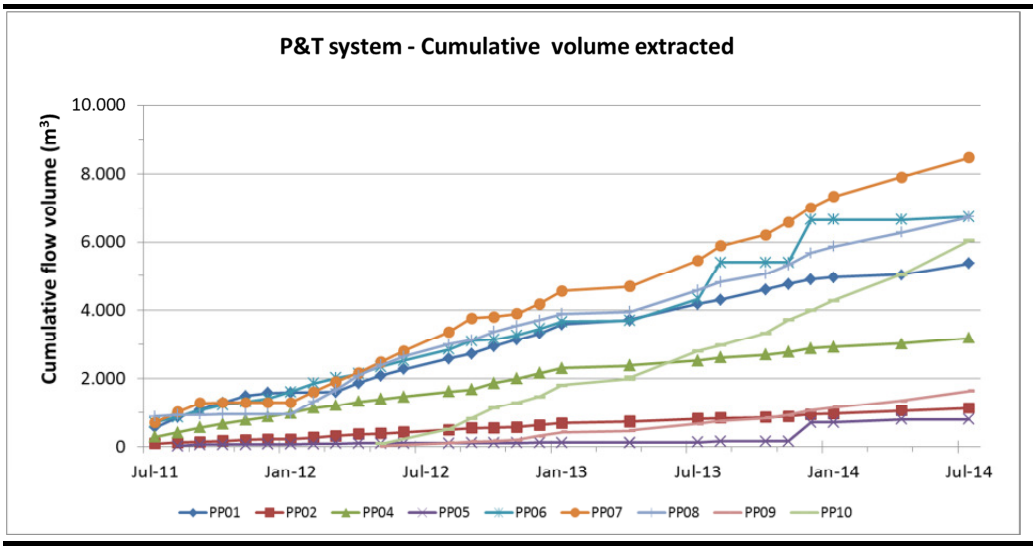
De onttrokken debieten

In onderstaande grafieken wordt getoond wat de gemiddelde verpompte debieten zijn en de cumulatieve verpompte debieten zijn van 1 juli 2013 tot 1 juli 2014.

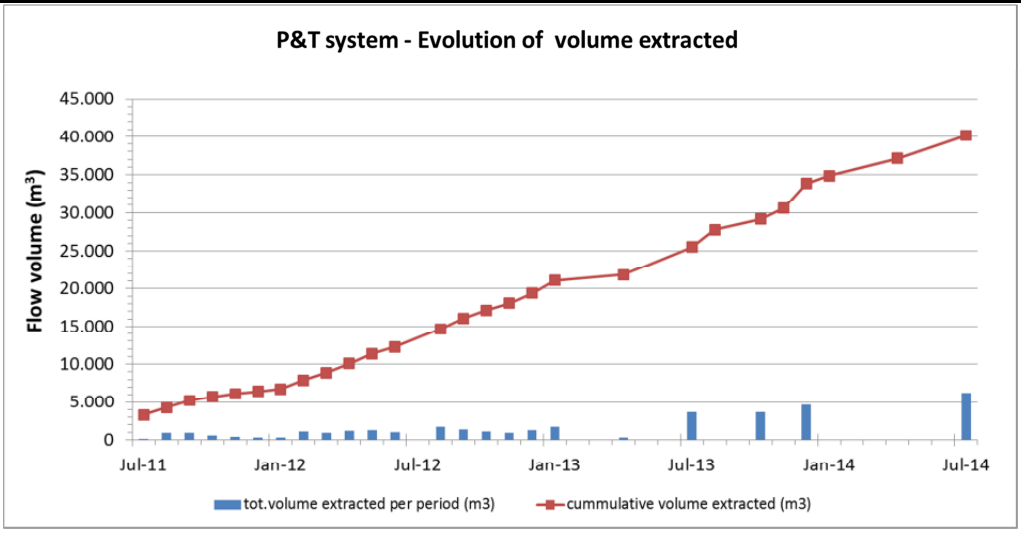
Grafiek 5.1 Gemiddeld debiet per onttrekkingsput(juli 2013 tot juli 2014)



Grafiek 5.2 Cumulatief debiet per onttrekkingsput



Grafiek 5.3 Cumulatief debiet van het totaal saneringssysteem



Uit hoger getoonde grafieken kunnen volgende zaken afgeleid worden:

- Het onttrokken debiet per put (uitgezonderd PP05 die intermitterend werkt) verschilt aanzienlijk en varieert tussen minimaal 0,3 m³/dag (PP02) en maximaal 19,5 m³/dag (PP10);

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

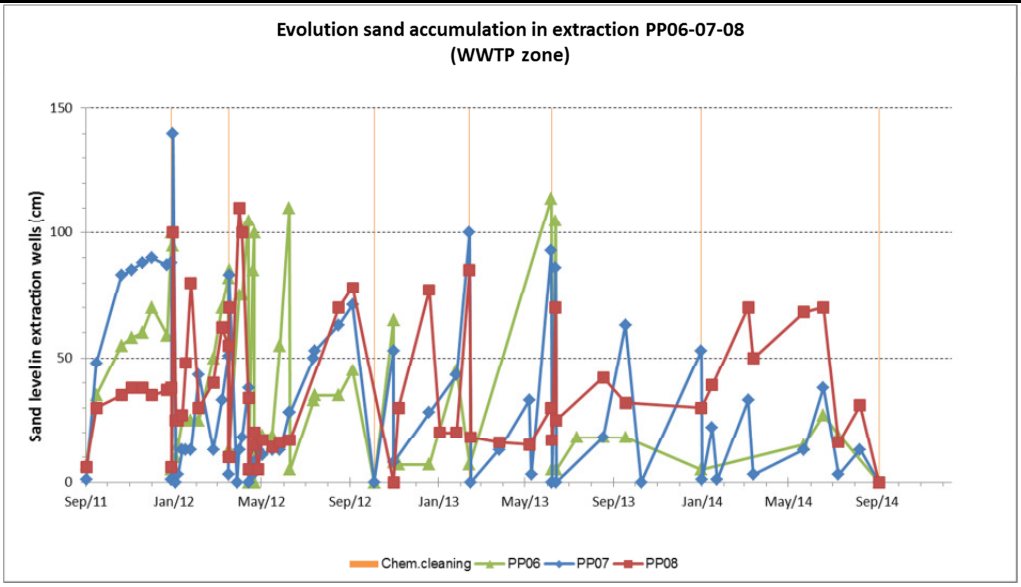
- Verder zijn nog een aantal kleinere onderbrekingen geweest van de installatie door technische storingen. De volgende storingen komen frequent voor:
  - Pompwaaiers en debietmeters verstopt, hetzij door fijn zand, hetzij door ijzerslib;
  - Afgaan van de thermische beveiliging van de pomp; en
  - Flexibele leidingen die los komen (in de pompkamer van verschillende pompputten).

- Tussen juli 2013 en juli 2014 is ongeveer 14.625 m³ verontreinigd grondwater onttrokken (tegen 13.500 m³ vorige periode). In totaal is sinds juli 2011 circa 40.000 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. 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.

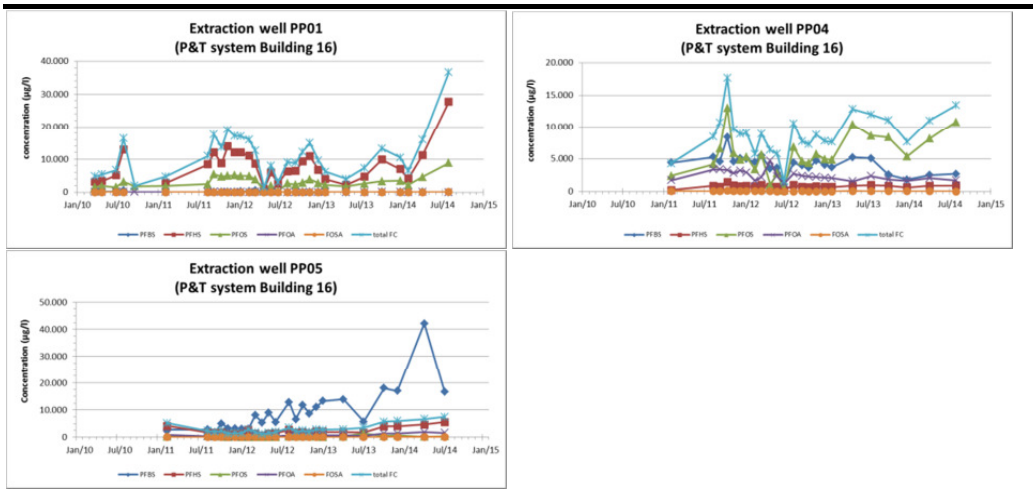
- Bij elektrische problemen of software problemen wordt de technische dienst van 3M gecontacteerd en gevraagd om de betreffende problemen op te lossen. Bij 3M is een vaste contactpersoon/technieker aangesteld om eventuele problemen op te lossen.

- 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 januari en september 2014 werd een chemische reiniging uitgevoerd.
  Het protocol van de reiniging is aangepast, zo worden alle putten nu eerst mechanische en daarna chemisch gereinigd (nadat eerst de pompen zijn verwijderd). Na de chemische reiniging worden de putten opnieuw gespoeld en worden de pompen teruggehangen.

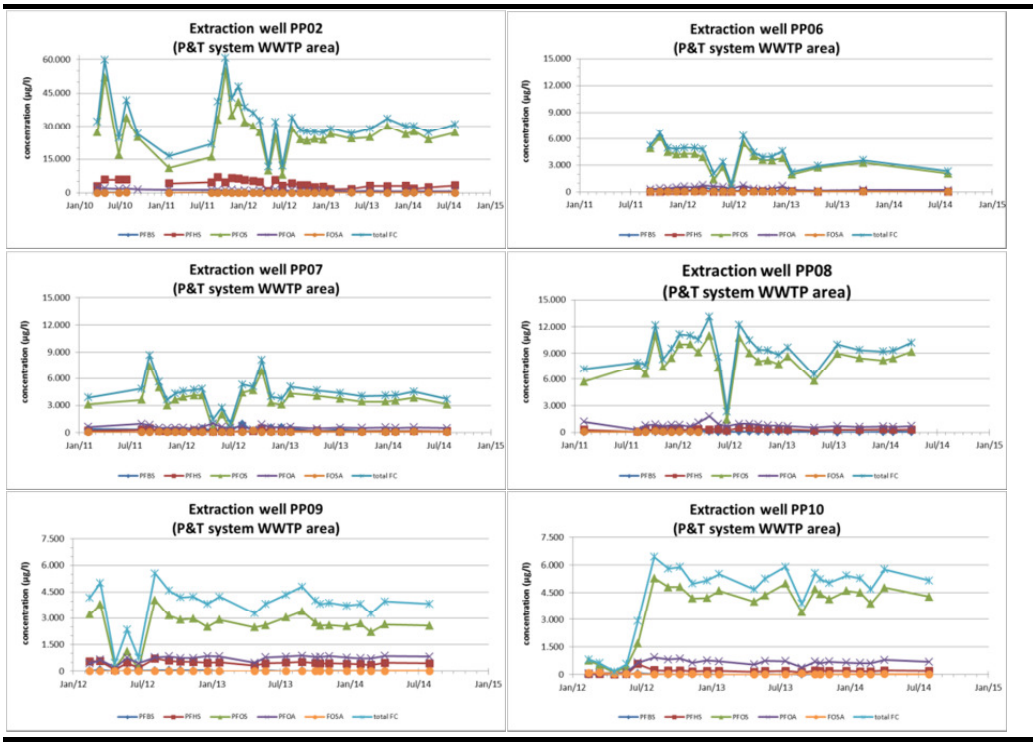
De concentraties in het onttrokken grondwater

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

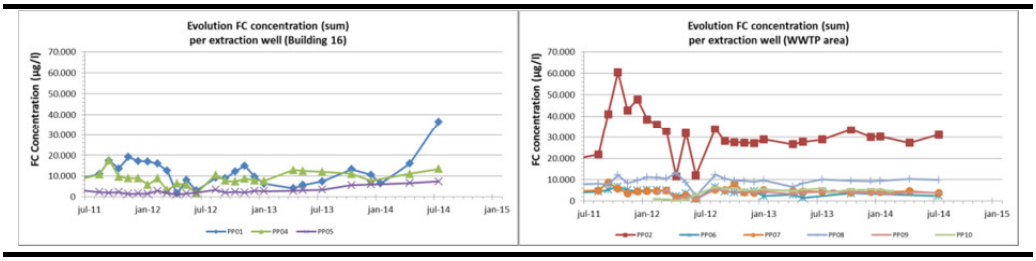
Grafiek 5.5 Evolutie FC concentraties t.h.v. gebouw 16 (pompput PP01, PP04 en PP05)



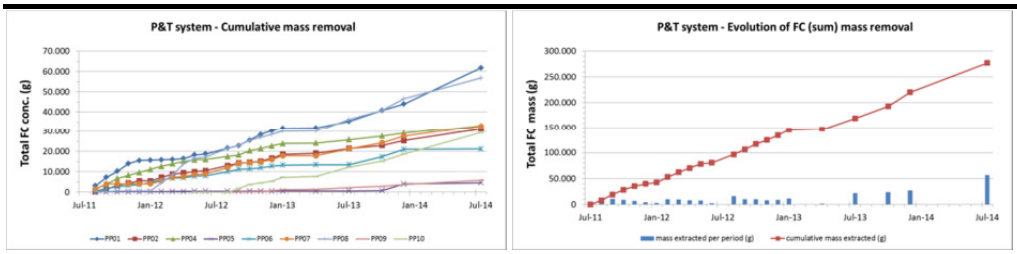
Grafiek 5.6 Evolutie FC concentraties t.h.v. WWTP zone (pompput PP02, PP06, PP07, PP08, PP09 en PP10)



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



Grafiek 5.8 Cumulatieve vrachtverwijdering per onttrekkingsput en van het totaal saneringssysteem



Uit bovenstaande grafieken kunnen volgende zaken afgeleid worden:

- Er is een verschil in samenstelling van het met FC's verontreinigde grondwater in de zone rond gebouw 16 (PP01, PP04 en PP05) en van het grondwater in de zone WWTP (overige pompputten):
  - In PP01 is PFHS de component met de hoogste concentraties.
  - In de overige pompputten 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 PP02.

- De opgepompte concentraties aan totale FC's (PFOS, PFOA en PFHS) fluctueren, maar in de meeste pompputten zijn de concentraties 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 2014 circa 37.000 µg/l en in pompput PP05 circa 7.000 µg/l.

- Tussen juli 2012 en juni 2013 is ongeveer 110 kg FC's verwijderd. In totaal is sinds juli 2011 circa 280 kg FC verwijderd.

- De lange-termijn trend van de FC-concentraties in de meeste pompputten is stabiel.

Stabiliteit

Uit de evaluatie (zie nota in xxx) door de onafhankelijk stabiliteitsexpert Grontmij van de zettingsmetingen uitgevoerd in de periode 2009 tot en met 2013 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 te stoppen. 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

Algemeen gezien werkt de onttrekking goed, met uitzondering van de onttrekking in pompput PP05. In totaal is in de periode juli 2011 tot juli 2013 ongeveer 40.000 m^3 verontreinigd grondwater onttrokken en circa 280 kg FC verwijderd. Uit de analyses blijkt dat PFOS is de belangrijkste verontreinigingsparameter is.

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 lekdichtheid van het leidingwerk in de pompkamers.

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.

De opgepompte concentraties zullen verder opgevolgd worden in de toekomst.

Het onttrekkingssysteem lijkt geen invloed te hebben op de zettingen op het terrein.

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 worden uitgevoerd. De staalname frequentie van de pompputten (met analyse van FC-concentraties) dient behouden te blijven op viermaandelijks. Verder wordt aanbevolen om nog eenmaal een stabiliteitsmonitoring uit te voeren en de resultaten te laten evalueren door een onafhankelijk stabiliteitsdeskundige om na te gaan of de monitoring kan worden stopgezet.

5.2 MONITORING GRONDWATERSTANDEN

Op 30 juni 2014 is een algemene dipronde geweest 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. De meetresultaten voor de eerste aquifer en de tweede aquifer zijn weergegeven op plan in Bijlage ***12***.

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]).

Om de seizoenale fluctuaties van het grondwaterpeil en de invloed van de pompputten beter te kunnen opvolgen, zal in vanaf 2015 elk kwartaal een dipronde worden uitgevoerd. Deze diprondes zullen samenvallen met de bemonsteringscampagnes voorzien in januari, april, juli en oktober. De algemene dipronde, waarbij het merendeel van de peilbuizen wordt ingemeten is voorzien voor april; In januari, juli en oktober zal enkel een representatieve selectie peilbuizen worden ingemeten.

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; en
  - Druk op de afvoerleiding.

- 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.

Op basis van de monitoringgegevens en de uiteindelijke omvang van de invloedstraal bij evenwicht na langdurig pompen, zal bepaald worden of een eventuele uitbreiding van het systeem noodzakelijk is.

6 VOORZIENE WERKEN IN VOLGENDE JAAR VAN DE SANERING

6.1 GRONDWATERSANERING

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

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

6.2 MONITORING GRONDWATER EN OPPERVLAKTEWATER

In Tabel 4.13 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.

Jaarlijks zal een waterniveau monitoringsronde uitgevoerd worden op het monitoringsnetwerk om de grondwaterstromingsrichting te bepalen en periodiek op te volgen.

6.3 PLANNING

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

- Grondwateronttrekking in de twee bronzones.
- Monitoring grondwaterconcentraties conform frequentie vermeld in Tabel 4.13.
- Viermaandelijkse opvolging van de grondwaterpeilen.
- September - november 2015: Opstellen en indienen 6e 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 2013) wordt verwezen naar paragraaf 3.3.

De wijzigingen in de saneringsaanpak van juli 2013 tot en met juni 2014 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 niet langer bruikbaar zijn van peilbuizen geselecteerd voor het monitoringsprogramma, de selectie van beter gelokaliseerde peilbuizen en aanpassing van de frequentie in functie van de concentratietrends. Een overzicht van het nieuw voorgestelde monitoringsprogramma is gegeven in Tabel 4.13.

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

8 CALAMITEITEN

8.1 PIPM INCIDENT TUSSEN GEBOUW 016 EN GEBOUW 003

In de nacht van 15 op 16 oktober 2013 heeft zich tussen gebouw 016 en gebouw 003 een lek voorgedaan in een bovengrondse leiding. Hierbij is vermoedelijk 1900 kg (ca. een duizendtal liter) van het product PIPM vrijgekomen onder vorm van een nevel, die vervolgens is neergedaald op een met steenslag verharde zone en een ingekuipte chemische riolering. Op dat moment was er een transfer bezig van het product PIPM tussen gebouw 016 en gebouw 003. Deze transfer is stopgezet en de transferlijn is doorgeblazen.

De dag nadien is in de zone waar het product was neergekomen, onder leiding van de erkend bodemsaneringsdeskundige ERM, een beperkte hoeveelheid grond (ca. 15 ton) afgegraven en zijn een zevental stalen genomen van de ontgravingsput en de afgegraven grond. In geen van de zeven stalen is een detecteerbare concentratie PIPM gemeten. Daarom is aangenomen dat er geen verder onderzoek van het grondwater noodzakelijk is en is, met toestemming van OVAM, besloten de afgegraven gravel en grond terug te storten in het ontgravingsvlak.

Uit verdere evaluatie blijkt dat er geen bodemverontreiniging tot stand is gekomen en er geen verdere maatregelen noodzakelijk zijn.

Het incident is op 24 oktober 2013 (binnen de 14 dagen na vaststelling) via het standaardformulier “Melding schadegeval bij klasse I-inrichtingen” gemeld aan OVAM en milieu-inspectie.

Meer informatie met betrekking tot het incident en de genomen maatregelen is opgenomen in het “Evaluatierapport schadegeval: PIPM Incident; Oktober 2013”; 3M Belgium bvba, Canadastraat 11, 2070 Zwijndrecht - december 2013' (Ref. R003-0188502-v2, dd. 19/12/13) dat op 17 februari 2014 digitaal aan is OVAM overgemaakt.

8.2 20% HF INCIDENT TER HOOGTE VAN GEBOUW 017

Op 11 augustus 2014 is in gebouw 17 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 fluoride concentratie 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 bijvoorbeeld 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 milieu-inspectie.

De resultaten en besluiten zullen, ter goedkeuring, in een evaluatierapport aan OVAM worden gerapporteerd.

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 perceelsgrens 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 vijfde tussentijds verslag (TTV5) van de saneringswerken, zoals beschreven in het door de OVAM conform verklaarde bodemsaneringsproject (1e fase) en de drie 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 2012 tot en met juli 2013.

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 2013 tot en met juni 2014 de volgende zaken uitgevoerd:

- Grondwatermonitoring ter hoogte van de kernzones (Gebouw 16 en WWTP), de 2e aquifer, de zuidelijke perceelsgrens, het gebied ten zuiden van de E34 expresweg (Zwijndrecht Z-peilbuizen), de zone met de ondergrondse tanks, het natuurgebied Blokkersdijk en, de monitoring van de regenwaterriolering, en van het oppervlaktewater van de Palingbeek, Tophatgracht, 3M vijver en Blokkersdijkvijver;

- Opvolging en onderhoud van het grondwatersaneringssysteem: de extractie in de negen onttrekkingsputten (PP01-02-04-05-06-07-08-09-10);

- Het uitvoeren en rapporteren van een partiële eind evaluatie voor de verontreiniging met BTEX ter hoogte van de zone met ondergrondse tanks en de FC verontreiniging ter hoogte van de Zwijndrecht Z-peilbuizen; en

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

Uit de onderzoeksverrichtingen en saneringswerken uitgevoerd in de voorbije periode (juli 2013-juli 2014) 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. Het voornaamste doel is om de horizontale en verticale verspreiding van de verontreiniging ter hoogte van de bronzones van Gebouw 16 en de WWTP tegen te gaan, wat bereikt lijkt te worden in de bronzones en ter hoogte van de zuidelijke perceelsgrens.
  Ter hoogte van de WWTP bronzone en het Blokkersdijk natuurreservaat zijn potentieel stijgende concentratietrends waargenomen. De potentiële stijgende trend ter hoogte van de WWTP bronzone moet verder geverifieerd worden door monitoring, en indien bevestigd zal geëvalueerd worden of een optimalisatie van het P&T systeem noodzakelijk is.
  De (potentiële) stijgende trend in enkele van de Blokkersdijk peilbuizen en de 3M vijver moet verder opgevolgd worden via monitoring en de reden voor de stijging moet verder onderzocht worden. Er is geen noodzaak voor bijkomende saneringsacties in deze zone aangezien er geen stijgende trend is waargenomen in de Blokkersdijk vijver.
  ERM raadt aan om de saneringsacties en regelmatige monitoring voort te zetten zoals samengevat in Tabel 4.13 (p.47).

- De gemiddelde berekende vuilvracht van PFOS naar de Schelde voor de periode 2013 – 2014 ligt in lijn met de vuilvrachten berekend in het eerste en tweede tussentijds rapport. De hogere berekende 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 2013 – juli 2014 hebben zich twee beperkte incidenten voorgedaan op de site van 3M. De analyseresultaten toonden geen impact in deze zone. Bijgevolg zijn er geen verdere acties vereist.

Het volgende interim rapport met de bespreking van de saneringswerken en monitoringsresultaten van de periode juli 2014 tot en met juli 2015 zal aan OVAM overgemaakt worden voor eind 2015.

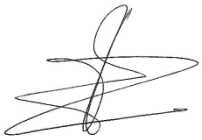
11 VERKLARING EN ONDERTEKENING

De bodemsaneringsdeskundige verklaart hierbij dat het voorliggende rapport representatief is voor de verontreinigingstoestand van de onderzoekslocatie.

Tevens verklaart de bodemsaneringsdeskundige dat de meegestuurde digitale gegevens overeenstemmen met de inhoud van het rapport.

Daarnaast verklaart de bodemsaneringsdeskundige dat alle analyses zijn uitgevoerd door een daartoe erkend laboratorium, dat de resultaten van alle uitgevoerde analyses zijn opgenomen in het rapport en dat analyseresultaten opgenomen in het rapport identiek zijn aan de analyseresultaten die zijn aangeleverd door het erkend laboratorium.

De bodemsaneringsdeskundige verklaart dat hij voor het uitvoeren van deze opdracht niet verkeert in een van de gevallen van onverenigbaarheid zoals bepaald in artikel 53/5 van het VLAREL.

Naam van de persoon die beschikt over de individuele handtekeningsbevoegdheid	**Module 2**	**Kwaliteits-verantwoordelijke**	**Handtekening**
Nanda Hermes	X		
Dirk Nuyens	X	X	
Naam van de persoon die de bodemsaneringsdeskundige rechtsgeldig kan vertegenwoordigen tegenover derden			
Dirk Nuyens			

Datum: 2 februari 2015

Bijlage 1

OVAM conformverklaring eerste gefaseerd bodemsaneringsproject

Bijlage 2

Situering onderzoekslocatie op topografische kaart

Bijlage 3

Overzichtsplan onderzoekslocatie

Bijlage 4

Detailplan ligging grondhopen

Bijlage 5

Detailplannen monsternamelocaties

Bijlage 6

Analysecertificaten

Bijlage 7

Overzicht veldmetingen en analyseresultaten

Bijlage 8

Statistische evaluatie analyseresultaten Blokkersdijk

Bijlage 9

Onttrekkingsfilters grondwateronttrekking

Bijlage 10

Schematische weergave pompput en inspectiekamer

Bijlage 11

Nota stabiliteitsdeskundige

Bijlage 12

Stijghoogtes en grondwaterisohypsen eerste en tweede aquifer

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

Situering onderzoekslocatie op topografische kaart


Onderzoekslocatie
Leopolddok
Vierde Havendok
Vijfde Havendok
Derde Havendok
Tweede Havendok
Hansadok
Industriedok
Amerikadok
Alberdok
SCHELDE
ZWIJNDRECHT
ANTWERPEN
Burcht
Antwerpen Linkeroever
Blokkersdijk, Nat. res.
Pijp Tabak
Sint-Annabos
Middenvijver
Galgenweel
Burchtse Weel
Petroleumbrug
Wilmarsdonkbrug
Oosterweelbrug
Noordkasteelbruggen
Zeevaartschool
Metropolis
Luchthal
Pomperhuis
Houtdok
Willemdok
Waaslandtunnel (voertuigen)
Kennedytunnel (voertuigen)
St-Annatunnel (voetgangers & fietsers)
Stadspark
Museum voor Schone Kunsten
Hobokense Polder
Groothandelsmarkt
Petroleum-Zuid
Kiel
Linkeroever
Het Vliet
Rot
Heidam
Neuzenberg
Slijkhoek
Oeverkant
Nieuwland
Heilige-Geesthoek
Kapellekouter
Kerkekouter
Melselepolder
Krankeloonpolder
Beemd
Schelewedi
Palingbeek
Blokkersdijk
Lauwe Hoeve
Fort van Kruibeke
Sleutelshof
Kapel Mill Domein
Hoge Akker
Schaarbeek
Heikant
Nieuwland
Krugerbrug
Wilmkse Plein
Bouwcentrum
Kliniek
Hertoghepark
Albertpark
Harmoniepark
Hof van Leysen
Sint-Antwerpen-Zuid
Kattendijkdok
Bonapartedok
Straatsburgdok
Noorderbrug
Lefebvrebrug
Siberiabrug
Brandweerkazerne
Voorm. kerk Austruweel
Noordkasteel, Dom
Verbindingsgeul
Wachtdok voor Lichters
Camping
St. Annastrand
Eurobaan
Steen, Mus.
Het Eilandje
Zuiderterminus
MUHKA
Konijnenwei
Pomp
Kattendijksluis
Kattendijkdok
Kempisch Dok
Asiadok
Oude Landen
Zwaaikom
Zwaaikom
Zwaaikom

Bijlage 3

Overzichtsplan onderzoekslocatie

SCHELDE

CANADASTRAAT

ONDERGRONDSE DIJK

TERREIN 3M

3M - PAD

3M VIJVER

3M Vijver

NATUURGEBIED BLOKKERSDIJK

BLOKKERSDIJKVIJVER

Bemalingsstation

Blokkersdijk Oost

Blokkerdijk standaard

TOPHATGRACHT

EXPRESWEG

EXPRESWEG

EXPRESWEG

PALINGBEEK

PALINGBEEK

EMC1 EMC2 EMC3 EMC4 EMC5-6.5m EMC5-12m

BR3 BR7 BR4-14m BR4-4.5m BR6-14m BR6-8.5m BR5-14m BR5-7m BR1-4m BR2-12m

BR33-4 BR33-18 BR33-17 BR33-35 BR33-33 BR33-32 BR33-29

P1 P2 P3 P4 P5 P6 P7 P8 P9 P10 P11 P12 P13 P14 P15 P16 P17 P18 P19 P20 P21 P22 P23 P24 P25 P26 P27 P28 P29 P30 P31 P32 P33 P34 P35 P36 P37 P38 P39 P40 P41 P42 P43 P44 P45 P46 P47 P48 P49 P50 P51 P52 P53 P54 P55 P56 P57 P58 P59 P60 P61 P62 P63 P64 P65 P66 P67 P68 P69 P70 P71 P72 P73 P74 P75 P76 P77 P78 P79 P80 P81 P82 P83 P84 P85 P86 P87 P88 P89 P90 P91 P92 P93 P94 P95 P96 P97 P98 P99 P100 P101 P102 P103 P104 P105 P106 P107 P108 P109 P110 P111 P112 P113 P114 P115 P116 P117A P117B P117C P118A P118B P118C P119A P119B P119C P120 P121 P205 P206 P207 P208 P209 P210 P211 P212 P213 P214 P215 P216 P217 P218 P219 P220 P221 P222 P223 P224 P225 P226 P227 P228 P229 P230 P231 P232 P233 P234 P235 P236 P237 P238 P239 P240 P241 P242 P243 P244 P245 P246 P247 P248 P249 P250 P251 P252 P253 P254 P255 P256 P257 P258 P259 P260 P261 P262 P263 P264 P265A P265B P265C P266A P266B P267 P268 P269 P270 P271 P272 P273 P274 P275 P276 P277 P278 P279 P280 P281 P282 P283 P284 P285 P286 P287 P288 P289 P290 P291 P292 P293 P294 P295 P296 P297 P298 P299 P300 P301 P302 P303 P304 P305 P306 P307 P308 P309 P310 P311 P312 P313 P314 P315 P316 P317 P318 P319 P320 P321 P322 P323 P324 P325 P326 P327 P328 P329 P330 P331 P332 P333 P334 P335 P336 P337 P338 P339 P340 P341 P342 P343 P344 P345 P346 P347 P348 P349 P350 P351 P352 P353 P354 P355 P356 P357 P358 P359 P360 P361 P362 P363 P364 P365 P366 P367 P368 P369 P370 P371 P372 P373 P374 P375 P376 P377 P378 P379 P380 P381 P382

L1 L2 L3 L4 L5 L6 L7 L8 L9 L10 L11 L12 L13 L14 L15 L16 L17 L18 L19 L20 L21 L22 L23 L24 L25 L26 L27 L28 L29 L30 L31 L32 L33

D1 D2 D3 D4 D5 D6 D7 D8 D9 D10 D11 D12 D13 D14 D15 D16 D17 D18 D19

PA101A PA101B PA102A PA102B PA103A PA103B PA104A PA104B PA105A PA105B PA106A PA106B PA107A PA107B PA108A PA108B PA109A PA109B PA110A PA110B PA111A PA111B PA112

BD24-4 BD24-3 BO-Pb12.2

Z1-3m Z1-10m Z2-3m Z2-10m Z3-3m Z3-10m Z4-8m Z5-8m Z6-8m

Legende:

- Peilbuis 2010
- Peilbuis
- Peilbuis - Buiten gebruik
- Rioolputten
- Oppervlaktewaterstaalnamepunt
- 3M Terrein
- Ondergrondse Dijk

0 20 40 60 80 100 (m)

Gefiatteerd: NVE	Gecontroleerd: NHE	Goedgekeurd: DNU	Versie: v1.0	☒ FINAL ☐ DRAFT

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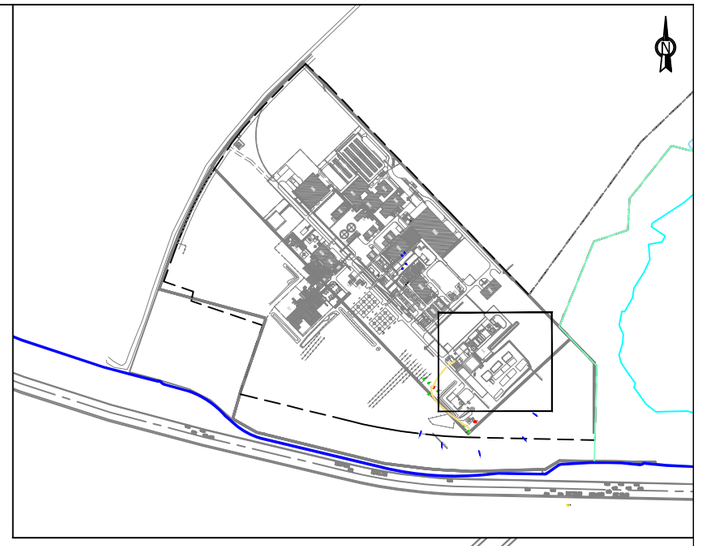
Place de l'Université 16, 1348 Louvain-la-Neuve, Tel.: 010/48.35.39, Fax.: 010/48.35.36

Klant / Projectnaam: 3M Belgium bvba	Locatie: Zwijndrecht
Projectnr.: 0243999	Titel: 5de Tussentijds verslag Bodemsaneringswerken
Figuur: 3	Beschrijving: Overzicht van de onderzoekslocatie met aanduiding van Peilbuizen
Fase: TTV-5	
Schaal: 1/3000	Formaat: A1
Bestandsnaam: 3M Zwijndrecht.dwg	Datum: 02/02/2015

Bijlage 4

Detailplan ligging grondhopen



0804
0801
0803
008
011
1
2
3
4
5
6
7
8
9
10
P384

Legende:

5 Nummer grondhoop

0 4 8 12 16 20 (m)

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
NVE	MCO	NHE	1	☐ FINAL ☒ DRAFT

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1348 Louvain-la-Neuve
Tel.: 010/48.35.39
Fax.: 010/48.35.36

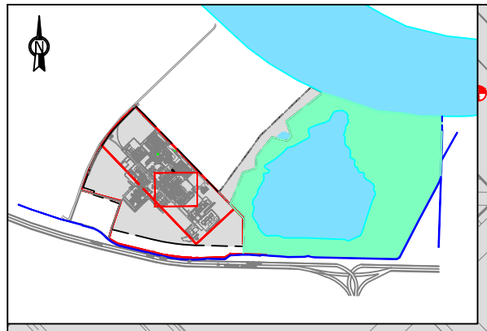
Klant / Projectnaam:	Locatie:
3M	Zwijndrecht

Projectnr.:	Titel:
0243999	5de Tussentijds Verslag Bodemsaneringswerken
4	Beschrijving: Overzicht Grondhopen
Fase: TTV-5	

Schaal:	Formaat:	Bestandsnaam:	Datum:
1/750	A3	3M Zwijndrecht.dwg	01/12/2014

Bijlage 5

Detailplannen monsternamelocaties



Legende:

- Peilbuis 2010
- Peilbuis
- Peilbuis - Buiten gebruik
- Pompput
- Regenwaterrioleringsput
- Oppervlaktewaterstaalnamepunt
- 3M Terrein
- Ondergrondse Dijk

0 4 8 12 16 20 (m)

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
NVE	NHE	DNU	v1.0	☒ FINAL ☐ DRAFT

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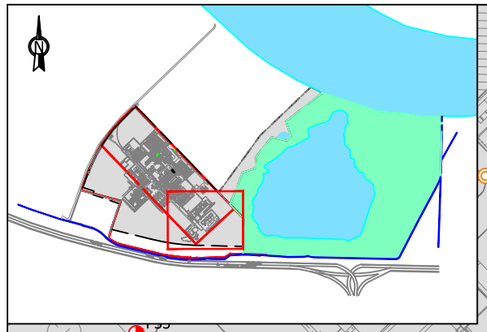
Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.:	Titel:
0243999	5de Tussentijds verslag Bodemsaneringswerken

Figuur:	Beschrijving:
5A	Overzicht monsternamelocaties
Fase: TTV-5	Bronzone Gebouw 16 en zone ondergrondse tanken

Schaal:	Formaat:	Bestandsnaam:	Datum:
1/750	A3	3M Zwijndrecht.dwg	02/02/2015



K2
P24
P304
P1064
P1065
P1062
P40
P216
P215
RW12
P225
P53
L29
P9
P54
P117A
P117B
P117C
P340
RW4
P50
L4
D10
P119A
P119B
P119C
P52
M4
P260
P11
L16
P261
P12
PP06-still
PP06
PP07
PP07-still
M1
P321
BD24-4
BD24-3
P341
Effluent WWTP
CollectorPit
M2
P11
L17
P262
L19
P266A
P266B
P361
M24
M21
M22
M23
PP02
P263
PP08
PP08-still
P264
L24
P55
PP10
D16
P51
L18
L5
P265A
P265B
P265C
P118A
P118B
P118C
P379
P343
P210
P31
P360A
P360B
P377
P376
P381
P382
P371
P370
P30
P120
L6
P373
P372
P32
D11
L28
P374
PP09
P375
P29
P380
L26

Legende:

- Peilbuis 2010
- Peilbuis
- Peilbuis - Buiten gebruik
- Pompput
- Regenwaterrioleringsput
- Oppervlaktewaterstaalnamepunt
- 3M Terrein
- Ondergrondse Dijk

0 10 20 30 40 50 (m)

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NVE	NHE	DNU	v1.0	

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Place de l'Université 16
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Tel.: 010/48.35.39
Fax.: 010/48.35.36

Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.:	Titel:
0243999	5de Tussentijds verslag Bodemsaneringswerken

Figuur:	Beschrijving:
5B	Overzicht monsternamelocaties Bronzone WWTP
Fase: TTV-5	

Schaal:	Formaat:	Bestandsnaam:	Datum:
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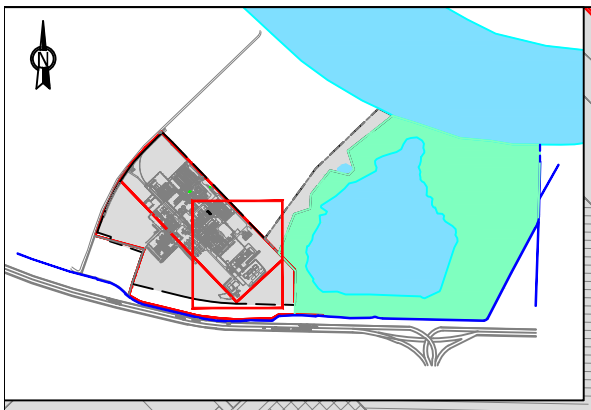

BR33-29
P46
L2
D14
P1026
P45
P218
P1025
P1022
P42
U11
U7
U8
U2
U14
U6
U5
U4
P1061
U3
U1
U9
U12
P22
P300
P219
L27
P56
P41
P39
S7
U13
D8
S6
S5
P20
K3
M14
M11
M13
PP01
M12
P28
PP05
PP05-still
S9
P18
S8
S1
P21B
S3
P21A
E1S2
D9
P5
P19
K4
PP04-still
P6
RW11
RW2
P15
P13
P26
P25
P14
P27
K2
P24
P304
P1064
P1065
P1062
P40
P216
P215
P121
L3
ND7
RW12
D17
K
P38
NP8
RW13
RW3
P225
P9
P53
L29
P33
P54
P117A
P117B
P117C
P340
RW4
P50
P119A
19B
P119C
P52
P260
M4
L16
P261
P11
P12
PP06-still
PP06
PP07
PP07-still
M1
P321
P341
Effluent WWTP
CollectorPit
M2
L17
P262
L19
P266A
P266B
P361
M24
M21
M22
P263
PP08
PP08-sti"
P264
P55
PP10
D16
L18
P379
P210
P31
P343
P118A
P118B
P118C
P360A
P360B
P377
P376
P381
P382
P371
P370
P30
P120
L6
P373
P372
P32
D11
P29
P374
PP09
P375
P380
L28
P378
B2
PA111A
PA111B
B7
B9
P121
L4
002
003
016
017
029
023
026
028
034
0801
0803
0804
008
011

Legende:

- Peilbuis 2010
- Peilbuis
- Peilbuis - Buiten gebruik
- Pompput
- Regenwaterrioleringsput
- Oppervlaktewaterstaalnamepunt
- 3M Terrein
- Ondergrondse Dijk

0 10 20 30 40 50 (m)

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
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Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.:	Titel:
0243999	5de Tussentijds verslag Bodemsaneringswerken

Figuur:	Beschrijving:
5C	Overzicht monsternamelocaties 2de Aquifer
Fase: TTV-5	

Schaal:	Formaat:	Bestandsnaam:	Datum:
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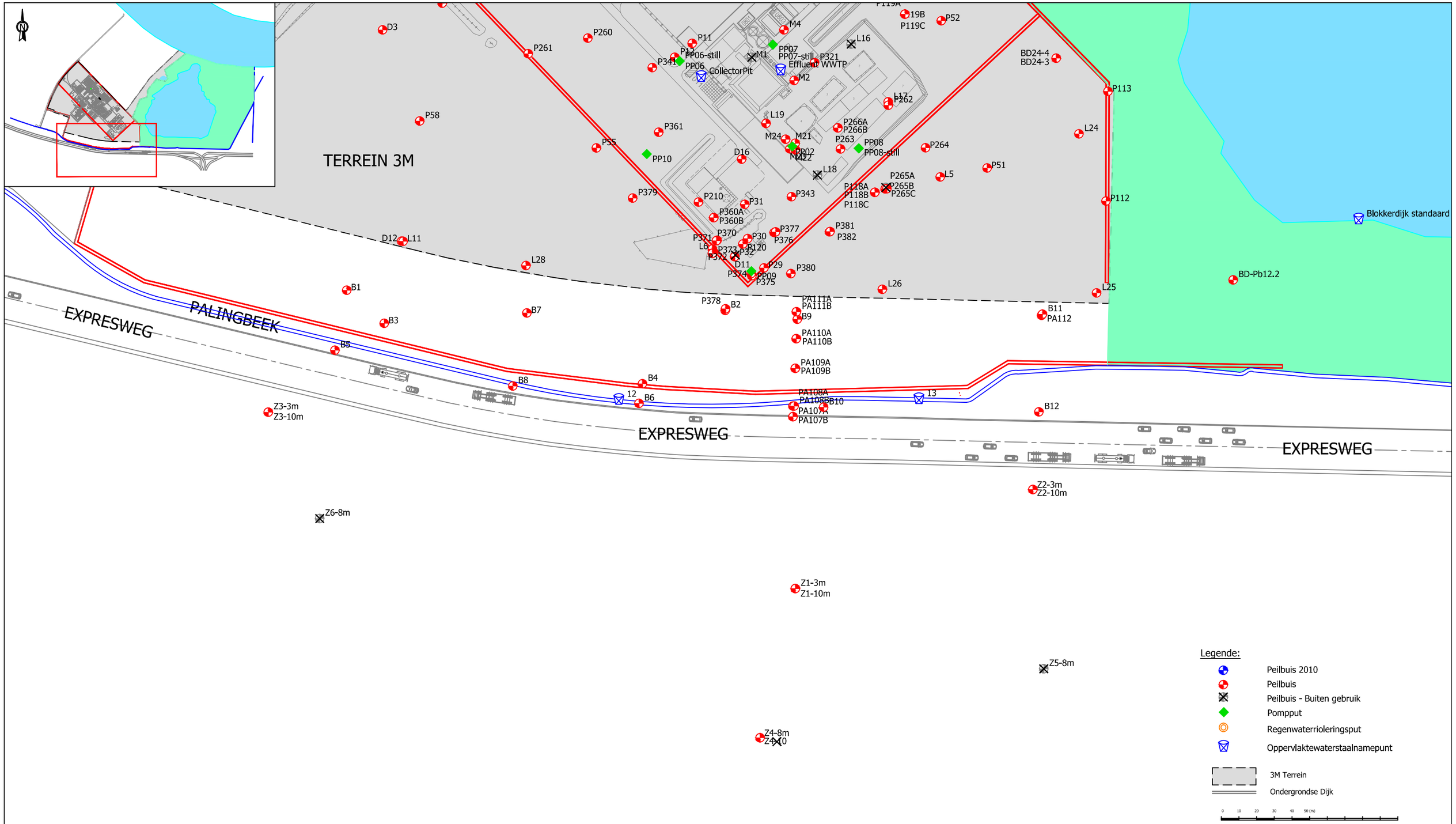

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
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ERM

Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.:	Titel:
0243999	5de Tussentijds verslag Bodemsaneringswerken

Figuur:	Beschrijving:
5D	Overzicht monsternamelocaties zuidelijke grens en Zwijndrecht wells
Fase: TTV-5	

Schaal:	Formaat:	Bestandsnaam:	Datum:
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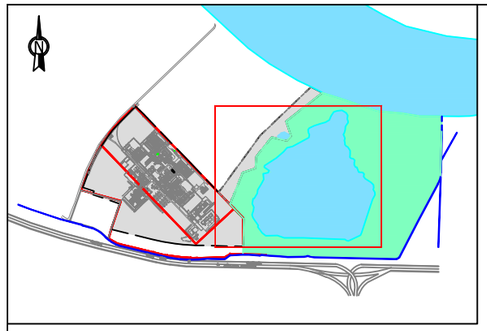

D1
L20
L32
BR4-14m
BR4-8.5m
L21
L31
3M Vijver
3M VIJVER
BR33-18
BR33-17
D2
25-3
3M - PAD
NATUURGEBIED BLOKKERSDIJK
L22
BR33-29
L30
P116
P115
BLOKKERSDIJKVIJVER
P121
L3
P53
Blokkersdijk Oost
L4
D10
P119A
P119B
P119C
P52
L16
BD24-4
BD24-3
P113
L17
P262
L24
PP08
PP08-still
P264
P51
P265A
L5
P265B
P265C
P112
Blokkerdijk standaard
BD-Pb12.2
L26
L25
B11

Legende:

- Peilbuis 2010
- Peilbuis
- Peilbuis - Buiten gebruik
- Pompput
- Regenwaterrioleringsput
- Oppervlaktewaterstaalnamepunt
- 3M Terrein
- Ondergrondse Dijk

0 20 40 60 80 100 (m)

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
NVE	NHE	DNU	v1.0	☒ FINAL ☐ DRAFT

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Fax.: 010/48.35.36

Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.:	Titel:
0243999	5de Tussentijds verslag Bodemsaneringswerken

Figuur:	Beschrijving:
5E	Overzicht monsternamelocaties Blokkersdijk
Fase: TTV-5	

Schaal:	Formaat:	Bestandsnaam:	Datum:
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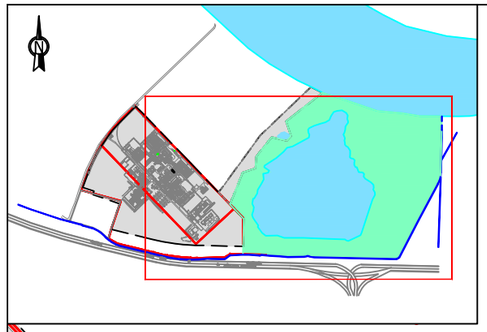

3M VIJVER

NATUURGEBIED BLOKKERSDIJK

BLOKKERSDIJKVIJVER

3M - PAD

TOPHATGRACHT

PALINGBEEK

EXPRESWEG

EXPRESWEG

3M Vijver

Blokkersdijk Oost

Blokkerdijk standaard

Bemalingsstation

Legende:

- Peilbuis 2010
- Peilbuis
- Peilbuis - Buiten gebruik
- Pompput
- Regenwaterrioleringsput
- Oppervlaktewaterstaalnamepunt
- 3M Terrein
- Ondergrondse Dijk

0 40 80 120 160 200 (m)

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
NVE	NHE	DNU	v1.0	☒ FINAL ☐ DRAFT

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Place de l'Université 16
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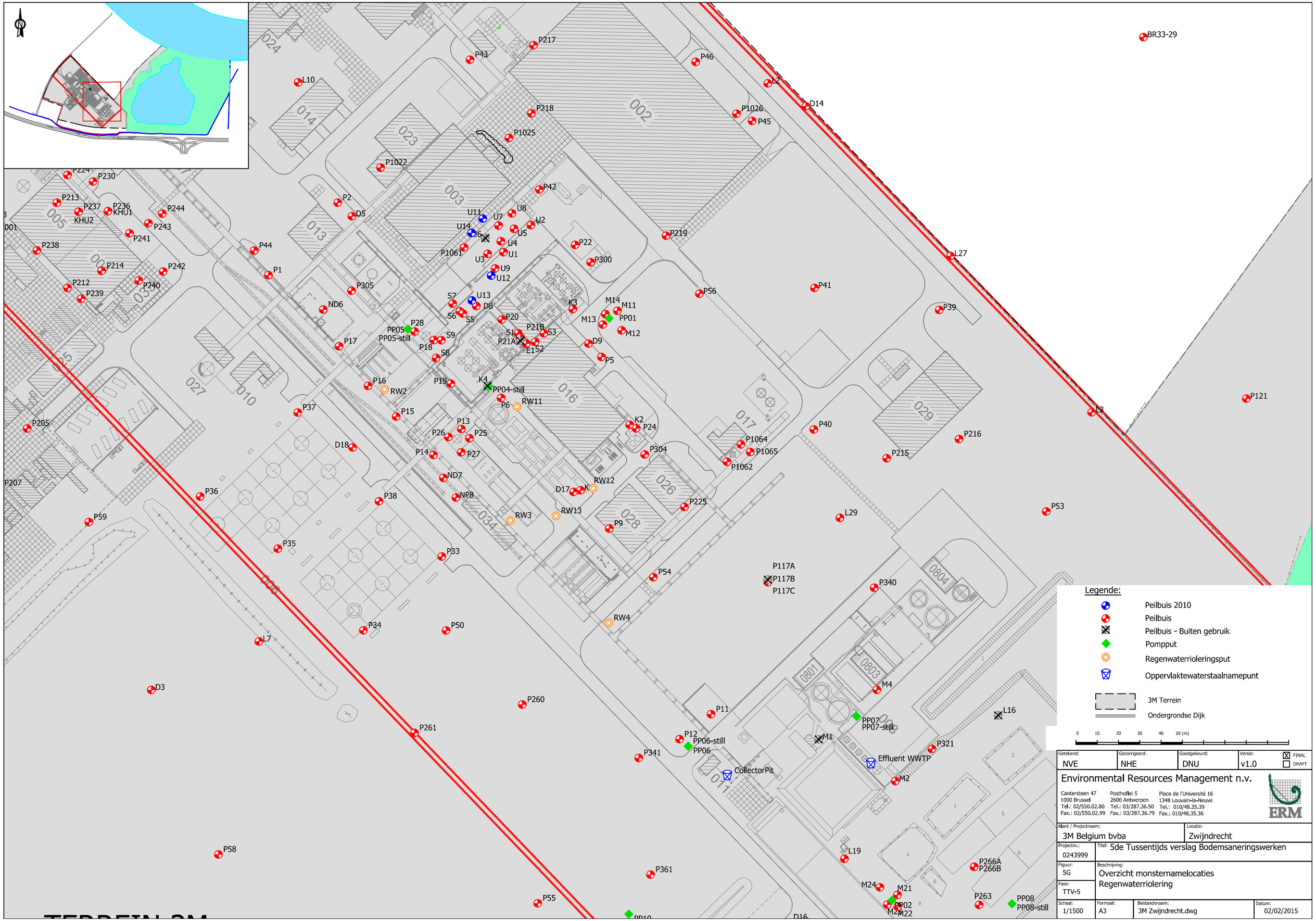
Tel.: 010/48.35.39
Fax.: 010/48.35.36

Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.:	Titel:
0243999	5de Tussentijds verslag Bodemsaneringswerken

Figuur:	Beschrijving:
5F	Overzicht monsternamelocaties Palingbeek
Fase: TTV-5	

Schaal:	Formaat:	Bestandsnaam:	Datum:
1/5000	A3	3M Zwijndrecht.dwg	02/02/2015



Legende:
Peilbuis 2010
Peilbuis
Peilbuis - Buiten gebruik
Pompput
Regenwaterrioleringsput
Oppervlaktewaterstaalnamepunt
3M Terrein
Ondergrondse Dijk
0 10 20 30 40 50 (m)
Getekend: NVE
Gecorrigeerd: NHE
Goedgekeurd: DNU
Versie: v1.0
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
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Place de l'Université 16
1348 Louvain-la-Neuve
Tel.: 010/48.35.39
Fax.: 010/48.35.36
ERM
Klant / Projectnaam: 3M Belgium bvba
Locatie: Zwijndrecht
Projectnr.: 0243999
Titel: 5de Tussentijds verslag Bodemsaneringswerken
Figuur: 5G
Beschrijving: Overzicht monsternamelocaties Regenwaterriolering
Fase: TTV-5
Schaal: 1/1500
Formaat: A3
Bestandsnaam: 3M Zwijndrecht.dwg
Datum: 02/02/2015
BR33-29
P217
P43
P46
L2
D14
P1026
P45
L10
P218
P1025
P1022
P42
P2
D5
U11
U7
U8
U2
U14
U6
U5
U4
U1
U3
U9
U12
P1061
P22
P300
P219
L27
P41
P56
P39
P224
P230
P213
P237
KHU2
P236
KHU1
P244
P243
P241
P238
P44
P214
P242
P1
P212
P240
P239
P305
ND6
S7
U13
D8
S6
S5
K3
M14
M11
M13
PP01
M12
P20
P21B
S3
S1
P21A
E1
S2
D9
P5
P28
PP05
PP05-still
S9
P18
S8
P17
P16
RW2
P19
K4
PP04-still
P6
RW11
P37
P15
P13
P26
P25
P27
P14
D18
K2
P24
P304
P1064
P1065
P1062
P40
P216
P215
L3
P121
P205
P207
P36
P38
ND7
NP8
D17
K
RW12
RW13
RW3
P225
P9
L29
P53
P59
P35
P33
P54
P117A
P117B
P117C
P340
RW4
P34
P50
L7
D3
P260
P261
P11
P12
PP06-still
PP06
P341
CollectorPit
M1
PP07
PP07-still
M4
L16
P321
Effluent WWTP
M2
P58
P361
P55
L19
P266A
P266B
M24
M21
PP02
M22
P263
PP08
PP08-still
D16

Bijlage 6

Analysecertificaten

SGS

3M Corporate Environmental Programs.
Robert A. Paschke
3M Center, 223-02-S-31
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USA

ANALYTICAL REPORT

IAC13-05052-2

3M Lab Request Number: E13-0714

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 15th, 2013

Requester

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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: E13-0714) of Robert A. Paschke, 3M Manager Corporate Environmental Programs, water samples were collected on October 14th and 15th 2013 from 5 locations (P114 not sampled) 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 sample containers under the direction of Geert De Smet. Sample containers for each sampling location included a field sample and field sample duplicate. 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 - standard (filtered)	0.492	1.30	0.669	<0.0283(b)
Blokkersdijkvijver - standard duplicate (filtered)	0.515	1.42	0.270	0.0335(b)
Blokkersdijkvijver - standard (not filtered)	0.392	1.35	<0.0756	<0.0283(a)
Blokkersdijkvijver - standard duplicate (not filtered)	0.502	1.37	0.198	<0.0283(a)
Blokkersdijkvijver - east (filtered)	0.721	1.54	8.05	0.161(c)
Blokkersdijkvijver - east duplicate (filtered)	0.799	1.67	8.69	0.162(c)
Blokkersdijkvijver - east (not filtered)	0.841	1.75	8.88	0.146(a)
Blokkersdijkvijver - east duplicate (not filtered)	0.734	1.60	7.98	0.146(a)
3M Pond	0.506	1.21	3.58	<0.0283(a)
3M Pond duplicate	0.507	1.28	3.61	<0.0283(a)
L31	0.660	1.72	10.5	0.0782(d)
L31 duplicate	0.761	0.790	12.6	0.0759(d)
P116	0.706	0.815	11.5	0.0822
P116 duplicate	0.767	1.81	10.6	0.0789

(a) The accuracy for FOSA for Blokkersdijkvijver-standard (not filtered), Blokkersdijkvijver-east (not filtered) and 3M Pond is ± 45%.
(b) The accuracy for FOSA for Blokkersdijkvijver standard (filtered) is ± 70%.
(c) The accuracy for FOSA for Blokkersdijkvijver east (filtered) is ± 65%.
(d) The accuracy for FOSA for L31 is ± 40%.

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 ($^{18}O_2$-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 - standard (filtered)	400	0.100	Solution A	0.25	-
		0.250	Solution B	-	1.0
Blokkersdijkvijver - standard duplicate (filtered)	400	0.100	Solution A	0.25	-
		0.250	Solution B	-	1.0
Blokkersdijkvijver - standard (not filtered)	400	0.100	Solution A	0.25	-
		0.250	Solution B	-	1.0
Blokkersdijkvijver - standard duplicate (not filtered)	400	0.100	Solution A	0.25	-
		0.250	Solution B	-	1.0
Blokkersdijkvijver - east (filtered) (*)	400	0.100	Solution A	0.25	-
		0.250	Solution B	-	1.0
Blokkersdijkvijver - east duplicate (filtered) (*)	400	0.100	Solution A	0.25	-
		0.250	Solution B	-	1.0
Blokkersdijkvijver - east (not filtered)(*)	400	0.100	Solution A	0.25	-
		0.250	Solution B	-	1.0
Blokkersdijkvijver - east duplicate (not filtered)(*)	400	0.100	Solution A	0.25	-
		0.250	Solution B	-	1.0
3M Pond	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
3M Pond duplicate	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
L31 duplicate	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
P114	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
P114 duplicate	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
P116	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
P116 duplicate	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 - standard (filtered)	40	8
Blokkersdijkvijver - standard duplicate (filtered)	40	8
Blokkersdijkvijver - standard (not filtered)	40	8
Blokkersdijkvijver - standard duplicate (not filtered)	40	8
Blokkersdijkvijver - east (filtered)	40	8
Blokkersdijkvijver - east duplicate (filtered)	40	8
Blokkersdijkvijver - east (not filtered)	40	8
Blokkersdijkvijver - east duplicate (not filtered)	40	8
3M Pond	40	8
3M Pond duplicate	40	8
L31	40	8
L31 duplicate	40	8
P116	40	8
P116 duplicate	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 en Blokkersdijkvijver - east.
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)	0.004
Blokkersdijkvijver - standard duplicate (filtered)	0.002
Blokkersdijkvijver - east (filtered)	0.005
Blokkersdijkvijver - east duplicate (filtered)	0.044

2.4. Analysis.

All solutions and extracts were analyzed for the PFCs (PFHS, PFOA, PFOS, FOSA) and the surrogate recovery standards ($^{18}O_2$-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
$^{18}O_2$-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 limit of quantification is 0.0272 ng/ml for PFHS, 0.0346 ng/ml for PFOA, 0.0756 ng/ml for PFOS and 0.0283 ng/ml for FOSA.

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.

Two method blanks were 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, and ± 15% RPD for precision, except for $^{13}C_7$-PFuDA (LCSL) and FOSA (LCSL) were the RPD is 33% and 29%.

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	$^{18}O_2$-PFHS	$^{13}C_7$-PFuDA	PFHS	PFOA	PFOS	FOSA
24 Oct 2013	LCSL1	0.125	115	119	98.4	87.1	93.2	101	121	91.8
	LCSL2	0.125	118	121	111	122	93.3	108	121	123
	Average		**117**	**120**	**105**	**104**	**93.3**	**105**	**121**	**107**
	%RPD		**2.7**	**1.3**	**12**	**33**	**0.1**	**7.0**	**0.2**	**29**
	LCSH1	2.5	107	98.3	100	112	88.7	97.6	115	104
	LCSH2	2.5	114	104	111	120	101	105	118	110
	Average		**110**	**101**	**105**	**116**	**94.9**	**101**	**116**	**107**
	%RPD		**5.9**	**5.3**	**9.6**	**7.1**	**13**	**7.2**	**2.3**	**4.8**

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.

3.8.1. Relative Percent Difference (RPD).

$$\text{RPD}\,(\%) = \frac{\text{ABSOLUTE concentration difference (sample - sample duplicate)}}{\text{AVERAGE sample concentration}} * 100$$

3.8.2. Recovery.

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

4. Data Summary.

Tabel 9 and 10 summarizes the results for the 13 sample locations.

Table 9. Sample Results PFHS and PFOA.

	PFHS			$^{18}O_2$-PFHS	PFOA			$^{13}C_4$-PFOA
	ng/ml	Sample average	RPD (%)	%Rec	ng/ml	Sample average	RPD (%)	%Rec
Field Trip Blank	<0.0272			104	<0.0346			116
Blokkersdijkvijver - standard (filtered)	0.492	0.503	4.5	110	1.30	1.36	8.6	106
Blokkersdijkvijver - standard duplicate (filtered)	0.515			104	1.42			116
Blokkersdijkvijver - standard (not filtered)	0.392	0.447	25(a)	96.3	1.35	1.36	1.5	108
Blokkersdijkvijver - standard duplicate (not filtered)	0.502			108	1.37			117
Blokkersdijkvijver - east (filtered)	0.721	0.760	10	96.7	1.54	1.61	8.2	112
Blokkersdijkvijver - east duplicate (filtered)	0.799			99.9	1.67			107
Blokkersdijkvijver - east (not filtered)	0.841	0.787	14	110	1.75	1.67	9.1	118
Blokkersdijkvijver - east duplicate (not filtered)	0.734			97.7	1.60			108
3M Pond	0.506	0.506	0.2	93.7	1.21	1.24	5.7	94.1
3M Pond duplicate	0.507			107	1.28			121
L31	0.660	0.711	14	89.9	1.72	1.25	74(a)	120
L31 duplicate	0.761			96.7	0.790			95.2
P116	0.706	0.737	8.3	111	0.815	1.31	76(a)	120
P116 duplicate	0.767			106	1.81			105

(a): The relative percent difference fell outside the method acceptance criterion of 15%.

Table 10. Sample Results PFOS and FOSA.

	PFOS			$^{13}C_4$-PFOS	FOSA			$^{13}C_7$-PFuDA
	ng/ml	Sample average	RPD (%)	%Rec	ng/ml	Sample average	RPD (%)	%Rec
Field Trip Blank	<0.0756			110	<0.0283			99.6
Blokkersdijkvijver - standard (filtered)	0.669	0.469	85(a)	113	<0.0283	NA	NA	42.4(b)
Blokkersdijkvijver - standard duplicate (filtered)	0.270			113	0.0335			31.6(b)
Blokkersdijkvijver - standard (not filtered)	<0.0756	NA	NA	97.1	<0.0283	NA	NA	60.3(b)
Blokkersdijkvijver - standard duplicate (not filtered)	0.198			104	<0.0283			56.9(b)
Blokkersdijkvijver - east (filtered)	8.05	8.37	7.6	112	0.161	0.162	0	40.7(b)
Blokkersdijkvijver - east duplicate (filtered)	8.69			101	0.162			34.9(b)
Blokkersdijkvijver - east (not filtered)	8.88	8.43	11	102	0.146	0.146	0	58.8(b)
Blokkersdijkvijver - east duplicate (not filtered)	7.98			97.8	0.146			63.3(b)
3M Pond	3.58	3.59	0.9	101	<0.0283	NA	NA	57.6(b)
3M Pond duplicate	3.61			113	<0.0283			62.1(b)
L31	10.5	11.6	18(a)	119	0.0782	0.0771	3.0	78.0
L31 duplicate	12.6			102	0.0759			64.7(b)
P116	11.5	11.0	8.1	108	0.0822	0.081	4.1	77.0
P116 duplicate	10.6			111	0.0789			74.5

(a): The relative percent difference fell outside the method acceptance criterion of 15%.
(b): 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-standard (not filtered), Blokkersdijkvijver-east (not filtered) and 3M Pond is ± 45%.
- The accuracy for FOSA for Blokkersdijkvijver standard (filtered) is ± 70%.
- The accuracy for FOSA for Blokkersdijkvijver east (filtered) is ± 65%.
- The accuracy for FOSA for L31 is ± 40%.

5. Conclusion.

- The relative percent difference exceeded 15% for three sample locations.
- 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.

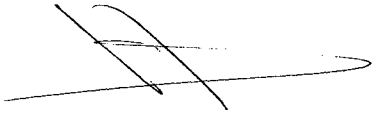
7. Addendum.

Addendum: e-mail from Ruben Maes (ERM Belgium).

8. Signatures.



Geert De Smet, Lab Operations Manager Date November 15th, 2013



Marc Van Ryckeghem, Division Manager Date November 15th, 2013

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

SGS

3M EHS
Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC13-05052-1

3M Lab Request Number: E13-0714

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

TESTING LABORATORY

SGS Belgium
Division IAC
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Author: Sandra Graré
Date issued: November 15th, 2013

Requester

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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: E13-0714) of Robert A. Paschke, 3M Manager Corporate Environmental Programs, water samples were collected on October 14th and 15th 2013 from 11 locations and analyzed for the following perfluorinated compounds:

- PFBS = perfluorobutane sulfonate ($C_4F_9SO_3^-$)
- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C-labeled analogue of PFOA ($C_6F_{13}{}^{13}CF_2{}^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C-labeled analogue of PFOS ($C_7F_{15}{}^{13}CF_2SO3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}\ SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Geert De Smet. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles.
Table 1 summarizes the sample results. All PFBS, PFHS, PFOA, PFOS and FOSA results have an accuracy of ± 30%.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
PP09	28.7	437	804	2574	<21.6
PP10	16.3	193	617	4416	<21.6
D10		102	301	20.6	<1.96
BD24-3		11.0	19.5	231	16.4
P321		1503	5626	1884	<5.89
5		7.61	13.0	27.8	<1.96
12		8.63	29.1	110	<1.96
13		9.45	30.0	106	<1.96
Pompstation		8.72	15.4	38.6	<1.96
Collector pit		13.1	48.2	183	12.8
Effluent WWTP		<1.82	<2.13	<1.13	<1.96

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)
PP09	100
PP10	100
D10	100
BD24-3	100
P321	100
5	100
12	100
13	100
Pompstation	100
Collector pit	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/037 "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
PP09	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP10	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
D10	1.0	0.100	Solution A	10.0	-
BD24-3	1.0	0.100	Solution A	10.0	-
P321	1.0	0.100	Solution A	10.0	1.0mL extract + 2.0mL MeOH (*)
5	1.0	0.100	Solution A	10.0	-
12	1.0	0.100	Solution A	10.0	-
13	1.0	0.100	Solution A	10.0	-
Pompstation	1.0	0.100	Solution A	10.0	-
Collector pit	1.0	0.100	Solution A	10.0	-
Effluent WWTP	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 0.102 to 0.192 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 1.13 to 23.5 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 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
18 Oct 2013	LCS1	80	102	104	101	94.1	109	102	99.2

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared and contains 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
27 Aug 2013	QC Lab Low inj1	10	102	104	107	101	102	98.9	104
	QC Lab High inj1	100	93.2	94.1	103	95.1	103	94.0	98.3
	QC Lab Low inj2	10	117	114	112	114	119	120	109
	QC Lab High inj2	100	106	107	111	107	124	111	102

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
PP09	28.7	437	804	2574	<21.6
PP10	16.3	193	617	4416	<21.6
D10		102	301	20.6	<1.96
BD24-3		11.0	19.5	231	16.4
P321		1503	5626	1884	<5.89
5		7.61	13.0	27.8	<1.96
12		8.63	29.1	110	<1.96
13		9.45	30.0	106	<1.96
Pompstation		8.72	15.4	38.6	<1.96
Collector pit		13.1	48.2	183	12.8
Effluent WWTP		<1.82	<2.13	<1.13	<1.96
Field Trip Blank	<1.13	<1.82	<2.13	<1.13	<1.96

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

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
PP09	126	105	125	102
PP10	126	105	130	106
D10	127	106	128	105
BD24-3	128	106	125	103
P321	114	94.3	126	103
5	127	105	132	108
12	130	108	131	107
13	134	111	126	104
Pompstation	129	107	132	108
Collector pit	133	110	126	103
Effluent WWTP	132	110	130	106
Field Trip Blank	123	102	126	103

5. Conclusion.

- The recovery of ^{13}C-PFOA and ^{13}C-PFOS fell within the method acceptance criterion of 70-130%.
- Lab control spike recoveries fell within the method acceptance criteria for all compounds, indicating that the analysis method is in control.
- QC Lab Standards recoveries fell within the method acceptance criteria for all compounds.

6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

7. Addendum.

Addendum: e-mail from Ruben Maes (ERM Belgium).

8. Signatures.

Geert De Smet, Lab Operations Manager Date November 15th, 2013

Marc Van Ryckeghem, Division Manager Date November 15th, 2013

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

Final Report

Groundwater and Surface Water Sample Analysis for PFOA, PFHS, PFOS, and PFOSA at 3M Belgium January 2014 Sampling

Laboratory Request Number: ISO14-0001

Report Date – Date of Last Signature

Testing Laboratory

3M EHS&S 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]

ACCREDITED
Testing Cert. # 2052.01

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

3M Environmental Laboratory

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

Analytical Report ISO14-0001

Groundwater and Surface Water Sample Analysis for PFOA, PFHS, PFOS, and PFOSA at 3M Belgium - January 2014 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M Environmental Laboratory prepared and analyzed groundwater and surface water samples collected by Environmental Resources Management (ERM) personnel at the 3M Belgium facility. Samples were collected on January 21-23, 2014. Samples were returned to the 3M Environmental Laboratory on January 28, 2014 at ambient temperature for the analysis of Perfluorooctanoic acid (PFOA), Perfluorohexane sulfonate (PFHS), Perfluorooctane sulfonate (PFOS), and Perfluorooctanesulfonamide (PFOSA) under laboratory project number ISO14-0001.

The 3M Environmental Laboratory prepared sample containers for thirty-six 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 and surrogate recovery standards prior to being sent to the field for sample collection. Locations B3 and P114bis were not collected during this sampling event. A field matrix spike sample was intended for locations 3M Pond and D10, however; the field matrix spike level was inadvertently excluded in Table 4 of the general project outline. As a result, the field matrix spike containers for these two locations were not fortified with the target analytes prior to being sent to the field for sample collection.

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

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

ACCREDITED
Testing Cert. # 2052.01

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

Table 1. Sample Results Summary [1]

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
ISO14-0001-001	12; Sample	65.7	18.5	208	0.378
ISO14-0001-002	12; Sample Dup	66.7	18.0	208	0.364
	Average	**66.2**	**18.3**	**208**	**0.371**
	%RPD Sample/Sample Dup	**1.5**	**2.7**	**0.0**	**3.8**
ISO14-0001-004	13; Sample	65.6	18.0	213	0.349
ISO14-0001-005	13; Sample Dup	64.0	18.2	209	0.346
	Average	**64.8**	**18.1**	**211**	**0.348**
	%RPD Sample/Sample Dup	**2.5**	**1.1**	**1.9**	**0.86**
ISO14-0001-007	3M pond; Sample	1.47	0.558	4.12	0.0252
ISO14-0001-008	3M pond; Sample Dup	1.49	0.574	4.14	<0.0250
	Average	**1.48**	**0.566**	**4.13**	**0.0252**
	%RPD Sample/Sample Dup	**1.4**	**2.8**	**0.48**	**NA**
ISO14-0001-010	5; Sample	28.1	19.1	65.1	0.130
ISO14-0001-011	5; Sample Dup	29.1	19.8	64.3	0.132
	Average	**28.6**	**19.5**	**64.7**	**0.131**
	%RPD Sample/Sample Dup	**3.5**	**3.6**	**1.2**	**1.5**
ISO14-0001-016	B7; Sample	1.91	0.839	33.6	0.0913
ISO14-0001-017	B7; Sample Dup	1.79	0.804	31.4	0.0790
	Average	**1.85**	**0.822**	**32.5**	**0.0852**
	%RPD Sample/Sample Dup	**6.5**	**4.3**	**6.8**	**14**
ISO14-0001-019	BD24-3; Sample	26.5	17.7	280	21.6
ISO14-0001-020	BD24-3; Sample Dup	29.8	19.7	295	20.6
	Average	**28.2**	**18.7**	**288**	**21.1**
	%RPD Sample/Sample Dup	**12**	**11**	**5.2**	**4.7**
ISO14-0001-022	Bemalingsstation; Sample	20.4	13.7	42.6	0.107
ISO14-0001-023	Bemalingsstation; Sample Dup	19.9	13.7	45.5	0.149
	Average	**20.2**	**13.7**	**44.1**	**0.128**
	%RPD Sample/Sample Dup	**2.5**	**0.0**	**6.6**	**33 [2]**
ISO14-0001-025	Blokkersdijkvijver std; Sample	1.27	0.663	1.09	<0.0250
ISO14-0001-026	Blokkersdijkvijver std; Sample Dup	1.26	0.668	1.03	<0.0250
	Average	**1.27**	**0.666**	**1.06**	**<0.0250**
	%RPD Sample/Sample Dup	**0.79**	**0.75**	**5.7**	**NA**
ISO14-0001-028	D10; Sample	439	148	40.8	0.0750
ISO14-0001-029	D10; Sample Dup	455	149	42.4	0.0770
	Average	**447**	**149**	**41.6**	**0.0760**
	%RPD Sample/Sample Dup	**3.6**	**0.67**	**3.8**	**2.6**

NA = Not Applicable

(1) The analytical method uncertainties associated with the reported results by internal standard calibration are as follows: PFOA ± 19%, PFHS ± 21%, PFOS ± 17%, and PFOSA ± 13%.

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

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

(4) Sample set was not bracketed by passing CCV. See section 3.4 for details.

Table 1 continued. Sample Results Summary [1]

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
ISO14-0001-031	L21; Sample	0.687	0.193	4.18	0.0333
ISO14-0001-032	L21; Sample Dup	0.668	0.184	4.14	0.0320
	Average	**0.678**	**0.189**	**4.16**	**0.0327**
	%RPD Sample/Sample Dup	**2.8**	**4.8**	**0.96**	**4.0**
ISO14-0001-034	L22; Sample	0.868	0.411	11.9	0.0384
ISO14-0001-035	L22; Sample Dup	0.890	0.391	12.2	0.0322
	Average	**0.879**	**0.401**	**12.1 [3]**	**0.0353**
	%RPD Sample/Sample Dup	**2.5**	**5.0**	**2.5**	**18**
ISO14-0001-037	L31; Sample	0.915	0.867	7.95	0.0354
ISO14-0001-038	L31; Sample Dup	0.923	0.851	8.18	0.0277
	Average	**0.919**	**0.859**	**8.07 [3]**	**0.0316**
	%RPD Sample/Sample Dup	**0.87**	**1.9**	**2.9**	**24 [2]**
ISO14-0001-040	L4; Sample	2.92	2.29	22.0	6.24
ISO14-0001-041	L4; Sample Dup	3.00	2.26	21.8	6.25
	Average	**2.96**	**2.28**	**21.9 [4]**	**6.25**
	%RPD Sample/Sample Dup	**2.7**	**1.3**	**0.91**	**0.16**
ISO14-0001-046	P115; Sample	4.49	2.14	0.518	<0.0250
ISO14-0001-047	P115; Sample Dup	4.51	2.06	0.517	<0.0250
	Average	**4.50**	**2.10**	**0.518 [4]**	**<0.0250**
	%RPD Sample/Sample Dup	**0.44**	**3.8**	**0.19**	**NA**
ISO14-0001-049	P116; Sample	2.26	0.854	9.18	0.0685
ISO14-0001-050	P116; Sample Dup	2.20	0.810	9.16	0.0684
	Average	**2.23**	**0.832**	**9.17 [4]**	**0.0685**
	%RPD Sample/Sample Dup	**2.7**	**5.3**	**0.22**	**0.15**
ISO14-0001-052	P118C; Sample	818	548	2240	57.7
ISO14-0001-053	P118C; Sample Dup	873	569	2430	56.5
	Average	**846**	**559**	**2340 [4]**	**57.1**
	%RPD Sample/Sample Dup	**6.5**	**3.8**	**8.1**	**2.1**
ISO14-0001-055	P119C; Sample	217	113	1740	60.9
ISO14-0001-056	P119C; Sample Dup	224	117	1710	61.9
	Average	**221**	**115**	**1730 [4]**	**61.4**
	%RPD Sample/Sample Dup	**3.2**	**3.5**	**1.7**	**1.6**
ISO14-0001-058	P264; Sample	392	260	1910	52.2
ISO14-0001-059	P264; Sample Dup	387	258	1920	51.0
	Average	**390**	**259**	**1920 [4]**	**51.6**
	%RPD Sample/Sample Dup	**1.3**	**0.77**	**0.52**	**2.3**

NA = Not Applicable

(1) The analytical method uncertainties associated with the reported results by internal standard calibration are as follows: PFOA ± 19%, PFHS ± 21%, PFOS ± 17%, and PFOSA ± 13%.

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

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

(4) Sample set was not bracketed by passing CCV. See section 3.4 for details.

Table 1 continued. Sample Results Summary [1]

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
ISO14-0001-061	P265C; Sample	109	34.0	1140	111
ISO14-0001-062	P265C; Sample Dup	132	54.1	1200	108
	Average	**121**	**44.1**	**1170**	**110**
	%RPD Sample/Sample Dup	**19**	**46 [2]**	**5.1**	**2.7**
ISO14-0001-064	P321; Sample	6260	1330	2520	1.13
ISO14-0001-065	P321; Sample Dup	6350	1400	2490	6.18
	Average	**6310**	**1370**	**2510**	**3.66**
	%RPD Sample/Sample Dup	**1.4**	**5.1**	**1.2**	**138 [2]**
ISO14-0001-067	P372; Sample	98.2	63.7	1790	50.4
ISO14-0001-068	P372; Sample Dup	102	65.4	1760	50.7
	Average	**100**	**64.6**	**1780**	**50.6**
	%RPD Sample/Sample Dup	**3.8**	**2.6**	**1.7**	**0.59**
ISO14-0001-070	P374; Sample	783	678	2580	33.1
ISO14-0001-071	P374; Sample Dup	773	657	2950	32.5
	Average	**778**	**668**	**2770**	**32.8**
	%RPD Sample/Sample Dup	**1.3**	**3.1**	**13**	**1.8**
ISO14-0001-073	P378; Sample	351	347	1180	1.79
ISO14-0001-074	P378; Sample Dup	333	345	1200	1.78
	Average	**342**	**346**	**1190**	**1.79**
	%RPD Sample/Sample Dup	**5.3**	**0.58**	**1.7**	**0.56**
ISO14-0001-076	P381; Sample	667	191	1710	20.7
ISO14-0001-077	P381; Sample Dup	645	176	1590	13.7
	Average	**656**	**184**	**1650**	**17.2**
	%RPD Sample/Sample Dup	**3.4**	**8.2**	**7.3**	**41 [2]**
ISO14-0001-079	P382; Sample	596	168	981	80.5
ISO14-0001-080	P382; Sample Dup	649	181	972	78.7
	Average	**623**	**175**	**977**	**79.6**
	%RPD Sample/Sample Dup	**8.5**	**7.4**	**0.92**	**2.3**
ISO14-0001-082	PA109A; Sample	302	299	496	1.21
ISO14-0001-083	PA109A; Sample Dup	310	309	536	1.12
	Average	**306**	**304**	**516**	**1.17**
	%RPD Sample/Sample Dup	**2.6**	**3.3**	**7.8**	**7.7**
ISO14-0001-085	PA111A; Sample	823	1060	2200	12.3
ISO14-0001-086	PA111A; Sample Dup	824	1070	2390	11.9
	Average	**824**	**1070**	**2300**	**12.1**
	%RPD Sample/Sample Dup	**0.12**	**0.93**	**8.3**	**3.3**

NA = Not Applicable

(1) The analytical method uncertainties associated with the reported results by internal standard calibration are as follows: PFOA ± 19%, PFHS ± 21%, PFOS ± 17%, and PFOSA ± 13%.

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

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

(4) Sample set was not bracketed by passing CCV. See section 3.4 for details.

Table 1 continued. Sample Results Summary [1]

		Concentration (ng/mL)			
3M LIMS ID	**Sample Description**	**PFOA**	**PFHS**	**PFOS**	**PFOSA**
ISO14-0001-088	PA112; Sample	154	31.7	341	48.3
ISO14-0001-089	PA112; Sample Dup	156	32.7	331	48.4
	Average	**155**	**32.2**	**336**	**48.4**
	%RPD Sample/Sample Dup	**1.3**	**3.1**	**3.0**	**0.21**
ISO14-0001-091	Z1-3m; Sample	0.877	0.741	0.395	0.0335
ISO14-0001-092	Z1-3m; Sample Dup	0.878	0.725	0.523	<0.0250
	Average	**0.878**	**0.733**	**0.459**	**0.0335**
	%RPD Sample/Sample Dup	**0.11**	**2.2**	**28** [2]	**NA**
ISO14-0001-094	Z2-10m; Sample	1.28	1.36	1.47	0.0475
ISO14-0001-095	Z2-10m; Sample Dup	1.31	1.38	1.45	0.0467
	Average	**1.30**	**1.37**	**1.46**	**0.0471**
	%RPD Sample/Sample Dup	**2.3**	**1.5**	**1.4**	**1.7**
ISO14-0001-097	Z2-3m; Sample	1.38	1.17	0.458	<0.0250
ISO14-0001-098	Z2-3m; Sample Dup	1.38	1.20	0.454	<0.0250
	Average	**1.38**	**1.19**	**0.456**	**<0.0250**
	%RPD Sample/Sample Dup	**0.0**	**2.5**	**0.88**	**NA**
ISO14-0001-100	P118A; Sample	2610	5030	1930	11.5
ISO14-0001-101	P118A; Sample Dup	2590	5300	1840	8.04
	Average	**2600**	**5170**	**1890**	**9.77**
	%RPD Sample/Sample Dup	**0.77**	**5.2**	**4.8**	**35** [2]
ISO14-0001-103	P121; Sample	0.284	0.153	5.21	0.337
ISO14-0001-104	P121; Sample Dup	0.255	0.153	4.69	0.283
	Average	**0.270**	**0.153**	**4.95**	**0.310**
	%RPD Sample/Sample Dup	**11**	**0.0**	**11**	**17**
ISO14-0001-106	P371; Sample	81.2	31.9	929	47.2
ISO14-0001-107	P371; Sample Dup	76.4	30.6	789	47.4
	Average	**78.8**	**31.3**	**859**	**47.3**
	%RPD Sample/Sample Dup	**6.1**	**4.2**	**16**	**0.42**

NA = Not Applicable

(1) The analytical method uncertainties associated with the reported results by internal standard calibration are as follows: PFOA ± 19%, PFHS ± 21%, PFOS ± 17%, and PFOSA ± 13%.

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

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

(4) Sample set was not bracketed by passing CCV. See section 3.4 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".

Table 2. Target Analytes

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

2.2 Sample Collection

Samples were collected on January 21-23, 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. The field matrix spike samples for sampling locations 3M Pond and D10 were not spiked during bottle preparation. All sample locations were spiked with a mixture of mass-labeled internal standards at a nominal concentration of 1 ng/mL and 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 January 28, 2014. Locations B3 and P114bis were not collected during this sampling event.

2.3 Sample Preparation

PFOA, PFHS, and PFOS Analysis: Samples not requiring a dilution were prepared by removing an aliquot of the well mixed sample and transferring it into an autovial. 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. Diluted samples were also fortified with internal standards and surrogate recovery standards at a nominal concentration of 1 ng/mL.

PFOSA Analysis: All 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).

Re-Analysis of Sample Location P121 for PFOA, PFHS, and PFOS: Sample set was 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.

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. 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 generate the reported results:

2/4/14; Direct Inject/Water Dilution Analysis – reported results for PFOA, PFHS, and PFOS.

2/6/14; Methanol Dilution Analysis – reported results for PFOSA.

2/10/14; Methanol Dilution Analysis – reported results for PFOA, PFHS, and PFOS for location P121, confirmed PFOSA results for location P321.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk	ETS DaVinci
Liquid Chromatograph	Agilent 1260	Agilent 1290
Analysis Method	ETS-8-044.1	ETS-8-044.1
Analysis Date	2/4/14, 2/10/14	2/6/14
Guard column	Betasil C18 (4.6 mm X 100 mm), 5 µ	Betasil C18 (2.1 mm X 100 mm), 5 µ
Analytical column	Betasil C18 (4.6 mm X 100 mm), 5 µ	Betasil C18 (2.1 mm X 100 mm), 5 µ
Injection Volume	5 µL	5 µL
Mass Spectrometer	AB Sciex Triple Quad 5500	AB Sciex Triple Quad 6500
Ion Source	Turbo Spray	Turbo Spray
Polarity	Negative	Negative
Software	Analyst 1.6.1	Analyst 1.6.1

Table 4. Liquid Chromatography Gradient Program.

ETS-8-044.1 Analysis (2/4/14 and 2/10/14)				
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-8-044.1 Analysis (2/6/14)				
Step Number	**Total Time (min)**	**Flow Rate (µL/min)**	**Percent A (2 mM ammonium acetate)**	**Percent B (Methanol)**
0	0.00	300	90.0	10.0
1	0.50	300	90.0	10.0
2	4.00	300	70.0	30.0
3	6.00	300	70.0	30.0
4	11.0	300	20.0	80.0
5	13.0	300	20.0	80.0
6	13.5	300	10.0	90.0
7	16.0	300	10.0	90.0
8	16.5	300	90.0	10.0
9	19.0	300	90.0	10.0

Table 5. Mass Transitions

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

3 Data Analysis

3.1 Calibration

2/4/14 Analysis - Direct Inject/Water Dilution: 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 laboratory prepared reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 1 ng/mL. A total of sixteen calibration standards ranging from 0.025 ng/mL to 100 ng/mL (nominal) were analyzed with the prepared samples. Of these sixteen calibration standards, eleven contained the surrogates at concentrations ranging from 0.025 ng/mL to 10 ng/mL nominal. A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve point.

2/6/14 and 2/10/14 Analysis - Methanol Dilution: 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 to 100 ng/mL (nominal) were analyzed (0.0125 ng/mL to 10 ng/mL (nominal) for the SRSs). 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 each calibration curve, the reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers. Each curve point was quantitated using the overall calibration curve and reviewed for accuracy. Method calibration accuracy requirements of 100±25% (100±30% for the lowest curve point) were met for all analytes. The correlation coefficient (r) was greater than 0.995 for all analytes.

3.2 System Suitability

A calibration standard was analyzed four times at the beginning of the analytical sequence to demonstrate overall system suitability. The acceptance criteria for system suitability samples of less than or equal to 5% relative standard deviation (RSD) for peak area ratio and retention time criteria of less than or equal to 2% RSD were met for all analytes.

3.3 Limit of Quantitation (LOQ)

The LOQ as defined in method ETS-8-044.1 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] 2/4/14 Analysis	LOQ, ng/mL [2] 2/6/14 Analysis	LOQ, ng/mL [2] 2/10/14 Analysis
PFOA	0.0240	NA	0.0240
PFHS	0.0250	NA	0.0250
PFOS	0.232	NA	0.0232
PFOSA	NA	0.0250	0.0250

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

3.4 Continuing Calibration

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

2/4/14 Analysis: Thirteen CCVs were analyzed with the reported samples. One CCV for PFOS had a recovery of 127%. The following samples were bracketed by this CCV: L4, P115, P116, P118C, P119C, and P264.

A method deviation is included with the raw data.

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 exception of LCSs that were spiked outside the resulting calibration range. These included the low level LCSs for PFOS analyzed on 2/4/14 and the high level LCSs for PFOSA analyzed on 2/10/14. 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 Analyzed 2/4/14	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-140203-1	0.190	0.183	96.4	0.198	0.200	101
LCS-140203-2	0.190	0.181	95.2	0.198	0.191	96.5
LCS-140203-3	0.190	0.184	96.6	0.198	0.194	97.8
Average ± %RSD	**96.1% ± 0.79%**			**98.4% ± 2.4%**		
LCS-140203-4	9.50	10.1	107	9.92	10.9	110
LCS-140203-5	9.50	10.2	108	9.92	10.7	108
LCS-140203-6	9.50	10.0	105	9.92	10.8	109
Average ± %RSD	**107% ± 1.4%**			**109% ± 0.92%**		
LCS-140203-7	75.7	79.1	104	79.0	80.8	102
LCS-140203-8	75.7	80.2	106	79.0	82.0	104
LCS-140203-9	75.7	79.1	105	79.0	84.0	106
Average ± %RSD	**105% ± 0.95%**			**104% ± 1.9%**		

ETS-8-044.1 Analyzed 2/4/14	PFOS (Linear + Branched)		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-140203-1	0.184	<0.232	NA
LCS-140203-2	0.184	<0.232	NA
LCS-140203-3	0.184	<0.232	NA
Average ± %RSD	**NA** (1)		
LCS-140203-4	9.20	9.78	106
LCS-140203-5	9.20	9.94	108
LCS-140203-6	9.20	9.62	105
Average ± %RSD	**106% ± 1.4%**		
LCS-140203-7	73.2	77.2	106
LCS-140203-8	73.2	75.9	104
LCS-140203-9	73.2	74.8	102
Average ± %RSD	**104% ± 1.9%**		

NA = Not Applicable
(1) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Analyzed 2/4/14	$^{13}C_4$-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-140203-1	0.198	0.227	115	0.189	0.207	110
LCS-140203-2	0.198	0.221	111	0.189	0.210	111
LCS-140203-3	0.198	0.224	113	0.189	0.213	113
Average ± %RSD	**113% ±1.8%**			**111% ± 1.4%**		
LCS-140203-4	1.98	2.16	109	1.90	2.04	107
LCS-140203-5	1.98	2.21	112	1.90	2.07	109
LCS-140203-6	1.98	2.20	111	1.90	2.01	106
Average ± %RSD	**111% ± 1.4%**			**107% ± 1.4%**		

ETS-8-044.1 Analyzed 2/6/14	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-140205-1	0.198	0.227	114
LCS-140205-2	0.198	0.218	110
LCS-140205-3	0.198	0.210	106
Average ± %RSD	**110% ± 3.6%**		
LCS-140205-4	9.92	10.5	106
LCS-140205-5	9.92	10.9	110
LCS-140205-6	9.92	10.7	108
Average ± %RSD	**108% ± 1.9%**		
LCS-140205-7	79.0	84.3	107
LCS-140205-8	79.0	86.2	109
LCS-140205-9	79.0	84.3	107
Average ± %RSD	**108% ± 1.1%**		

NA = Not Applicable
(1) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Analyzed 2/10/14	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-140205-1	0.190	0.174	91.5	0.198	0.197	99.4
LCS-140205-2	0.190	0.186	98.2	0.198	0.211	107
LCS-140205-3	0.190	0.152	79.9	0.198	0.188	94.9
Average ± %RSD	**89.9% ± 10%**			**100% ± 6.1%**		
LCS-140205-4	9.50	10.2	108	9.92	10.9	110
LCS-140205-5	9.50	10.7	113	9.92	11.0	110
LCS-140205-6	9.50	11.0	116	9.92	11.7	118
Average ± %RSD	**112% ± 3.6%**			**113% ± 4.1%**		
LCS-140205-7	75.7	77.2	102	79.0	79.6	101
LCS-140205-8	75.7	80.1	106	79.0	79.4	100
LCS-140205-9	75.7	82.8	109	79.0	79.7	101
Average ± %RSD	**106% ± 3.3%**			**101% ± 0.57%**		

ETS-8-044.1 Analyzed 2/10/14	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-140205-1	0.184	0.194	105	0.198	0.207	104
LCS-140205-2	0.184	0.199	108	0.198	0.217	110
LCS-140205-3	0.184	0.183	99.3	0.198	0.198	100
Average ± %RSD	**104% ± 4.2%**			**105% ± 4.8%**		
LCS-140205-4	9.20	10.1	110	9.92	10.9	110
LCS-140205-5	9.20	10.0	109	9.92	11.3	114
LCS-140205-6	9.20	10.9	119	9.92	11.9	120
Average ± %RSD	**113% ± 4.9%**			**115% ± 4.4%**		
LCS-140205-7	73.2	75.1	103	79.0	>50.0	NA
LCS-140205-8	73.2	77.6	106	79.0	>50.0	NA
LCS-140205-9	73.2	78.8	108	79.0	>50.0	NA
Average ± %RSD	**106% ± 2.4%**			**NA (1)**		

NA = Not Applicable
(1) LCSs spiked outside the calibration range.

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Analyzed 2/10/14	$^{13}C_4$-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-140205-1	0.198	0.221	112	0.189	0.204	108
LCS-140205-2	0.198	0.213	108	0.189	0.212	112
LCS-140205-3	0.198	0.223	113	0.189	0.204	108
Average ± %RSD	**111% ± 2.4%**			**109% ± 2.1%**		
LCS-140205-4	1.98	2.14	108	1.90	1.98	104
LCS-140205-5	1.98	2.29	116	1.90	2.07	109
LCS-140205-6	1.98	2.21	112	1.90	2.07	109
Average ± %RSD	**112% ± 3.6%**			**107% ± 2.7%**		

NA = Not Applicable
(1) LCSs spiked outside the calibration range.

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	Standard Deviation (%)	Method Uncertainty
PFOA	9.33	±19%
PFHS	10.6	±21%
PFOS	8.51	±17%
PFOSA	6.72	±13%

3.8 Field Matrix Spikes (FMS)

A target analyte field matrix spike sample(s) was collected at each sampling point, except for sampling locations 3M Pond and D10, 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 bottle 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 all 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 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	PFHS	PFOS	PFOSA
Z1-3m, Z2-10m, and Z2-3m	FMS	1.98	2.00	2.00	2.00
5,B3, B7, Blokkersdijkvijver – standard, L21, L22, L31, L4, P114bis, P115, P116, and P121	FMS	9.92	10.0	9.98	10.0
12, 13, and Bemalingsstation	FMS	101	98.2	99.8	10.0
BD24-3, PA109A, and PA112	FMS	507	491	499	50.1
P118A, P118C, P119C, P264, P265C, P321, P371, P372, P374, P378, P381, P382, and PA111A	FMS	1010	982	998	100
Trip Blank	Low	1.98	2.00	2.00	2.00
	Mid	101	98.2	99.8	10.0
	High	1010	982	998	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.

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. The following sampling locations had field QC samples outside method acceptance criteria.

Bemalingsstation: Sample/sample duplicate RPD was 33% for PFOSA.

L22: The FMS recovery for PFOS was 136%. The method uncertainty has been expanded for PFOS to ± 36%, based on the recovery of the FMS sample.

L31: The FMS recovery for PFOS was 131%. The method uncertainty has been expanded for PFOS to ± 31%, based on the recovery of the FMS sample. Sample/sample duplicate RPD was 24% for PFOSA.

P265C: Sample/sample duplicate RPD was 46% for PFHS. The FMS recovery is an estimated value for PFOSA due to the FMS sample concentration (218 ng/mL) being just above the upper limit of quantitation (ULOQ - 200 ng/mL).

P321: Sample/sample duplicate RPD was 138% for PFOSA. The sample set was re-prepared and re-analyzed on 2/10/14 for PFOSA to verify the initial sample results. The re-analysis confirmed the high RPD value; therefore, the initial sample results are reported.

P381: Sample/sample duplicate RPD was 41% for PFOSA.

Z1-3m: Sample/sample duplicate RPD was 28% for PFOS.

P118A: Sample/sample duplicate RPD was 35% for PFOSA.

Table 10. Location ID: 12

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-001	12; Sample	65.7	NA	18.5	NA	208	NA
ISO14-0001-002	12; Sample Dup	66.7	NA	18.0	NA	208	NA
ISO14-0001-003	12; FMS	169	102	111	94.4	312	104
Average Concentration (ng/mL) ± %RPD		**66.2 ng/mL ± 1.5%**		**18.3 ng/mL ± 2.7%**		**208 ng/mL ± 0.0%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-001	12; Sample	0.378	NA	111	110
ISO14-0001-002	12; Sample Dup	0.364	NA	106	102
ISO14-0001-003	12; FMS	11.7	113	108	105
Average Concentration (ng/mL) ± %RPD/RSD		**0.371 ng/mL ± 3.8%**		**108% ± 2.3%**	**106% ± 3.8%**

NA = Not Applicable

Table 11. Location ID: 13

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-004	13; Sample	65.6	NA	18.0	NA	213	NA
ISO14-0001-005	13; Sample Dup	64.0	NA	18.2	NA	209	NA
ISO14-0001-006	13; FMS	167	101	113	96.6	315	104
Average Concentration (ng/mL) ± %RPD		**64.8 ng/mL ± 2.5%**		**18.1 ng/mL ± 1.1%**		**211 ng/mL ± 1.9%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-004	13; Sample	0.349	NA	110	108
ISO14-0001-005	13; Sample Dup	0.346	NA	107	107
ISO14-0001-006	13; FMS	11.3	110	122	118
Average Concentration (ng/mL) ± %RPD/RSD		**0.348 ng/mL ± 0.86%**		**113% ± 7.0%**	**111% ± 5.5%**

NA = Not Applicable

Table 12. Location ID: 3M Pond

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-007	3M pond; Sample	1.47	NA	0.558	NA	4.12	NA
ISO14-0001-008	3M pond; Sample Dup	1.49	NA	0.574	NA	4.14	NA
ISO14-0001-009	3M pond; FMS	1.48	NA (1)	0.556	NA (1)	3.96	NA (1)
Average Concentration (ng/mL) ± %RPD		**1.48 ng/mL ± 1.4%**		**0.566 ng/mL ± 7.6%**		**4.13 ng/mL ± 0.48%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-007	3M pond; Sample	0.0252	NA	105	108
ISO14-0001-008	3M pond; Sample Dup	<0.0250	NA	109	108
ISO14-0001-009	3M pond; FMS	NA	NA (2)	111	104
Average Concentration (ng/mL) ± %RPD/RSD		**0.0252 ng/mL**		**108% ± 2.8%**	**107% ± 2.2%**

NA = Not Applicable
(1) Sample container was not fortified with the target analytes prior to being sent to the field for sample collection.
(2) Sample was not included in the analysis of PFOSA on 2/6/14.

Table 13. Location ID: 5

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-010	5; Sample	28.1	NA	19.1	NA	65.1	NA
ISO14-0001-011	5; Sample Dup	29.1	NA	19.8	NA	64.3	NA
ISO14-0001-012	5; FMS	41.3	NC	31.3	118	82.9	NC
Average Concentration (ng/mL) ± %RPD		**28.6 ng/mL ± 3.5%**		**19.5 ng/mL ± 3.6%**		**64.7 ng/mL ± 1.2%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-010	5; Sample	0.130	NA	101	102
ISO14-0001-011	5; Sample Dup	0.132	NA	104	97.4
ISO14-0001-012	5; FMS	11.2	111	98.8	108
Average Concentration (ng/mL) ± %RPD/RSD		**0.131 ng/mL ± 1.5%**		**101% ± 2.6%**	**102% ± 5.2%**

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

Table 14. Location ID: B7

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-016	B7; Sample	1.91	NA	0.839	NA	33.6	NA
ISO14-0001-017	B7; Sample Dup	1.79	NA	0.804	NA	31.4	NA
ISO14-0001-018	B7; FMS	13.1	113	12.0	112	46.4	NC
Average Concentration (ng/mL) ± %RPD		**1.85 ng/mL ± 6.5%**		**0.822 ng/mL ± 4.3%**		**32.5 ng/mL ± 3.8%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-016	B7; Sample	0.0913	NA	107	102
ISO14-0001-017	B7; Sample Dup	0.0790	NA	107	96.9
ISO14-0001-018	B7; FMS	10.9	108	108	101
Average Concentration (ng/mL) ± %RPD/RSD		**0.0852 ng/mL ± 14%**		**107% ± 0.54%**	**100% ± 2.7%**

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

Table 15. Location ID: BD24-3

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-019	BD24-3; Sample	26.5	NA	17.7	NA	280	NA
ISO14-0001-020	BD24-3; Sample Dup	29.8	NA	19.7	NA	295	NA
ISO14-0001-021	BD24-3; FMS	510	95.0	474	92.7	736	89.8
Average Concentration (ng/mL) ± %RPD		**28.2 ng/mL ± 12%**		**18.7 ng/mL ± 11%**		**288 ng/mL ± 5.2%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-019	BD24-3; Sample	21.6	NA	115	112
ISO14-0001-020	BD24-3; Sample Dup	20.6	NA	102	101
ISO14-0001-021	BD24-3; FMS	78.3	114	108	109
Average Concentration (ng/mL) ± %RPD/RSD		**21.1 ng/mL ± 4.7%**		**108% ± 6.0%**	**107% ± 5.3%**

NA = Not Applicable

Table 16. Location ID: Bemalingsstation

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-022	Bemalingsstation; Sample	20.4	NA	13.7	NA	42.6	NA
ISO14-0001-023	Bemalingsstation; Sample Dup	19.9	NA	13.7	NA	45.5	NA
ISO14-0001-024	Bemalingsstation; FMS	125	104	111	99.1	146	102
Average Concentration (ng/mL) ± %RPD		**20.2 ng/mL ± 2.5%**		**13.7 ng/mL ± 0.0%**		**44.1 ng/mL ± 6.6%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-022	Bemalingsstation; Sample	0.107	NA	100	97.2
ISO14-0001-023	Bemalingsstation; Sample Dup	0.149	NA	108	104
ISO14-0001-024	Bemalingsstation; FMS	11.1	110	110	105
Average Concentration (ng/mL) ± %RPD/RSD		**0.128 ng/mL ± 33%** (1)		**106% ± 5.0%**	**102% ± 4.2%**

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

Table 17. Location ID: Blokkersdijkvijver std

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-025	Blokkersdijkvijver std; Sample	1.27	NA	0.663	NA	1.09	NA
ISO14-0001-026	Blokkersdijkvijver std; Sample Dup	1.26	NA	0.668	NA	1.03	NA
ISO14-0001-027	Blokkersdijkvijver std; FMS	12.5	113	11.9	112	12.7	117
Average Concentration (ng/mL) ± %RPD		**1.27 ng/mL ± 0.79%**		**0.666 ng/mL ± 0.75%**		**1.06 ng/mL ± 5.7%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-025	Blokkersdijkvijver std; Sample	<0.0250	NA	107	112
ISO14-0001-026	Blokkersdijkvijver std; Sample Dup	<0.0250	NA	106	101
ISO14-0001-027	Blokkersdijkvijver std; FMS	11.0	110	111	105
Average Concentration (ng/mL) ± %RPD/RSD		**<0.0250 ng/mL**		**108% ± 2.4%**	**106% ± 5.3%**

NA = Not Applicable

Table 18. Location ID: D10

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-028	D10; Sample	439	NA	148	NA	40.8	NA
ISO14-0001-029	D10; Sample Dup	455	NA	149	NA	42.4	NA
ISO14-0001-030	D10; FMS	442	NA (1)	151	NA (1)	38.9	NA (1)
Average Concentration (ng/mL) ± %RPD		**1.27 ng/mL ± 0.79%**		**0.666 ng/mL ± 0.75%**		**1.06 ng/mL ± 5.7%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-028	D10; Sample	0.0750	NA	105	108
ISO14-0001-029	D10; Sample Dup	0.0770	NA	101	103
ISO14-0001-030	D10; FMS	NA (2)	NA (1)	120	114
Average Concentration (ng/mL) ± %RPD/RSD		**0.0760 ng/mL ± 2.7%**		**109% ± 9.2%**	**108% ± 5.1%**

NA = Not Applicable
(1) Sample container was not fortified with the target analytes prior to being sent to the field for sample collection.
(2) Sample was not included in the analysis of PFOSA on 2/6/14.

Table 19. Location ID: L21

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-031	L21; Sample	0.687	NA	0.193	NA	4.18	NA
ISO14-0001-032	L21; Sample Dup	0.668	NA	0.184	NA	4.14	NA
ISO14-0001-033	L21; FMS	12.0	114	11.4	112	16.3	122
Average Concentration (ng/mL) ± %RPD		**0.678 ng/mL ± 2.8%**		**0.189 ng/mL ± 4.8%**		**4.16 ng/mL ± 0.96%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-031	L21; Sample	0.0333	NA	104	104
ISO14-0001-032	L21; Sample Dup	0.0320	NA	108	100
ISO14-0001-033	L21; FMS	11.1	111	104	105
Average Concentration (ng/mL) ± %RPD/RSD		**0.0327 ng/mL ± 4.0%**		**105% ± 2.2%**	**103% ± 2.6%**

NA = Not Applicable

Table 20. Location ID: L22

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-034	L22; Sample	0.868	NA	0.411	NA	11.9	NA
ISO14-0001-035	L22; Sample Dup	0.890	NA	0.391	NA	12.2	NA
ISO14-0001-036	L22; FMS	11.7	109	11.5	111	25.7	136 (1)
Average Concentration (ng/mL) ± %RPD		**0.879 ng/mL ± 2.5%**		**0.401 ng/mL ± 5.0%**		**12.1 ng/mL ± 2.5%** (2)	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-034	L22; Sample	0.0384	NA	104	98.8
ISO14-0001-035	L22; Sample Dup	0.0322	NA	101	105
ISO14-0001-036	L22; FMS	11.1	111	102	112
Average Concentration (ng/mL) ± %RPD/RSD		**0.0353 ng/mL ± 18%**		**102% ± 1.5%**	**105% ± 6.3%**

NA = Not Applicable
(1) The field matrix spike recovery did not meet method acceptance criteria of 100%±30%.
(2) The analytical method uncertainty for PFOS has been adjusted to ±36%.

Table 21. Location ID: L31

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-037	L31; Sample	0.915	NA	0.867	NA	7.95	NA
ISO14-0001-038	L31; Sample Dup	0.923	NA	0.851	NA	8.18	NA
ISO14-0001-039	L31; FMS	12.0	112	11.9	110	21.1	131 (1)
Average Concentration (ng/mL) ± %RPD		**0.919 ng/mL ± 0.87%**		**0.859 ng/mL ± 1.9%**		**8.07 ng/mL ± 2.9%** (2)	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-037	L31; Sample	0.0354	NA	105	103
ISO14-0001-038	L31; Sample Dup	0.0277	NA	102	110
ISO14-0001-039	L31; FMS	11.1	111	102	109
Average Concentration (ng/mL) ± %RPD/RSD		**0.0316 ng/mL ± 24%** (3)		**103% ± 1.7%**	**107% ± 3.5%**

NA = Not Applicable
(2) The field matrix spike recovery did not meet method acceptance criteria of 100%±30%.
(3) The analytical method uncertainty for PFOS has been adjusted to ±31%.
(4) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 22. Location ID: L4

		PFOA		PFHS		PFOS [1]	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-040	L4; Sample	2.92	NA	2.29	NA	22.0	NA
ISO14-0001-041	L4; Sample Dup	3.00	NA	2.26	NA	21.8	NA
ISO14-0001-042	L4; FMS	13.9	110	13.5	112	35.3	NC
Average Concentration (ng/mL) ± %RPD		**2.96 ng/mL ± 2.7%**		**2.28 ng/mL ± 1.3%**		**21.9 ng/mL ± 0.91%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-040	L4; Sample	6.24	NA	104	106
ISO14-0001-041	L4; Sample Dup	6.25	NA	105	105
ISO14-0001-042	L4; FMS	17.6	114	101	103
Average Concentration (ng/mL) ± %RPD/RSD		**6.25 ng/mL ± 0.16%**		**103% ± 2.0%**	**105% ± 1.5%**

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Samples were bracketed by a non-compliant CCV for PFOS. See section 3.4 for details.

Table 23. Location ID: P115

		PFOA		PFHS		PFOS [1]	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-046	P115; Sample	4.49	NA	2.14	NA	0.518	NA
ISO14-0001-047	P115; Sample Dup	4.51	NA	2.06	NA	0.517	NA
ISO14-0001-048	P115; FMS	15.7	113	13.6	115	12.2	117
Average Concentration (ng/mL) ± %RPD		**4.50 ng/mL ± 0.44%**		**2.10 ng/mL ± 3.8%**		**0.518 ng/mL ± 0.19%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-046	P115; Sample	<0.0250	NA	102	104
ISO14-0001-047	P115; Sample Dup	<0.0250	NA	114	100
ISO14-0001-048	P115; FMS	11.1	111	100	111
Average Concentration (ng/mL) ± %RPD/RSD		**<0.0250 ng/mL**		**105% ± 7.2%**	**105% ± 5.3%**

NA = Not Applicable
(1) Samples were bracketed by a non-compliant CCV for PFOS. See section 3.4 for details.

Table 24. Location ID: P116

		PFOA		PFHS		PFOS [1]	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-049	P116; Sample	2.26	NA	0.854	NA	9.18	NA
ISO14-0001-050	P116; Sample Dup	2.20	NA	0.810	NA	9.16	NA
ISO14-0001-051	P116; FMS	13.0	109	12.1	113	21.5	124
Average Concentration (ng/mL) ± %RPD		**2.23 ng/mL ± 2.7%**		**0.832 ng/mL ± 5.3%**		**9.17 ng/mL ± 0.22%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-049	P116; Sample	0.0685	NA	107	104
ISO14-0001-050	P116; Sample Dup	0.0684	NA	110	100
ISO14-0001-051	P116; FMS	11.2	111	102	110
Average Concentration (ng/mL) ± %RPD/RSD		**0.0685 ng/mL ± 0.15%**		**106% ± 3.8%**	**105% ± 4.8%**

NA = Not Applicable
(1) Samples were bracketed by a non-compliant CCV for PFOS. See section 3.4 for details.

Table 25. Location ID: P118C

		PFOA		PFHS		PFOS [1]	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-052	P118C; Sample	818	NA	548	NA	2240	NA
ISO14-0001-053	P118C; Sample Dup	873	NA	569	NA	2430	NA
ISO14-0001-054	P118C; FMS	1880	102	1520	97.9	3510	NC
Average Concentration (ng/mL) ± %RPD		**846 ng/mL ± 6.5%**		**559 ng/mL ± 3.8%**		**2340 ng/mL ± 8.1%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-052	P118C; Sample	57.7	NA	113	113
ISO14-0001-053	P118C; Sample Dup	56.5	NA	120	117
ISO14-0001-054	P118C; FMS	169	112	105	103
Average Concentration (ng/mL) ± %RPD/RSD		**57.1 ng/mL ± 2.1%**		**113% ± 6.6%**	**111% ± 6.5%**

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Samples were bracketed by a non-compliant CCV for PFOS. See section 3.4 for details.

Table 26. Location ID: P119C

		PFOA		PFHS		PFOS [1]	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-055	P119C; Sample	217	NA	113	NA	1740	NA
ISO14-0001-056	P119C; Sample Dup	224	NA	117	NA	1710	NA
ISO14-0001-057	P119C; FMS	1230	99.9	1080	98.3	2750	102
Average Concentration (ng/mL) ± %RPD		**221 ng/mL ± 3.2%**		**115 ng/mL ± 3.5%**		**1730 ng/mL ± 1.7%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-055	P119C; Sample	60.9	NA	108	103
ISO14-0001-056	P119C; Sample Dup	61.9	NA	109	110
ISO14-0001-057	P119C; FMS	175	114	101	97.9
Average Concentration (ng/mL) ± %RPD/RSD		**61.4 ng/mL ± 1.6%**		**106% ± 4.1%**	**104% ± 5.8%**

NA = Not Applicable
(1) Samples were bracketed by a non-compliant CCV for PFOS. See section 3.4 for details.

Table 27. Location ID: P264

		PFOA		PFHS		PFOS [1]	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-058	P264; Sample	392	NA	260	NA	1910	NA
ISO14-0001-059	P264; Sample Dup	387	NA	258	NA	1920	NA
ISO14-0001-060	P264; FMS	1430	103	1230	98.9	2980	106
Average Concentration (ng/mL) ± %RPD		**390 ng/mL ± 1.3%**		**259 ng/mL ± 0.77%**		**1920 ng/mL ± 0.52%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-058	P264; Sample	52.2	NA	105	106
ISO14-0001-059	P264; Sample Dup	51.0	NA	111	108
ISO14-0001-060	P264; FMS	166	114	108	101
Average Concentration (ng/mL) ± %RPD/RSD		**51.6 ng/mL ± 2.3%**		**108% ± 2.8%**	**105% ± 3.4%**

NA = Not Applicable
(1) Samples were bracketed by a non-compliant CCV for PFOS. See section 3.4 for details.

Table 28. Location ID: P265C

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-061	P265C; Sample	109	NA	34.0	NA	1140	NA
ISO14-0001-062	P265C; Sample Dup	132	NA	54.1	NA	1200	NA
ISO14-0001-063	P265C; FMS	1110	97.9	1000	97.3	2160	99.2
Average Concentration (ng/mL) ± %RPD		**121 ng/mL ± 19%**		**44.1 ng/mL ± 46% (1)**		**1170 ng/mL ± 5.1%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-061	P265C; Sample	111	NA	107	108
ISO14-0001-062	P265C; Sample Dup	108	NA	112	107
ISO14-0001-063	P265C; FMS	218	108 (2)	115	111
Average Concentration (ng/mL) ± %RPD/RSD		**110 ng/mL ± 2.7%**		**111% ± 3.6%**	**109% ± 1.9%**

NA = Not Applicable
(1) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.
(2) Field matrix spike recovery is an estimated value due to the concentration exceeding the upper limit of quantification (200 ng/mL).

Table 29. Location ID: P321

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-064	P321; Sample	6260	NA	1330	NA	2520	NA
ISO14-0001-065	P321; Sample Dup	6350	NA	1400	NA	2490	NA
ISO14-0001-066	P321; FMS	7010	NC	2280	92.7	3640	NC
Average Concentration (ng/mL) ± %RPD		**6310 ng/mL ± 1.4%**		**1370 ng/mL ± 5.1%**		**2510 ng/mL ± 1.2%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-064	P321; Sample	1.13	NA	117	115
ISO14-0001-065	P321; Sample Dup	6.18	NA	109	106
ISO14-0001-066	P321; FMS	103	99.3	114	112
Average Concentration (ng/mL) ± %RPD/RSD		**3.66 ng/mL ± 138% (1)**		**113% ± 3.6%**	**111% ± 4.1%**

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%. Sample was re-analyzed on 2/10/14 and confirmed the results.

Table 30. Location ID: P372

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-067	P372; Sample	98.2	NA	63.7	NA	1790	NA
ISO14-0001-068	P372; Sample Dup	102	NA	65.4	NA	1760	NA
ISO14-0001-069	P372; FMS	1100	99.0	1040	99.3	2870	109
Average Concentration (ng/mL) ± %RPD		**100 ng/mL ± 3.8%**		**64.6 ng/mL ± 2.6%**		**1780 ng/mL ± 1.7%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-067	P372; Sample	50.4	NA	106	109
ISO14-0001-068	P372; Sample Dup	50.7	NA	109	107
ISO14-0001-069	P372; FMS	170	119	103	108
Average Concentration (ng/mL) ± %RPD/RSD		**50.6 ng/mL ± 0.59%**		**106% ± 2.8%**	**108% ± 0.93%**

NA = Not Applicable

Table 31. Location ID: P374

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-070	P374; Sample	783	NA	678	NA	2580	NA
ISO14-0001-071	P374; Sample Dup	773	NA	657	NA	2950	NA
ISO14-0001-072	P374; FMS	1730	94.3	1620	96.9	3700	NC
Average Concentration (ng/mL) ± %RPD		**778 ng/mL ± 1.3%**		**668 ng/mL ± 3.1%**		**2770 ng/mL ± 13%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-070	P374; Sample	33.1	NA	112	105
ISO14-0001-071	P374; Sample Dup	32.5	NA	104	105
ISO14-0001-072	P374; FMS	149	116	107	106
Average Concentration (ng/mL) ± %RPD/RSD		**32.8 ng/mL ± 1.8%**		**108% ± 3.7%**	**105% ± 0.55%**

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

Table 32. Location ID: P378

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-073	P378; Sample	351	NA	347	NA	1180	NA
ISO14-0001-074	P378; Sample Dup	333	NA	345	NA	1200	NA
ISO14-0001-075	P378; FMS	1340	98.8	1310	98.2	2150	96.2
Average Concentration (ng/mL) ± %RPD		**342 ng/mL ± 5.3%**		**346 ng/mL ± 0.58%**		**1190 ng/mL ± 1.7%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-073	P378; Sample	1.79	NA	111	109
ISO14-0001-074	P378; Sample Dup	1.78	NA	108	106
ISO14-0001-075	P378; FMS	114	112	112	111
Average Concentration (ng/mL) ± %RPD/RSD		**1.79 ng/mL ± 0.56%**		**110% ± 1.9%**	**109% ± 2.3%**

NA = Not Applicable

Table 33. Location ID: P381

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-076	P381; Sample	667	NA	191	NA	1710	NA
ISO14-0001-077	P381; Sample Dup	645	NA	176	NA	1590	NA
ISO14-0001-078	P381; FMS	1650	98.4	1120	95.3	2700	105
Average Concentration (ng/mL) ± %RPD		**656 ng/mL ± 3.4%**		**184 ng/mL ± 8.2%**		**1650 ng/mL ± 7.3%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-076	P381; Sample	20.7	NA	107	104
ISO14-0001-077	P381; Sample Dup	13.7	NA	106	106
ISO14-0001-078	P381; FMS	128	111	106	104
Average Concentration (ng/mL) ± %RPD/RSD		**17.2 ng/mL ± 41%** (1)		**106% ± 0.54%**	**105% ± 1.1%**

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

Table 34. Location ID: P382

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-079	P382; Sample	596	NA	168	NA	981	NA
ISO14-0001-080	P382; Sample Dup	649	NA	181	NA	972	NA
ISO14-0001-081	P382; FMS	1640	101	1080	92.2	1950	97.5
Average Concentration (ng/mL) ± %RPD		**623 ng/mL ± 8.5%**		**175 ng/mL ± 7.4%**		**977 ng/mL ± 0.92%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-079	P382; Sample	80.5	NA	105	107
ISO14-0001-080	P382; Sample Dup	78.7	NA	111	104
ISO14-0001-081	P382; FMS	197	117	106	102
Average Concentration (ng/mL) ± %RPD/RSD		**79.6 ng/mL ± 2.3%**		**107% ± 3.0%**	**104% ± 2.4%**

NA = Not Applicable

Table 35. Location ID: PA109A

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-082	PA109A; Sample	302	NA	299	NA	496	NA
ISO14-0001-083	PA109A; Sample Dup	310	NA	309	NA	536	NA
ISO14-0001-084	PA109A; FMS	831	104	784	97.8	1000	97.0
Average Concentration (ng/mL) ± %RPD		**306 ng/mL ± 2.6%**		**304 ng/mL ± 3.3%**		**516 ng/mL ± 7.8%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-082	PA109A; Sample	1.21	NA	105	101
ISO14-0001-083	PA109A; Sample Dup	1.12	NA	108	110
ISO14-0001-084	PA109A; FMS	56.9	111	108	103
Average Concentration (ng/mL) ± %RPD/RSD		**1.17 ng/mL ± 7.7%**		**107% ± 1.6%**	**105% ± 4.5%**

NA = Not Applicable

Table 36. Location ID: PA111A

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-085	PA111A; Sample	823	NA	1060	NA	2200	NA
ISO14-0001-086	PA111A; Sample Dup	824	NA	1070	NA	2390	NA
ISO14-0001-087	PA111A; FMS	1860	103	2040	98.8	3750	NC
Average Concentration (ng/mL) ± %RPD		**824 ng/mL ± 0.12%**		**1070 ng/mL ± 0.93%**		**2300 ng/mL ± 8.3%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-085	PA111A; Sample	12.3	NA	108	105
ISO14-0001-086	PA111A; Sample Dup	11.9	NA	108	106
ISO14-0001-087	PA111A; FMS	127	115	107	106
Average Concentration (ng/mL) ± %RPD/RSD		**12.1 ng/mL ± 3.3%**		**108% ± 0.53%**	**106% ± 0.54%**

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

Table 37. Location ID: PA112

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-088	PA112; Sample	154	NA	31.7	NA	341	NA
ISO14-0001-089	PA112; Sample Dup	156	NA	32.7	NA	331	NA
ISO14-0001-090	PA112; FMS	659	99.4	526	101	845	102
Average Concentration (ng/mL) ± %RPD		**155 ng/mL ± 1.3%**		**32.2 ng/mL ± 3.1%**		**336 ng/mL ± 3.0%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-088	PA112; Sample	48.3	NA	113	110
ISO14-0001-089	PA112; Sample Dup	48.4	NA	107	108
ISO14-0001-090	PA112; FMS	108	119	109	106
Average Concentration (ng/mL) ± %RPD/RSD		**48.4 ng/mL ± 0.21%**		**110% ± 2.8%**	**108% ± 1.9%**

NA = Not Applicable

Table 38. Location ID: Z1-3m

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-091	Z1-3m; Sample	0.877	NA	0.741	NA	0.395	NA
ISO14-0001-092	Z1-3m; Sample Dup	0.878	NA	0.725	NA	0.523	NA
ISO14-0001-093	Z1-3m; FMS	2.99	107	3.05	116	3.00	127
Average Concentration (ng/mL) ± %RPD		**0.878 ng/mL ± 0.11%**		**0.733 ng/mL ± 2.2%**		**0.459 ng/mL ± 28% (1)**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-091	Z1-3m; Sample	0.0335	NA	107	108
ISO14-0001-092	Z1-3m; Sample Dup	<0.0250	NA	104	109
ISO14-0001-093	Z1-3m; FMS	2.22	109	99.6	108
Average Concentration (ng/mL) ± %RPD/RSD		**0.0335 ng/mL**		**104% ± 3.6%**	**108% ± 0.53%**

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

Table 39. Location ID: Z2-10m

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-094	Z2-10m; Sample	1.28	NA	1.36	NA	1.47	NA
ISO14-0001-095	Z2-10m; Sample Dup	1.31	NA	1.38	NA	1.45	NA
ISO14-0001-096	Z2-10m; FMS	3.53	113	3.60	112	3.78	116
Average Concentration (ng/mL) ± %RPD		**1.30 ng/mL ± 2.3%**		**1.37 ng/mL ± 1.5%**		**1.46 ng/mL ± 1.4%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-094	Z2-10m; Sample	0.0475	NA	98.7	114
ISO14-0001-095	Z2-10m; Sample Dup	0.0467	NA	108	106
ISO14-0001-096	Z2-10m; FMS	2.26	111	105	102
Average Concentration (ng/mL) ± %RPD/RSD		**0.0471 ng/mL ± 1.7%**		**104% ± 4.6%**	**107% ± 5.7%**

NA = Not Applicable

Table 40. Location ID: Z2-3m

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-097	Z2-3m; Sample	1.38	NA	1.17	NA	0.458	NA
ISO14-0001-098	Z2-3m; Sample Dup	1.38	NA	1.20	NA	0.454	NA
ISO14-0001-099	Z2-3m; FMS	3.54	109	3.47	114	3.01	128
Average Concentration (ng/mL) ± %RPD		**1.38 ng/mL ± 0.0%**		**1.19 ng/mL ± 2.5%**		**0.456 ng/mL ± 0.88%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-097	Z2-3m; Sample	<0.0250	NA	102	111
ISO14-0001-098	Z2-3m; Sample Dup	<0.0250	NA	107	116
ISO14-0001-099	Z2-3m; FMS	2.22	111	105	121
Average Concentration (ng/mL) ± %RPD/RSD		**<0.0250 ng/mL**		**105% ± 2.4%**	**116% ± 4.3%**

NA = Not Applicable

Table 41. Location ID: P118A

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-100	P118A; Sample	2610	NA	5030	NA	1930	NA
ISO14-0001-101	P118A; Sample Dup	2590	NA	5300	NA	1840	NA
ISO14-0001-102	P118A; FMS	3450	NC	6590	NC	3180	129
Average Concentration (ng/mL) ± %RPD		**2600 ng/mL ± 0.77%**		**5170 ng/mL ± 5.2%**		**1890 ng/mL ± 4.8%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-100	P118A; Sample	11.5	NA	112	108
ISO14-0001-101	P118A; Sample Dup	8.04	NA	106	104
ISO14-0001-102	P118A; FMS	113	103	104	108
Average Concentration (ng/mL) ± %RPD/RSD		**9.77 ng/mL ± 35% (1)**		**107% ± 3.9%**	**107% ± 2.2%**

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

Table 42. Location ID: P121

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-103	P121; Sample	0.284	NA	0.153	NA	5.21	NA
ISO14-0001-104	P121; Sample Dup	0.255	NA	0.153	NA	4.69	NA
ISO14-0001-105	P121; FMS	11.6	114	11.7	115	17.4	125
Average Concentration (ng/mL) ± %RPD		**0.270 ng/mL ± 11%**		**0.153 ng/mL ± 0.0%**		**4.95 ng/mL ± 11%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-103	P121; Sample	0.337	NA	92.0	98.5
ISO14-0001-104	P121; Sample Dup	0.283	NA	89.6	101
ISO14-0001-105	P121; FMS	11.3	110	99.2	103
Average Concentration (ng/mL) ± %RPD/RSD		**0.310 ng/mL ± 17%**		**93.6% ± 5.3%**	**101% ± 2.2%**

NA = Not Applicable

Table 43. Location ID: P371

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-106	P371; Sample	81.2	NA	31.9	NA	929	NA
ISO14-0001-107	P371; Sample Dup	76.4	NA	30.6	NA	789	NA
ISO14-0001-108	P371; FMS	1130	104	1030	102	2000	114
Average Concentration (ng/mL) ± %RPD		**78.8 ng/mL ± 6.1%**		**31.3 ng/mL ± 4.2%**		**859 ng/mL ± 16%**	

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-106	P371; Sample	47.2	NA	103	101
ISO14-0001-107	P371; Sample Dup	47.4	NA	106	105
ISO14-0001-108	P371; FMS	162	115	111	105
Average Concentration (ng/mL) ± %RPD/RSD		**47.3 ng/mL ± 0.42%**		**107% ± 3.8%**	**104% ± 2.2%**

NA = Not Applicable

Table 44. Trip Blank

		PFOA		PFHS		PFOS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0001-109	Trip Blank; Sample	<0.0240	NA	<0.0250	NA	<0.232	NA
ISO14-0001-110	Trip Blank; FMS Low	2.21	112	2.25	113	2.18	109
ISO14-0001-111	Trip Blank; FMS Mid	100	99.0	97.0	98.8	102	102
ISO14-0001-112	Trip Blank; FMS High	965	95.5	965	98.3	1080	108

		PFOSA		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	%Recovery	%Recovery
ISO14-0001-109	Trip Blank; Sample	<0.0250	NA	96.1	104
ISO14-0001-110	Trip Blank; FMS Low	2.22	111	107	93.4
ISO14-0001-111	Trip Blank; FMS Mid	11.1	111	109	105
ISO14-0001-112	Trip Blank; FMS High	111	111	104	105

NA = Not Applicable

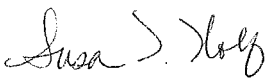
5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M 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 10-44 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: 2014.02.25 07:38:59 -06'00'

Susan T. Wolf, 3M Principal Analytical Investigator



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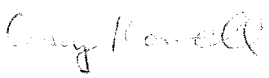
Reason: I am approving this document
Date: 2014.02.26 11:28:06 -06'00'

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 Casey Howell
DN: c=US, st=MN, l=St. Paul, ou=Quality Assurance Unit, ou=3M Environmental Laboratory - authenticated by LRA, email=[redacted] o=3M, cn=Casey Howell
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 EHS
Robert A. Paschke
3M Center, 223-02-S-31
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USA

ANALYTICAL REPORT

IAC14-00221

3M Lab Request Number: E14-0040

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

TESTING LABORATORY

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Author: Sandra Graré
Date issued: February 6th, 2014

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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: E14-0040) of Robert A. Paschke, 3M Manager Corporate Environmental Programs, water samples were collected on Jan 28th 2014 from 8 locations and analyzed for the following perfluorinated compounds (locations PP04, 05 and 06 are not sampled):

- 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
PP01	72.2	4133	55.0	2202	18.8
PP02	<26.2	1803	753	27617	33.5
PP07	144	120	490	3523	55.4
PP08	39.6	284	599	8436	46.2
PP09	29.2	399	722	2693	17.6
PP10	16.0	167	619	4485	17.6
Effluent WWTP	NR	<0.614	<1.03	<1.28	<0.655
Collector Pit	NR	15.2	54.9	303	12.7

NR: sample analysis included PFBS, however results for PFBS were not requested and therefore not reported.

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)
PP01	100
PP02	100
PP04	-
PP05	-
PP06	-
PP07	100
PP08	100
PP09	100
PP10	100
Effluent WWTP	100
Collector Pit	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/037 "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
PP01	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP02	1.0	0.100	Solution A	10.0	0.5mL extract + 10.0mL MeOH (*)
PP07	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP08	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 (*)
PP10	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
Effluent WWTP	1.0	0.100	Solution A	5.0	-
Collector Pit	1.0	0.100	Solution A	5.0	-
Field Trip Blank	1.0	0.100	Solution A	10.0	-

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

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (PFBS, PFHS, PFOA, ^{13}C-PFOA, PFOS, FOSA and ^{13}C-PFOS) using ultra performance liquid chromatography / tandem mass spectrometry (UPLC/MS/MS). Pertinent instrument parameters, the liquid chromatography gradient program, and the specific mass transitions analyzed are described in the tables 4 to 6.

Table 4. Instrument parameters.

Liquid Chromatograph	Acquity UPLC
Guard Column	Symmetrie C18 50 x 2.1mm, 3.5µm
Analytical Column	Acquity UPLC BEH C18 50 mm x 2.1mm, 1.7µm
Injection volume	5 µL
Mass Spectrometer	Quattro Premier XE
Ion source	Electrospray
Polarity	Negative

Table 5. Liquid Chromatography Gradient Program.

Step Number	Total time (min)	Flow Rate (µL/min)	Percent A (2mM ammonium acetate)	Percent B (Methanol)
0	0	400	95	5
1	0.50	400	95	5
2	1.00	400	47	53
3	4.00	400	32	68
4	5.00	400	0	100
5	5.50	400	0	100
6	5.55	400	95	5
7	6.50	400	95	5

Table 6. Mass transitions.

Analyte	Time window (min)	Mass Transition Q1/Q3	Dwell time (msec)	Collision Energy (eV)	Cone Voltage (eV)
^{13}C-PFOA	2.91 - 3.80	416.8 /371.8	60	11	14
PFBS	2.08 - 2.50	298.93/79.82	40	28	45
		298.93/98.78	40	29	45
PFHS	2.45 - 3.05	398.86/79.83	40	38	51
		398.86/98.79	40	35	51
PFOA	2.80 - 3.65	412.9/218.98	40	11	14
		412.9/368.87	40	11	14
PFOS	2.92 - 4.20	498.78/79.77	50	48	56
		498.78/98.73	50	39	56
FOSA	4.00 - 5.60	497.8 /77.82	125	34	44
^{13}C-PFOS	3.40 – 4.20	503.0 /80.0	60	48	56

For all analytes the sum of all mass transitions was used for quantification.

3. Data Analysis.

3.1. Solvent Calibration Standard.

A total of 12 solvent standards ranging from 0.1 ng/mL to 400 ng/mL (nominal) were prepared and contains PFBS, PFHS, PFOA, ^{13}C-PFOA, PFOS, FOSA and ^{13}C-PFOS. A quadratic, 1/x weighted, calibration curve was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentration using the peak area counts and the resultant calibration curve confirmed accuracy of each curve point. Each calibration standard (at least 6 points in total) used to generate the final calibration curve met the method calibration accuracy requirements of ± 25% (± 30% for LLOQ level). Coefficients of determination (r^2) were greater than 0.990 for all analytes.

3.2. Limit of Quantitation.

The LOQ for this analysis is based on the lowest non-zero active calibration standard in the curve, which was 0.101 to 0.210 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 12.3 to 49.0 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 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
03 Feb 2014	LCS1	80	90.5	92.5	93.4	91.3	105	101	88.7

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
27 Aug 2013	QC Lab Low inj1	10	104	102	105	104	109	104	105
	QC Lab High inj1	100	93.7	93.1	103	95.2	110	102	92.6
	QC Lab Low inj2	10	114	107	114	114	125	124	108
	QC Lab High inj2	100	109	103	113	105	125	125	97.5

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
PP01	72.2	4133	55.0	2202	18.8
PP02	<26.2	1803	753	27617	33.5
PP07	144	120	490	3523	55.4
PP08	39.6	284	599	8436	46.2
PP09	29.2	399	722	2693	17.6
PP10	16.0	167	619	4485	17.6
Effluent WWTP	NR	<0.614	<1.03	<1.28	<0.655
Collector Pit	NR	15.2	54.9	303	12.7
Field Trip Blank	<1.25	<1.12	<1.87	<2.33	<1.19

NR: sample analysis included PFBS, however results for PFBS were not requested and therefore not reported.

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

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
PP01	117	96.8	123	101
PP02	140	116	101	82.9
PP07	125	103	116	95.0
PP08	133	111	113	92.3
PP09	132	110	119	97.4
PP10	124	103	114	92.9
Effluent WWTP	133	111	125	102
Collector Pit	124	103	117	96.0
Field Trip Blank	124	103	120	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 Ruben Maes (ERM Belgium).

8. Signatures.

Geert De Smet, Lab Operations Manager Date February 6th, 2014

Marc Van Ryckeghem, Division Manager Date February 6th, 2014

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

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

SGS

3M Corporate Environmental Programs.
Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC14-00370

3M Lab Request Number: E14-0130

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

TESTING LABORATORY

SGS Belgium
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Author: Sandra Graré
Date issued: February 17th, 2014

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1. Introduction - Summary.

At the request (Lab Request Number: E14-0130) of Robert A. Paschke, 3M Manager Corporate Environmental Programs, water samples were collected on February 4th 2014 from 1 location and analyzed for the following perfluorinated compounds:

- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$) (and its ^{13}C-labeled analogues)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$) (and its ^{13}C-labeled analogues)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$) (and its ^{13}C-labeled analogues)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}\ SO_2NH_2$) (and its ^{13}C-labeled analogue)
- ^{13}C-labeled analogue of PFUdA ($C_4F_{21}{}^{13}C_6F_2{}^{13}COO^-$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Geert De Smet. Sample containers for each sampling location included a field sample and field sample duplicate. 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 - east (filtered)	0.404	0.958	1.29	0.0920 (b)
Blokkersdijkvijver - east duplicate (filtered)	0.412	0.899	1.37	0.0956 (b)
Blokkersdijkvijver - east (not filtered)	0.551	1.21	1.47	0.100 (a)
Blokkersdijkvijver - east duplicate (not filtered)	0.479	1.07	1.41	0.0721 (a)

(a) The accuracy for FOSA for Blokkersdijkvijver-east (not filtered) is ± 45%.
(b) The accuracy for FOSA for Blokkersdijkvijver-east (filtered) is ± 60%.

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 ($^{18}O_2$-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 - east (filtered)	400	0.100	Solution A	0.25	-
		0.250	Solution B	-	1.0
Blokkersdijkvijver - east duplicate (filtered)	400	0.100	Solution A	0.25	-
		0.250	Solution B	-	1.0
Blokkersdijkvijver - east (not filtered)	400	0.100	Solution A	0.25	-
		0.250	Solution B	-	1.0
Blokkersdijkvijver - east duplicate (not filtered)	400	0.100	Solution A	0.25	-
		0.250	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 - east (filtered)	40	8
Blokkersdijkvijver - east duplicate (filtered)	40	8
Blokkersdijkvijver - east (not filtered)	40	8
Blokkersdijkvijver - east duplicate (not filtered)	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 location Blokkersdijkvijver - east.
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 - east (filtered)	0.001
Blokkersdijkvijver - east duplicate (filtered)	0.005

2.4. Analysis.

All solutions and extracts were analyzed for the PFCs (PFHS, PFOA, PFOS, FOSA) and the surrogate recovery standards ($^{18}O_2$-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
$^{18}O_2$-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.

Two method blanks were 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 400 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, and ± 15% RPD for precision.

Table 8 summarize the LCS recovery results.

Table 8. Lab Control Spike Results.

Extraction date	Description	Nominal Spike Level (ng/mL)	Percent Recovery							
			$^{13}C_4$-PFOA	$^{13}C_4$-PFOS	$^{18}O_2$-PFHS	$^{13}C_7$-PFuDA	PFHS	PFOA	PFOS	FOSA
10 Feb 2014	LCSL1	0.125	98.7	92.6	110	101	101	106	114	98.2
	LCSL2	0.125	96.4	104	98.0	92.5	91.7	104	109	100
	Average		**97.6**	**98.3**	**104**	**96.9**	**96.1**	**105**	**111**	**99.3**
	%RPD		**2.4**	**12**	**12**	**9.1**	**9.2**	**1.2**	**5.0**	**2.2**
	LCSH1	2.5	105	98.2	96.2	95.4	88.8	106	100	102
	LCSH2	2.5	107	103	99.4	104	85.9	100	101	100
	Average		**106**	**97.9**	**97.8**	**100**	**84.9**	**103**	**100**	**101**
	%RPD		**1.9**	**9.5**	**3.3**	**8.7**	**2.5**	**5.3**	**1.0**	**2.0**

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.

3.8.1. Relative Percent Difference (RPD).

$$\text{RPD (\%)} = \frac{\text{ABSOLUTE concentration difference (sample - sample duplicate)}}{\text{AVERAGE sample concentration}} * 100$$

3.8.2. Recovery.

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

4. Data Summary.

Tabel 9 and 10 summarizes the results for the sample location.

Table 9. Sample Results PFHS and PFOA.

	PFHS			$^{18}O_2$-PFHS	PFOA			$^{13}C_4$-PFOA
	ng/ml	Sample average	RPD (%)	%Rec	ng/ml	Sample average	RPD (%)	%Rec
Field Trip Blank	<0.025			89.7	<0.025			88.2
Blokkersdijkvijver - east (filtered)	0.404	0.408	1.8	99.6	0.958	0.929	6.4	97.3
Blokkersdijkvijver - east duplicate (filtered)	0.412			86.9	0.899			95.6
Blokkersdijkvijver - east (not filtered)	0.551	0.515	14	93.2	1.21	1.14	12	93.3
Blokkersdijkvijver - east duplicate (not filtered)	0.479			85.7	1.07			95.3

Table 10. Sample Results PFOS and FOSA.

	PFOS			$^{13}C_4$-PFOS	FOSA			$^{13}C_7$-PFuDA
	ng/ml	Sample average	RPD (%)	%Rec	ng/ml	Sample average	RPD (%)	%Rec
Field Trip Blank	<0.025			90.5	<0.025			74.1
Blokkersdijkvijver - east (filtered)	1.29	1.33	5.7	94.4	0.0920	0.0938	3.8	43.3 (b)
Blokkersdijkvijver - east duplicate (filtered)	1.37			100	0.0956			43.6 (b)
Blokkersdijkvijver - east (not filtered)	1.47	1.44	4.6	94.5	0.100	0.0862	33 (a)	58.8 (b)
Blokkersdijkvijver - east duplicate (not filtered)	1.41			96.3	0.0721			56.6 (b)

(a): The relative percent difference fell outside the method acceptance criterion of 15%.
(b): 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-east (not filtered) is ± 45%.
- The accuracy for FOSA for Blokkersdijkvijver-east (filtered) is ± 60%.

5. Conclusion.

- The relative percent difference exceeded 15% for one sample location.
- The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130% for one sample location.
- Lab control spike recoveries fell within the method acceptance criteria of 25%, indicating that the analysis method is in control.

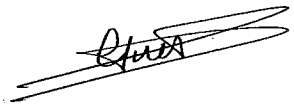
6. Data / Sample Retention.

All hardcopy and electronic data will be archived according to SGS Belgium NV standard operating procedures. A paper or electronic copy of all necessary raw data will be archived at 3M. The remaining volume of samples will be retained for 3 months. The empty sample bottles are not retained.

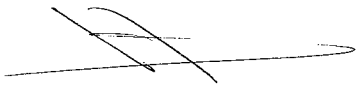
7. Addendum.

Addendum: e-mail from Ruben Maes (ERM Belgium).

8. Signatures.



Geert De Smet, Lab Operations Manager Date February 17th, 2014



Marc Van Ryckeghem, Division Manager Date February 17th, 2014

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

SGS

3M EHS
Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC14-00716

3M Lab Request Number: E14-0212

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

TESTING LABORATORY

SGS Belgium
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Author: Sandra Graré
Date issued: March 26th, 2014

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All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction - Summary.

At the request (Lab Request Number: E14-0212) of Robert A. Paschke, 3M Manager Corporate Environmental Programs, water samples were collected on Feb 25th 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
PP09	32.6	345	715	2208	<10.9
PP10	19.2	169	601	3892	<10.9

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)
PP09	100
PP10	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/037 "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
PP09	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP10	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
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 0.0527 to 0.172 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 6.43 to 21.0 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 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
03 Feb 2014	LCS1	80	82.1	84.3	91.0	85.6	96.8	83.6	78.2

3.6. QC Lab Standards (QCs)

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

Table 8. QC Lab Standards Results.

Preparing date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C-PFOA	PFOS	FOSA	^{13}C-PFOS
27 Aug 2013	QC Lab Low inj1	10	103	103	113	106	108	101	97.7
	QC Lab High inj1	100	85.6	84.9	100	92.8	96.5	82.8	80.5
	QC Lab Low inj2	10	97.7	98.9	113	100	105	100	95.0
	QC Lab High inj2	100	87.1	86.3	101	93.2	100	87.7	79.3

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
PP09	32.6	345	715	2208	<10.9
PP10	19.2	169	601	3892	<10.9
Field Trip Blank	<0.585	<0.992	<1.85	<1.91	<0.995

NR: sample analysis included PFBS, however results for PFBS were not requested and therefore not reported.

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

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
PP09	112	92.6	99.3	81.2
PP10	96.4	80.0	100	81.9
Field Trip Blank	118	98.1	115	93.8

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.

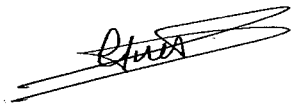
6. Data / Sample Retention.

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.

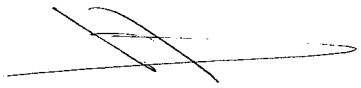
7. Addendum.

Addendum: e-mail from Ruben Maes (ERM Belgium).

8. Signatures.



Geert De Smet, Lab Operations Manager Date March 27th, 2014



Marc Van Ryckeghem, Division Manager Date March 27th, 2014

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

Final Report

Groundwater and Surface Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Belgium April 2014 Sampling

Laboratory Request Number: ISO14-0010

Report Date – Date of Last Signature

Testing Laboratory

3M EHS&S 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]

ACCREDITED
Testing Cert. # 2052.01

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

3M Environmental Laboratory

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

Analytical Report ISO14-0010

Groundwater and Surface Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Belgium - April 2014 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M Environmental Laboratory prepared and analyzed groundwater and surface water samples collected by Environmental Resources Management (ERM) personnel at the 3M Belgium facility. Samples were collected on April 2, 2014. Samples were returned to the 3M Environmental Laboratory on April 7, 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 ISO14-0010.

The 3M Environmental Laboratory prepared sample containers for nineteen 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. Sample location PP01 was inadvertently spiked with a standard spiking solution that did not contain the analytes PFBS or PFOSA. Therefore, the surrogate recovery standards or a lab matrix spike sample were used assess the recovery for PFBS and PFOSA. All sample bottles were fortified with internal standards and surrogate recovery standards prior to being sent to the field for sample collection. Sample location PP06 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.

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**
ISO14-0010-001	P321; Sample	1940	674	3620	10.3
ISO14-0010-002	P321; Sample Dup	1590	539	3810	13.7
	Average	**1770**	**607**	**3720**	**12.0** [2]
	%RPD Sample/Sample Dup	**20**	**22** [3]	**5.1**	**28** [3]
ISO14-0010-004	L4; Sample	2.57	2.04	23.7	2.33
ISO14-0010-005	L4; Sample Dup	2.62	2.09	22.7	2.33
	Average	**2.60**	**2.07**	**23.2**	**2.33**
	%RPD Sample/Sample Dup	**1.9**	**2.4**	**4.3**	**0.0**
ISO14-0010-007	P114bis; Sample	4.15	1.67	16.1	0.0735
ISO14-0010-008	P114bis; Sample Dup	4.05	1.67	16.4	NR [4]
	Average	**4.10**	**1.67**	**16.3**	**0.0735** [2]
	%RPD Sample/Sample Dup	**2.4**	**0.0**	**1.8**	**NA**
ISO14-0010-010	P116; Sample	2.02	0.795	7.76	0.0916
ISO14-0010-011	P116; Sample Dup	1.94	0.773	7.16	0.0921
	Average	**1.98**	**0.784**	**7.46**	**0.0919** [2]
	%RPD Sample/Sample Dup	**4.0**	**2.8**	**8.0**	**0.54**
ISO14-0010-013	3M Pond; Sample	1.63	0.728	4.20	<0.0500
ISO14-0010-014	3M Pond; Sample Dup	1.77	0.776	3.80	<0.0500
	Average	**1.70**	**0.752**	**4.00**	**<0.0500** [2]
	%RPD Sample/Sample Dup	**8.2**	**6.4**	**10**	**NA**
ISO14-0010-016	Blokkersdijkvijver std; Sample	1.33	0.678	1.19	<0.0500
ISO14-0010-017	Blokkersdijkvijver std; Sample Dup	1.29	0.653	1.15	<0.0500
	Average	**1.31**	**0.666**	**1.17**	**<0.0500** [2]
	%RPD Sample/Sample Dup	**3.1**	**3.8**	**3.4**	**NA**
ISO14-0010-019	12; Sample	120	31.4	329	0.328
ISO14-0010-020	12; Sample Dup	126	31.9	313	0.297
	Average	**123**	**31.7**	**321**	**0.313**
	%RPD Sample/Sample Dup	**4.9**	**1.6**	**5.0**	**9.9**
ISO14-0010-022	13; Sample	113	30.6	292	0.330
ISO14-0010-023	13; Sample Dup	108	29.7	290	0.349
	Average	**111**	**30.2**	**291**	**0.340**
	%RPD Sample/Sample Dup	**4.5**	**3.0**	**0.69**	**5.6**
ISO14-0010-025	5; Sample	48.2	31.6	81.0	0.230
ISO14-0010-026	5; Sample Dup	47.8	32.0	81.1	0.199
	Average	**48.0**	**31.8**	**81.1**	**0.215** [2]
	%RPD Sample/Sample Dup	**0.83**	**1.3**	**0.12**	**14**
ISO14-0010-028	Bemlingsstation; Sample	37.6	25.6	66.9	0.244
ISO14-0010-029	Bemlingsstation; Sample Dup	38.4	25.6	64.3	0.220
	Average	**38.0**	**25.6**	**65.6**	**0.232** [2]
	%RPD Sample/Sample Dup	**2.1**	**0.0**	**4.0**	**10**

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 ± 14%, PFBS ± 18%, PFHS ± 10%, PFOS ± 8.0%, and PFOSA ± 26%.

(2) Samples reported using internal calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 16%.

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

(4) NR = Not reportable; asee 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**
ISO14-0010-031	PP01; Sample	73.4	79.5	10700	4100	24.3
ISO14-0010-032	PP01; Sample Dup	73.9	81.8	13200	5150	30.9
	Average	**73.7**	**80.7**	**11200**	**4630**	**27.6**
	%RPD Sample/Sample Dup	**0.68**	**2.9**	**21** [3]	**23** [3]	**24** [3]
ISO14-0010-034	PP02; Sample	701	25.7	2520	23100	25.7
ISO14-0010-035	PP02; Sample Dup	669	24.0	2380	24800	24.4
	Average	**685**	**24.9**	**2450**	**24000**	**25.1**
	%RPD Sample/Sample Dup	**4.7**	**6.8**	**5.7**	**7.1**	**5.2**
ISO14-0010-037	PP04; Sample	1960	2400	782	7640	42.4
ISO14-0010-038	PP04; Sample Dup	2130	2620	927	8630	39.4
	Average	**2050**	**2510**	**855** [5]	**8140**	**40.9** [2]
	%RPD Sample/Sample Dup	**8.3**	**8.8**	**17**	**12**	**7.3**
ISO14-0010-040	PP05; Sample	1760	39500	4730	200	45.0
ISO14-0010-041	PP05; Sample Dup	1830	44800	4510	130	31.4
	Average	**1800**	**42200**	**4620**	**165**	**38.2** [5]
	%RPD Sample/Sample Dup	**3.9**	**13**	**4.8**	**42** [3]	**36** [3]
ISO14-0010-043	PP07; Sample	537	140	142	3790	73.3
ISO14-0010-044	PP07; Sample Dup	550	131	141	3890	72.1
	Average	**544**	**136**	**142**	**3840**	**72.7**
	%RPD Sample/Sample Dup	**2.4**	**6.6**	**0.70**	**2.6**	**1.7**
ISO14-0010-046	PP08; Sample	721	43.4	316	9220	52.1
ISO14-0010-047	PP08; Sample Dup	718	41.8	322	9120	51.5
	Average	**720**	**42.6**	**319**	**9170**	**51.8**
	%RPD Sample/Sample Dup	**0.42**	**3.8**	**1.9**	**1.1**	**1.2**
ISO14-0010-049	PP09; Sample	870	32.4	475	2710	16.7
ISO14-0010-050	PP09; Sample Dup	846	30.9	460	2570	14.6
	Average	**858**	**31.7**	**468**	**2640**	**15.7**
	%RPD Sample/Sample Dup	**2.8**	**4.7**	**3.2**	**5.3**	**13**
ISO14-0010-052	PP10; Sample	781	23.1	210	4700	13.4
ISO14-0010-053	PP10; Sample Dup	789	23.5	213	4810	18.5
	Average	**785**	**23.3**	**212**	**4760**	**16.0**
	%RPD Sample/Sample Dup	**1.0**	**1.7**	**1.4**	**2.3**	**32** [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 ± 14%, PFBS ± 18%, PFHS ± 10%, PFOS ± 8.0%, and PFOSA ± 26%.

(2) Samples reported using internal calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 16%.

(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". Sampling

locations P321, L4, P114bis, P116, 3M Pond, Blokkersdijkvijver – standard, 12, 13, 5, and Bemalingsstation 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 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 2, 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 sample locations were spiked with a mixture of mass-labeled internal standards at a nominal concentration of 1 ng/mL and 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 April 7, 2014.

2.3 Sample Preparation

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 for PFOSA: 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.

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.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk
Liquid Chromatograph	Agilent 1200
Analysis Method	ETS-8-044.1
Analysis Date	4/23/14, 4/25/14, 4/30/14, 5/5/14
Guard column	Betasil C18 (4.6 mm X 100 mm), 5 µ
Analytical column	Betasil C18 (4.6 mm X 100 mm), 5 µ
Injection Volume	2 or 5 µL
Mass Spectrometer	AB Sciex Triple Quad 5500
Ion Source	Turbo Spray
Polarity	Negative
Software	Analyst 1.6.1

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 4/30/14 and 5/5/14.

3 Data Analysis

3.1 Calibration

4/23/14 and 4/25/14 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.

4/30/14 and 5/5/14 Analysis of PFOSA: 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.

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] 4/23/14 Analysis	LOQ, ng/mL [1] 4/25/14 Analysis	LOQ, ng/mL [2] 4/30/14 Analysis	LOQ, ng/mL [2] 5/5/14 Analysis
PFOA	0.0479	NA	NA	NA
PFBS	0.0200	0.0200	NA	NA
PFHS	0.0500	0.0200	NA	NA
PFOS	0.0464	0.0185	NA	NA
PFOSA	0.0200	NA	0.0500	2.00

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 exception of the low level LCSs prepared on 5/5/14 and analyzed externally for PFOSA, which had an average recovery of 74/3%. 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 4/23/14	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-140423-1	0.191	0.193	101	0.199	0.212	107
LCS-140423-2	0.191	0.192	100	0.199	0.209	105
LCS-140423-3	0.191	0.193	101	0.199	0.207	104
Average ± %RSD	**101% ± 0.57%**			**105% ± 1.5%**		
LCS-140423-4	4.74	4.92	104	4.95	5.1	104
LCS-140423-5	4.74	4.95	104	4.95	5.2	104
LCS-140423-6	4.74	4.96	105	4.95	5.2	105
Average ± %RSD	**104% ± 0.56%**			**104% ± 0.56%**		
LCS-140423-7	38.2	41.9	110	39.8	42.8	108
LCS-140423-8	38.2	41.4	108	39.8	42.4	107
LCS-140423-9	38.2	42.4	111	39.8	43.5	109
Average ± %RSD	**110% ± 1.4%**			**108% ± 0.93%**		

ETS-8-044.1 External Calibration Analyzed 4/23/14	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-140423-1	0.199	0.208	105	0.185	0.193	104
LCS-140423-2	0.199	0.216	109	0.185	0.194	105
LCS-140423-3	0.199	0.211	106	0.185	0.202	109
Average ± %RSD	**107% ± 1.9%**			**106% ± 2.5%**		
LCS-140423-4	4.95	5.17	104	4.59	4.84	105
LCS-140423-5	4.95	5.23	106	4.59	4.83	105
LCS-140423-6	4.95	5.34	108	4.59	4.94	108
Average ± %RSD	**106% ± 1.9%**			**106% ± 1.6%**		
LCS-140423-7	39.8	42.8	108	36.9	40.2	109
LCS-140423-8	39.8	41.6	105	36.9	39.7	107
LCS-140423-9	39.8	42.8	108	36.9	40.8	110
Average ± %RSD	**107% ± 1.6%**			**109% ± 1.4%**		

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

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External Calibration Analyzed 4/23/14	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-140423-1	0.199	0.209	105
LCS-140423-2	0.199	0.211	106
LCS-140423-3	0.199	0.211	106
Average ± %RSD	**106% ± 0.54%**		
LCS-140423-4	4.95	5.18	105
LCS-140423-5	4.95	5.41	109
LCS-140423-6	4.95	5.37	108
Average ± %RSD	**107% ± 1.9%**		
LCS-140423-7	39.8	43.0	108
LCS-140423-8	39.8	42.6	107
LCS-140423-9	39.8	44.4	111
Average ± %RSD	**109% ± 1.9%**		

ETS-8-044.1 External Calibration Analyzed 4/23/14	$^{13}C_4$-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-140423-1	0.199	0.218	109	0.190	0.209	110
LCS-140423-2	0.199	0.222	111	0.190	0.208	109
LCS-140423-3	0.199	0.221	111	0.190	0.208	109
Average ± %RSD	**110% ±1.0%**			**109% ± 0.53%**		
LCS-140423-4	4.94	5.39	109	4.73	5.09	108
LCS-140423-5	4.94	5.29	107	4.73	5.12	108
LCS-140423-6	4.94	5.43	110	4.73	5.17	109
Average ± %RSD	**109% ± 1.4%**			**108% ± 0.53%**		

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

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External Calibration Analyzed 4/25/14	PFBS			PFHS		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-140425-1	0.199	0.212	107	0.199	0.196	98.4
LCS-140425-2	0.199	0.195	98.2	0.199	0.200	100
LCS-140425-3	0.199	0.210	105	0.199	0.200	100
Average ± %RSD	**103% ± 4.5%**			**99.5% ± 0.93%**		
LCS-140425-4	4.95	5.08	103	4.95	5.14	104
LCS-140425-5	4.95	5.12	103	4.95	5.25	106
LCS-140425-6	4.95	5.05	102	4.95	5.25	106
Average ± %RSD	**103% ± 0.56%**			**105% ± 1.1%**		
LCS-140425-7	39.8	42.9	108	39.8	43.4	109
LCS-140425-8	39.8	41.3	104	39.8	41.8	105
LCS-140425-9	39.8	41.8	105	39.8	42.5	107
Average ± %RSD	**106% ± 2.0%**			**107% ± 1.9%**		

ETS-8-044.1 External Calibration Analyzed 4/25/14	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-140425-1	0.185	0.179	96.6	0.190	0.195	103
LCS-140425-2	0.185	0.175	94.6	0.190	0.190	99.9
LCS-140425-3	0.185	0.179	96.5	0.190	0.195	103
Average ± %RSD	**95.9% ± 1.2%**			**102% ± 1.8%**		
LCS-140425-4	4.59	4.64	101	4.73	4.84	102
LCS-140425-5	4.59	4.73	103	4.73	4.93	104
LCS-140425-6	4.59	4.68	102	4.73	4.97	105
Average ± %RSD	**102% ± 0.98%**			**104% ± 1.5%**		
LCS-140425-7	36.9	40.2	109			
LCS-140425-8	36.9	38.9	106			
LCS-140425-9	36.9	40.2	109			
Average ± %RSD	**108% ± 1.6%**					

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

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal Calibration Analyzed 4/30/14	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-140428-1	0.198	0.223	112
LCS-140428-2	0.198	0.226	114
LCS-140428-3	0.198	0.219	111
Average ± %RSD	**112% ± 1.4%**		
LCS-140428-4	19.8	20.1	102
LCS-140428-5	19.8	19.6	99.2
LCS-140428-6	19.8	20.1	101
Average ± %RSD	**101% ± 1.4%**		
LCS-140428-7	79.0	83.8	106
LCS-140428-8	79.0	80.9	102
LCS-140428-9	79.0	81.5	103
Average ± %RSD	**104% ± 2.0%**		

ETS-8-044.1 External Calibration Analyzed 5/5/14	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-140505-1	1.99	1.44	72.4
LCS-140505-2	1.99	1.48	74.4
LCS-140505-3	1.99	1.51	76.1
Average ± %RSD	**74.3% ± 2.5%** (1)		
LCS-140505-4	19.9	16.3	81.7
LCS-140505-5	19.9	16.8	84.6
LCS-140505-6	19.9	15.3	77.0
Average ± %RSD	**81.1% ± 4.7%**		
LCS-140505-7	79.0	65.9	83.4
LCS-140505-8	79.0	61.0	77.2
LCS-140505-9	79.0	69.2	87.6
Average ± %RSD	**82.7% ± 6.3%**		

(1) 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%. The method uncertainty for PFOSA using external calibration was calculated at ±24%, however; due to the non-compliant low level LCSs analyzed on 5/5/14, the method uncertainty has been expanded to ±26% for samples analyzed using external calibration based on the %bias.

Table 8. Analytical Method Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	7.04	±14%
PFBS	External	9.03	±18%
PFHS	External	5.05	±10%
PFOS	External	4.02	±8.0%
PFOSA	External	NA	±26%
PFOSA	Internal	8.16	±16%

NA = Not Applicable

3.8 Field Matrix Spikes (FMS)

A target analyte field matrix spike sample(s) 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 sample bottle 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 all 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
P114bis, P116, 3M Pond, Blokkersdijkvijver – standard	FMS	10.0	NA	10.0	9.99	10.0
L4	FMS	20.0	NA	20.0	20.0	20.0
12, 13, 5, and Bemalingsstation	FMS	100	NA	100	100	10.0
P321	FMS	2010	NA	2010	2010	10.0
PP01	FMS	501	NA	500	501	NA
PP09, PP10	FMS	501	25.0	500	501	25.0
PP08	FMS	501	50.0	500	501	50.0
PP07	FMS	501	100	500	501	100
PP02, PP04	FMS	1000	25.0	1000	1000	25.0
PP05	FMS	1000	100	1000	1000	100
Trip Blank	Low	10.0	10.0	10.0	9.99	10.0
	Mid	501	50.0	500	501	50.0
	High	1000	100	1000	1000	100

NA = Not Applicable

3.9 Lab Matrix Spikes (LMS)

The field matrix spike solution used to prepare the field matrix spike for sampling location PP01did not contain PFOSA or PFBS. Due to the higher RPD value for PFOSA for this location, 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 at 49.8 ng/mL for PFOSA. The lab matrix spike results are presented in section 4 of this report.

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

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

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.

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:

P321: Sample/sample duplicate RPD was 22% for PFHS and 28% for PFOSA.

PP01: Sample/sample duplicate RPD was 21% for PFHS, 23% for PFOS, and 24% for PFOSA.

PP04: The FMS recovery for PFHS was 65.5%. The method uncertainty has been expanded for PFHS to ± 35%, based on the recovery of the FMS sample.

PP05: The FMS recovery for PFOSA was 148%. The method uncertainty has been expanded for PFOSA to ± 48%, based on the recovery of the FMS sample. Sample/sample duplicate RPD was 36% for PFOSA and 42% for PFOS. Samples were re-analyzed for PFOSA to confirm the results. The results from the initial analysis are reported.

PP10: Sample/sample duplicate RPD was 32% for PFOSA.

The PFOSA sample result for location P114bis; Sample Dup (ISO14-0010-008) 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. In addition, the sample target analyte peak area counts were also less than twice the average area counts for the method blanks, making the sample result not reportable.

Table 10. Location ID: P321

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-001	P321; Sample	1940	NA	674	NA
ISO14-0010-002	P321; Sample Dup	1590	NA	539	NA
ISO14-0010-003	P321; FMS	3680	95.3	2570	97.7
Average Concentration (ng/mL) ± %RPD		**1770 ng/mL ± 20%**		**607 ng/mL ± 22%** (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
ISO14-0010-001	P321; Sample	3620	NA	10.3	NA	90.8	84.1
ISO14-0010-002	P321; Sample Dup	3810	NA	13.7	NA	93.4	87.5
ISO14-0010-003	P321; FMS	5560	91.8	21.3	93.0	91.1	91.2
Average Concentration (ng/mL) ± %RPD		**3720 ng/mL ± 5.1%**		**12.0 ng/mL ± 28%** (1)		**91.8% ± 1.5%**	**87.6% ± 4.1%**

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

Table 11. Location ID: L4

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-004	L4; Sample	2.57	NA	2.04	NA
ISO14-0010-005	L4; Sample Dup	2.62	NA	2.09	NA
ISO14-0010-006	L4; FMS	24.3	109	22.7	103
Average Concentration (ng/mL) ± %RPD		**2.60 ng/mL ± 1.9%**		**2.07 ng/mL ± 2.4%**	

		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
ISO14-0010-004	L4; Sample	23.7	NA	2.33	NA	92.1	90.1
ISO14-0010-005	L4; Sample Dup	22.7	NA	2.33	NA	96.5	93.7
ISO14-0010-006	L4; FMS	44.0	104	18.6	81.4	93.8	94.9
Average Concentration (ng/mL) ± %RPD		**23.2 ng/mL ± 4.3%**		**2.33 ng/mL ± 0.0%**		**94.1% ± 2.4%**	**92.9% ± 2.7%**

NA = Not Applicable

Table 12. Location ID: P114bis

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0010-007	P114bis; Sample	4.15	NA	1.67	NA
ISO14-0010-008	P114bis; Sample Dup	4.05	NA	1.67	NA
ISO14-0010-009	P114bis; FMS	15.8	117	12.7	110
Average Concentration (ng/mL) ± %RPD		**4.10 ng/mL ± 2.4%**		**1.67 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**
ISO14-0010-007	P114bis; Sample	16.1	NA	0.0735	NA	96.7	92.8
ISO14-0010-008	P114bis; Sample Dup	16.4	NA	NR (1)	NA	98.2	93.5
ISO14-0010-009	P114bis; FMS	27.3	110	10.1	100	100	94.4
Average Concentration (ng/mL) ± %RPD		**16.3 ng/mL ± 1.8%**		**0.0735 ng/mL**		**98.3% ± 1.7%**	**93.6% ± 0.86%**

NA = Not Applicable
(1) NR = Not reportable; analyte area counts below the area counts of the LOQ and sample did not meet method blank criteria.

Table 13. Location ID: P116

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0010-010	P116; Sample	2.02	NA	0.795	NA
ISO14-0010-011	P116; Sample Dup	1.94	NA	0.773	NA
ISO14-0010-012	P116; FMS	12.3	103	10.7	99.2
Average Concentration (ng/mL) ± %RPD		**1.98 ng/mL ± 4.0%**		**0.784 ng/mL ± 2.8%**	

		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**
ISO14-0010-010	P116; Sample	7.76	NA	0.0916	NA	95.5	94.5
ISO14-0010-011	P116; Sample Dup	7.16	NA	0.0921	NA	97.8	92.4
ISO14-0010-012	P116; FMS	17.1	96.5	9.57	94.8	100	93.7
Average Concentration (ng/mL) ± %RPD		**7.46 ng/mL ± 8.0%**		**0.0919 ng/mL ± 0.54%**		**97.8% ± 2.3%**	**93.5% ± 1.1%**

NA = Not Applicable

Table 14. Location ID: 3M Pond

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-013	3M pond; Sample	1.63	NA	0.728	NA
ISO14-0010-014	3M pond; Sample Dup	1.77	NA	0.776	NA
ISO14-0010-015	3M pond; FMS	12.5	108	11.0	102
Average Concentration (ng/mL) ± %RPD		**1.70 ng/mL ± 8.2%**		**0.752 ng/mL ± 6.4%**	

		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
ISO14-0010-013	3M pond; Sample	4.20	NA	<0.0500	NA	98.4	94.1
ISO14-0010-014	3M pond; Sample Dup	3.80	NA	<0.0500	NA	97.5	96.8
ISO14-0010-015	3M pond; FMS	14.5	105	9.82	98.2	112	106
Average Concentration (ng/mL) ± %RPD		**4.00 ng/mL ± 10%**		**<0.0500 ng/mL**		**103% ± 7.9%**	**99.0% ± 6.3%**

NA = Not Applicable

Table 15. Location ID: Blokkersdijkvijver std

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-016	Blokkersdijkvijver std; Sample	1.33	NA	0.678	NA
ISO14-0010-017	Blokkersdijkvijver std; Sample Dup	1.29	NA	0.653	NA
ISO14-0010-018	Blokkersdijkvijver std; FMS	13.8	125	11.7	110
Average Concentration (ng/mL) ± %RPD		**1.31 ng/mL ± 3.1%**		**0.666 ng/mL ± 3.8%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-016	Blokkersdijkvijver std; Sample	1.19	NA	<0.0500	NA
ISO14-0010-017	Blokkersdijkvijver std; Sample Dup	1.15	NA	<0.0500	NA
ISO14-0010-018	Blokkersdijkvijver std; FMS	12.2	110	9.81	98.1
Average Concentration (ng/mL) ± %RPD		**1.17 ng/mL ± 3.4%**		**<0.0500 ng/mL**	

		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	Sample Description	%Recovery	%Recovery
ISO14-0010-016	Blokkersdijkvijver std; Sample	114	105
ISO14-0010-017	Blokkersdijkvijver std; Sample Dup	110	105
ISO14-0010-018	Blokkersdijkvijver std; FMS	122	111
Average Concentration (ng/mL) ± %RPD		**115% ± 5.3%**	**107% ± 3.2%**

NA = Not Applicable

Table 16. Location ID: 12

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-019	12; Sample	120	NA	31.4	NA
ISO14-0010-020	12; Sample Dup	126	NA	31.9	NA
ISO14-0010-021	12; FMS	238	115	137	105
Average Concentration (ng/mL) ± %RPD		**123 ng/mL ± 4.9%**		**31.7 ng/mL ± 1.6%**	

		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
ISO14-0010-019	12; Sample	329	NA	0.328	NA	115	105
ISO14-0010-020	12; Sample Dup	313	NA	0.297	NA	113	104
ISO14-0010-021	12; FMS	435	NC	8.43	81.2	113	100
Average Concentration (ng/mL) ± %RPD		**321 ng/mL ± 5.0%**		**0.313 ng/mL ± 9.9%**		**114% ± 1.0%**	**103% ± 2.6%**

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

Table 17. Location ID: 13

		PFOA		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-022	13; Sample	113	NA	30.6	NA
ISO14-0010-023	13; Sample Dup	108	NA	29.7	NA
ISO14-0010-024	13; FMS	215	104	126	95.8
Average Concentration (ng/mL) ± %RPD		**111 ng/mL ± 4.5%**		**30.2 ng/mL ± 3.0%**	

		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
ISO14-0010-022	13; Sample	292	NA	0.330	NA	114	106
ISO14-0010-023	13; Sample Dup	290	NA	0.349	NA	112	104
ISO14-0010-024	13; FMS	395	NC	8.45	81.1	110	99.2
Average Concentration (ng/mL) ± %RPD		**291 ng/mL ± 0.69%**		**0.340 ng/mL ± 5.6%**		**112% ± 1.8%**	**103% ± 3.4%**

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

Table 18. Location ID: 5

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0010-025	5; Sample	48.2	NA	31.6	NA
ISO14-0010-026	5; Sample Dup	47.8	NA	32.0	NA
ISO14-0010-027	5; FMS	139	91.0	114	82.2
Average Concentration (ng/mL) ± %RPD		**48.0 ng/mL ± 0.83%**		**31.8 ng/mL ± 1.3%**	

		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**
ISO14-0010-025	5; Sample	81.0	NA	0.230	NA	116	103
ISO14-0010-026	5; Sample Dup	81.1	NA	0.199	NA	115	106
ISO14-0010-027	5; FMS	165	83.9	10.0	97.9	113	101
Average Concentration (ng/mL) ± %RPD		**81.1 ng/mL ± 0.12%**		**0.215 ng/mL ± 14%**		**115% ± 1.3%**	**103% ± 2.4%**

NA = Not Applicable

Table 19. Location ID: Bemlingsstation

		PFOA		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0010-028	Bemalingsstation; Sample	37.6	NA	25.6	NA
ISO14-0010-029	Bemalingsstation; Sample Dup	38.4	NA	25.6	NA
ISO14-0010-030	Bemalingsstation; FMS	149	111	126	100
Average Concentration (ng/mL) ± %RPD		**38.0 ng/mL ± 2.1%**		**25.6 ng/mL ± 0.0%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0010-028	Bemalingsstation; Sample	66.9	NA	0.244	NA
ISO14-0010-029	Bemalingsstation; Sample Dup	64.3	NA	0.220	NA
ISO14-0010-030	Bemalingsstation; FMS	169	103	9.81	95.8
Average Concentration (ng/mL) ± %RPD		**65.6 ng/mL ± 4.0%**		**0.232 ng/mL ± 10%**	

		$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
3M LIMS ID	**Sample Description**	**%Recovery**	**%Recovery**
ISO14-0010-028	Bemalingsstation; Sample	114	102
ISO14-0010-029	Bemalingsstation; Sample Dup	110	101
ISO14-0010-030	Bemalingsstation; FMS	113	103
Average Concentration (ng/mL) ± %RPD		**112% ± 1.9%**	**102% ± 0.98%**

NA = Not Applicable

Table 20. Location ID: PP01

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-031	PP01; Sample	73.4	NA	79.5	NA	10700	NA
ISO14-0010-032	PP01; Sample Dup	73.9	NA	81.8	NA	13200	NA
ISO14-0010-033	PP01; FMS	627	110	83.2	NA (1)	NA (2)	NA (2)
Average Concentration (ng/mL) ± %RPD		**73.7 ng/mL ± 0.68%**		**80.7 ng/mL ± 2.9%**		**12000 ng/mL ± 21% (3)**	

		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
ISO14-0010-031	PP01; Sample	4100	NA	24.3	NA	107	102
ISO14-0010-032	PP01; Sample Dup	5150	NA	30.9	NA	107	104
ISO14-0010-033	PP01; FMS	NA (2)	NA (2)	NA (1)	NA (1)	110	NA (2)
ISO14-0010-031; LMS	PP01; Sample LMS	NA	NA	69.6	91.0	NA	NA
Average Concentration (ng/mL) ± %RPD		**4630 ng/mL ± 23% (3)**		**27.6 ng/mL ± 24% (3)**		**108% ± 1.6%**	**103% ± 1.9%**

NA = Not Applicable
(1) The field matrix spike did not contain PFBS or PFOSA. A lab matrix spike was prepared for PFOSA only.
(2) Sample concentration exceeded calibration range; sample was not re-analyzed since field matrix spike level was not appropriate.
(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 21. Location ID: PP02

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-034	PP02; Sample	701	NA	25.7	NA	2520	NA
ISO14-0010-035	PP02; Sample Dup	669	NA	24.0	NA	2380	NA
ISO14-0010-036	PP02; FMS	1540	85.5	45.5	82.4	3320	NC
Average Concentration (ng/mL) ± %RPD		**685 ng/mL ± 4.7%**		**24.9 ng/mL ± 6.8%**		**2450 ng/mL ± 5.7%**	

		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
ISO14-0010-034	PP02; Sample	23100	NA	25.7	NA	95.3	105
ISO14-0010-035	PP02; Sample Dup	24800	NA	24.4	NA	96.6	106
ISO14-0010-036	PP02; FMS	NA (1)	NA (1)	44.2	76.4	96.4	NA (1)
Average Concentration (ng/mL) ± %RPD		**24000 ng/mL ± 7.1%**		**25.1 ng/mL ± 5.2%**		**96.1% ± 0.73%**	**106% ± 0.94%**

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

Table 22. Location ID: PP04

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-037	PP04; Sample	1960	NA	2400	NA	782	NA
ISO14-0010-038	PP04; Sample Dup	2130	NA	2620	NA	927	NA
ISO14-0010-039	PP04; FMS	2680	NC	2420	NC	1510	65.5 (1)
Average Concentration (ng/mL) ± %RPD		**2050 ng/mL ± 8.3%**		**2510 ng/mL ± 8.8%**		**855 ng/mL ± 17%**	

		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
ISO14-0010-037	PP04; Sample	7640	NA	42.4	NA	95.6	109
ISO14-0010-038	PP04; Sample Dup	8630	NA	39.4	NA	94.4	110
ISO14-0010-039	PP04; FMS	NA (2)	NA (2)	73.3	130	94.1	NA (2)
Average Concentration (ng/mL) ± %RPD		**8140 ng/mL ± 12%**		**40.9 ng/mL ± 7.3%**		**94.7% ± 0.84%**	**110% ± 0.91%**

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%. Method uncertainty has been expanded to ±35%.
(2) Sample concentration exceeded calibration range; sample was not re-analyzed since field matrix spike level was not appropriate.

Table 23. Location ID: PP05

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-040	PP05; Sample	1760	NA	39500	NA	4730	NA
ISO14-0010-041	PP05; Sample Dup	1830	NA	44800	NA	4510	NA
ISO14-0010-042	PP05; FMS	2670	87.5	NA (1)	NA (1)	NA (1)	NA (1)
Average Concentration (ng/mL) ± %RPD		**1800 ng/mL ± 3.9%**		**42200 ng/mL ± 13%**		**4620 ng/mL ± 4.8%**	

		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
ISO14-0010-040	PP05; Sample	200	NA	45.0	NA	96.5	84.6
ISO14-0010-041	PP05; Sample Dup	130	NA	31.4	NA	96.8	83.6
ISO14-0010-042	PP05; FMS	1310	115	186	148 (2)	95.4	85.5
Average Concentration (ng/mL) ± %RPD		**165 ng/mL ± 42% (3)**		**38.2 ng/mL ± 36% (3)**		**96.2% ± 0.77%**	**84.6% ± 1.1%**

NA = Not Applicable
(1) Sample concentration exceeded calibration range; sample was not re-analyzed since field matrix spike level was not appropriate.
(2) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%. Method uncertainty has been expanded to ±48%.
(3) Sample/sample duplicate RPD did not meet acceptance criteria of ≤20%.

Table 24. Location ID: PP07

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-046	PP07; Sample	537	NA	140	NA	142	NA
ISO14-0010-047	PP07; Sample Dup	550	NA	131	NA	141	NA
ISO14-0010-048	PP07; FMS	1030	97.0	216	80.0	592	90.0
Average Concentration (ng/mL) ± %RPD		**544 ng/mL ± 2.4%**		**136 ng/mL ± 6.6%**		**142 ng/mL ± 0.70%**	

		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
ISO14-0010-046	PP07; Sample	3790	NA	73.3	NA	98.3	83.4
ISO14-0010-047	PP07; Sample Dup	3890	NA	72.1	NA	98.1	86.1
ISO14-0010-048	PP07; FMS	4200	NC	148	75.3	98.0	82.8
Average Concentration (ng/mL) ± %RPD		**3840 ng/mL ± 2.6%**		**72.7 ng/mL ± 1.7%**		**98.1% ± 0.16%**	**84.1% ± 2.1%**

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

Table 25. Location ID: PP08

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-049	PP08; Sample	721	NA	43.4	NA	316	NA
ISO14-0010-050	PP08; Sample Dup	718	NA	41.8	NA	322	NA
ISO14-0010-051	PP08; FMS	1270	110	93.2	101	819	100
Average Concentration (ng/mL) ± %RPD		**720 ng/mL ± 0.42%**		**42.6 ng/mL ± 3.8%**		**319 ng/mL ± 1.9%**	

		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
ISO14-0010-049	PP08; Sample	9220	NA	52.1	NA	95.8	109
ISO14-0010-050	PP08; Sample Dup	9120	NA	51.5	NA	97.2	112
ISO14-0010-051	PP08; FMS	NA (1)	NA (1)	91.2	78.8	98.4	NA (1)
Average Concentration (ng/mL) ± %RPD		**9170 ng/mL ± 1.1%**		**51.8 ng/mL ± 1.2%**		**97.1% ± 1.3%**	**111% ± 2.7%**

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

Table 26. Location ID: PP09

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-052	PP09; Sample	870	NA	32.4	NA	475	NA
ISO14-0010-053	PP09; Sample Dup	846	NA	30.9	NA	460	NA
ISO14-0010-054	PP09; FMS	1400	108	56.1	97.6	968	100
Average Concentration (ng/mL) ± %RPD		**858 ng/mL ± 2.8%**		**31.7 ng/mL ± 4.7%**		**468 ng/mL ± 3.2%**	

		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
ISO14-0010-052	PP09; Sample	2710	NA	16.7	NA	97.4	90.1
ISO14-0010-053	PP09; Sample Dup	2570	NA	14.6	NA	98.1	87.1
ISO14-0010-054	PP09; FMS	3160	NC	36.0	81.2	97.6	88.8
Average Concentration (ng/mL) ± %RPD		**2640 ng/mL ± 5.3%**		**15.7 ng/mL ± 13%**		**97.7% ± 0.37%**	**88.7% ± 1.7%**

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

Table 27. Location ID: PP10

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-055	PP10; Sample	781	NA	23.1	NA	210	NA
ISO14-0010-056	PP10; Sample Dup	789	NA	23.5	NA	213	NA
ISO14-0010-057	PP10; FMS	1350	113	47.9	98.4	696	96.8
Average Concentration (ng/mL) ± %RPD		**785 ng/mL ± 1.0%**		**23.3 ng/mL ± 1.7%**		**212 ng/mL ± 1.4%**	

		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
ISO14-0010-055	PP10; Sample	4700	NA	13.4	NA	97.6	110
ISO14-0010-056	PP10; Sample Dup	4810	NA	18.5	NA	97.3	111
ISO14-0010-057	PP10; FMS	NA (1)	NA (1)	36.0	80.0	96.9	NA (1)
Average Concentration (ng/mL) ± %RPD		**4760 ng/mL ± 2.3%**		**16.0 ng/mL ± 32% (2)**		**97.3% ± 0.36%**	**111% ± 0.90%**

NA = Not Applicable
(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 28. Trip Blank

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0010-058	Trip Blank; Sample	<0.479	NA	<0.200	NA	<0.500	NA
ISO14-0010-059	Trip Blank; FMS Low	11.8	118	10.9	109	10.8	108
ISO14-0010-060	Trip Blank; FMS Mid	569	114	51.1	102	506	101
ISO14-0010-061	Trip Blank; FMS High	1160	116	105	105	1020	102

		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
ISO14-0010-058	Trip Blank; Sample	<0.464	NA	<0.200	NA	113	105
ISO14-0010-059	Trip Blank; FMS Low	10.9	109	8.80	88.0	118	104
ISO14-0010-060	Trip Blank; FMS Mid	516	103	43.2	86.4	101	91.1
ISO14-0010-061	Trip Blank; FMS High	1070	107	87.2	87.2	100	93.3

NA = Not Applicable

5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M 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 10-28 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: 2014.05.12 07:20:06 -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: 2014.05.12 14:02:49 -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 Daryl K. Peterson
DN: c=US, st=MN, l=St. Paul, ou=3M Environmental Laboratory - authenticated by LRA, [redacted] o=3M, cn=Daryl K. Peterson
Reason: I have reviewed this document
Date: 2014.05.12 13:16:29 -05'00'

Quality Assurance Representative

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SGS

3M EHS
Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC14-01522

3M Lab Request Number: E14-0234

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

TESTING LABORATORY

SGS Belgium
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Author: Sandra Graré
Date issued: April 18th, 2014

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All orders are executed only in accordance with our General Conditions, deposited with the Antwerp Chamber of Commerce and Industry.

1. Introduction - Summary.

At the request (Lab Request Number: E14-0234) of Robert A. Paschke, 3M Manager Corporate Environmental Programs, water samples were collected on April 2nd 2014 from 2 locations and analyzed for the following perfluorinated compounds:

- PFHS = perfluorohexane sulfonate ($C_6F_{13}SO_3^-$)
- PFOA = perfluorooctanoate ($C_7F_{15}COO^-$)
- Surrogate Spike = ^{13}C-labeled analogue of PFOA ($C_6F_{13}{}^{13}CF_2{}^{13}COO^-$)
- PFOS = perfluorooctane sulfonate ($C_8F_{17}SO_3^-$)
- Surrogate Spike = ^{13}C-labeled analogue of PFOS ($C_7F_{15}{}^{13}CF_2SO_3^-$)
- FOSA = perfluorooctane sulfonamide ($C_8F_{17}\ SO_2NH_2$)

Professional services personnel at the SGS Belgium NV, division IAC laboratory prepared sample containers under the direction of Geert De Smet. For each sampling location, one field sample container was prepared. There were no spikes added to the sampling bottles.
Table 1 summarizes the sample results. All PFHS, PFOA, PFOS and FOSA results have an accuracy of ± 30%.

Table 1. Sample Results Summary.

Sample identification	Concentration (in ng/mL, ppb)			
	PFHS	PFOA	PFOS	FOSA
Effluent WWTP	0.883	2.00	1.04	<0.503
Collector Pit	21.4	75.1	310	14.6

2. Methods – Analytical and Preparatory.

2.1. Sample collection.

Samples were collected in polyethylene bottles prepared at the SGS Belgium NV, Division IAC Laboratory.
Table 2 below details the samples collected.

Table 2. Sample Collection.

Sample Identification	Nominal Final Volume Collected (ml)
Effluent WWTP	100
Collector Pit	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 5.0 mL MeOH:LCMS-water (60:40) and shaked for 30 minutes. The dilution was filtered if necessary and transferred to an autosampler vial. The sample dilutions were injected directly into the UPLC/MS/MS, and analyzed versus a solvent curve. Details for the used dilutions are given in table 3.

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
Effluent WWTP	1.0	0.100	Solution A	5.0	-
Collector Pit	1.0	0.100	Solution A	5.0	-
Field Trip Blank	1.0	0.100	Solution A	10.0	-

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

2.3. Analysis.

All solutions and dilutions were analyzed for the PFCs (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
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 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 0.0824 to 0.252 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.503 to 1.54 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					
			PFHS	PFOA	^{13}C-PFOA	PFOS	FOSA	^{13}C-PFOS
07 April 2014	LCS1	80	101	93.4	86.6	114	98.5	85.5

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing 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					
			PFHS	PFOA	^{13}C-PFOA	PFOS	FOSA	^{13}C-PFOS
27 Aug 2013	QC Lab Low inj1	10	99.1	107	102	124	111	103
	QC Lab High inj1	100	98.9	105	95.0	111	96.9	90.6
	QC Lab Low inj2	10	112	120	109	118	122	111
	QC Lab High inj2	100	125	109	98.2	121	120	94.0

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 PFHS, PFOA, PFOS and FOSA.

	PFHS	PFOA	PFOS	FOSA
	ng/ml	ng/ml	ng/ml	ng/ml
Effluent WWTP	0.883	2.00	1.04	<0.503
Collector Pit	21.4	75.1	310	14.6
Field Trip Blank	<1.52	<2.80	<1.60	<0.915

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

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
Effluent WWTP	121	100	127	104
Collector Pit	116	96.2	114	93.6
Field Trip Blank	118	97.8	124	101

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 April 18th, 2014

Marc Van Ryckeghem, Division Manager Date April 18th, 2014

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

Reports are established on behalf of and for the account of the principal, who expressly accepts that these reports purely represent the situation at a given time and that they must always be presented and/or mentioned in their totality and in their particular context. SGS Belgium N.V., issuer of the reports, cannot be held liable for errors or modification of results during electronic or fax transmission. Only the originally signed report is binding.
The analytical report can only be used within the specific context of the order and is only valid for the samples analysed.

Final Report

Groundwater and Surface Water Sample Analysis for PFOA, PFBS, PFHS, PFOS, and PFOSA at 3M Belgium July 2014 Sampling

Laboratory Request Number: ISO14-0020

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]

ACCREDITED
Testing Cert. # 2052.01

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

3M Environmental Laboratory

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

Analytical Report ISO14-0020

Groundwater and Surface Water Sample Analysis at 3M Belgium
July 2014 Sampling

Report Date: Date of Last Signature

1 Introduction/Summary

The 3M Environmental Laboratory prepared and analyzed groundwater and surface water samples collected by Environmental Resources Management (ERM) personnel at the 3M Belgium facility. Samples were collected between July 29, 2014 and August 1, 2014. Samples were returned to the 3M Environmental Laboratory on August 7, 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 ISO14-0020.

The 3M Environmental Laboratory prepared sample containers for seventy-seven 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 fortified with a mass-labeled PFOSA internal standard and surrogate recovery standards $^{13}C_4$PFOA and $^{13}C_4$PFOS prior to being sent to the field for sample collection. Sample locations 12 and P16 were not collected along with samples SSB-3 thru SSB-10. Sample location P14 was replaced with P27 and samples SSB-1 and SSB-2 were collected from PA109B and PA111B, respectively.

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.

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**	**PFBS**	**PFHS**	**PFOS**	**PFOSA**
ISO14-0020-001	BD24-3; Sample	202	12.9	126	1670	2.41
ISO14-0020-002	BD24-3; Sample Dup	201	12.9	128	1640	2.54
	Average	**202**	**12.9**	**127**	**1660**	**2.48**
	%RPD Sample/Sample Dup	**0.50**	**0.0**	**1.6**	**1.8**	**5.3**
ISO14-0020-003	BD24-4; Sample	11.8	20.8	4.68	8.01	0.189
ISO14-0020-004	BD24-4; Sample Dup	11.8	20.8	4.71	8.82	0.205
	Average	**11.8**	**20.8**	**4.70**	**8.42**	**0.197** [2]
	%RPD Sample/Sample Dup	**0.0**	**0.0**	**0.64**	**9.6**	**8.1**
ISO14-0020-006	D09; Sample	243	412	10800	1930	34.8
ISO14-0020-007	D09; Sample Dup	237	414	10800	1920	40.8
	Average	**240**	**413**	**10800**	**1930**	**37.8** [2]
	%RPD Sample/Sample Dup	**2.5**	**0.48**	**0.0**	**0.52**	**16**
ISO14-0020-009	D10; Sample	499	7.43	174	26.6	0.171
ISO14-0020-010	D10; Sample Dup	507	7.39	173	26.9	0.140
	Average	**503**	**7.41**	**174**	**26.8**	**0.156** [2]
	%RPD Sample/Sample Dup	**1.6**	**0.54**	**0.58**	**1.1**	**20**
ISO14-0020-012	D11; Sample	478	19.6	212	548	15.7
ISO14-0020-013	D11; Sample Dup	461	19.3	212	573	15.8
	Average	**470**	**19.5**	**212**	**561**	**15.8** [5]
	%RPD Sample/Sample Dup	**3.6**	**1.5**	**0.0**	**4.5**	**0.63**
ISO14-0020-015	D14; Sample	2.24	0.322	1.44	1.06	0.0838
ISO14-0020-016	D14; Sample Dup	2.24	0.328	1.38	0.992	NR [4]
	Average	**2.24**	**0.325**	**1.41**	**1.03**	**0.0838** [2]
	%RPD Sample/Sample Dup	**0.0**	**1.8**	**4.3**	**6.6**	**NA**
ISO14-0020-018	P118A; Sample	2220	719	4360	2170	5.39
ISO14-0020-019	P118A; Sample Dup	2250	726	4370	2190	5.40
	Average	**2240**	**723**	**4370**	**2180**	**5.40**
	%RPD Sample/Sample Dup	**1.3**	**0.97**	**0.23**	**0.92**	**0.19**
ISO14-0020-020	P118B; Sample	2540	293	4210	12900	3.54
ISO14-0020-021	P118B; Sample Dup	2510	291	4170	12900	4.29
	Average	**2530**	**292**	**4190**	**12900**	**3.92** [2]
	%RPD Sample/Sample Dup	**1.2**	**0.68**	**0.95**	**0.0**	**19**
ISO14-0020-023	P121; Sample	<0.192	4.47	<0.200	3.27	<0.200
ISO14-0020-024	P121; Sample Dup	0.225	4.73	<0.200	3.64	<0.200
	Average	**0.225**	**4.60**	**<0.200**	**3.46**	**<0.200**
	%RPD Sample/Sample Dup	**NA**	**5.7**	**NA**	**11**	**NA**

NA = Not Applicable

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(2) Sample set reported using internal standard calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 9.8%.

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

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

(5) The field matrix spike did not meet acceptance criteria of 100 ± 30%. See section 4 for details.

(6) Sample results are qualitative due to the field matrix spike recovery being less than 50%. 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**
ISO14-0020-025	P321; Sample	3360	477	867	2520	<2.00
ISO14-0020-026	P321; Sample Dup	3320	489	844	2510	<2.00
	Average	**3340**	**483**	**856**	**2520**	**<2.00**
	%RPD Sample/Sample Dup	**1.2**	**2.5**	**2.7**	**0.40**	**NA**
ISO14-0020-027	3M pond; Sample	1.31	2.45	0.429	3.89	<0.200
ISO14-0020-028	3M pond; Sample Dup	1.26	2.40	0.462	3.81	<0.200
	Average	**1.29**	**2.43**	**0.446**	**3.85**	**<0.200**
	%RPD Sample/Sample Dup	**3.9**	**2.1**	**7.4**	**2.1**	**NA**
ISO14-0020-029	Blokkersdijkvijver std; Sample	1.31	1.95	0.594	0.891	<0.200
ISO14-0020-030	Blokkersdijkvijver std;Sample Dup	1.23	1.92	0.597	0.828	<0.200
	Average	**1.27**	**1.94**	**0.596**	**0.860**	**<0.200**
	%RPD Sample/Sample Dup	**6.3**	**1.6**	**0.50**	**7.3**	**NA**
ISO14-0020-031	L21; Sample	0.408	1.75	<0.200	3.64	<0.200
ISO14-0020-032	L21; Sample Dup	0.429	1.72	<0.200	3.52	<0.200
	Average	**0.418**	**1.74**	**<0.200**	**3.58**	**<0.200**
	%RPD Sample/Sample Dup	**4.8**	**1.7**	**NA**	**3.4**	**NA**
ISO14-0020-033	L22; Sample	0.515	2.56	0.221	11.7	0.114
ISO14-0020-034	L22; Sample Dup	0.508	2.45	0.202	12.0	0.106
	Average	**0.512**	**2.51**	**0.212**	**11.9**	**0.110 [2]**
	%RPD Sample/Sample Dup	**1.4**	**4.4**	**9.0**	**2.5**	**7.3**
ISO14-0020-036	L31; Sample	0.932	3.99	0.479	8.04	<0.200
ISO14-0020-037	L31; Sample Dup	0.918	3.67	0.509	8.02	<0.200
	Average	**0.925**	**3.83**	**0.494**	**8.03**	**<0.200**
	%RPD Sample/Sample Dup	**1.5**	**8.4**	**6.1**	**0.25**	**NA**
ISO14-0020-038	L4; Sample	6.65	8.34	4.02	28.1	0.220
ISO14-0020-039	L4; Sample Dup	6.48	8.21	3.95	26.8	0.216
	Average	**6.57**	**8.28**	**3.99**	**27.5**	**0.218**
	%RPD Sample/Sample Dup	**2.6**	**1.6**	**1.8**	**4.7**	**1.8**
ISO14-0020-040	P114bis; Sample	4.95	3.39	1.54	11.0	<0.200
ISO14-0020-041	P114bis; Sample Dup	4.90	3.22	1.52	11.9	<0.200
	Average	**4.93**	**3.31**	**1.53**	**11.5**	**<0.200**
	%RPD Sample/Sample Dup	**1.0**	**5.1**	**1.3**	**7.9**	**NA**
ISO14-0020-042	P115; Sample	3.75	0.838	1.75	<0.464	<0.200
ISO14-0020-043	P115; Sample Dup	3.79	0.838	1.76	<0.464	<0.200
	Average	**3.77**	**0.838**	**1.76**	**<0.464**	**<0.200**
	%RPD Sample/Sample Dup	**1.1**	**0.0**	**0.57**	**NA**	**NA**

NA = Not Applicable

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(2) Sample set reported using internal standard calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 9.8%.

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

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

(5) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%. See section 4 for details.

(6) Sample results are qualitative due to the field matrix spike recovery being less than 50%. 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**
ISO14-0020-044	P116; Sample	1.51	4.12	0.716	7.61	<0.200
ISO14-0020-045	P116; Sample Dup	1.47	3.94	0.575	7.23	<0.200
	Average	**1.49**	**4.03**	**0.646**	**7.42**	**<0.200**
	%RPD Sample/Sample Dup	**2.7**	**4.5**	**22** [3]	**5.1**	**NA**
ISO14-0020-046	P119C; Sample	335	8.75	183	2200	37.5
ISO14-0020-047	P119C; Sample Dup	336	8.84	186	2290	36.9
	Average	**336**	**8.80**	**185**	**2250**	**37.2**
	%RPD Sample/Sample Dup	**0.30**	**1.0**	**1.6**	**4.0**	**1.6**
ISO14-0020-048	PP01; Sample	55.9	18.0	28300	8930	63.0
ISO14-0020-049	PP01; Sample Dup	54.4	17.9	27000	8780	71.2
	Average	**55.2**	**18.0**	**27700**	**8860**	**67.1** [5]
	%RPD Sample/Sample Dup	**2.7**	**0.56**	**4.7**	**1.7**	**12**
ISO14-0020-051	PP02; Sample	699	28.6	3150	29000	21.8
ISO14-0020-052	PP02; Sample Dup	704	29.7	3280	25200	22.9
	Average	**702**	**29.2**	**3220**	**27100**	**22.4**
	%RPD Sample/Sample Dup	**0.71**	**3.8**	**4.0**	**14**	**4.9**
ISO14-0020-053	PP04; Sample	1740	2760	904	10600	NR
ISO14-0020-054	PP04; Sample Dup	1680	2630	879	11000	NR
	Average	**1710**	**2700**	**892**	**10800**	**NR** [4]
	%RPD Sample/Sample Dup	**3.5**	**4.8**	**2.8**	**3.7**	**NA**
ISO14-0020-055	PP05; Sample	1530	16500	5500	310	61.6
ISO14-0020-056	PP05; Sample Dup	1560	16800	5510	316	66.3
	Average	**1550**	**16700**	**5510**	**313**	**64.0**
	%RPD Sample/Sample Dup	**1.9**	**1.8**	**0.18**	**1.9**	**7.3**
ISO14-0020-058	PP06; Sample	199	16.0	47.8	2030	25.6
ISO14-0020-059	PP06; Sample Dup	196	14.9	49.8	2100	25.6
	Average	**198**	**15.5**	**48.8**	**2070**	**25.6** [5]
	%RPD Sample/Sample Dup	**1.5**	**7.1**	**4.1**	**3.4**	**0.0**
ISO14-0020-061	PP07; Sample	457	121	114	3070	44.1
ISO14-0020-062	PP07; Sample Dup	455	120	113	3120	49.8
	Average	**456**	**121**	**114**	**3100**	**47.0**
	%RPD Sample/Sample Dup	**0.44**	**0.83**	**0.88**	**1.6**	**12**
ISO14-0020-063	PP08; Sample	678	34.5	319	8990	63.0
ISO14-0020-064	PP08; Sample Dup	657	33.4	310	8590	49.5
	Average	**668**	**34.0**	**315**	**8790**	**56.3**
	%RPD Sample/Sample Dup	**3.1**	**3.2**	**2.9**	**4.6**	**24** [3]

NA = Not Applicable

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(5) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%. See section 4 for details.

(6) Sample results are qualitative due to the field matrix spike recovery being less than 50%. 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**
ISO14-0020-065	PP09; Sample	815	28.5	449	2580	10.3
ISO14-0020-066	PP09; Sample Dup	827	28.2	435	2560	10.4
	Average	**821**	**28.4**	**442**	**2570**	**10.4**
	%RPD Sample/Sample Dup	**1.5**	**1.1**	**3.2**	**0.78**	**0.97**
ISO14-0020-067	PP10; Sample	684	18.5	191	4220	11.9
ISO14-0020-068	PP10; Sample Dup	677	17.9	191	4320	12.0
	Average	**681**	**18.2**	**191**	**4270**	**12.0**
	%RPD Sample/Sample Dup	**1.0**	**3.3**	**0.0**	**2.3**	**0.84**
ISO14-0020-071	13; Sample	93.0	4.99	25.4	207	<0.200
ISO14-0020-072	13; Sample Dup	93.6	5.07	25.8	204	<0.200
	Average	**93.3**	**5.03**	**25.6**	**206**	**<0.200**
	%RPD Sample/Sample Dup	**0.64**	**1.6**	**1.6**	**1.5**	**NA**
ISO14-0020-073	5; Sample	32.1	4.20	21.6	55.0	<0.200
ISO14-0020-074	5; Sample Dup	31.4	4.17	21.2	50.9	<0.200
	Average	**31.8**	**4.19**	**21.4**	**53.0**	**<0.200**
	%RPD Sample/Sample Dup	**2.2**	**0.72**	**1.9**	**7.7**	**NA**
ISO14-0020-075	Bemalingsstation; Sample	37.4	4.95	26.0	60.5	<0.200
ISO14-0020-076	Bemalingsstation; Sample Dup	37.2	5.01	25.7	62.6	<0.200
	Average	**37.3**	**4.98**	**25.9**	**61.6**	**<0.200**
	%RPD Sample/Sample Dup	**0.54**	**1.2**	**1.2**	**3.4**	**NA**
ISO14-0020-077	K1; Sample	2.56	4.45	13.2	16.1	2.03
ISO14-0020-078	K1; Sample Dup	2.52	4.44	12.9	15.8	2.03
	Average	**2.54**	**4.45**	**13.1**	**16.0**	**2.03 [6]**
	%RPD Sample/Sample Dup	**1.6**	**0.22**	**2.3**	**1.9**	**0.0**
ISO14-0020-080	K3; Sample	103	846	55700	3320	20.6
ISO14-0020-081	K3; Sample Dup	99.5	785	53100	3230	23.1
	Average	**101**	**816**	**54400**	**3280**	**21.9 [2]**
	%RPD Sample/Sample Dup	**3.5**	**7.5**	**4.8**	**2.7**	**11**
ISO14-0020-083	P21B; Sample	14000	5360	10700	82700	12.2
ISO14-0020-084	P21B; Sample Dup	14100	5280	10600	72800	16.0
	Average	**14100**	**5320**	**10700**	**77800**	**14.1 [2, 5]**
	%RPD Sample/Sample Dup	**0.71**	**1.5**	**0.94**	**13**	**27 [3]**
ISO14-0020-086	P304; Sample	585	130	90.5	878	16.0
ISO14-0020-087	P304; Sample Dup	582	128	89.4	857	14.7
	Average	**584**	**129**	**90.0**	**868**	**15.4**
	%RPD Sample/Sample Dup	**0.51**	**1.6**	**1.2**	**2.4**	**8.5**

NA = Not Applicable

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(6) Sample results are qualitative due to the field matrix spike recovery being less than 50%. 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**
ISO14-0020-089	P305; Sample	290	130	318	654	9.45
ISO14-0020-090	P305; Sample Dup	279	126	306	666	10.2
	Average	**285**	**128**	**312 [5]**	**660**	**9.83 [5]**
	%RPD Sample/Sample Dup	**3.9**	**3.1**	**3.8**	**1.8**	**7.6**
ISO14-0020-092	P42; Sample	533	46.6	510	4760	14.6
ISO14-0020-093	P42; Sample Dup	582	44.7	500	5130	16.6
	Average	**558 [5]**	**45.7 [5]**	**505 [5]**	**4950**	**15.6 [5]**
	%RPD Sample/Sample Dup	**8.8**	**4.2**	**2.0**	**7.5**	**13**
ISO14-0020-095	P56; Sample	60.3	10.6	16.0	183	7.76
ISO14-0020-096	P56; Sample Dup	59.7	10.7	16.0	184	7.99
	Average	**60.0**	**10.7**	**16.0**	**184**	**7.88**
	%RPD Sample/Sample Dup	**1.0**	**0.94**	**0.0**	**0.54**	**2.9**
ISO14-0020-098	P2; Sample	1810	55.2	221	2260	108
ISO14-0020-099	P2; Sample Dup	1760	55.7	222	2210	112
	Average	**1790**	**55.5**	**222**	**2240**	**110**
	%RPD Sample/Sample Dup	**2.8**	**0.90**	**0.45**	**2.2**	**3.6**
ISO14-0020-101	P27; Sample	370	6190	48.4	2300	107
ISO14-0020-102	P27; Sample Dup	372	6260	48.3	2290	106
	Average	**371**	**6230**	**48.4**	**2300**	**107**
	%RPD Sample/Sample Dup	**0.54**	**1.1**	**0.21**	**0.44**	**0.94**
ISO14-0020-104	P1065; Sample	9.14	12.5	3.08	63.7	3.88
ISO14-0020-105	P1065; Sample Dup	9.43	12.0	3.20	65.5	4.05
	Average	**9.29 [5]**	**12.3**	**3.14**	**64.6**	**3.97 [5]**
	%RPD Sample/Sample Dup	**3.1**	**4.1**	**3.8**	**2.8**	**4.3**
ISO14-0020-110	L19; Sample	263	18.0	153	3500	49.4
ISO14-0020-111	L19; Sample Dup	270	19.5	158	3490	46.8
	Average	**267**	**18.8**	**156**	**3500**	**48.1**
	%RPD Sample/Sample Dup	**2.6**	**8.0**	**3.2**	**0.29**	**5.4**
ISO14-0020-113	M4; Sample	375	111	142	5730	140
ISO14-0020-114	M4; Sample Dup	334	114	127	5090	110
	Average	**355**	**113**	**135**	**5410**	**125**
	%RPD Sample/Sample Dup	**12**	**2.7**	**11**	**12**	**24 [3]**
ISO14-0020-116	P118C; Sample	115	5.21	41.2	1510	55.3
ISO14-0020-117	P118C; Sample Dup	117	5.62	41.2	1520	51.1
	Average	**116**	**5.42**	**41.2**	**1520**	**53.2 [5]**
	%RPD Sample/Sample Dup	**1.7**	**7.6**	**0.0**	**0.66**	**7.9**

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(2) Sample set reported using internal standard calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 9.8%.

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(5) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%. See section 4 for details.

(6) Sample results are qualitative due to the field matrix spike recovery being less than 50%. 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**
ISO14-0020-119	P262; Sample	661	22.0	272	2220	21.9
ISO14-0020-120	P262; Sample Dup	644	21.9	268	2150	20.5
	Average	**653**	**22.0**	**270**	**2190**	**21.2**
	%RPD Sample/Sample Dup	**2.6**	**0.46**	**1.5**	**3.2**	**6.6**
ISO14-0020-122	P263; Sample	2820	87.8	947	12100	8.34
ISO14-0020-123	P263; Sample Dup	2830	84.3	922	12300	8.32
	Average	**2830**	**86.1**	**935**	**12200**	**8.33** [5]
	%RPD Sample/Sample Dup	**0.35**	**4.1**	**2.7**	**1.6**	**0.24**
ISO14-0020-125	P264; Sample	493	44.9	350	1850	28.7
ISO14-0020-126	P264; Sample Dup	480	44.1	341	1840	26.7
	Average	**487**	**44.5**	**346**	**1850**	**27.7**
	%RPD Sample/Sample Dup	**2.7**	**1.8**	**2.6**	**0.54**	**7.2**
ISO14-0020-127	P265C; Sample	97.3	5.04	30.1	1030	84.4
ISO14-0020-128	P265C; Sample Dup	101	5.13	31.0	1030	77.9
	Average	**99.2**	**5.09**	**30.6**	**1030**	**81.2**
	%RPD Sample/Sample Dup	**3.7**	**1.8**	**2.9**	**0.0**	**8.0**
ISO14-0020-129	P340; Sample	836	164	319	3470	26.4
ISO14-0020-130	P340; Sample Dup	799	165	318	3370	26.8
	Average	**818**	**165**	**319**	**3420**	**26.6** [5]
	%RPD Sample/Sample Dup	**4.5**	**0.61**	**0.31**	**2.9**	**1.5**
ISO14-0020-132	P341; Sample	1640	40.9	612	5240	8.81
ISO14-0020-133	P341; Sample Dup	1610	40.0	601	4950	8.95
	Average	**1630**	**40.5**	**607**	**5100**	**8.88**
	%RPD Sample/Sample Dup	**1.8**	**2.2**	**1.8**	**5.7**	**1.6**
ISO14-0020-135	P343; Sample	994	47.6	701	8060	12.0
ISO14-0020-136	P343; Sample Dup	1030	46.5	705	8140	11.7
	Average	**1010**	**47.1**	**703**	**8100**	**11.9**
	%RPD Sample/Sample Dup	**3.6**	**2.3**	**0.57**	**0.99**	**2.5**
ISO14-0020-138	P371; Sample	140	7.14	49.4	799	35.6
ISO14-0020-139	P371; Sample Dup	136	7.20	50.2	799	33.8
	Average	**138**	**7.17**	**49.8**	**799**	**34.7**
	%RPD Sample/Sample Dup	**2.9**	**0.84**	**1.6**	**0.0**	**5.2**
ISO14-0020-140	P374; Sample	481	36.2	461	2010	22.1
ISO14-0020-141	P374; Sample Dup	484	35.0	451	2010	22.2
	Average	**483**	**35.6**	**456**	**2010**	**22.2**
	%RPD Sample/Sample Dup	**0.62**	**3.4**	**2.2**	**0.0**	**0.45**

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 ± 12%, PFHS ± 10%, PFOS ± 12%, and PFOSA ± 38%.

(2) Sample set reported using internal standard calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 9.8%.

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

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

(5) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%. See section 4 for details.

(6) Sample results are qualitative due to the field matrix spike recovery being less than 50%. 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**
ISO14-0020-142	P379; Sample	128	4.26	33.3	1090	32.3
ISO14-0020-143	P379; Sample Dup	131	3.77	34.1	1110	35.2
	Average	**130**	**4.02**	**33.7**	**1100**	**33.8**
	%RPD Sample/Sample Dup	**2.3**	**12**	**2.4**	**1.8**	**8.6**
ISO14-0020-145	P380; Sample	534	23.2	439	1700	62.2
ISO14-0020-146	P380; Sample Dup	567	25.1	482	1750	63.9
	Average	**551**	**24.2**	**461**	**1730**	**63.1 [6]**
	%RPD Sample/Sample Dup	**6.0**	**7.9**	**9.3**	**2.9**	**2.7**
ISO14-0020-148	P381; Sample	671	8.63	178	1480	7.95
ISO14-0020-149	P381; Sample Dup	682	9.12	179	1550	7.66
	Average	**677**	**8.88**	**179**	**1520**	**7.81**
	%RPD Sample/Sample Dup	**1.6**	**5.5**	**0.56**	**4.6**	**3.7**
ISO14-0020-150	P382; Sample	417	12.8	101	1000	57.7
ISO14-0020-151	P382; Sample Dup	415	13.1	104	1010	57.8
	Average	**416**	**13.0**	**103**	**1010**	**57.8**
	%RPD Sample/Sample Dup	**0.48**	**2.3**	**2.9**	**1.0**	**0.17**
ISO14-0020-152	B3; Sample	255	9.21	99.3	1260	48.9
ISO14-0020-153	B3; Sample Dup	240	7.62	92.2	1220	44.4
	Average	**248**	**8.42**	**95.8**	**1240**	**46.7 [5]**
	%RPD Sample/Sample Dup	**6.1**	**19**	**7.4**	**3.2**	**9.6**
ISO14-0020-155	B7; Sample	4.91	1.28	1.07	179	0.580
ISO14-0020-156	B7; Sample Dup	4.79	1.34	1.09	187	0.604
	Average	**4.85**	**1.31**	**1.08**	**183**	**0.592**
	%RPD Sample/Sample Dup	**2.5**	**4.6**	**1.9**	**4.4**	**4.1**
ISO14-0020-157	P372; Sample	98.4	7.39	67.1	1750	26.3
ISO14-0020-158	P372; Sample Dup	99.1	7.03	67.3	1730	22.1
	Average	**98.8**	**7.21**	**67.2**	**1740**	**24.2**
	%RPD Sample/Sample Dup	**0.71**	**5.0**	**0.30**	**1.1**	**17**
ISO14-0020-159	P378; Sample	244	13.1	246	750	<2.00
ISO14-0020-160	P378; Sample Dup	240	13.4	248	730	<2.00
	Average	**242**	**13.3**	**247**	**740**	**<2.00**
	%RPD Sample/Sample Dup	**1.7**	**2.3**	**0.81**	**2.7**	**NA**
ISO14-0020-161	PA109A; Sample	89.5	6.51	88.9	240	0.952
ISO14-0020-162	PA109A; Sample Dup	88.6	6.50	89.4	232	1.03
	Average	**89.1**	**6.51**	**89.2**	**236**	**0.991**
	%RPD Sample/Sample Dup	**1.0**	**0.15**	**0.56**	**3.4**	**7.9**

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 ± 12%, PFHS ± 10%, PFOS ± 12%, and PFOSA ± 38%.

(2) Sample set reported using internal standard calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 9.8%.

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

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

(5) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%. See section 4 for details.

(6) Sample results are qualitative due to the field matrix spike recovery being less than 50%. 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**
ISO14-0020-163	PA111A; Sample	918	68.0	1230	2420	11.0
ISO14-0020-164	PA111A; Sample Dup	914	66.7	1210	2360	8.95
	Average	**916**	**67.4**	**1220**	**2390**	**9.98**
	%RPD Sample/Sample Dup	**0.44**	**1.9**	**1.6**	**2.5**	**21 [3]**
ISO14-0020-165	PA112; Sample	258	3.15	45.4	398	29.5
ISO14-0020-166	PA112; Sample Dup	253	3.13	44.4	387	28.0
	Average	**256**	**3.14**	**44.9**	**393**	**28.8**
	%RPD Sample/Sample Dup	**2.0**	**0.64**	**2.2**	**2.8**	**5.2**
ISO14-0020-171	PA109B; Sample	0.903	1.06	0.267	4.74	<0.200
ISO14-0020-172	PA109B; Sample Dup	0.861	1.24	0.242	4.69	<0.200
	Average	**0.882**	**1.15**	**0.255**	**4.72**	**<0.200 [5]**
	%RPD Sample/Sample Dup	**4.8**	**16**	**9.8**	**1.1**	**NA**
ISO14-0020-174	PA111B; Sample	NR	NR	NR	9.65	NR
ISO14-0020-175	PA111B; Sample Dup	NR	NR	NR	10.2	NR
	Average	**NR [4]**	**NR [4]**	**NR [4]**	**9.93**	**NR [4]**
	%RPD Sample/Sample Dup	**NA**	**NA**	**NA**	**5.5**	**NA**

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 ± 12%, PFHS ± 10%, PFOS ± 12%, and PFOSA ± 38%.

(2) Sample set reported using internal standard calibration. The analytical method uncertainty associated with the reported results for PFOSA is ± 9.8%.

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

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

(5) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%. See section 4 for details.

(6) Sample results are qualitative due to the field matrix spike recovery being less than 50%. 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".

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 29, 2014 and August 1, 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 sample locations were spiked with a mass-labeled PFOSA internal standard at a nominal concentration of 1 ng/mL and 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 7, 2014.

2.3 Sample Preparation

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 diluting 0.01 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 for PFOSA using internal standard calibration: 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.

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.

Table 3. Instrument Parameters.

Instrument Name	ETS Kirk
Liquid Chromatograph	Agilent 1260
Analysis Method	ETS-8-044.1
Analysis Date	8/18/14, 8/21/14, 8/25/14, 8/27/14, 9/4/14, 9/5/14, 9/9/14
Guard column	Betasil C18 (4.6 mm X 100 mm), 5 µ
Analytical column	Betasil C18 (4.6 mm X 100 mm), 5 µ
Injection Volume	2 or 5 µL
Mass Spectrometer	AB Sciex Triple Quad 5500
Ion Source	Turbo Spray
Polarity	Negative
Software	Analyst 1.6.1

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 8/27/14, 8/29/14, and 9/4/14.

3 Data Analysis

3.1 Calibration

8/18/14, 8/21/14, 8/25/14, 9/5/14, and 9/9/14 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. Calibrations standards ranging from 0.02 ng/mL to 100 ng/mL (nominal) were analyzed. The standards also contained the surrogates at concentrations ranging from 0.02 ng/mL to 25 ng/mL nominal. A quadratic, 1/x weighted, calibration curve of the ratio of the standard peak area counts over the internal standard peak area counts was used to fit the data for each analyte. The data were not forced through zero during the fitting process. Calculating the standard concentrations using the peak area ratios and the resultant calibration curve confirmed accuracy of each curve

point. The reference standards of PFOA and PFOS used to prepare the calibration standards consisted of both linear and branched isomers.

8/27/14 and 9/4/14 Analysis of PFOSA: Samples were analyzed against a matrix-matched stable isotope internal standard calibration curve. Calibration standards were prepared by spiking known amounts of stock solutions into 50 mL of 50:50 methanol:laboratory reagent water. The calibration standards contained an internal standard mix at a nominal concentration of 0.5 ng/mL. Calibration standards ranging from 0.0125 ng/mL to 150 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.

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] 8/18/14, 8/21/14 Analysis	LOQ, ng/mL [1] 8/25/14 Analysis	LOQ, ng/mL [2] 8/27/14 Analysis	LOQ, ng/mL [2] 9/4/14 Analysis	LOQ, ng/mL [1] 9/5/14 Analysis	LOQ, ng/mL [1] 9/9/14 Analysis
PFOA	0.0192	0.0192	NA	NA	NA	NA
PFBS	0.0200	0.0500	NA	NA	NA	NA
PFHS	0.0200	0.0200	NA	NA	NA	NA
PFOS	0.0464	0.0185	NA	NA	NA	NA
PFOSA	0.0200	NA	0.0500	0.0250	0.0500	0.0200

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 exception of the high level LCSs analyzed for PFHS on 8/18/14, 8/21/14, and 8/25/14 and for PFOSA on 8/18/14, 8/21/14, 8/27/14, and 9/5/14, which were spiked above the resulting upper limit of quantification.

The mid level and high level LCSs prepared on 8/27/14 for PFOSA were prepared with a spiking solution that was suspected to be concentrated due to being left uncapped for an unknown period of time. The mid level recovery was 132% and the high level LCSs were not within the calibration range. The mid and high level LCSs were re-prepared on 8/29/14 using a new stock spiking solution and added to the analytical batch. The re-prepared mid level LCSs met acceptance criteria. The re-prepared high level LCSs were not within the calibration range. The LCS samples results presented in Table 7 are reported from the mid and high level LCSs prepared on 8/29/14, as the non-compliant mid level LCSs appear to be a result of the concentrated spiking solution and not related to the method performance.

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 8/18/14	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-140818-1	0.191	0.194	102	0.199	0.205	103
LCS-140818-2	0.191	0.198	104	0.199	0.198	99.7
LCS-140818-3	0.191	0.205	107	0.199	0.206	104
Average ± %RSD	**104% ± 2.4%**			**102% ± 2.2%**		
LCS-140818-4	19.1	21.1	110	19.9	21.7	109
LCS-140818-5	19.1	21.0	110	19.9	21.4	108
LCS-140818-6	19.1	22.2	116	19.9	22.5	113
Average ± %RSD	**112% ± 3.1%**			**110% ± 2.4%**		
LCS-140818-7	76.0	80.1	105	79.4	83.0	105
LCS-140818-8	76.0	79.0	104	79.4	83.6	105
LCS-140818-9	76.0	78.8	104	79.4	83.2	105
Average ± %RSD	**104% ± 0.55%**			**105% ± 0.0%**		

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

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External Calibration Analyzed 8/18/14	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-140818-1	0.199	0.200	101	0.185	0.191	103
LCS-140818-2	0.199	0.201	101	0.185	0.188	102
LCS-140818-3	0.199	0.208	105	0.185	0.191	104
Average ± %RSD	**102% ± 2.3%**			**103% ± 0.97%**		
LCS-140818-4	19.9	21.4	108	18.5	20.4	110
LCS-140818-5	19.9	21.3	107	18.5	20.2	109
LCS-140818-6	19.9	22.0	110	18.5	21.0	113
Average ± %RSD	**108% ± 1.4%**			**111% ± 1.9%**		
LCS-140818-7	79.4	>ULOQ	NA	73.6	75.4	102
LCS-140818-8	79.4	>ULOQ	NA	73.6	75.8	103
LCS-140818-9	79.4	>ULOQ	NA	73.6	75.4	102
Average ± %RSD	**NA (1)**			**102% ± 0.56%**		

ETS-8-044.1 External Calibration Analyzed 8/18/14	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-140818-1	0.199	0.208	104
LCS-140818-2	0.199	0.211	106
LCS-140818-3	0.199	0.207	104
Average ± %RSD	**105% ± 1.1%**		
LCS-140818-4	19.9	21.9	110
LCS-140818-5	19.9	21.9	110
LCS-140818-6	19.9	22.4	113
Average ± %RSD	**111% ± 1.6%**		
LCS-140818-7	79.4	>ULOQ	NA
LCS-140818-8	79.4	>ULOQ	NA
LCS-140818-9	79.4	>ULOQ	NA
Average ± %RSD	**NA (1)**		

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

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External Calibration Analyzed 8/18/14	$^{13}C_4$-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-140818-1	0.199	0.223	112	0.190	0.214	113
LCS-140818-2	0.199	0.224	113	0.190	0.223	117
LCS-140818-3	0.199	0.225	113	0.190	0.216	114
Average ± %RSD	**113% ± 0.51%**			**115% ± 1.8%**		
LCS-140818-4	1.99	2.08	105	1.90	1.98	104
LCS-140818-5	1.99	2.06	103	1.90	2.02	106
LCS-140818-6	1.99	2.06	104	1.90	1.96	103
Average ± %RSD	**104% ± 0.96%**			**104% ± 1.5%**		

ETS-8-044.1 External Calibration Analyzed 8/21/14	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-140821-1	0.191	0.191	100	0.199	0.198	99.3
LCS-140821-2	0.191	0.196	103	0.199	0.208	105
LCS-140821-3	0.191	0.193	101	0.199	0.199	99.8
Average ± %RSD	**101% ± 1.5%**			**101% ± 3.1%**		
LCS-140821-4	19.1	21.5	113	19.9	22.3	112
LCS-140821-5	19.1	20.8	109	19.9	21.4	107
LCS-140821-6	19.1	21.5	113	19.9	22.3	112
Average ± %RSD	**112% ± 2.1%**			**110% ± 2.6%**		
LCS-140821-7	76.0	79.4	104	79.4	82.5	104
LCS-140821-8	76.0	77.3	102	79.4	80.1	101
LCS-140821-9	76.0	78.1	103	79.4	82.9	104
Average ± %RSD	**103% ± 0.97%**			**103% ± 1.7%**		

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

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External Calibration Analyzed 8/21/14	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-140821-1	0.199	0.210	106	0.185	0.189	102
LCS-140821-2	0.199	0.213	107	0.185	0.189	102
LCS-140821-3	0.199	0.210	105	0.185	0.191	103
Average ± %RSD	**106% ± 0.94%**			**102% ± 0.56%**		
LCS-140821-4	19.9	22.1	111	18.5	21.1	114
LCS-140821-5	19.9	21.5	108	18.5	20.2	109
LCS-140821-6	19.9	21.9	110	18.5	21.1	114
Average ± %RSD	**110% ± 1.4%**			**112% ± 2.6%**		
LCS-140821-7	79.4	>ULOQ	NA	73.6	77.1	105
LCS-140821-8	79.4	>ULOQ	NA	73.6	74.3	101
LCS-140821-9	79.4	>ULOQ	NA	73.6	75.6	103
Average ± %RSD	**NA (1)**			**103% ± 1.9%**		

ETS-8-044.1 External Calibration Analyzed 8/21/14	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-140821-1	0.199	0.211	106
LCS-140821-2	0.199	0.210	105
LCS-140821-3	0.199	0.212	107
Average ± %RSD	**106% ± 0.94%**		
LCS-140821-4	19.9	22.1	111
LCS-140821-5	19.9	21.5	108
LCS-140821-6	19.9	22.2	111
Average ± %RSD	**110% ± 1.6%**		
LCS-140821-7	79.4	>ULOQ	NA
LCS-140821-8	79.4	>ULOQ	NA
LCS-140821-9	79.4	>ULOQ	NA
Average ± %RSD	**NA (1)**		

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

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External Calibration Analyzed 8/21/14	$^{13}C_4$-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-140821-1	0.199	0.229	115	0.190	0.214	112
LCS-140821-2	0.199	0.224	112	0.190	0.215	113
LCS-140821-3	0.199	0.238	120	0.190	0.220	116
Average ± %RSD	**116% ± 3.5%**			**114% ± 1.8%**		
LCS-140821-4	1.99	2.07	104	1.90	2.04	107
LCS-140821-5	1.99	2.07	104	1.90	1.99	105
LCS-140821-6	1.99	2.14	107	1.90	2.05	108
Average ± %RSD	**105% ± 1.6%**			**107% ± 1.4%**		

ETS-8-044.1 External Calibration Analyzed 8/25/14	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-140825-1	0.191	0.198	103	0.199	0.201	101
LCS-140825-2	0.191	0.196	102	0.199	0.202	101
LCS-140825-3	0.191	0.202	106	0.199	0.205	103
Average ± %RSD	**104% ± 2.0%**			**102% ± 1.1%**		
LCS-140825-4	19.1	21.4	112	19.9	22.4	112
LCS-140825-5	19.1	21.0	110	19.9	22.3	112
LCS-140825-6	19.1	21.1	110	19.9	22.5	113
Average ± %RSD	**111% ± 1.0%**			**112% ± 0.51%**		
LCS-140825-7	76.0	80.0	105	79.4	85.4	108
LCS-140825-8	76.0	79.8	105	79.4	85.5	108
LCS-140825-9	76.0	79.9	105	79.4	85.4	108
Average ± %RSD	**105% ± 0.0%**			**108% ± 0.0%**		

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

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External Calibration Analyzed 8/25/14	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-140825-1	0.199	0.208	104	0.185	0.191	103
LCS-140825-2	0.199	0.209	105	0.185	0.184	99.3
LCS-140825-3	0.199	0.211	106	0.185	0.188	102
Average ± %RSD	**105% ± 0.95%**			**101% ± 1.9%**		
LCS-140825-4	19.9	21.5	108	18.5	20.1	109
LCS-140825-5	19.9	21.2	106	18.5	19.5	106
LCS-140825-6	19.9	21.2	107	18.5	19.7	107
Average ± %RSD	**107% ± 0.93%**			**107% ± 1.4%**		
LCS-140825-7	79.4	>ULOQ	NA	73.6	75.5	103
LCS-140825-8	79.4	>ULOQ	NA	73.6	75.0	102
LCS-140825-9	79.4	>ULOQ	NA	73.6	74.0	101
Average ± %RSD	**NA (1)**			**102% ± 0.98%**		

ETS-8-044.1 External Calibration Analyzed 8/25/14	$^{13}C_4$-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-140825-1	0.199	0.239	120	0.190	0.215	113
LCS-140825-2	0.199	0.221	111	0.190	0.199	105
LCS-140825-3	0.199	0.241	121	0.190	0.223	117
Average ± %RSD	**117% ± 4.7%**			**112% ± 5.5%**		
LCS-140825-4	1.99	2.12	107	1.90	2.06	109
LCS-140825-5	1.99	2.13	107	1.90	2.02	106
LCS-140825-6	1.99	2.13	107	1.90	2.02	106
Average ± %RSD	**107% ± 0.0%**			**107% ± 1.6%**		

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

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 Internal Calibration Analyzed 8/27/14	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-140827-1	0.198	0.206	104
LCS-140827-2	0.198	0.194	98.0
LCS-140827-3	0.198	0.192	97.0
Average ± %RSD	**99.7% ± 3.8%**		
LCS-140829-4	19.8	17.2	86.7
LCS-140829-5	19.8	18.9	95.6
LCS-140829-6	19.8	17.5	88.2
Average ± %RSD	**90.2% ± 5.3%**		
LCS-140829-7	157	>ULOQ	NA
LCS-140829-8	157	>ULOQ	NA
LCS-140829-9	157	>ULOQ	NA
Average ± %RSD	**NA (1)**		

ETS-8-044.1 External Calibration Analyzed 9/5/14	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-140905-1	0.200	0.193	96.6
LCS-140905-2	0.200	0.190	95.2
LCS-140905-3	0.200	0.209	104
Average ± %RSD	**98.6% ± 4.8%**		
LCS-140905-4	20.0	20.5	102
LCS-140905-5	20.0	20.4	102
LCS-140905-6	20.0	21.0	105
Average ± %RSD	**103% ± 1.7%**		
LCS-140905-7	79.4	>ULOQ	NA
LCS-140905-8	79.4	>ULOQ	NA
LCS-140905-9	79.4	>ULOQ	NA
Average ± %RSD	**NA (1)**		

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

Table 7 continued. Laboratory Control Spike Results.

ETS-8-044.1 External Calibration Analyzed 9/9/14	PFOSA		
Lab ID	**Spiked Concentration (ng/mL)**	**Calculated Concentration (ng/mL)**	**%Recovery**
LCS-140909-1	0.200	0.199	99.6
LCS-140909-2	0.200	0.183	91.4
LCS-140909-3	0.200	0.195	97.3
Average ± %RSD	**96.1% ± 4.4%**		
LCS-140909-4	9.99	10.3	103
LCS-140909-5	9.99	9.95	99.6
LCS-140909-6	9.99	10.4	104
Average ± %RSD	**102% ± 2.3%**		
LCS-140909-7	39.8	39.4	99.0
LCS-140909-8	39.8	37.7	94.8
LCS-140909-9	39.8	40.5	102
Average ± %RSD	**98.6% ± 3.7%**		

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

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%.

The method uncertainty for PFOSA using external calibration was calculated at ±24% following ETS-12-012.2; however, due to multiple non-compliant field matrix spike recoveries, including the trip blank spikes (recoveries between 62.5% and 62.6%), the method uncertainty has been expanded to ±38% based on the %bias of the trip blank spikes which were greater than the average pooled field matrix spike recovery of the samples analyzed by external calibration (69.4%). Since only select samples had field matrix spikes, the expanded method uncertainty will apply to all samples reported by external calibration.

Table 8. Analytical Method Uncertainty.

Analyte	Calibration	Standard Deviation (%)	Method Uncertainty
PFOA	External	6.80	±14%
PFBS	External	5.99	±12%
PFHS	External	5.25	±10%
PFOS	External	6.02	±12%
PFOSA	External	NA	±38%
PFOSA	Internal	4.88	±9.8%

NA = Not Applicable

3.8 Field Matrix Spikes (FMS)

A target analyte field matrix spike sample(s) 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. 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, each sample bottle 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 all 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
BD24-4, D14, L22	FMS	10.0	10.0	10.0	9.99	10.0
P118B, P263, K1, K3, P21B, SSB-1, SSB-2	FMS	20.0	20.0	20.0	20.0	20.0
D11, P56, P2, P14, P1065, P16	FMS	50.0	50.0	50.0	50.0	50.0
L19, P379, P380, B3, PP01, PP05, PP06	FMS	100	100	100	99.9	10.0
D09, D10, P262	FMS	100	10.0	100	100	10.0
P340, P341, P343, P304, P305	FMS	496	20.0	495	496	20.0
P42	FMS	501	50.0	500	501	50.0
M4, P118C	FMS	501	100	10.0	9.99	10.0
Trip Blank	Low	10.0	10.0	10.0	9.99	10.0
	Mid	100	20.0	100	100	20.0
	High	501	50.0	500	501	50.0

4 Data Summary and Discussion

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

Since many of these samples had not been previously analyzed by the 3M Environmental Laboratory, every effort was made to try and appropriately match the FMS spike levels with the expected sample concentrations. There are instances where the FMS the spike level was outside the recommended spike range of being less than 10 times the endogenous sample concentration, and the FMS recovery value is reported. While the target analyte field matrix spike sample is intended to verify that the analytical method is applicable for the collected matrix, it also can provide useful information on holding time stability. In all instances where the spike level was greater than 10x the endogenous levels, the concentrations were within the calibration range and no additional dilution of the FMS sample was performed in an effort to the report the FMS result.

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. A method deviation is included with the raw data.

PP04: Sample/sample duplicate RPD was 122% for PFOSA. Since the RPD value was greater than 50%, the sample results will not be reported for this location.

PP06: The FMS recovery for PFOSA was 60.8%. The method uncertainty has been expanded for PFOSA to ± 39%, based on the recovery of the FMS sample.

P305: The FMS recovery for PFHS was 68.9% and for PFOSA 58.4%. The method uncertainty has been expanded for PFHS to ± 31% and for PFOSA to ± 42%, based on the recovery of the FMS sample.

P42: The FMS recoveries for PFOA (64.8%), PFBS (60.5%), PFHS (66.2%), and PFOSA (52.4%) did not meet acceptance criteria. The method uncertainty has been expanded for these analytes based on the recovery of the FMS sample to ±35% for PFOA, ±40% for PFBS, ±34% for PFHS, and ±48% for PFOSA.

P1065: The FMS recovery for PFOA was 54.4% and for PFOSA 58.1%. The method uncertainty has been expanded for PFOA to ± 46% and for PFOSA to ± 42%, based on the recovery of the FMS sample. Samples were re-analyzed for PFOA and confirmed the initial results. The results from the initial analysis are reported.

PA111B: Sample/sample duplicate RPD was 57% for PFOA, 88% for PFBS, 80% for PFHS, and 97% for PFOSA. Samples were re-analyzed and confirmed the initial results. Since the RPD values are greater than 50%, the sample results will not be reported for this location.

P262: The FMS recovery for PFOSA was 55.0%. The method uncertainty has been expanded for PFOSA to ± 45%, based on the recovery of the FMS sample.

P340: The FMS recovery for PFOSA was 56.0%. The method uncertainty has been expanded for PFOSA to ± 44%, based on the recovery of the FMS sample.

B3: The FMS recovery for PFOSA was 50.3%. The method uncertainty has been expanded for PFOSA to ± 50%, based on the recovery of the FMS sample.

PA109B: The FMS recovery for PFOSA was 61.0%. The method uncertainty has been expanded for PFOSA to ± 39%, based on the recovery of the FMS sample.

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 two times higher than the area count response that was produced for the primary sample and sample duplicate, resulting in low FMS recoveries. Results for PFOSA were reported by internal standard calibration for any sample set that included a FMS sample, where the area count response for $^{13}C_8$-PFOSA was consistent with the primary sample and sample duplicate. Sample sets reported by internal standard calibration include: BD24-4, D09, D10, D14, P118B, L22, K3, P21B, and the Trip Blank set. All other sample sets were reported by external standard calibration.

- **Sample locations P2, P27, P262**: 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 locations P2, P27, and P262, the spike level for PFOSA was just below the method recommendation at between 45% and 47% of the endogenous level. To evaluate sample recovery the FMS recovery was calculated for PFOSA and the FMS recoveries are flagged as being below the recommended spike level.

- **Sample locations D11, PP01, PP06, K1, P305, P42, P1065, P118C, P263, P340, P380, B3, PA109B, Trip blank:** Samples were analyzed using external calibration for PFOSA and had FMS recoveries outside the acceptance criteria of 100 ± 30%. Since only select sample sets contained a FMS samples, all FMS recoveries were pooled to determine an overall method uncertainly for samples analyzed for PFOSA by external calibration. The average pooled FMS recoveries using external calibration was 69.4% with trip blank recoveries between 62.5% and 62.6%. Based on these recoveries, the method uncertainty has been expanded to ±38% for all samples analyzed for PFOSA by external calibration. Samples with FMS recoveries below 50% will be reported but flagged as qualitative. Qualitative results for PFOSA are reported for sampling locations K1 and P380.

- **Sample location D14**; Sample Dup (ISO14-0020-016) analyzed for PFOSA 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 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.

Table 10. Location ID: BD24-4

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-003	BD24-4; Sample	11.8	NA	20.8	NA	4.68	NA
ISO14-0020-004	BD24-4; Sample Dup	11.8	NA	20.8	NA	4.71	NA
ISO14-0020-005	BD24-4; FMS	21.2	94.0	29.2	NC	13.8	91.1
Average Concentration (ng/mL) ± %RPD		**11.8 ng/mL ± 0.0%**		**20.8 ng/mL ± 0.0%**		**4.70 ng/mL ± 0.64%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-003	BD24-4; Sample	8.01	NA	0.189	NA
ISO14-0020-004	BD24-4; Sample Dup	8.82	NA	0.205	NA
ISO14-0020-005	BD24-4; FMS	17.5	90.9	10.3	101 (1)
Average Concentration (ng/mL) ± %RPD		**8.42 ng/mL ± 9.6%**		**0.197 ng/mL ± 8.1%**	

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

Table 11. Location ID: D09

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-006	D09; Sample	243	NA	412	NA	10800	NA
ISO14-0020-007	D09; Sample Dup	237	NA	414	NA	10800	NA
ISO14-0020-008	D09; FMS	271	NC	428	NC	11000	NC
Average Concentration (ng/mL) ± %RPD		**240 ng/mL ± 2.5%**		**413 ng/mL ± 0.48%**		**10800 ng/mL ± 0.0%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-006	D09; Sample	1930	NA	34.8	NA
ISO14-0020-007	D09; Sample Dup	1920	NA	40.8	NA
ISO14-0020-008	D09; FMS	2020	NC	41.1	NC
Average Concentration (ng/mL) ± %RPD		**1930 ng/mL ± 0.52%**		**37.8 ng/mL ± 16%**	

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

Table 12. Location ID: D10

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-009	D10; Sample	499	NA	7.43	NA	174	NA
ISO14-0020-010	D10; Sample Dup	507	NA	7.39	NA	173	NA
ISO14-0020-011	D10; FMS	533	NC	17.0	95.9	266	92.5
Average Concentration (ng/mL) ± %RPD		**503 ng/mL ± 1.6%**		**7.41 ng/mL ± 0.54%**		**174 ng/mL ± 0.58%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-009	D10; Sample	26.6	NA	0.171	NA
ISO14-0020-010	D10; Sample Dup	26.9	NA	0.140	NA
ISO14-0020-011	D10; FMS	116	89.1	9.51	93.5 (1)
Average Concentration (ng/mL) ± %RPD		**26.8 ng/mL ± 1.1%**		**0.156 ng/mL ± 20%**	

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

Table 13. Location ID: D11

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-012	D11; Sample	478	NA	19.6	NA	212	NA
ISO14-0020-013	D11; Sample Dup	461	NA	19.3	NA	212	NA
ISO14-0020-014	D11; FMS	488	NC	63.5	88.1	250	NC
Average Concentration (ng/mL) ± %RPD		**470 ng/mL ± 3.6%**		**19.5 ng/mL ± 1.5%**		**212 ng/mL ± 0.0%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-012	D11; Sample	548	NA	15.7	NA
ISO14-0020-013	D11; Sample Dup	573	NA	15.8	NA
ISO14-0020-014	D11; FMS	650	NC	48.8	66.1 (1)
Average Concentration (ng/mL) ± %RPD		**561 ng/mL ± 4.5%**		**15.8 ng/mL ± 0.63%**	

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%. The recovery was within the expanded uncertainty for PFOSA; therefore no additional adjustment was made to the analytical uncertainty.

Table 14. Location ID: D14

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-015	D14; Sample	2.24	NA	0.322	NA	1.44	NA
ISO14-0020-016	D14; Sample Dup	2.24	NA	0.328	NA	1.38	NA
ISO14-0020-017	D14; FMS	11.5	92.6	9.83	95.1 (1)	10.8	93.9
Average Concentration (ng/mL) ± %RPD		**2.24 ng/mL ± 0.0%**		**0.325 ng/mL ± 1.8%**		**1.41 ng/mL ± 4.3%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-015	D14; Sample	1.06	NA	0.0838	NA
ISO14-0020-016	D14; Sample Dup	0.992	NA	NR (2)	NA
ISO14-0020-017	D14; FMS	8.91	78.9	10.0	99.2 (1)
Average Concentration (ng/mL) ± %RPD		**1.03 ng/mL ± 6.6%**		**0.0838 ng/mL**	

NA = Not Applicable
(1) FMS concentration greater than 10 times the sample concentration.
(2) NR = Not Reportable; did not meet method blank criteria.

Table 15. Location ID: P118B

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-020	P118B; Sample	2540	NA	293	NA	4210	NA
ISO14-0020-021	P118B; Sample Dup	2510	NA	291	NA	4170	NA
ISO14-0020-022	P118B; FMS	2500	NC	310	NC	4260	NC
Average Concentration (ng/mL) ± %RPD		**2530 ng/mL ± 1.2%**		**292 ng/mL ± 0.68%**		**4190 ng/mL ± 0.95%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-020	P118B; Sample	12900	NA	3.54	NA
ISO14-0020-021	P118B; Sample Dup	12900	NA	4.29	NA
ISO14-0020-022	P118B; FMS	13000	NC	23.8	99.4
Average Concentration (ng/mL) ± %RPD		**12900 ng/mL ± 0.0%**		**3.92 ng/mL ± 19%**	

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

Table 16. Location ID: L22

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-033	L22; Sample	0.515	NA	2.56	NA	0.221	NA
ISO14-0020-034	L22; Sample Dup	0.508	NA	2.45	NA	0.202	NA
ISO14-0020-035	L22; FMS	9.86	93.5 (1)	11.7	92.0	9.65	94.4 (1)
Average Concentration (ng/mL) ± %RPD		**0.512 ng/mL ± 1.4%**		**2.51 ng/mL ± 4.4%**		**0.212 ng/mL ± 9.0%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-033	L22; Sample	11.7	NA	0.114	NA
ISO14-0020-034	L22; Sample Dup	12.0	NA	0.106	NA
ISO14-0020-035	L22; FMS	20.7	88.6	9.58	94.7
Average Concentration (ng/mL) ± %RPD		**11.9 ng/mL ± 2.5%**		**0.110 ng/mL ± 7.3%**	

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

Table 17. Location ID: PP01

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-048	PP01; Sample	55.9	NA	18.0	NA	28300	NA
ISO14-0020-049	PP01; Sample Dup	54.4	NA	17.9	NA	27000	NA
ISO14-0020-050	PP01; FMS	133	77.9	93.5	75.6	NA (1)	NA (1)
Average Concentration (ng/mL) ± %RPD		**55.2 ng/mL ± 2.7%**		**18.0 ng/mL ± 0.56%**		**27700 ng/mL ± 4.7%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-048	PP01; Sample	8930	NA	63.0	NA
ISO14-0020-049	PP01; Sample Dup	8780	NA	71.2	NA
ISO14-0020-050	PP01; FMS	8540	NC	136	68.9 (2)
Average Concentration (ng/mL) ± %RPD		**8860 ng/mL ± 1.7%**		**67.1 ng/mL ± 12%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Sample concentration exceeded calibration range; sample was not re-analyzed since field matrix spike level was not appropriate.
(2) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%. The recovery was within the expanded uncertainty for PFOSA; therefore no additional adjustment was made to the analytical uncertainty.

Table 18. Location ID: PP05

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-055	PP05; Sample	1530	NA	16500	NA	5500	NA
ISO14-0020-056	PP05; Sample Dup	1560	NA	16800	NA	5510	NA
ISO14-0020-057	PP05; FMS	1660	NC	17000	NC	5760	NC
Average Concentration (ng/mL) ± %RPD		**1550 ng/mL ± 1.9%**		**16700 ng/mL ± 1.8%**		**5510 ng/mL ± 0.18%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-055	PP05; Sample	310	NA	61.6	NA
ISO14-0020-056	PP05; Sample Dup	316	NA	66.3	NA
ISO14-0020-057	PP05; FMS	390	NC	140	76.1
Average Concentration (ng/mL) ± %RPD		**313 ng/mL ± 1.9%**		**64.0 ng/mL ± 7.3%**	

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

Table 19. Location ID: PP06

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-058	PP06; Sample	199	NA	16.0	NA	47.8	NA
ISO14-0020-059	PP06; Sample Dup	196	NA	14.9	NA	49.8	NA
ISO14-0020-060	PP06; FMS	289	91.5	96.0	80.6	135	86.2
Average Concentration (ng/mL) ± %RPD		**198 ng/mL ± 1.5%**		**15.5 ng/mL ± 7.1%**		**48.8 ng/mL ± 4.1%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-058	PP06; Sample	2030	NA	25.6	NA
ISO14-0020-059	PP06; Sample Dup	2100	NA	25.6	NA
ISO14-0020-060	PP06; FMS	2200	NC	86.4	60.8 (1)
Average Concentration (ng/mL) ± %RPD		**2070 ng/mL ± 3.4%**		**25.6 ng/mL ± 0.0%** (2)	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%.
(2) The method uncertainty has been expanded to ±39% for PFOSA.

Table 20. Location ID: K1

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-077	K1; Sample	2.56	NA	4.45	NA	13.2	NA
ISO14-0020-078	K1; Sample Dup	2.52	NA	4.44	NA	12.9	NA
ISO14-0020-079	K1; FMS	19.5	84.8	21.9	87.3	31.0	89.8
Average Concentration (ng/mL) ± %RPD		**2.54 ng/mL ± 1.6%**		**4.45 ng/mL ± 0.22%**		**13.1 ng/mL ± 2.3%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-077	K1; Sample	16.1	NA	2.03	NA
ISO14-0020-078	K1; Sample Dup	15.8	NA	2.03	NA
ISO14-0020-079	K1; FMS	33.3	86.8	10.5	42.4 (1)
Average Concentration (ng/mL) ± %RPD		**16.0 ng/mL ± 1.9%**		**2.03 ng/mL ± 0.0%** (2)	

NA = Not Applicable
(1) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%.
(2) Sample results should be considered qualitative due to field matrix spike recovery below 50%.

Table 21. Location ID: K3

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-080	K3; Sample	103	NA	846	NA	55700	NA
ISO14-0020-081	K3; Sample Dup	99.5	NA	785	NA	53100	NA
ISO14-0020-082	K3; FMS	120	NC	885	NC	NA (1)	NA (1)
Average Concentration (ng/mL) ± %RPD		**101 ng/mL ± 3.5%**		**816 ng/mL ± 7.5%**		**54400 ng/mL ± 4.8%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-080	K3; Sample	3320	NA	20.6	NA
ISO14-0020-081	K3; Sample Dup	3230	NA	23.1	NA
ISO14-0020-082	K3; FMS	3380	NC	35.8	69.8
Average Concentration (ng/mL) ± %RPD		**3280 ng/mL ± 2.7%**		**21.9 ng/mL ± 11%**	

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

Table 22. Location ID: P21B

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-083	P21B; Sample	14000	NA	5360	NA	10700	NA
ISO14-0020-084	P21B; Sample Dup	14100	NA	5280	NA	10600	NA
ISO14-0020-085	P21B; FMS	14100	NC	5600	NC	10400	NC
Average Concentration (ng/mL) ± %RPD		**14100 ng/mL ± 0.71%**		**5320 ng/mL ± 1.5%**		**10700 ng/mL ± 0.94%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-083	P21B; Sample	82700	NA	12.2	NA
ISO14-0020-084	P21B; Sample Dup	72800	NA	16.0	NA
ISO14-0020-085	P21B; FMS	NA (1)	NA (1)	26.4	61.5 (2)
Average Concentration (ng/mL) ± %RPD		**77800 ng/mL ± 13%**		**14.1 ng/mL ± 27% (3)**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Sample concentration exceeded calibration range; sample was not re-analyzed since field matrix spike level was not appropriate.
(2) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%. The recovery was within the expanded uncertainty for PFOSA; therefore no additional adjustment was made to the analytical uncertainty.
(3) Sample/sample duplicate RPD does not meet acceptance criteria of ≤20%.

Table 23. Location ID: P304

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-086	P304; Sample	585	NA	130	NA	90.5	NA
ISO14-0020-087	P304; Sample Dup	582	NA	128	NA	89.4	NA
ISO14-0020-088	P304; FMS	1050	94.1	148	NC	512	85.3
Average Concentration (ng/mL) ± %RPD		**584 ng/mL ± 0.51%**		**129 ng/mL ± 1.6%**		**90.0 ng/mL ± 1.2%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-086	P304; Sample	878	NA	16.0	NA
ISO14-0020-087	P304; Sample Dup	857	NA	14.7	NA
ISO14-0020-088	P304; FMS	1320	91.2	41.1	129
Average Concentration (ng/mL) ± %RPD		**868 ng/mL ± 2.4%**		**15.4 ng/mL ± 8.5%**	

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

Table 24. Location ID: P305

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-089	P305; Sample	290	NA	130	NA	318	NA
ISO14-0020-090	P305; Sample Dup	279	NA	126	NA	306	NA
ISO14-0020-091	P305; FMS	637	71.1	140	NC	653	68.9 (1)
Average Concentration (ng/mL) ± %RPD		**285 ng/mL ± 3.9%**		**128 ng/mL ± 3.1%**		**312 ng/mL ± 3.8% (2)**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-089	P305; Sample	654	NA	9.45	NA
ISO14-0020-090	P305; Sample Dup	666	NA	10.2	NA
ISO14-0020-091	P305; FMS	1090	86.7	21.5	58.4 (1)
Average Concentration (ng/mL) ± %RPD		**660 ng/mL ± 1.8%**		**9.83 ng/mL ± 7.6% (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) The method uncertainty has been expanded to ±31% for PFHS and ±42% for PFOSA.

Table 25. Location ID: P42

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-092	P42; Sample	533	NA	46.6	NA	510	NA
ISO14-0020-093	P42; Sample Dup	582	NA	44.7	NA	500	NA
ISO14-0020-094	P42; FMS	882	64.8 (1)	75.9	60.5 (1)	836	66.2 (1)
Average Concentration (ng/mL) ± %RPD		**558 ng/mL ± 8.8% (2)**		**45.7 ng/mL ± 4.2% (2)**		**505 ng/mL ± 2.0% (2)**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-092	P42; Sample	4760	NA	14.6	NA
ISO14-0020-093	P42; Sample Dup	5130	NA	16.6	NA
ISO14-0020-094	P42; FMS	5210	NC	41.8	52.4 (1)
Average Concentration (ng/mL) ± %RPD		**4950 ng/mL ± 7.5%**		**15.6 ng/mL ± 13% (2)**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%.
(2) The method uncertainty has been expanded to ±35% for PFOA, ±40% for PFBS, ±34% for PFHS, and ±48% for PFOSA.

Table 26. Location ID: P56

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-095	P56; Sample	60.3	NA	10.6	NA	16.0	NA
ISO14-0020-096	P56; Sample Dup	59.7	NA	10.7	NA	16.0	NA
ISO14-0020-097	P56; FMS	94.8	69.6	57.8	94.3	65.1	98.2
Average Concentration (ng/mL) ± %RPD		**60.0 ng/mL ± 1.0%**		**10.7 ng/mL ± 0.94%**		**16.0 ng/mL ± 0.0%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-095	P56; Sample	183	NA	7.76	NA
ISO14-0020-096	P56; Sample Dup	184	NA	7.99	NA
ISO14-0020-097	P56; FMS	245	NC	45.5	75.3
Average Concentration (ng/mL) ± %RPD		**184 ng/mL ± 0.54%**		**7.88 ng/mL ± 2.9%**	

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

Table 27. Location ID: P2

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-098	P2; Sample	1810	NA	55.2	NA	221	NA
ISO14-0020-099	P2; Sample Dup	1760	NA	55.7	NA	222	NA
ISO14-0020-100	P2; FMS	NA (1)	NA (1)	106	101	272	NC
Average Concentration (ng/mL) ± %RPD		**1790 ng/mL ± 2.8%**		**55.5 ng/mL ± 0.90%**		**222 ng/mL ± 0.45%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-098	P2; Sample	2260	NA	108	NA
ISO14-0020-099	P2; Sample Dup	2210	NA	112	NA
ISO14-0020-100	P2; FMS	NA (1)	NA (1)	156	92.0 (2)
Average Concentration (ng/mL) ± %RPD		**2240 ng/mL ± 2.2%**		**110 ng/mL ± 3.6%**	

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

Table 28. Location ID: P27

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-101	P27; Sample	370	NA	6190	NA	48.4	NA
ISO14-0020-102	P27; Sample Dup	372	NA	6260	NA	48.3	NA
ISO14-0020-103	P27; FMS	399	NC	NA [1]	NA [1]	98.9	101
Average Concentration (ng/mL) ± %RPD		**371 ng/mL ± 0.54%**		**6230 ng/mL ± 1.1%**		**48.4 ng/mL ± 0.21%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-101	P27; Sample	2300	NA	107	NA
ISO14-0020-102	P27; Sample Dup	2290	NA	106	NA
ISO14-0020-103	P27; FMS	NA [1]	NA [1]	143	73.0 [2]
Average Concentration (ng/mL) ± %RPD		**2300 ng/mL ± 0.44%**		**107 ng/mL ± 0.94%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Sample concentration exceeded calibration range; sample was not re-analyzed since field matrix spike level was not appropriate.
(2) Although the FMS spike level was less than 50% of the endogenous level (47%), the FMS spike recovery was calculated was used to assess accuracy.

Table 29. Location ID: P1065

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-104	P1065; Sample	9.14	NA	12.5	NA	3.08	NA
ISO14-0020-105	P1065; Sample Dup	9.43	NA	12.0	NA	3.20	NA
ISO14-0020-106	P1065; FMS	36.5	54.4 [1]	59.3	94.1	51.9	97.5 [2]
Average Concentration (ng/mL) ± %RPD		**9.29 ng/mL ± 3.1% [3]**		**12.3 ng/mL ± 4.1%**		**3.14 ng/mL ± 3.8%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-104	P1065; Sample	63.7	NA	3.88	NA
ISO14-0020-105	P1065; Sample Dup	65.5	NA	4.05	NA
ISO14-0020-106	P1065; FMS	115	101	33.0	58.1 [1, 2]
Average Concentration (ng/mL) ± %RPD		**64.6 ng/mL ± 2.8%**		**3.97 ng/mL ± 4.3% [3]**	

NA = Not Applicable
(1) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%.
(2) FMS concentration greater than 10 times the sample concentration.
(3) The method uncertainty has been expanded to ±46% for PFOA and ±42% for PFOSA.

Table 30. Location ID: L19

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-110	L19; Sample	263	NA	18.0	NA	153	NA
ISO14-0020-111	L19; Sample Dup	270	NA	19.5	NA	158	NA
ISO14-0020-112	L19; FMS	372	NC	110	91.3	251	95.5
Average Concentration (ng/mL) ± %RPD		**267 ng/mL ± 2.6%**		**18.8 ng/mL ± 8.0%**		**156 ng/mL ± 3.2%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-110	L19; Sample	3500	NA	49.4	NA
ISO14-0020-111	L19; Sample Dup	3490	NA	46.8	NA
ISO14-0020-112	L19; FMS	3630	NC	136	87.9
Average Concentration (ng/mL) ± %RPD		**3500 ng/mL ± 0.29%**		**48.1 ng/mL ± 5.4%**	

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

Table 31. Location ID: M4

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-113	M4; Sample	375	NA	111	NA	142	NA
ISO14-0020-114	M4; Sample Dup	334	NA	114	NA	127	NA
ISO14-0020-115	M4; FMS	928	115	196	83.5	639	101
Average Concentration (ng/mL) ± %RPD		**355 ng/mL ± 12%**		**113 ng/mL ± 2.7%**		**135 ng/mL ± 11%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-113	M4; Sample	5730	NA	140	NA
ISO14-0020-114	M4; Sample Dup	5090	NA	110	NA
ISO14-0020-115	M4; FMS	6720	NC	241	116
Average Concentration (ng/mL) ± %RPD		**5410 ng/mL ± 12%**		**125 ng/mL ± 24%** [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 32. Location ID: P118C

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-116	P118C; Sample	115	NA	5.21	NA	41.2	NA
ISO14-0020-117	P118C; Sample Dup	117	NA	5.62	NA	41.2	NA
ISO14-0020-118	P118C; FMS	625	102	94.3	88.9 (1)	512	94.2 (1)
Average Concentration (ng/mL) ± %RPD		**116 ng/mL ± 1.7%**		**5.42 ng/mL ± 7.6%**		**41.2 ng/mL ± 0.0%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-116	P118C; Sample	1510	NA	55.3	NA
ISO14-0020-117	P118C; Sample Dup	1520	NA	51.1	NA
ISO14-0020-118	P118C; FMS	2160	NC	118	64.8 (2)
Average Concentration (ng/mL) ± %RPD		**1520 ng/mL ± 0.66%**		**53.2 ng/mL ± 7.9%**	

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%. The recovery was within the expanded uncertainty for PFOSA; therefore no additional adjustment was made to the analytical uncertainty.

Table 33. Location ID: P262

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-119	P262; Sample	661	NA	22.0	NA	272	NA
ISO14-0020-120	P262; Sample Dup	644	NA	21.9	NA	268	NA
ISO14-0020-121	P262; FMS	711	NC	32.1	NC	382	NC
Average Concentration (ng/mL) ± %RPD		**653 ng/mL ± 2.6%**		**22.0 ng/mL ± 0.46%**		**270 ng/mL ± 1.5%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-119	P262; Sample	2220	NA	21.9	NA
ISO14-0020-120	P262; Sample Dup	2150	NA	20.5	NA
ISO14-0020-121	P262; FMS	2400	NC	26.7	55.0 (1,2)
Average Concentration (ng/mL) ± %RPD		**2190 ng/mL ± 3.2%**		**21.2 ng/mL ± 6.6%** (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 (47%), the FMS spike recovery was calculated was used to assess accuracy.
(3) The method uncertainty has been expanded to ±45% for PFOSA.

Table 34. Location ID: P263

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-122	P263; Sample	2820	NA	87.8	NA	947	NA
ISO14-0020-123	P263; Sample Dup	2830	NA	84.3	NA	922	NA
ISO14-0020-124	P263; FMS	2810	NC	103	NC	962	NC
Average Concentration (ng/mL) ± %RPD		**2830 ng/mL ± 0.35%**		**86.1 ng/mL ± 4.1%**		**935 ng/mL ± 2.7%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-122	P263; Sample	12100	NA	8.34	NA
ISO14-0020-123	P263; Sample Dup	12300	NA	8.32	NA
ISO14-0020-124	P263; FMS	NA (1)	NA (1)	20.8	62.4 (2)
Average Concentration (ng/mL) ± %RPD		**12200 ng/mL ± 1.6%**		**8.33 ng/mL ± 0.24%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) Sample concentration exceeded calibration range; sample was not re-analyzed since field matrix spike level was not appropriate.
(2) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%. The recovery was within the expanded uncertainty for PFOSA; therefore no additional adjustment was made to the analytical uncertainty.

Table 35. Location ID: P340

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-129	P340; Sample	836	NA	164	NA	319	NA
ISO14-0020-130	P340; Sample Dup	799	NA	165	NA	318	NA
ISO14-0020-131	P340; FMS	1240	85.2	178	NC	782	93.6
Average Concentration (ng/mL) ± %RPD		**818 ng/mL ± 4.5%**		**165 ng/mL ± 0.61%**		**319 ng/mL ± 0.31%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-129	P340; Sample	3470	NA	26.4	NA
ISO14-0020-130	P340; Sample Dup	3370	NA	26.8	NA
ISO14-0020-131	P340; FMS	3990	NC	37.8	56.0 (1)
Average Concentration (ng/mL) ± %RPD		**3420 ng/mL ± 2.9%**		**26.6 ng/mL ± 1.5%** (2)	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%.
(2) The method uncertainty has been expanded to ±44% for PFOSA.

Table 36. Location ID: P341

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-132	P341; Sample	1640	NA	40.9	NA	612	NA
ISO14-0020-133	P341; Sample Dup	1610	NA	40.0	NA	601	NA
ISO14-0020-134	P341; FMS	1980	NC	55.7	NC	1040	87.6
Average Concentration (ng/mL) ± %RPD		**1630 ng/mL ± 1.8%**		**40.5 ng/mL ± 2.2%**		**607 ng/mL ± 1.8%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-132	P341; Sample	5240	NA	8.81	NA
ISO14-0020-133	P341; Sample Dup	4950	NA	8.95	NA
ISO14-0020-134	P341; FMS	5320	NC	22.8	69.6
Average Concentration (ng/mL) ± %RPD		**5100 ng/mL ± 5.7%**		**8.88 ng/mL ± 1.6%**	

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

Table 37. Location ID: P343

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-135	P343; Sample	994	NA	47.6	NA	701	NA
ISO14-0020-136	P343; Sample Dup	1030	NA	46.5	NA	705	NA
ISO14-0020-137	P343; FMS	1510	NC	65.3	NC	1180	96.4
Average Concentration (ng/mL) ± %RPD		**1010 ng/mL ± 3.6%**		**47.1 ng/mL ± 2.3%**		**703 ng/mL ± 0.57%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-135	P343; Sample	8060	NA	12.0	NA
ISO14-0020-136	P343; Sample Dup	8140	NA	11.7	NA
ISO14-0020-137	P343; FMS	8890	NC	27.4	77.8
Average Concentration (ng/mL) ± %RPD		**8100 ng/mL ± 0.99%**		**11.9 ng/mL ± 2.5%**	

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

Table 38. Location ID: P379

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-142	P379; Sample	128	NA	4.26	NA	33.3	NA
ISO14-0020-143	P379; Sample Dup	131	NA	3.77	NA	34.1	NA
ISO14-0020-144	P379; FMS	220	90.5	91.3	87.3	123	89.3
Average Concentration (ng/mL) ± %RPD		**130 ng/mL ± 2.3%**		**4.02 ng/mL ± 12%**		**33.7 ng/mL ± 2.4%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-142	P379; Sample	1090	NA	32.3	NA
ISO14-0020-143	P379; Sample Dup	1110	NA	35.2	NA
ISO14-0020-144	P379; FMS	1200	NC	110	76.3
Average Concentration (ng/mL) ± %RPD		**1100 ng/mL ± 1.8%**		**33.8 ng/mL ± 8.6%**	

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

Table 39. Location ID: P380

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-145	P380; Sample	534	NA	23.2	NA	439	NA
ISO14-0020-146	P380; Sample Dup	567	NA	25.1	NA	482	NA
ISO14-0020-147	P380; FMS	664	NC	95.7	71.6	563	NC
Average Concentration (ng/mL) ± %RPD		**551 ng/mL ± 6.0%**		**24.2 ng/mL ± 7.9%**		**461 ng/mL ± 9.3%**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-145	P380; Sample	1700	NA	62.2	NA
ISO14-0020-146	P380; Sample Dup	1750	NA	63.9	NA
ISO14-0020-147	P380; FMS	1820	NC	111	48.0 [1]
Average Concentration (ng/mL) ± %RPD		**1730 ng/mL ± 2.9%**		**63.1 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) Sample results should be considered qualitative due to field matrix spike recovery below 50%

Table 40. Location ID: B3

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-152	B3; Sample	255	NA	9.21	NA	99.3	NA
ISO14-0020-153	B3; Sample Dup	240	NA	7.62	NA	92.2	NA
ISO14-0020-154	B3; FMS	340	NC	97.7	89.3	188	92.3
Average Concentration (ng/mL) ± %RPD		**248 ng/mL ± 6.1%**		**8.42 ng/mL ± 19%**		**95.8 ng/mL ± 7.4%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-152	B3; Sample	1260	NA	48.9	NA
ISO14-0020-153	B3; Sample Dup	1220	NA	44.4	NA
ISO14-0020-154	B3; FMS	1290	NC	96.9	50.3 (1)
Average Concentration (ng/mL) ± %RPD		**1240 ng/mL ± 3.2%**		**46.7 ng/mL ± 9.6%**	

NA = Not Applicable
NC = Not Calculated; Spike level was less than 0.5x the endogenous sample concentration.
(1) The field matrix spike sample did not meet acceptance criteria of 100 ± 30%.
(2) The method uncertainty has been expanded to ±50% for PFOSA.

Table 41. Location ID: PA109B

		PFOA		PFBS		PFHS	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-171	PA109B; Sample	0.903	NA	1.06	NA	0.267	NA
ISO14-0020-172	PA109B; Sample Dup	0.861	NA	1.24	NA	0.242	NA
ISO14-0020-173	PA109B; FMS	18.7	89.1 (1)	20.4	96.3 (1)	19.9	98.2 (1)
Average Concentration (ng/mL) ± %RPD		**0.882 ng/mL ± 4.8%**		**1.15 ng/mL ± 16%**		**0.255 ng/mL ± 9.8%**	

		PFOS		PFOSA	
3M LIMS ID	**Sample Description**	**Concentration (ng/mL)**	**%Recovery**	**Concentration (ng/mL)**	**%Recovery**
ISO14-0020-171	PA109B; Sample	4.74	NA	<0.200	NA
ISO14-0020-172	PA109B; Sample Dup	4.69	NA	<0.200	NA
ISO14-0020-173	PA109B; FMS	23.4	93.5	12.2	61.0 (2)
Average Concentration (ng/mL) ± %RPD		**4.72 ng/mL ± 1.1%**		**<0.200 ng/mL (3)**	

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) The method uncertainty has been expanded to ±39% for PFOSA.

Table 42. Location ID: PA111B

		PFOA		PFBS		PFHS	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-174	PA111B; Sample	Not Reportable		Not Reportable		Not Reportable	
ISO14-0020-175	PA111B; Sample Dup						
ISO14-0020-176	PA111B; FMS						
Average Concentration (ng/mL) ± %RPD		**NR (1)**		**NR (1)**		**NR (1)**	

		PFOS		PFOSA	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-174	PA111B; Sample	9.65	NA	Not Reportable	
ISO14-0020-175	PA111B; Sample Dup	10.2	NA		
ISO14-0020-176	PA111B; FMS	25.3	77.0		
Average Concentration (ng/mL) ± %RPD		**9.93 ng/mL ± 5.5%**		**NR (1)**	

NA = Not Applicable
(1) NR = Not Reportable; sample not reported due to sample/sample duplicate RPD being greater than 50%.

Table 43. 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
ISO14-0020-167	Trip Blank; Sample	<0.192	NA	<0.200	NA	<0.200	NA
ISO14-0020-168	Trip Blank; FMS Low	10.6	106	9.75	97.5	9.98	99.8
ISO14-0020-169	Trip Blank; FMS Mid	106	106	19.4	97.0	99.7	99.7
ISO14-0020-170	Trip Blank; FMS High	512	102	46.6	93.2	487	97.4

		PFOS (1)		PFOSA (1)		PFOSA (2)	
3M LIMS ID	Sample Description	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery	Concentration (ng/mL)	%Recovery
ISO14-0020-167	Trip Blank; Sample	<0.464	NA	<0.200	NA	<0.200	NA
ISO14-0020-168	Trip Blank; FMS Low	10.3	103	6.25	62.5 (3)	9.41	94.1
ISO14-0020-169	Trip Blank; FMS Mid	105	105	12.5	62.5 (3)	19.2	96.0
ISO14-0020-170	Trip Blank; FMS High	503	100	31.3	62.6 (3)	52.4	105

NA = Not Applicable
(1) Results by external standard calibration.
(2) Results by internal standard calibration.
(3) The field matrix spike sample did not meet acceptance criteria of 100 ± 30% using external calibration.

Table 44. Surrogate Recovery Standard Results [1]

		Percent Recovery (%)	
3M LIMS ID	Sample Description	$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
ISO14-0020-001	BD24-3; Sample	108	106
ISO14-0020-002	BD24-3; Sample Dup	109	105
ISO14-0020-003	BD24-4; Sample	108	107
ISO14-0020-004	BD24-4; Sample Dup	108	107
ISO14-0020-005	BD24-4; FMS	105	105
ISO14-0020-006	D09; Sample	110	109
ISO14-0020-007	D09; Sample Dup	108	106
ISO14-0020-008	D09; FMS	106	106
ISO14-0020-009	D10; Sample	106	108
ISO14-0020-010	D10; Sample Dup	106	109
ISO14-0020-011	D10; FMS	109	110
ISO14-0020-012	D11; Sample	108	109
ISO14-0020-013	D11; Sample Dup	105	108
ISO14-0020-014	D11; FMS	106	107
ISO14-0020-015	D14; Sample	109	108
ISO14-0020-016	D14; Sample Dup	108	108
ISO14-0020-017	D14; FMS	107	105
ISO14-0020-018	P118A; Sample	103	108
ISO14-0020-019	P118A; Sample Dup	106	109
ISO14-0020-020	P118B; Sample	105	104
ISO14-0020-021	P118B; Sample Dup	106	100
ISO14-0020-022	P118B; FMS	106	104
ISO14-0020-023	P121; Sample	104	106
ISO14-0020-024	P121; Sample Dup	105	108
ISO14-0020-025	P321; Sample	101	106
ISO14-0020-026	P321; Sample Dup	101	108
ISO14-0020-027	3M pond; Sample	105	106
ISO14-0020-028	3M pond; Sample Dup	108	108
ISO14-0020-029	Blokkersdijkvijver std; Sample	107	108
ISO14-0020-030	Blokkersdijkvijver std;Sample Dup	107	107
ISO14-0020-031	L21; Sample	110	109
ISO14-0020-032	L21; Sample Dup	108	106
ISO14-0020-033	L22; Sample	108	107
ISO14-0020-034	L22; Sample Dup	104	107
ISO14-0020-035	L22; FMS	106	105
ISO14-0020-036	L31; Sample	107	108
ISO14-0020-037	L31; Sample Dup	107	105
ISO14-0020-038	L4; Sample	105	107
ISO14-0020-039	L4; Sample Dup	104	103
ISO14-0020-040	P114bis; Sample	106	106
ISO14-0020-041	P114bis; Sample Dup	106	106
ISO14-0020-042	P115; Sample	107	105
ISO14-0020-043	P115; Sample Dup	106	107
ISO14-0020-044	P116; Sample	105	106
ISO14-0020-045	P116; Sample Dup	104	105
ISO14-0020-046	P119C; Sample	101	94.6
ISO14-0020-047	P119C; Sample Dup	101	94.6

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

Table 45 continued. Surrogate Recovery Standard Results [1]

		Percent Recovery (%)	
3M LIMS ID	**Sample Description**	**$^{13}C_4$-PFOA**	**$^{13}C_4$-PFOS**
ISO14-0020-048	PP01; Sample	108	98.9
ISO14-0020-049	PP01; Sample Dup	105	98.9
ISO14-0020-050	PP01; FMS	106	96.9
ISO14-0020-051	PP02; Sample	105	94.0
ISO14-0020-052	PP02; Sample Dup	104	91.4
ISO14-0020-053	PP04; Sample	106	102
ISO14-0020-054	PP04; Sample Dup	105	104
ISO14-0020-055	PP05; Sample	103	102
ISO14-0020-056	PP05; Sample Dup	104	104
ISO14-0020-057	PP05; FMS	103	104
ISO14-0020-058	PP06; Sample	106	102
ISO14-0020-059	PP06; Sample Dup	105	104
ISO14-0020-060	PP06; FMS	106	104
ISO14-0020-061	PP07; Sample	108	106
ISO14-0020-062	PP07; Sample Dup	107	104
ISO14-0020-063	PP08; Sample	108	99.3
ISO14-0020-064	PP08; Sample Dup	107	97.6
ISO14-0020-065	PP09; Sample	105	106
ISO14-0020-066	PP09; Sample Dup	106	103
ISO14-0020-067	PP10; Sample	107	102
ISO14-0020-068	PP10; Sample Dup	106	104
ISO14-0020-071	13; Sample	99.0	98.0
ISO14-0020-072	13; Sample Dup	101	99.9
ISO14-0020-073	5; Sample	100	104
ISO14-0020-074	5; Sample Dup	101	103
ISO14-0020-075	Bemalingsstation; Sample	102	102
ISO14-0020-076	Bemalingsstation; Sample Dup	102	102
ISO14-0020-077	K1; Sample	103	103
ISO14-0020-078	K1; Sample Dup	102	102
ISO14-0020-079	K1; FMS	101	103
ISO14-0020-080	K3; Sample	105	101
ISO14-0020-081	K3; Sample Dup	105	104
ISO14-0020-082	K3; FMS	103	102
ISO14-0020-083	P21B; Sample	102	95.7
ISO14-0020-084	P21B; Sample Dup	102	97.8
ISO14-0020-085	P21B; FMS	101	99.4
ISO14-0020-086	P304; Sample	108	108
ISO14-0020-087	P304; Sample Dup	102	104
ISO14-0020-088	P304; FMS	105	104
ISO14-0020-089	P305; Sample	105	109
ISO14-0020-090	P305; Sample Dup	111	111
ISO14-0020-091	P305; FMS	104	111
ISO14-0020-092	P42; Sample	112	108
ISO14-0020-093	P42; Sample Dup	113	104
ISO14-0020-094	P42; FMS	110	107

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

Table 46 continued. Surrogate Recovery Standard Results [1]

		Percent Recovery (%)	
3M LIMS ID	Sample Description	$^{13}C_4$-PFOA	$^{13}C_4$-PFOS
ISO14-0020-095	P56; Sample	107	105
ISO14-0020-096	P56; Sample Dup	109	107
ISO14-0020-097	P56; FMS	112	110
ISO14-0020-098	P2; Sample	100	104
ISO14-0020-099	P2; Sample Dup	101	107
ISO14-0020-100	P2; FMS	89.8	83
ISO14-0020-101	P27; Sample	100	104
ISO14-0020-102	P27; Sample Dup	101	107
ISO14-0020-103	P27; FMS	99.0	78.1
ISO14-0020-104	P1065; Sample	111	108
ISO14-0020-105	P1065; Sample Dup	109	114
ISO14-0020-106	P1065; FMS	109	111
ISO14-0020-110	L19; Sample	111	107
ISO14-0020-111	L19; Sample Dup	112	108
ISO14-0020-112	L19; FMS	111	106
ISO14-0020-113	M4; Sample	113	104
ISO14-0020-114	M4; Sample Dup	112	104
ISO14-0020-115	M4; FMS	110	102
ISO14-0020-116	P118C; Sample	112	111
ISO14-0020-117	P118C; Sample Dup	114	113
ISO14-0020-118	P118C; FMS	115	111
ISO14-0020-119	P262; Sample	110	108
ISO14-0020-120	P262; Sample Dup	114	112
ISO14-0020-121	P262; FMS	113	110
ISO14-0020-122	P263; Sample	109	111
ISO14-0020-123	P263; Sample Dup	108	113
ISO14-0020-124	P263; FMS	109	104
ISO14-0020-125	P264; Sample	114	111
ISO14-0020-126	P264; Sample Dup	109	110
ISO14-0020-127	P265C; Sample	111	109
ISO14-0020-128	P265C; Sample Dup	116	114
ISO14-0020-129	P340; Sample	108	108
ISO14-0020-130	P340; Sample Dup	111	109
ISO14-0020-131	P340; FMS	110	109
ISO14-0020-132	P341; Sample	110	108
ISO14-0020-133	P341; Sample Dup	110	109
ISO14-0020-134	P341; FMS	109	108
ISO14-0020-135	P343; Sample	112	103
ISO14-0020-136	P343; Sample Dup	111	106
ISO14-0020-137	P343; FMS	109	104
ISO14-0020-138	P371; Sample	113	110
ISO14-0020-139	P371; Sample Dup	111	110
ISO14-0020-140	P374; Sample	111	113
ISO14-0020-141	P374; Sample Dup	111	109
ISO14-0020-142	P379; Sample	110	111
ISO14-0020-143	P379; Sample Dup	110	110
ISO14-0020-144	P379; FMS	111	115

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

Table 47 continued. Surrogate Recovery Standard Results [1]

		Percent Recovery (%)	
3M LIMS ID	**Sample Description**	**$^{13}C_4$-PFOA**	**$^{13}C_4$-PFOS**
ISO14-0020-145	P380; Sample	113	111
ISO14-0020-146	P380; Sample Dup	110	111
ISO14-0020-147	P380; FMS	111	110
ISO14-0020-148	P381; Sample	111	111
ISO14-0020-149	P381; Sample Dup	107	112
ISO14-0020-150	P382; Sample	111	110
ISO14-0020-151	P382; Sample Dup	112	111
ISO14-0020-152	B3; Sample	111	109
ISO14-0020-153	B3; Sample Dup	111	113
ISO14-0020-154	B3; FMS	110	110
ISO14-0020-155	B7; Sample	109	110
ISO14-0020-156	B7; Sample Dup	109	110
ISO14-0020-157	P372; Sample	109	109
ISO14-0020-158	P372; Sample Dup	110	108
ISO14-0020-159	P378; Sample	109	109
ISO14-0020-160	P378; Sample Dup	109	111
ISO14-0020-161	PA109A; Sample	107	112
ISO14-0020-162	PA109A; Sample Dup	107	111
ISO14-0020-163	PA111A; Sample	108	110
ISO14-0020-164	PA111A; Sample Dup	109	110
ISO14-0020-165	PA112; Sample	108	109
ISO14-0020-166	PA112; Sample Dup	109	107
ISO14-0020-167	Trip Blan; Sample	111	110
ISO14-0020-168	Trip Blank; FMS Low	112	111
ISO14-0020-169	Trip Blank; FMS Mid	110	112
ISO14-0020-170	Trip Blank; FMS High	109	110
ISO14-0020-171	PA109B; Sample	110	110
ISO14-0020-172	PA109B; Sample Dup	111	112
ISO14-0020-173	PA109B; FMS	109	112
ISO14-0020-174	PA111B; Sample	112	111
ISO14-0020-175	PA111B; Sample Dup	111	111
ISO14-0020-176	PA111B; FMS	111	110

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

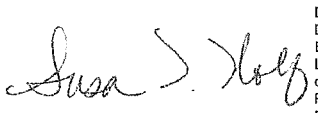
5 Conclusion

Laboratory control spikes were used to determine the analytical method accuracy and precision for all analytes. The accuracy and precision were then used to estimate the method uncertainty for the results. Field matrix spike recoveries demonstrated that the analytical method was appropriate for the given sample matrix except where noted. In those instances where the field matrix spike recovery did not meet method acceptance criteria, the method uncertainty has been adjusted accordingly. Analysis was completed using 3M Environmental Laboratory method ETS-8-044.1 "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 10-43 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: 2014.09.26 12:45:24 -05'00'

Susan T. Wolf, 3M Principal Analytical Investigator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William K. Reagen, Ph.D., 3M Environmental Laboratory Technical Director

The 3M Environmental Laboratory's Quality Assurance Unit has audited the data and report for this project.


Digitally signed by Matthew D. Naruko-Stewart
DN: c=US, st=MN, l=St. Paul, ou=Quality Assurance Unit, ou=3M Environmental Laboratory - authenticated by LRA, [redacted] o=3M, cn=Matthew D. Naruko-Stewart
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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

IAC14-03673-001

3M Lab Request Number: E14-1908

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

TESTING LABORATORY

SGS Belgium
Division IAC
Polderdijkweg 16
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Author: Sandra Graré
Date issued: September 18th, 2014

Requester

Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
Tel: [redacted]

1. Introduction - Summary.

At the request (Lab Request Number: E14-1908) of Robert A. Paschke, 3M Manager Corporate Environmental Programs, water samples were collected between July 29th and August 1st, 2014 from 57 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 add to 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
PP01	14.6	22759	38.8	8975	80.9
PP02	<16.7	3741	950	28848	<16.4
PP04	2955	960	2008	13963	164
PP05	18141	4614	1562	358	93.5
PP06	<8.75	48.4	218	3128	38.2
PP07	132	109	496	4156	64.5
PP08	29.4	320	734	9955	70.0
PP09	35.2	499	940	3344	7.33
PP10	16.0	214	759	5686	16.4
L4		2.38	6.03	41.0	<0.781
5		24.7	35.1	92.9	<0.781
Bemalingsstation		32.3	41.9	97.6	<0.781
K1	4.88	18.6	<1.70	33.4	5.70
Collector Put	43.4	29.1	95.4	506	32.5
P121		<1.98	<1.70	4.38	<0.781
BD24-3		111	204	1623	2.07
D09		10415	260	2696	20.3
D10		169	564	20.7	<1.56
D11		225	576	1047	38.0
P118A		4336	2577	3106	8.28
P118B		4130	2781	17205	<8.6
P321		913	3650	3501	<1.56
P119C		154	332	3228	66.1
13		24.1	110	330	<1.56

Sample identification	Concentration (in ng/mL, ppb)				
	PFBS	PFHS	PFOA	PFOS	FOSA
K3	1069	53016	101	5091	<8.60
P21B	6660	10842	15864	60918	<8.60
P304	159	90.8	652	1454	35.6
P305	194	428	358	1346	13.6
P42	44.8	546	642	7622	19.5
P56	13.7	16.1	70.2	301	14.6
P2	61.1	228	1920	3566	216
P27	7669	54.1	431	3910	192
P1065	<8.75	<21.8	<18.8	113	<8.60
L19		174	308	5345	61.1
M4		142	378	7859	204
P118C		36.7	141	2573	106
P262		444	1036	4902	52.4
P263		1040	3193	16254	11.1
P264		379	532	2828	66.2
P265C		31.1	89.7	1636	189
P340		328	907	5110	66.8
P341		647	1719	7193	<8.6
P343		816	1134	11581	19.2
P371		61.0	171	1379	64.9
P374		480	532	3096	37.2
P379		36.2	148	1803	86.5
P380		512	584	2531	130
P381		196	789	2239	17.7
P382		117	474	1674	139
B3		109	272	2149	115
B7		<1.98	4.63	309	<0.781
P372		97.3	144	4426	57.7
P378		229	245	856	<4.69
PA109A		95.7	94.8	345	4.04
PA111A		1361	1034	3220	12.5
PA112		45.1	285	632	79.4
Effluent WWTP	2410	(*)	(*)	(*)	(*)

(*) see results in analytical report IAC14-03673-002

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)
PP01	100
PP02	100
PP04	100
PP05	100
PP06	100
PP07	100
PP08	100
PP09	100
PP10	100
L4	100
5	100
Bemalingsstation	100
K1	100
Collector Put	100
P121	100
BD24-3	100
D09	100
D10	100
D11	100
P118A	100
P118B	100
P321	100
P119C	100
13	100
K3	100
P21B	100
P304	100
P305	100
P42	100
P56	100
P2	100
P27	100
P1065	100
L19	100
M4	100
P118C	100
P262	100
P263	100
P264	100
P265C	100
P340	100
P341	100
P343	100
P371	100
P374	100
P379	100
P380	100
P381	100

Sample Identification	Nominal Final Volume Collected *(ml)*
P382	100
B3	100
B7	100
P372	100
P378	100
PA109A	100
PA111A	100
PA112	100
Effluent WWTP	100
Field Trip Blank	100

2.2. Direct injection.

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

Table 3. Sample dilutions.

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
PP01	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP02	1.0	0.100	Solution A	10.0	0.5mL extract + 10.0mL MeOH (*)
PP04	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
PP05	1.0	0.100	Solution A	10.0	0.5mL extract + 10.0mL MeOH (*)
PP06	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 (*)
PP08	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 (*)
PP10	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
L4	1.0	0.100	Solution A	10.0	-
5	1.0	0.100	Solution A	10.0	-
Bemalingsstation	1.0	0.100	Solution A	10.0	-
K1	1.0	0.100	Solution A	10.0	-

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	10.0	-
P121	1.0	0.100	Solution A	10.0	-
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 (*)
D10	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
D11	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
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 (*)
P321	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
P119C	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
13	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
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 (*)
P304	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P305	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P42	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P56	1.0	0.100	Solution A	10.0	-
P2	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P27	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P1065	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
L19	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 (*)
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 (*)
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 (*)
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 (*)
P371	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)

Sample	Amount of Sample used (mL)	Volume of Spike Solution (mL)	Spike Solution Description	Volume of Solvent (*) (mL)	Extra Dilution
P374	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
P379	1.0	0.100	Solution A	10.0	-
P380	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
P381	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P382	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
B3	1.0	0.100	Solution A	10.0	1.0mL extract + 10.0mL MeOH (*)
B7	1.0	0.100	Solution A	10.0	-
P372	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
P378	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
PA109A	1.0	0.100	Solution A	10.0	-
PA111A	1.0	0.100	Solution A	10.0	1.0mL extract + 5.0mL MeOH (*)
PA112	1.0	0.100	Solution A	10.0	1.0mL extract + 1.0mL MeOH (*)
Effluent WWTP	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 0.0704 to 0.178 ng/mL for this project. Taking into account the dilution steps, as defined in ECO/AV/IAC/037, the LOQ was 0.781 to 41.6 ng/mL.

3.3. Continuing Calibration.

During the course of the analytical sequences, several continuing calibration verification samples (CCVs) were analyzed to confirm that the instrument response and the initial calibration curve was still in control. All CCVs, for all analytes, produced recoveries within ± 25%, which met method criteria.

3.4. Blanks.

Three types of blanks were prepared and analyzed with the samples: method blanks, solvent blanks, and field/trip blanks. Each blank type is described below. Analyte peak area counts in all blank samples must be less than half the area counts of the lowest valid calibration standard to meet the method criteria.

3.4.1. Method Blanks.

One method blank was prepared by diluting 1 mL water with 10.0 mL MeOH:LCMS-water (60:40). Method blanks were prepared to evaluate the levels of background contamination in the overall dilution process (glassware, etc.).

3.4.2. Solvent Blanks.

Several methanol blanks were analyzed to assess system contamination and/or instrument carryover.

3.4.3. Field /Trip Blank.

Prior to sample collection, one sample container was filled with 100 mL of LCMS-water, sealed, and shipped to the sample collection site along with the empty containers. This sample was analyzed as field/trip blank.

3.5. Lab Control Spikes (LCS)

One lab control spike was prepared and analyzed.
LCS was prepared by spiking known amounts of the analytes into 1.0 mL of LCMS-water to produce the desired concentration.
The spiked water sample was diluted with 10.0 mL MeOH:LCMS-water (60:40) and analyzed in the same manner as the samples.
The LCS produced recoveries within the method acceptance criteria of ± 25% for accuracy.
Table 7 summarizes the LCS recovery results

Table 7. Lab Control Spike Results.

Dilution date	Description	Nominal Spike Level (ng/mL)	Percent Recovery						
			PFBS	PFHS	PFOA	^{13}C-PFOA	PFOS	FOSA	^{13}C-PFOS
14 August 2014	LCS1	80	111	112	116	110	122	122	97.1

3.6. QC Lab Standards (QCs)

Two QC lab standards were prepared containing PFBS, PFHS, PFOA, ^{13}C-PFOA, PFOS, FOSA and ^{13}C-PFOS (QC Lab Low and QC Lab High).
The QCs produced recoveries within the method acceptance criteria of ± 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	107	92.8	123	111	119	109	109
	QC Lab High inj1	100	100	94.0	124	106	112	109	103
	QC Lab Low inj2	10	125	123	122	121	125	117	113
	QC Lab High inj2	100	123	121	124	118	120	120	108

3.7. Equations.

Recovery.

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

4. Data Summary.

The following tables summarize the results for the sample locations.

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

	PFBS	PFHS	PFOA	PFOS	FOSA
	ng/ml	ng/ml	ng/ml	ng/ml	ng/ml
PP01	14.6	22759	38.8	8975	80.9
PP02	<16.7	3741	950	28848	<16.4
PP04	2955	960	2008	13963	164
PP05	18141	4614	1562	358	93.5
PP06	<8.75	48.4	218	3128	38.2
PP07	132	109	496	4156	64.5
PP08	29.4	320	734	9955	70.0
PP09	35.2	499	940	3344	7.33
PP10	16.0	214	759	5686	16.4
L4		2.38	6.03	41.0	<0.781
5		24.7	35.1	92.9	<0.781
Bemalingsstation		32.3	41.9	97.6	<0.781
K1	4.88	18.6	<1.70	33.4	5.70
Collector Put	43.4	29.1	95.4	506	32.5
P121		<1.98	<1.70	4.38	<0.781
BD24-3		111	204	1623	2.07
D09		10415	260	2696	20.3
D10		169	564	20.7	<1.56
D11		225	576	1047	38.0
P118A		4336	2577	3106	8.28
P118B		4130	2781	17205	<8.6
P321		913	3650	3501	<1.56
P119C		154	332	3228	66.1
13		24.1	110	330	<1.56
K3	1069	53016	101	5091	<8.60
P21B	6660	10842	15864	60918	<8.60
P304	159	90.8	652	1454	35.6
P305	194	428	358	1346	13.6
P42	44.8	546	642	7622	19.5
P56	13.7	16.1	70.2	301	14.6
P2	61.1	228	1920	3566	216
P27	7669	54.1	431	3910	192
P1065	<8.75	<21.8	<18.8	113	<8.60
L19		174	308	5345	61.1
M4		142	378	7859	204
P118C		36.7	141	2573	106
P262		444	1036	4902	52.4
P263		1040	3193	16254	11.1
P264		379	532	2828	66.2
P265C		31.1	89.7	1636	189
P340		328	907	5110	66.8

	PFBS	PFHS	PFOA	PFOS	FOSA
	ng/ml	ng/ml	ng/ml	ng/ml	ng/ml
P341		647	1719	7193	<8.6
P343		816	1134	11581	19.2
P371		61.0	171	1379	64.9
P374		480	532	3096	37.2
P379		36.2	148	1803	86.5
P380		512	584	2531	130
P381		196	789	2239	17.7
P382		117	474	1674	139
B3		109	272	2149	115
B7		<1.98	4.63	309	<0.781
P372		97.3	144	4426	57.7
P378		229	245	856	<4.69
PA109A		95.7	94.8	345	4.04
PA111A		1361	1034	3220	12.5
PA112		45.1	285	632	79.4
Effluent WWTP	2410	*	*	*	*
Field Trip Blank	<0.796	<1.98	<1.70	<0.889	<0.781

* see results in analytical report IAC14-03673-002

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

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
PP01	142	118	120	97.5
PP02	146	122	107	86.6
PP04	152	127	128	104
PP05	146	122	128	104
PP06	142	119	117	95.4
PP07	128	107	119	96.7
PP08	135	113	119	96.3
PP09	139	116	118	95.8
PP10	152	127	140	114
L4	145	121	139	113
5	153	128	137	111
Bemalingsstation	144	120	138	112
K1	148	123	137	112
Collector Put	148	123	135	110
P121	148	124	138	112
BD24-3	136	114	124	101
D09	143	120	131	106
D10	127	106	128	104
D11	150	125	155	126
P118A	146	122	126	102
P118B	141	118	113	91.6
P321	132	110	126	102
P119C	122	102	133	108
13	136	113	129	105
K3	123	103	135	110

	^{13}C-PFOA		^{13}C-PFOS	
	ng/ml	% Rec	ng/ml	% Rec
P21B	133	111	116	94.1
P304	154	129	127	103
P305	154	128	133	108
P42	145	121	124	101
P56	152	127	147	119
P2	147	123	131	106
P14 (P27)	133	112	141	114
P1065	128	107	130	106
L19	152	127	136	111
M4	152	127	127	103
P118C	143	119	130	106
P262	153	128	141	115
P263	144	120	128	104
P264	153	128	123	100
P265C	148	124	114	92.7
P340	122	102	151	122
P341	134	112	129	105
P343	155	130	137	111
P371	137	115	123	100
P374	149	125	130	106
P379	155	129	130	105
P380	151	126	130	106
P381	152	127	138	112
P382	147	123	126	102
B3	152	127	124	101
B7	153	128	140	114
P372	152	127	124	101
P378	114	95.1	131	106
PA109A	155	130	140	114
PA111A	146	122	121	97.9
PA112	154	129	132	108
Effluent WWTP	249	108	251	113
Field Trip Blank	147	123	131	107

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,
Lab Operations Manager Date September 18th, 2014

Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories Date September 18th, 2014

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

SGS

3M Corporate Environmental Programs.
Robert A. Paschke
3M Center, 223-02-S-31
St. Paul, MN 55144-1000
USA

ANALYTICAL REPORT

IAC14-03673-002

3M Lab Request Number: E14-1908

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

TESTING LABORATORY

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1. Introduction - Summary.

At the request (Lab Request Number: E14-1908) of Robert A. Paschke, 3M Manager Corporate Environmental Programs, water samples were collected between July 29th and July 31st, 2014 from 11 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 - standard (filtered)	0.542	1.19	0.976	<0.025 (b)
Blokkersdijkvijver - standard (not filtered)	0.534	1.29	0.960	0.025 (a)
3M Vijver	0.378	1.17	5.10	0.108 (a)
L21	0.124	0.376	2.47	<0.025 (c)
L22	0.177	0.402	10.2	<0.025 (b)
L31	0.436	0.830	6.30	0.0375 (d)
P116	0.382	1.18	5.25	0.0396 (b)
P115	1.50	3.69	0.911	<0.025 (b)
P114 bis	1.15	4.72	11.0	<0.025 (d)
Effluent WWTP	28.3	39.9	19.7	0.133 (e)
BD24-4	3.37	9.95	7.78	0.0498 (f)
D14	1.12	2.18	1.64	<0.025 (g)

(a) The accuracy for FOSA for Blokkersdijkvijver-standard (not filtered) and 3M vijver is ± 40%.
(b) The accuracy for FOSA for Blokkersdijkvijver-standard (filtered), L22, P116 and P115 is ± 50%.
(c) The accuracy for FOSA for L21 is ± 45%.
(d) The accuracy for FOSA for L31 and P114 bis is ± 55%.
(e) The accuracy for FOSA for Effluent WWTP is ± 60%.
(f) The accuracy for FOSA for BD24-4 is ± 65%.
(g) The accuracy for FOSA for D14 is ± 75%.

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 - standard (filtered)	400	0.100	Solution A	0.25	-
		0.250	Solution B	-	1.0
Blokkersdijkvijver - standard (not filtered)	400	0.100	Solution A	0.25	-
		0.250	Solution B	-	1.0
3M Vijver	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
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
P116	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
P114bis	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
Effluent WWTP	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
BD24-4	100	0.025	Solution A	0.25	-
		0.070	Solution B	-	1.0
D14	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 - standard (filtered)	40	8
Blokkersdijkvijver - standard (not filtered)	40	8
3M Vijver	40	8
L21	40	8
L22	40	8
L31	40	8
P116	40	8
P115	40	8
P114 bis	40	8
Effluent WWTP	40	8
BD24-4	40	8
D14	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)	0.042

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.

Two method blanks were 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, except for FOSA LCSL02 and LCSH02 were the recoveries are 74.2% and 72.9%.
All LCSs produced recoveries within the method acceptance criteria of ± 15% RPD for precision.

Table 8 summarize the LCS recovery results.

Table 8. Lab Control Spike Results.

Extraction date	Description	Nominal Spike Level (ng/mL)	Percent Recovery							
			$^{13}C_4$-PFOA	$^{13}C_4$-PFOS	$^{18}O_2$-PFHS	$^{13}C_7$-PFuDA	PFHS	PFOA	PFOS	FOSA
14 August 2014	LCSL01	0.125	92.3	89.3	83.9	92.6	76.0	119	83.2	76.7
	LCSL02	0.125	80.8	90.8	81.7	87.9	76.4	112	86.6	74.2
	Average		**86.6**	**90.1**	**82.8**	**90.3**	**76.2**	**116**	**84.9**	**75.5**
	%RPD		**13**	**1.7**	**2.7**	**5.2**	**0.5**	**6.6**	**4.0**	**3.3**
	LCSH 01	2.5	100	97.7	99.4	111	84.6	116	79.3	81.7
	LCSH 02	2.5	86.0	87.8	91.0	97.0	78.7	100	76.0	72.9
	Average		**93.2**	**92.8**	**95.2**	**104**	**81.7**	**108**	**77.7**	**77.3**
	%RPD		**15**	**11**	**8.8**	**13**	**7.2**	**14**	**4.2**	**11**

3.6. Surrogates.

Surrogate recovery standards were added to all samples to evaluate overall method performance.

3.7. Internal Standards.

Internal standards were added to all samples to calculate the concentration of PFCs in the samples by using internal standard calibration.

3.8. Equations.

Recovery.

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

4. Data Summary.

Tabel 9 and 10 summarizes the results for the 11 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	89.9	<0.025	89.7
Blokkersdijkvijver – standard (filtered)	0.542	104	1.19	101
Blokkersdijkvijver – standard (not filtered)	0.534	99.5	1.29	107
3M Vijver	0.378	84.5	1.17	86.4
L21	0.124	73.2	0.376	82.3
L22	0.177	85.9	0.402	80.8
L31	0.436	84.1	0.830	78.7
P116	0.382	75.9	1.18	81.6
P115	1.50	80.6	3.69	79.9
P114bis	1.15	78.6	4.72	81.7
Effluent WWTP	28.3	75.8	39.9	76.3
BD24-4	3.37	74.0	9.95	74.4
D14	1.12	75.2	2.18	83.9

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	89.2	<0.025	95.6
Blokkersdijkvijver – standard (filtered)	0.976	103	<0.025	53.6 (a)
Blokkersdijkvijver – standard (not filtered)	0.960	96.1	0.025	64.4 (a)
3M Vijver	5.10	87.9	0.108	62.6 (a)
L21	2.47	76.4	<0.025	57.0 (a)
L22	10.2	87.1	<0.025	51.6 (a)
L31	6.30	85.1	0.0375	48.8 (a)
P116	5.25	78.1	0.0396	50.2 (a)
P115	0.911	82.1	<0.025	50.0 (a)
P114bis	11.0	95.0	<0.025	47.1 (a)
Effluent WWTP	19.7	85.0	0.133	43.2 (a)
BD24-4	7.78	87.4	0.0498	36.7 (a)
D14	1.64	79.9	<0.025	29.1 (a)

(a): The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130%.

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

- The accuracy for FOSA for Blokkersdijkvijver-standard (not filtered) and 3M vijver is ± 40%.
- The accuracy for FOSA for Blokkersdijkvijver-standard (filtered), L22, P116 and P¨115 is ± 50%.
- The accuracy for FOSA for L21 is ± 45%.
- The accuracy for FOSA for L31 and P114 bis is ± 55%.
- The accuracy for FOSA for Effluent WWTP is ± 60%.
- The accuracy for FOSA for BD24-4 is ± 65%.
- The accuracy for FOSA for D14 is ± 75%.

5. Conclusion.

- The recovery of the surrogate recovery standard fell outside the method acceptance criterion of 70-130% for eleven sample locations.
- Lab control spike recoveries fell within the method acceptance criteria of 25%.

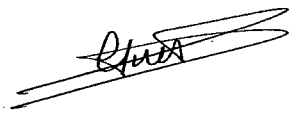
6. Data / Sample Retention.

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.

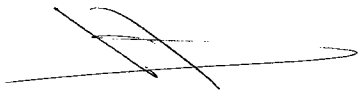
7. Addendum.

Addendum: e-mail from Marleen Coetsiers (ERM Belgium).

8. Signatures.



Geert De Smet,
Lab Operations Manager Date September 18th, 2014



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories Date September 18th, 2014

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3M Corporate Environmental Programs.
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3M Center, 223-02-S-31
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USA

ANALYTICAL REPORT

IAC14-03674

3M Lab Request Number: E14-1908

Analysis of pH, Conductivity and Mineral Oil in Aqueous Samples
3M, Zwijndrecht, Belgium

TESTING LABORATORY

SGS Belgium
Division IAC
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Date issued: September 18th, 2014

Requester

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1. Introduction.

At the request (Lab Request Number: E14-1908) of Robert A. Paschke, 3M Manager Corporate Environmental Programs, water samples were collected on July 31st and August 1st 2014 from 8 locations and analyzed for :

- pH
- Conductivity
- Mineral oil; fractions C-10 – C-40

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 send by SGS Belgium NV, Division IAC Laboratory.

2.2. Analysis.

2.2.1. pH.

Samples were analyzed according to CMA/2/I/A.1 and WAC/III/A/005, the official Dutch method.

2.2.2. Conductivity.

Samples were analyzed according to CMA/2/I/A.2 and WAC/III/A/004, the official Dutch method.

2.2.3. Mineral oil.

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

3. Results.

Table 1-3 summarizes the sample results.

Table 1. pH results.

Sample identification	Temp (°C)	pH
P21B	23.8	7.4
P305	24.3	7.1
P16	24.7	7.3
P17	24.8	7.2
P13	24.9	7.2
U14	25.0	7.0
P18	25.0	12.4 (*)
P28	25.0	13.0 (*)

(*): semi-kwantitative result

Table 2. Conductivity results.

Sample identification	Temp (°C)	Conductivity (µS/cm)
P21B	20.1	2400
P305	20.1	970
P16	20.1	710
P17	20.1	570
P13	20.1	640
U14	20.2	1700
P18	20.2	58000
P28	20.1	76000

Table 3. 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
P21B	< 25	25	< 25	< 25	< 100
P305	< 25	< 25	< 25	< 25	< 100
P16	< 25	< 25	< 25	< 25	< 100
P17	< 25	< 25	< 25	< 25	< 100
P13	< 25	< 25	< 25	< 25	< 100
U14	< 25	< 25	< 25	< 25	< 100
P18	170	1000	740	31	2000
P28	360	1000	340	< 250	1800

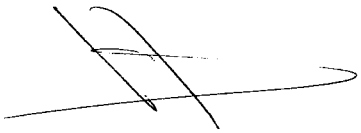
4. Data / Sample Retention.

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 18th, 2014



Marc Van Ryckeghem,
Business Unit Manager Environmental Laboratories — Date September 18th, 2014

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.

Bijlage 7

Overzicht veldmetingen en analyseresultaten

PuntlocatieId	PP09	PP10	PP01	PP02	PP04	PP05	PP06	PP07	PP08	PP09	PP10	BD24-3	D10	L31
BemonsteringsDatum	27-08-13	27-08-13	01-10-13	01-10-13	01-10-13	01-10-13	01-10-13	01-10-13	01-10-13	01-10-13	01-10-13	14-10-13	14-10-13	14-10-13
TopFilter	3	3	2	3,2	2	1,7	1,3	2	3,2	3	3	17	15	1,2
BodemFilter	6	6	5,5	6,2	6	5,7	5,3	6	7,2	6	6	18	16	3,2
Grondwaterpeil (cm-TOP)												3,26	4,34	2,05
Temperatuur (°C)												11,4	11,7	13,29
pH												7,59	7,03	7,21
Geleidbaarheid (µS/cm)												360	1617	519
Minerale olie (GCMS) (µg/l)														
Minerale olie C10 - C12 (µg/l)														
Minerale olie C12 - C20 (µg/l)														
Minerale olie C20 - C30 (µg/l)														
Minerale olie C30 - C40 (µg/l)														
PFBS (µg/l)														
PFHS (µg/l)	529	128	9840	2983	860	3928	60,4	129	282	458	196	11	102	0,711
PFOA (µg/l)	875	372	61,6	591	1819	1314	227	499	615	799	690	19,5	301	1,25
PFOS (µg/l)	3391	3394	3353	29989	8384	375	3273	3397	8469	2757	4666	231	20,6	11,6
PFOSA (µg/l)	< 23,8	57,6	19,1	<20,2	29,7	76,2	46,2	57,3	42,5	<10,6	13,9	16,4	<1,96	0,0771

PuntlocatieId	P116	P321	PP09	PP10	PP09	PP10	PP01	PP02	PP04	PP05	PP07	PP08	PP09	PP10
BemonsteringsDatum	14-10-13	14-10-13	14-10-13	14-10-13	07-11-13	07-11-13	23-12-13	23-12-13	23-12-13	23-12-13	23-12-13	23-12-13	23-12-13	23-12-13
TopFilter	2,5	15,5	3	3	3	3	2	3,2	2	1,7	2	3,2	3	3
BodemFilter	3,5	16,5	6	6	6	6	5,5	6,2	6	5,7	6	7,2	6	6
Grondwaterpeil (cm-TOP)	2,255	4,035												
Temperatuur (°C)	12,99	13,12												
pH	7,45	7,07												
Geleidbaarheid (µS/cm)	529	1703												
Minerale olie (GCMS) (µg/l)														
Minerale olie C10 - C12 (µg/l)														
Minerale olie C12 - C20 (µg/l)														
Minerale olie C20 - C30 (µg/l)														
Minerale olie C30 - C40 (µg/l)														
PFBS (µg/l)														
PFHS (µg/l)	0,737	1503	437	193	446	188	7031	3016	620	4021	142	343	420	188
PFOA (µg/l)	1,31	5626	804	617	851	700	67	639	1651	1326	548	667	769	646
PFOS (µg/l)	11	1884	2574	4416	2595	4124	3443	26323	5390	555	3392	8201	2534	4586
PFOSA (µg/l)	0,081	<5,89	<21,6	<21,6	<12,9	13,5	20,5	24,8	25,3	94,5	45,1	45	12,2	20,1

PuntlocatieId	BD24-3	D10	L21	L22	L31	L4	P115	P116	P118A	P118C	P119C	P121	P265C	P381
BemonsteringsDatum	21-01-14	21-01-14	21-01-14	21-01-14	21-01-14	21-01-14	21-01-14	21-01-14	21-01-14	21-01-14	21-01-14	21-01-14	21-01-14	21-01-14
TopFilter	17	15	3	2	1,2	2	3,3	2,5	22,5	2,5	2,5	23,5	4,5	5
BodemFilter	18	16	5	4	3,2	4	4,3	3,5	23,5	4,5	4,5	24,5	5,5	6
Grondwaterpeil (cm-TOP)	311	425	148	125	192	158	111	204	273	235	125	452		265
Temperatuur (°C)	10,6	10,9	9,5	9,4	8,2	8,7	10,3	9,9	10,11	9,53	9,8	11		9,19
pH	7,48	7,18	7,65	7,56	7,44	7,54	7	7,67	6,91	7,09	7,5	6,46		6,98
Geleidbaarheid (µS/cm)	517	2115	419	540	523	362	3310	616	2407	1369	731	2630		909
Minerale olie (GCMS) (µg/l)														
Minerale olie C10 - C12 (µg/l)														
Minerale olie C12 - C20 (µg/l)														
Minerale olie C20 - C30 (µg/l)														
Minerale olie C30 - C40 (µg/l)														
PFBS (µg/l)														
PFHS (µg/l)	18,7	149	0,189	0,401	0,859	2,28	2,1	0,832	5170	559	115	0,153	44,1	184
PFOA (µg/l)	28,2	447	0,678	0,879	0,919	2,96	4,5	2,23	2600	846	221	0,27	121	656
PFOS (µg/l)	288	41,6	4,16	12,1	8,07	21,9	0,518	9,17	1890	2340	1730	4,95	1170	1650
PFOSA (µg/l)	21,1	0,076	0,0327	0,0353	0,0316	6,25	<0,025	0,0685	9,77	57,1	61,4	0,31	110	17,2

PuntlocatieId	P382	B7	P264	P321	P371	P372	P374	P378	PA109A	PA111A	PA112	Z1-3m	Z2-10m	Z2-3m
BemonsteringsDatum	21-01-14	22-01-14	22-01-14	22-01-14	22-01-14	22-01-14	22-01-14	22-01-14	22-01-14	22-01-14	22-01-14	22-01-14	22-01-14	22-01-14
TopFilter	2,5	1,8	5	15,5	1,5	4,8	5	3,5	5	4	3,45	2	8	3,5
BodemFilter	3,5	3,8	5,5	16,5	3,5	5,8	6	5,5	6	6	5,45	4	10	5,5
Grondwaterpeil (cm-TOP)	223	194	257	362	207	227	381	293	312	232	246	136	309	306
Temperatuur (°C)	8,94	8,8	8,67	11,11	7,1	8,9	8,3	11	9,6	8,9	9,2	8,91	11	9,9
pH	7,12	7,73	7,46	7,23	7,33	7,17	6,85	6,85	0,61	6,64	7,48	7,18	7	6,86
Geleidbaarheid (µS/cm)	831	333	870	2009	553	494	1140	1044	1400	1351	1092	660	1202	846
Minerale olie (GCMS) (µg/l)														
Minerale olie C10 - C12 (µg/l)														
Minerale olie C12 - C20 (µg/l)														
Minerale olie C20 - C30 (µg/l)														
Minerale olie C30 - C40 (µg/l)														
PFBS (µg/l)														
PFHS (µg/l)	175	0,822	259	1370	31,3	64,6	668	346	304	1070	32,2	0,733	1,37	1,19
PFOA (µg/l)	623	1,85	390	6310	78,8	100	778	342	306	824	155	0,878	1,3	1,38
PFOS (µg/l)	977	32,5	1920	2510	859	1780	2770	1190	516	2300	336	0,459	1,46	0,456
PFOSA (µg/l)	79,6	0,0852	51,6	3,66	47,3	50,6	32,8	1,79	1,17	12,1	48,4	0,0335	0,0471	<0,0250

PuntlocatieId	PP01	PP02	PP07	PP08	PP09	PP10	PP09	PP10	L4	P114bis	P116	P321	PP01	PP02
BemonsteringsDatum	28-01-14	28-01-14	28-01-14	28-01-14	28-01-14	28-01-14	25-02-14	25-02-14	02-04-14	02-04-14	02-04-14	02-04-14	02-04-14	02-04-14
TopFilter	2	3,2	2	3,2	3	3	3	3	2	3,6	2,5	15,5	2	3,2
BodemFilter	5,5	6,2	6	7,2	6	6	6	6	4	4,6	3,5	16,5	5,5	6,2
Grondwaterpeil (cm-TOP)									153	143	240	369		
Temperatuur (°C)									9,9	10,6	10,8	13,5		
pH									7,24	7,24	7,68	7,11		
Geleidbaarheid (µS/cm)									578	1291	754	1472		
Minerale olie (GCMS) (µg/l)														
Minerale olie C10 - C12 (µg/l)														
Minerale olie C12 - C20 (µg/l)														
Minerale olie C20 - C30 (µg/l)														
Minerale olie C30 - C40 (µg/l)														
PFBS (µg/l)	72,2	<26,2	144	39,6	29,2	16	32,6	19,2					80,7	24,9
PFHS (µg/l)	4133	1803	120	284	399	167	345	169	2,07	1,67	0,784	607	11200	2450
PFOA (µg/l)	55	753	490	599	722	619	715	601	2,6	4,1	1,98	1770	73,7	685
PFOS (µg/l)	2202	27617	3523	8436	2693	4485	2208	3892	23,2	16,3	7,46	3720	4630	24000
PFOSA (µg/l)	18,8	33,5	55,4	46,2	17,6	17,6	<10,9	<10,9	2,33	0,0735	0,0919	12	27,6	25,1

PuntlocatieId	PP04	PP05	PP07	PP08	PP09	PP10	L19	M4	P118A	P118B	P118C	P2	P262	P263
BemonsteringsDatum	02-04-14	02-04-14	02-04-14	02-04-14	02-04-14	02-04-14	29-07-14	29-07-14	29-07-14	29-07-14	29-07-14	29-07-14	29-07-14	29-07-14
TopFilter	2	1,7	2	3,2	3	3	2,8	1	22,5	13	2,5		6,2	7,4
BodemFilter	6	5,7	6	7,2	6	6	4,8	3	23,5	14	4,5		7,2	8,4
Grondwaterpeil (cm-TOP)									316	316	291			
Temperatuur (°C)									14,2	14,4	16,5			
pH									6,87	7,04	7,19			
Geleidbaarheid (µS/cm)									2320	1806	608			
Minerale olie (GCMS) (µg/l)														
Minerale olie C10 - C12 (µg/l)														
Minerale olie C12 - C20 (µg/l)														
Minerale olie C20 - C30 (µg/l)														
Minerale olie C30 - C40 (µg/l)														
PFBS (µg/l)	2510	42200	136	42,6	31,7	23,3						55,5		
PFHS (µg/l)	855	4620	142	319	468	212	156	135	4370	4190	41,2	222	270	935
PFOA (µg/l)	2050	1800	544	720	858	785	267	355	2240	2530	116	1790	653	2830
PFOS (µg/l)	8140	165	3840	9170	2640	4760	3500	5410	2180	12900	1520	2240	2190	12200
PFOSA (µg/l)	40,9	38,2	72,7	51,8	15,7	16	48,1	125	5,4	3,92	53,2	110	21,2	8,33

PuntlocatieId	P264	P265C	P321	P343	P371	P372	P374	P380	P381	PP01	PP02	PP04	PP05	PP06
BemonsteringsDatum	29-07-14	29-07-14	29-07-14	29-07-14	29-07-14	29-07-14	29-07-14	29-07-14	29-07-14	29-07-14	29-07-14	29-07-14	29-07-14	29-07-14
TopFilter	5	4,5	15,5	4,8	1,5	4,8	5	3,5	5	2	3,2	2	1,7	1,3
BodemFilter	5,5	5,5	16,5	6,8	3,5	5,8	6	5,5	6	5,5	6,2	6	5,7	5,3
Grondwaterpeil (cm-TOP)	276	0		361,5	240,5	265,5	304,5	346	306,5					
Temperatuur (°C)	16,5	18,3		16,84	15,97	18,28	16,72	18,16	17,5					
pH	7,28	7,39		6,91	7,03	6,88	6,54	6,63	6,82	7,33	7,4	7,2		7,19
Geleidbaarheid (µS/cm)	821	531		1152	539	491	883	749	852	588	908	911		738
Minerale olie (GCMS) (µg/l)														
Minerale olie C10 - C12 (µg/l)														
Minerale olie C12 - C20 (µg/l)														
Minerale olie C20 - C30 (µg/l)														
Minerale olie C30 - C40 (µg/l)														
PFBS (µg/l)										18	29,2	2700	16700	15,5
PFHS (µg/l)	346	30,6	856	703	49,8	67,2	456	461	179	27700	3220	892	5510	48,8
PFOA (µg/l)	487	99,2	3340	1010	138	98,8	483	551	677	55,2	702	1710	1550	198
PFOS (µg/l)	1850	1030	2520	8100	799	1740	2010	1730	1520	8860	27100	10800	313	2070
PFOSA (µg/l)	27,7	81,2	<2	11,9	34,7	24,2	22,2	63,1	7,81	67,1	22,4		64	25,6

PuntlocatieId	PP07	PP08	PP09	PP10	BD24-3	BD24-4	D10	D14	L21	L22	L31	L4	P1065	P114bis
BemonsteringsDatum	29-07-14	29-07-14	29-07-14	29-07-14	30-07-14	30-07-14	30-07-14	30-07-14	30-07-14	30-07-14	30-07-14	30-07-14	30-07-14	30-07-14
TopFilter	2	3,2	3	3	17	22	15	15,5	3	2	1,2	2		3,6
BodemFilter	6	7,2	6	6	18	24	16	16,5	5	4	3,2	4		4,6
Grondwaterpeil (cm-TOP)					326,5	327		492,5	184	190	223		154,5	160
Temperatuur (°C)					12,56	13,51		17,1	16,6	16,5	16,2		22,53	14,1
pH	7,19	7,08	7,04	6,5	7,26	6,96		6,89	7,5	7,4	7,19		7,02	7,04
Geleidbaarheid (µS/cm)	829	932	859	882	757	1291		3450	382	356	582		469	1825
Minerale olie (GCMS) (µg/l)														
Minerale olie C10 - C12 (µg/l)														
Minerale olie C12 - C20 (µg/l)														
Minerale olie C20 - C30 (µg/l)														
Minerale olie C30 - C40 (µg/l)														
PFBS (µg/l)	121	34	28,4	18,2									12,3	
PFHS (µg/l)	114	315	442	191	127	4,7	174	1,41	<0,2	0,212	0,494	3,99	3,14	1,53
PFOA (µg/l)	456	668	821	681	202	11,8	503	2,24	0,418	0,512	0,925	6,57	9,29	4,93
PFOS (µg/l)	3100	8790	2570	4270	1660	8,42	26,8	1,03	3,58	11,9	8,03	27,5	64,6	11,5
PFOSA (µg/l)	47	56,3	10,4	12	2,48	0,197	0,156	0,0838	<0,2	0,11	<0,2	0,218	3,97	<0,2

PuntlocatieId	P115	P116	P119C	P340	P379	P382	D11	D9	K1	K3	P121	P21B	P304	P305
BemonsteringsDatum	30-07-14	30-07-14	30-07-14	30-07-14	30-07-14	30-07-14	31-07-14	31-07-14	31-07-14	31-07-14	31-07-14	31-07-14	31-07-14	31-07-14
TopFilter	3,3	2,5	2,5	4	3,5	2,5	14,9	13	2,3	3,6	23,5	3,5	4	3,5
BodemFilter	4,3	3,5	4,5	6	5,5	3,5	15,9	15	4,3	5,6	24,5	5,5	6	5,5
Grondwaterpeil (cm-TOP)	177	244	201		253	279,5	342,5	342	204	207		116	240,5	178
Temperatuur (°C)	14,8	14,8	13,76		18,91	14,8	14,87	19,1	19,7	19,9		18,1	17,5	18,9
pH	6,96	7,59	7,24		6,95	7,05	6,97	7,14	7,4	7,31		7,36	7,1	6,99
Geleidbaarheid (µS/cm)	3440	657	700		532	667	738	1167	1860	634		2095	1075	965
Minerale olie (GCMS) (µg/l)												<100		<100
Minerale olie C10 - C12 (µg/l)												<25		<25
Minerale olie C12 - C20 (µg/l)												25		<25
Minerale olie C20 - C30 (µg/l)												<25		<25
Minerale olie C30 - C40 (µg/l)												<25		<25
PFBS (µg/l)									4,45	816		5320	129	128
PFHS (µg/l)	1,76	0,646	185	319	33,7	103	212	10800	13,1	54400	<0,2	10700	90	312
PFOA (µg/l)	3,77	1,49	336	818	130	416	470	240	2,54	101	0,225	14100	584	285
PFOS (µg/l)	<0,464	7,42	2250	3420	1100	1010	561	1930	16	3280	3,46	77800	868	660
PFOSA (µg/l)	<0,2	<0,2	37,2	26,6	33,8	57,8	15,8	37,8	2,03	21,9	<0,2	14,1	15,4	9,83

PuntlocatieId	P341	P28	B3	B7	P27	P378	P42	P56	PA109A	PA109B	PA111A	PA111B	PA112	U14
BemonsteringsDatum	31-07-14	31-07-14	01-08-14	01-08-14	01-08-14	01-08-14	01-08-14	01-08-14	01-08-14	01-08-14	01-08-14	01-08-14	01-08-14	01-08-14
TopFilter	3,5	3,1	0,4	1,8		3,5	3	1,2	5	2	4	1,5	3,45	1
BodemFilter	5,5	3,1	1,4	3,8	3,44	5,5	5	3,2	6	3	6	3,5	5,45	3
Grondwaterpeil (cm-TOP)	273	195	121	258	148	246	274,5	166	337	280	274	276	278	2,14
Temperatuur (°C)	16,78		16,63	17,21	22,1	16,63	18,6	18,8	17,5	22,38	20,39	21,41	14,84	21
pH	6,84		6,84	7,33	7,37	6,66	6,94	7,52	6,9	7,56	6,48	7,14	7,06	6,85
Geleidbaarheid (µS/cm)	656		655	293	824	798	1692	500	548	331	1313	323	676	1659
Minerale olie (GCMS) (µg/l)		1800												<100
Minerale olie C10 - C12 (µg/l)		360												<25
Minerale olie C12 - C20 (µg/l)		1000												<25
Minerale olie C20 - C30 (µg/l)		340												<25
Minerale olie C30 - C40 (µg/l)		<250												<25
PFBS (µg/l)					6230		45,7	10,7						
PFHS (µg/l)	607		95,8	1,08	48,4	247	505	16	89,2	0,255	1220		44,9	
PFOA (µg/l)	1630		248	4,85	371	242	558	60	89,1	0,882	916		256	
PFOS (µg/l)	5100		1240	183	2300	740	4950	184	236	4,72	2390	9,93	393	
PFOSA (µg/l)	8,88		46,7	0,592	107	<2	15,6	7,88	0,991	<0,2	9,98		28,8	

PuntlocatieId	P13	P16	P17	P18
BemonsteringsDatum	01-08-14	01-08-14	01-08-14	01-08-14
TopFilter	onbekend	4	4	1,4
BodemFilter	5	6	6	3,4
Grondwaterpeil (cm-TOP)	2,04	2,21	2,26	
Temperatuur (°C)	17,3	17,1	16,1	
pH	7,33	7,38	7,41	
Geleidbaarheid (µS/cm)	595	721	616	
Minerale olie (GCMS) (µg/l)	<100	<100	<100	2000
Minerale olie C10 - C12 (µg/l)	<25	<25	<25	170
Minerale olie C12 - C20 (µg/l)	<25	<25	<25	1000
Minerale olie C20 - C30 (µg/l)	<25	<25	<25	740
Minerale olie C30 - C40 (µg/l)	<25	<25	<25	31
PFBS (µg/l)				
PFHS (µg/l)				
PFOA (µg/l)				
PFOS (µg/l)				
PFOSA (µg/l)				

PuntlocatieId	Collector put	Collector put	Collector put	Collector put	Effluent WWTP	Effluent WWTP	Effluent WWTP	Effluent WWTP
BemonsteringsDatum	15-10-13	28-01-14	2-04-14	29-07-14	14-10-13	28-01-14	2-04-14	29-07-14
PFHS (µg/l)	13,1	15,2	21,4	29,1	<1,82	<0,614	0,883	28,3
PFOA (µg/l)	48,2	54,9	75,1	95,4	<2,13	<1,03	2,00	39,9
PFOS (µg/l)	183	303	310	506	<1,13	<1,28	1,04	19,7
PFOSA (µg/l)	12,8	12,7	14,6	32,5	<1,96	<0,655	<0,503	0,13

PuntlocatieId	5	5	5	5	12	12	12	13	13	13	13	Bemalingsstation	Bemalingsstation	Bemalingsstation
BemonsteringsDatum	15-10-13	23-01-14	2-04-14	31-07-14	15-10-13	23-01-14	2-04-14	14-10-13	23-01-14	2-04-14	31-07-14	15-10-13	23-01-14	2-04-14
PFHS (µg/l)	7,61	19,5	31,8	21,4	8,63	18,3	31,7	9,45	18,1	30,2	25,6	8,72	13,7	25,6
PFOA (µg/l)	13	28,6	48	31,8	29,1	66,2	123	30	64,8	111	93,3	15,4	20,2	38
PFOS (µg/l)	27,8	64,7	81,1	53	110	208	321	106	211	291	206	38,6	44,1	65,6
PFOSA (µg/l)	<1,96	0,131	0,215	0,2	<1,96	0,371	0,313	<1,96	0,348	0,34	0,2	<1,96	0,128	0,232

PuntlocatieId	Bemalingsstation	3M vijver	3M vijver	3M vijver	3M vijver	Blokkersdijkvijver standaard	Blokkersdijkvijver standaard	Blokkersdijkvijver standaard	Blokkersdijkvijver standaard	Blokkersdijkvijver oost	Blokkersdijkvijver oost
BemonsteringsDatum	31-07-14	14-10-13	23-01-14	2-04-14	31-07-14	14-10-13	23-01-14	2-04-14	31-07-14	15-10-13	4-02-14
PFHS (µg/l)	25,9	0,506	0,566	0,752	0,446	0,447	0,666	0,666	0,596	0,787	0,515
PFOA (µg/l)	37,3	1,24	1,48	1,7	1,29	1,36	1,27	1,31	1,27	1,67	1,14
PFOS (µg/l)	61,6	3,59	4,13	4	3,85	0,198	1,06	1,17	0,86	8,43	1,44
PFOSA (µg/l)	0,2	<0,0283	0,0252	0,05	0,2	<0,0283	<0,025	0,05	0,2	0,146	0,0861

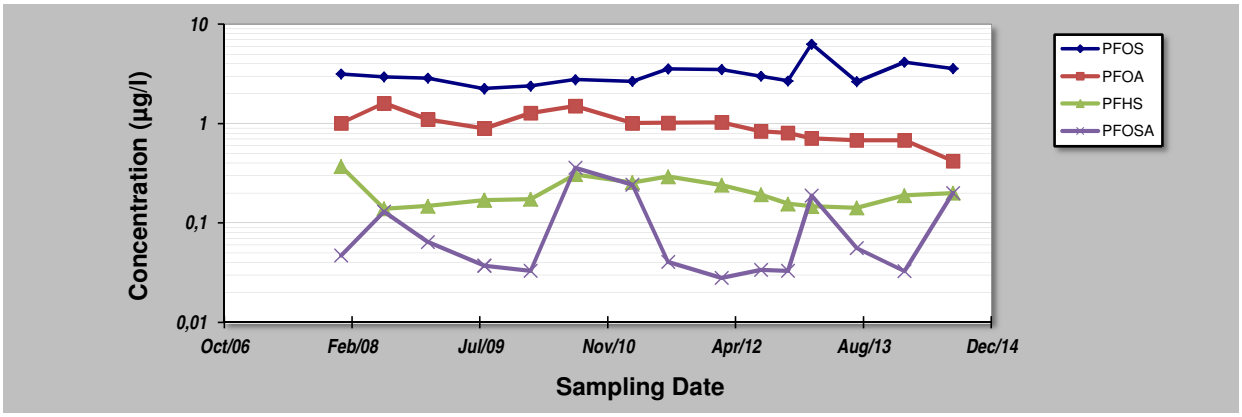
Bijlage 8

Statistische evaluatie analyseresultaten Blokkersdijk

GSI MANN-KENDALL TOOLKIT
for Constituent Trend Analysis

Evaluation Date:	06-okt-14	Job ID:	0243999
Facility Name:	3M	Constituent:	L21
Conducted By:	MCO	Concentration Units:	µg/l

Sampling Point ID:	PFOS	PFOA	PFHS	PFOSA			
Sampling Event	Sampling Date	L21 CONCENTRATION (µg/l)						
1	10-Jan-08	3,16	1,01	0,369	0,047			
2	26-Jun-08	2,95	1,6	0,139	0,13			
3	15-Dec-08	2,86	1,1	0,148	0,0642			
4	23-Jul-09	2,245	0,8935	0,17	0,03705			
5	24-Jul-09	2,25	0,894	0,17	0,0371			
6	20-Jan-10	2,39	1,28	0,174	0,0329			
7	14-Jul-10	2,77	1,5	0,307	0,36			
8	22-Feb-11	2,66	1,01	0,256	0,242			
9	12-Jul-11	3,555	1,0175	0,293	0,0405			
10	6-Feb-12	3,51	1,03	0,24	0,028			
11	9-Jul-12	3	0,838	0,193	0,0337			
12	23-Oct-12	2,69	0,804	0,156	0,0329			
13	23-Jan-13	6,31	0,709	0,147	0,188			
14	19-Jul-13	2,64	0,678	0,142	0,0557			
15	21-Jan-14	4,16	0,678	0,189	0,0327			
16	30-Jul-14	3,58	0,42	0,2	0,2			
17								
18								
19								
20								
Coefficient of Variation:		0,31	0,32	0,33	1,01			
Mann-Kendall Statistic (S):		34	-72	-5	-13			
Confidence Factor:		93,0%	100,0%	57,1%	70,3%			
Concentration Trend:		Prob. Increasing	Decreasing	Stable	No Trend			



Notes:

1. At least four independent sampling events per well are required for calculating the trend. *Methodology is valid for 4 to 40 samples.*
2. Confidence in Trend = Confidence (in percent) that constituent concentration is increasing (S>0) or decreasing (S<0): >95% = Increasing or Decreasing; ≥ 90% = Probably Increasing or Probably Decreasing; < 90% and S>0 = No Trend; < 90%, S≤0, and COV ≥ 1 = No Trend; < 90% and COV < 1 = Stable.
3. Methodology based on "MAROS: A Decision Support System for Optimizing Monitoring Plans", J.J. Aziz, M. Ling, H.S. Rifai, C.J. Newell, and J.R. Gonzales, *Ground Water*, 41(3):355-367, 2003.

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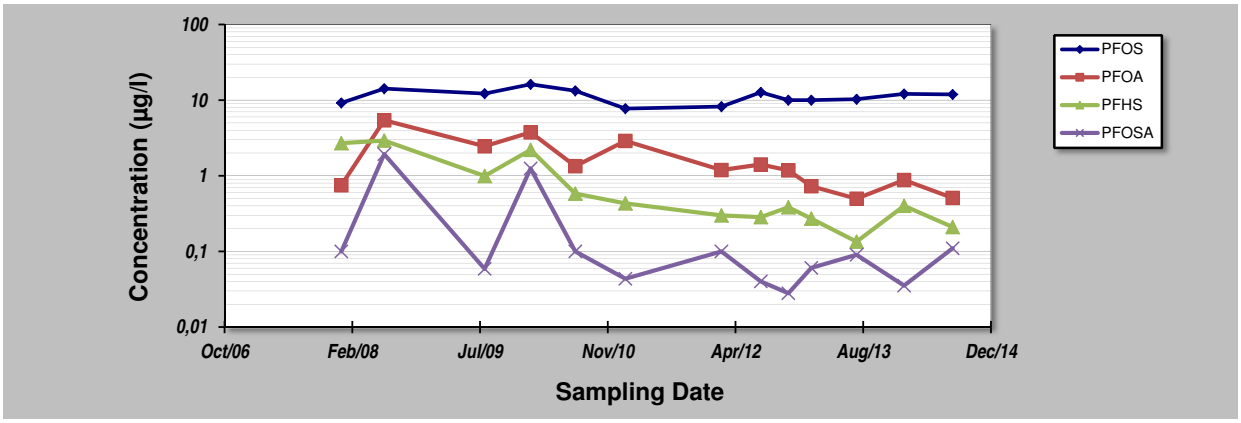
GSI MANN-KENDALL TOOLKIT
for Constituent Trend Analysis

Evaluation Date:	06-okt-14	Job ID:	0243999
Facility Name:	3M	Constituent:	L22
Conducted By:	MCO	Concentration Units:	µg/l

Sampling Point ID:	PFOS	PFOA	PFHS	PFOSA			
Sampling Event	Sampling Date	L22 CONCENTRATION (µg/l)						
1	10-Jan-08	9,2	0,75	2,7	0,1			
2	26-Jun-08	14,2	5,42	2,92	1,93			
3	24-Jul-09	12,2	2,46	0,993	0,0588			
4	20-Jan-10	16,2	3,75	2,2	1,25			
5	14-Jul-10	13,3	1,34	0,581	0,1			
6	26-Jan-11	7,73	2,89	0,432	0,0435			
7	6-Feb-12	8,22	1,19	0,299	0,1			
8	9-Jul-12	12,7	1,41	0,284	0,0401			
9	25-Oct-12	10	1,18	0,383	0,028			
10	23-Jan-13	10	0,728	0,271	0,0606			
11	19-Jul-13	10,293	0,4975	0,1345	0,09025			
12	21-Jan-14	12,1	0,879	0,401	0,0353			
13	30-Jul-14	11,9	0,51	0,21	0,11			
14								
15								
16								
17								
18								
19								
20								
Coefficient of Variation:		0,21	0,83	1,10	1,94			
Mann-Kendall Statistic (S):		-7	-44	-58	-23			
Confidence Factor:		64,0%	99,7%	>99.9%	90,8%			
Concentration Trend:		Stable	Decreasing	Decreasing	Prob. Decreasing			



Notes:

1. At least four independent sampling events per well are required for calculating the trend. *Methodology is valid for 4 to 40 samples.*
2. Confidence in Trend = Confidence (in percent) that constituent concentration is increasing (S>0) or decreasing (S<0): >95% = Increasing or Decreasing; ≥ 90% = Probably Increasing or Probably Decreasing; < 90% and S>0 = No Trend; < 90%, S≤0, and COV ≥ 1 = No Trend; < 90% and COV < 1 = Stable.
3. Methodology based on "MAROS: A Decision Support System for Optimizing Monitoring Plans", J.J. Aziz, M. Ling, H.S. Rifai, C.J. Newell, and J.R. Gonzales, *Ground Water*, 41(3):355-367, 2003.

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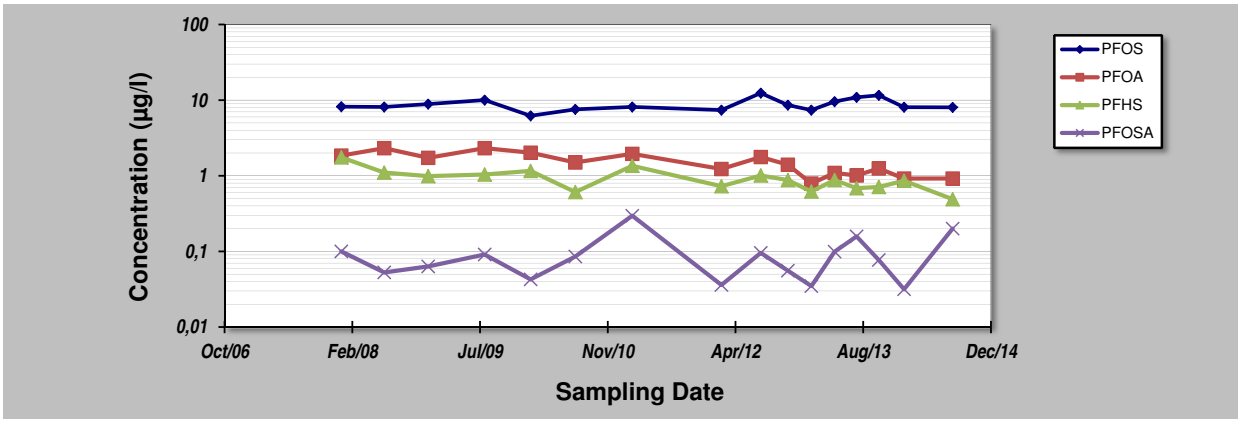
GSI MANN-KENDALL TOOLKIT
for Constituent Trend Analysis

Evaluation Date:	06-okt-14	Job ID:	0243999
Facility Name:	3M	Constituent:	L31
Conducted By:	MCO	Concentration Units:	µg/l

Sampling Point ID:	PFOS	PFOA	PFHS	PFOSA			
Sampling Event	Sampling Date	L31 CONCENTRATION (µg/l)						
1	10-Jan-08	8,2	1,85	1,75	0,1			
2	26-Jun-08	8,13	2,32	1,102	0,0527			
3	15-Dec-08	8,875	1,7271	0,989	0,0633			
4	24-Jul-09	10,025	2,32	1,04	0,091			
5	20-Jan-10	6,21	2,02	1,16	0,0426			
6	14-Jul-10	7,56	1,5	0,609	0,0856			
7	22-Feb-11	8,14	1,95	1,355	0,2965			
8	6-Feb-12	7,39	1,23	0,728	0,036			
9	9-Jul-12	12,4	1,77	1,01	0,0957			
10	23-Oct-12	8,6	1,4	0,877	0,0557			
11	23-Jan-13	7,39	0,79	0,619	0,0348			
12	24-Apr-13	9,57	1,09	0,879	0,0992			
13	19-Jul-13	10,9	1,01	0,685	0,158			
14	14-Oct-13	11,6	1,25	0,711	0,0771			
15	21-Jan-14	8,07	0,919	0,859	0,0316			
16	30-Jul-14	8,03	0,92	0,49	0,2			
17								
18								
19								
20								
Coefficient of Variation:		0,19	0,33	0,34	0,74			
Mann-Kendall Statistic (S):		11	-77	-60	2			
Confidence Factor:		67,1%	>99.9%	99,7%	51,8%			
Concentration Trend:		No Trend	Decreasing	Decreasing	No Trend			



Notes:

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2. Confidence in Trend = Confidence (in percent) that constituent concentration is increasing (S>0) or decreasing (S<0): >95% = Increasing or Decreasing; ≥ 90% = Probably Increasing or Probably Decreasing; < 90% and S>0 = No Trend; < 90%, S≤0, and COV ≥ 1 = No Trend; < 90% and COV < 1 = Stable.
3. Methodology based on "MAROS: A Decision Support System for Optimizing Monitoring Plans", J.J. Aziz, M. Ling, H.S. Rifai, C.J. Newell, and J.R. Gonzales, *Ground Water*, 41(3):355-367, 2003.

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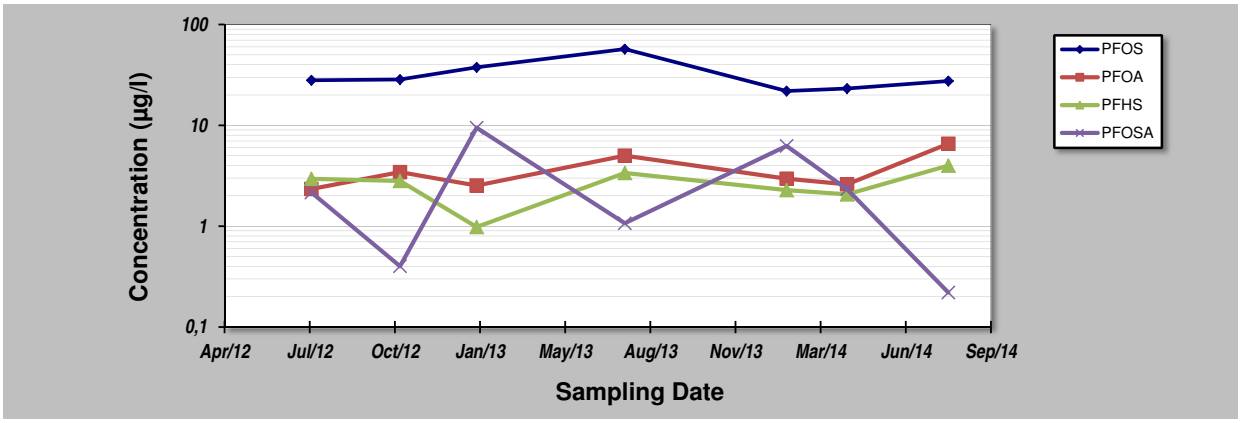
GSI MANN-KENDALL TOOLKIT
for Constituent Trend Analysis

Evaluation Date:	06-okt-14	Job ID:	0243999
Facility Name:	3M	Constituent:	L4
Conducted By:	MCO	Concentration Units:	µg/l

Sampling Point ID:	PFOS	PFOA	PFHS	PFOSA			
Sampling Event	Sampling Date	L4 CONCENTRATION (µg/l)						
1	12-Jul-12	28	2,34	2,95	2,16			
2	24-Oct-12	28,5	3,44	2,81	0,401			
3	22-Jan-13	37,6	2,53	0,982	9,47			
4	15-Jul-13	57,1	5,01	3,37	1,07			
5	21-Jan-14	21,9	2,96	2,28	6,25			
6	2-Apr-14	23,2	2,6	2,07	2,33			
7	30-Jul-14	27,5	6,57	3,99	0,22			
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
Coefficient of Variation:		0,38	0,43	0,37	1,10			
Mann-Kendall Statistic (S):		-3	9	1	-3			
Confidence Factor:		61,4%	88,1%	50,0%	61,4%			
Concentration Trend:		Stable	No Trend	No Trend	No Trend			



Notes:

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2. Confidence in Trend = Confidence (in percent) that constituent concentration is increasing (S>0) or decreasing (S<0): >95% = Increasing or Decreasing; ≥ 90% = Probably Increasing or Probably Decreasing; < 90% and S>0 = No Trend; < 90%, S≤0, and COV ≥ 1 = No Trend; < 90% and COV < 1 = Stable.
3. Methodology based on "MAROS: A Decision Support System for Optimizing Monitoring Plans", J.J. Aziz, M. Ling, H.S. Rifai, C.J. Newell, and J.R. Gonzales, *Ground Water*, 41(3):355-367, 2003.

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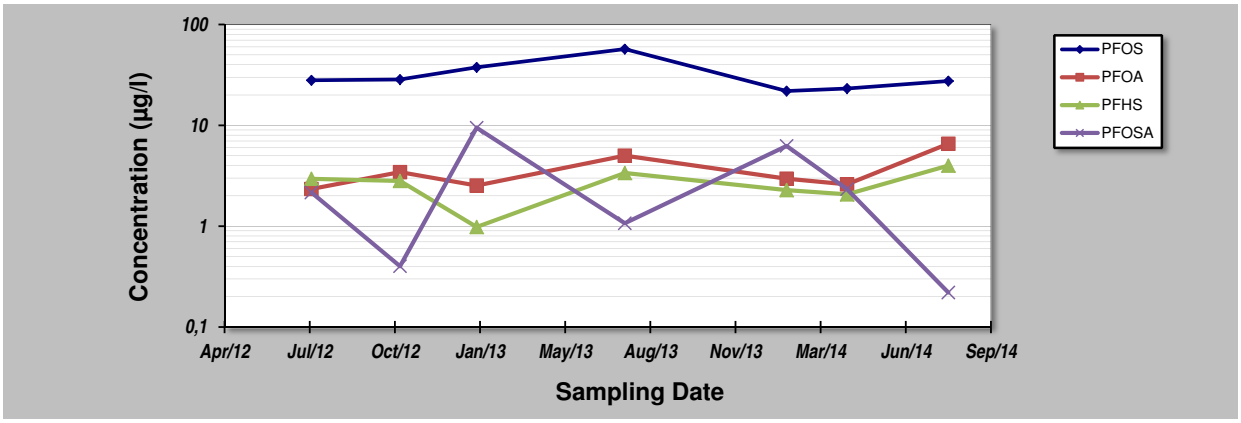
GSI MANN-KENDALL TOOLKIT
for Constituent Trend Analysis

Evaluation Date:	06-okt-14	Job ID:	0243999
Facility Name:	3M	Constituent:	L4
Conducted By:	MCO	Concentration Units:	µg/l

Sampling Point ID:	PFOS	PFOA	PFHS	PFOSA			
Sampling Event	Sampling Date	L4 CONCENTRATION (µg/l)						
1	12-Jul-12	28	2,34	2,95	2,16			
2	24-Oct-12	28,5	3,44	2,81	0,401			
3	22-Jan-13	37,6	2,53	0,982	9,47			
4	15-Jul-13	57,1	5,01	3,37	1,07			
5	21-Jan-14	21,9	2,96	2,28	6,25			
6	2-Apr-14	23,2	2,6	2,07	2,33			
7	30-Jul-14	27,5	6,57	3,99	0,22			
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
Coefficient of Variation:		0,38	0,43	0,37	1,10			
Mann-Kendall Statistic (S):		-3	9	1	-3			
Confidence Factor:		61,4%	88,1%	50,0%	61,4%			
Concentration Trend:		Stable	No Trend	No Trend	No Trend			



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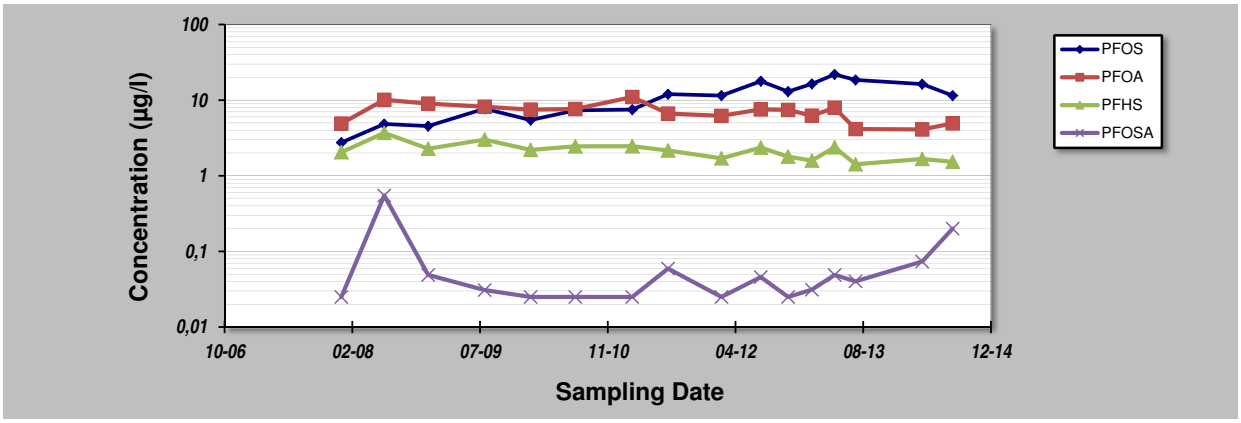
GSI MANN-KENDALL TOOLKIT
for Constituent Trend Analysis

Evaluation Date:	06-okt-14	Job ID:	0243999
Facility Name:	3M	Constituent:	P114
Conducted By:	MCO	Concentration Units:	µg/l

Sampling Point ID:	PFOS	PFOA	PFHS	PFOSA			
Sampling Event	Sampling Date	P114 CONCENTRATION (µg/l)						
1	10-Jan-08	2,75	4,9	2,05	0,025			
2	26-Jun-08	4,84	10,1	3,69	0,548			
3	15-Dec-08	4,54	9	2,28	0,0488			
4	24-Jul-09	7,71	8,18	3,01	0,0308			
5	20-Jan-10	5,45	7,5	2,2	0,025			
6	14-Jul-10	7,33	7,63	2,45	0,025			
7	22-Feb-11	7,5	11	2,46	0,025			
8	12-Jul-11	12	6,64	2,16	0,0593			
9	6-Feb-12	11,5	6,2	1,7	0,025			
10	9-Jul-12	17,8	7,55	2,36	0,0458			
11	24-Oct-12	13	7,43	1,79	0,025			
12	25-Jan-13	16,4	6,23	1,59	0,0312			
13	24-Apr-13	21,9	7,92	2,38	0,0488			
14	15-Jul-13	18,5	4,15	1,42	0,0402			
15	2-Apr-14	16,3	4,1	1,67	0,0735			
16	30-Jul-14	11,5	4,93	1,53	0,2			
17								
18								
19								
20								
Coefficient of Variation:		0,52	0,28	0,27	1,66			
Mann-Kendall Statistic (S):		81	-52	-60	28			
Confidence Factor:		>99.9%	99,0%	99,7%	88,6%			
Concentration Trend:		Increasing	Decreasing	Decreasing	No Trend			



Notes:

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3. Methodology based on "MAROS: A Decision Support System for Optimizing Monitoring Plans", J.J. Aziz, M. Ling, H.S. Rifai, C.J. Newell, and J.R. Gonzales, *Ground Water*, 41(3):355-367, 2003.

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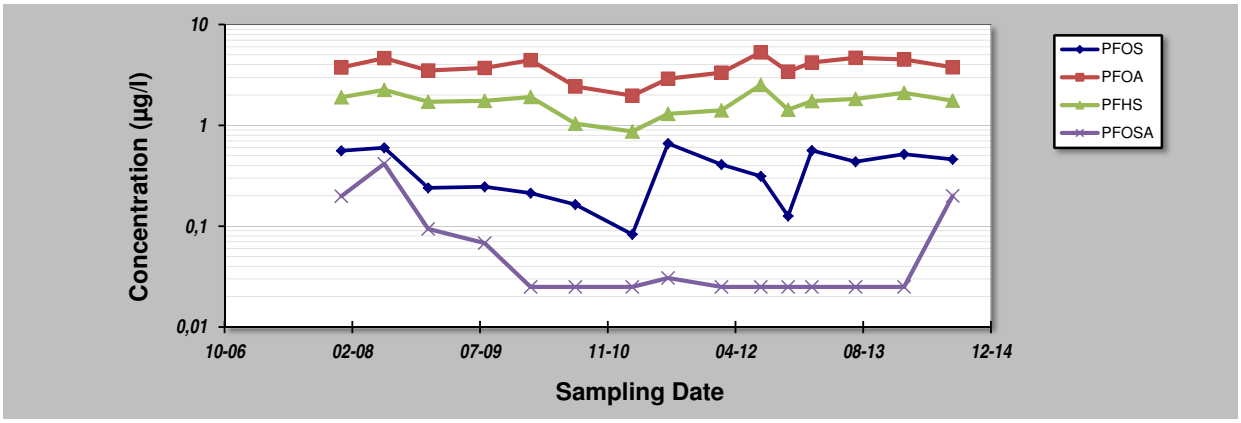
GSI MANN-KENDALL TOOLKIT
for Constituent Trend Analysis

Evaluation Date:	06-okt-14	Job ID:	0243999
Facility Name:	3M	Constituent:	P115
Conducted By:	MCO	Concentration Units:	µg/l

Sampling Point ID:	PFOS	PFOA	PFHS	PFOSA			
Sampling Event	Sampling Date	P115 CONCENTRATION (µg/l)						
1	10-Jan-08	0,56	3,75	1,9	0,198			
2	26-Jun-08	0,6	4,64	2,25	0,416			
3	15-Dec-08	0,24	3,5	1,714	0,094			
4	24-Jul-09	0,246	3,71	1,75	0,068			
5	20-Jan-10	0,213	4,43	1,91	0,025			
6	14-Jul-10	0,164	2,43	1,04	0,025			
7	22-Feb-11	0,083	1,97	0,87	0,025			
8	12-Jul-11	0,661	2,9	1,3	0,03065			
9	6-Feb-12	0,409	3,33	1,41	0,025			
10	9-Jul-12	0,313	5,3	2,52	0,025			
11	24-Oct-12	0,126	3,41	1,43	0,025			
12	25-Jan-13	0,564	4,2	1,74	0,025			
13	15-Jul-13	0,436	4,675	1,83	0,025			
14	21-Jan-14	0,518	4,5	2,1	0,025			
15	30-Jul-14	0,46	3,77	1,76	0,2			
16								
17								
18								
19								
20								
Coefficient of Variation:		0,50	0,24	0,26	1,35			
Mann-Kendall Statistic (S):		5	15	7	-35			
Confidence Factor:		57,7%	75,2%	61,5%	95,4%			
Concentration Trend:		No Trend	No Trend	No Trend	Decreasing			



Notes:

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2. Confidence in Trend = Confidence (in percent) that constituent concentration is increasing (S>0) or decreasing (S<0): >95% = Increasing or Decreasing; ≥ 90% = Probably Increasing or Probably Decreasing; < 90% and S>0 = No Trend; < 90%, S≤0, and COV ≥ 1 = No Trend; < 90% and COV < 1 = Stable.
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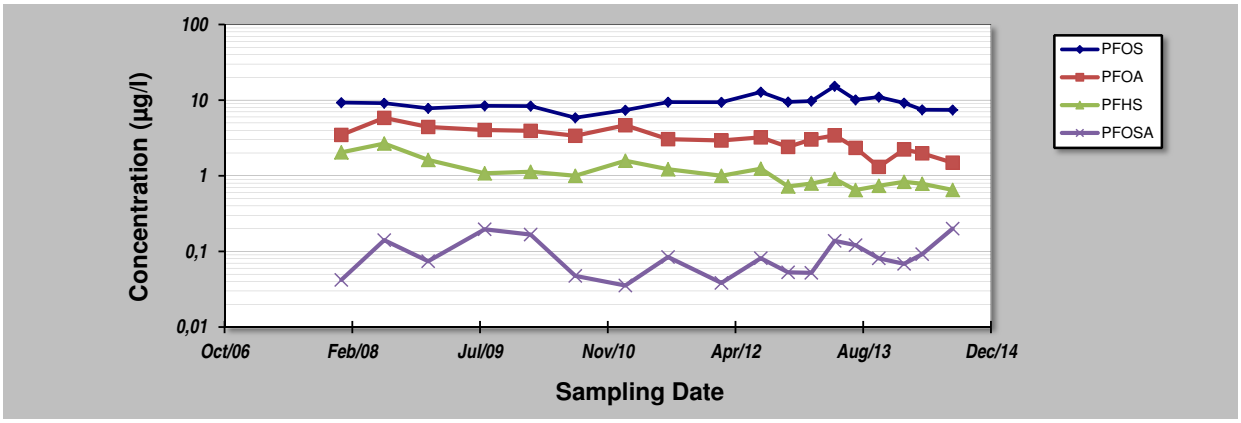
GSI MANN-KENDALL TOOLKIT
for Constituent Trend Analysis

Evaluation Date:	06-okt-14	Job ID:	0243999
Facility Name:	3M	Constituent:	P116
Conducted By:	MCO	Concentration Units:	µg/l

Sampling Point ID:	PFOS	PFOA	PFHS	PFOSA			
Sampling Event	Sampling Date	P116 CONCENTRATION (µg/l)						
1	10-Jan-08	9,28	3,47	2,04	0,042			
2	26-Jun-08	9,12	5,83	2,67	0,141			
3	15-Dec-08	7,8	4,42	1,62	0,0742			
4	24-Jul-09	8,41	4,03	1,08	0,196			
5	20-Jan-10	8,35	3,93	1,13	0,167			
6	14-Jul-10	5,86	3,38	1	0,0475			
7	26-Jan-11	7,38	4,66	1,58	0,0354			
8	12-Jul-11	9,42	3,05	1,22	0,0844			
9	6-Feb-12	9,39	2,93	1	0,0383			
10	9-Jul-12	12,8	3,22	1,24	0,0813			
11	24-Oct-12	9,48	2,41	0,723	0,0529			
12	23-Jan-13	9,73	3,02	0,789	0,0521			
13	24-Apr-13	15,3	3,42	0,91	0,138			
14	15-Jul-13	10,1	2,34	0,648	0,121			
15	14-Oct-13	11	1,31	0,737	0,081			
16	21-Jan-14	9,17	2,23	0,832	0,0685			
17	2-Apr-14	7,46	1,98	0,784	0,0919			
18	30-Jul-14	7,42	1,49	0,65	0,2			
19								
20								
Coefficient of Variation:		0,23	0,36	0,47	0,56			
Mann-Kendall Statistic (S):		27	-109	-100	19			
Confidence Factor:		83,5%	>99.9%	>99.9%	75,0%			
Concentration Trend:		No Trend	Decreasing	Decreasing	No Trend			



Notes:

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GSI MANN-KENDALL TOOLKIT
for Constituent Trend Analysis

Evaluation Date:	06-okt-14	Job ID:	0243999
Facility Name:	3M	Constituent:	P119C
Conducted By:	MCO	Concentration Units:	µg/l

Sampling Point ID:	PFOS	PFOA	PFHS	PFOSA			
Sampling Event	Sampling Date	P119C CONCENTRATION (µg/l)						
1	12-Jul-12	2355,5	452,5	175	3,14			
2	25-Oct-12	1174	609	307	41,8			
3	22-Jan-13	1168	197	94,5	39,5			
4	15-Jul-13	1892	371	154	37,3			
5	21-Jan-14	1730	221	115	61,4			
6	30-Jul-14	2250	336	185	37,2			
7								
8								
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14								
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17								
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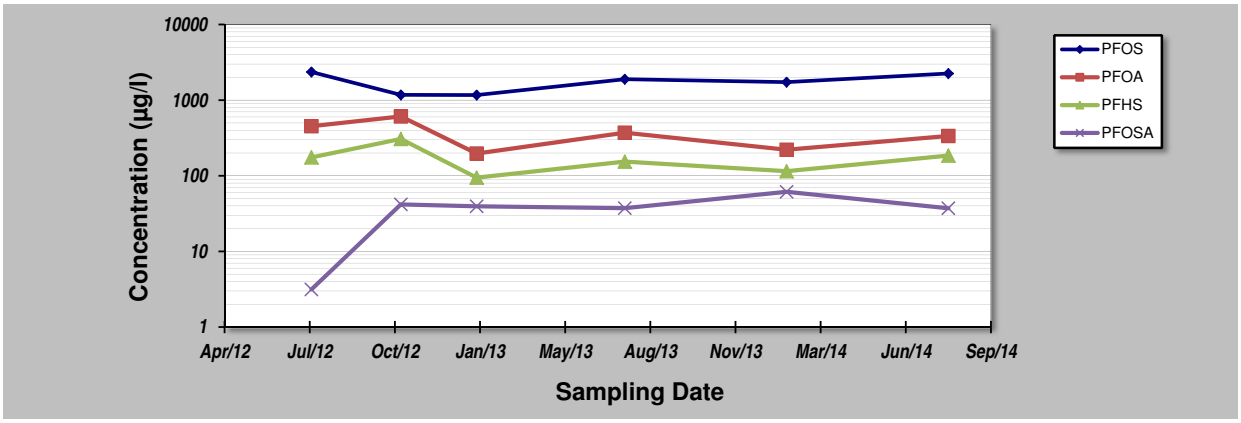
20								
Coefficient of Variation:		0,29	0,42	0,44	0,51			
Mann-Kendall Statistic (S):		1	-5	-1	1			
Confidence Factor:		50,0%	76,5%	50,0%	50,0%			
Concentration Trend:		No Trend	Stable	Stable	No Trend			



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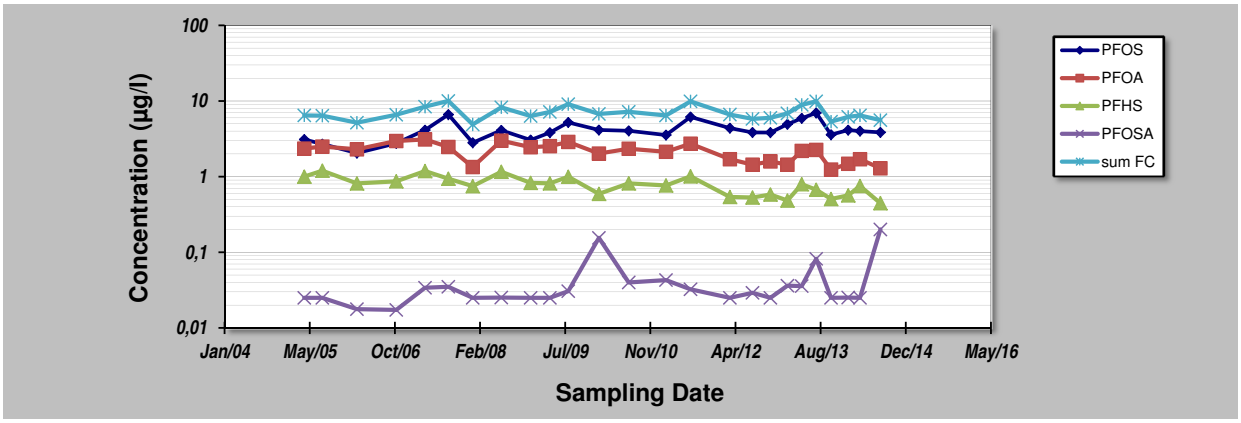
GSI MANN-KENDALL TOOLKIT
for Constituent Trend Analysis

Evaluation Date:	06-okt-14	Job ID:	0243999
Facility Name:	3M	Constituent:	3M vijver
Conducted By:	MCO	Concentration Units:	µg/l

Sampling Point ID:	PFOS	PFOA	PFHS	PFOSA	sum FC		
Sampling Event	Sampling Date	3M VIJVER CONCENTRATION (µg/l)						
1	26-Apr-05	3,1	2,35	1	0,025	6,45		
2	10-Aug-05	2,7	2,5	1,2	0,025	6,4		
3	1-Mar-06	2,05	2,3	0,814	0,0177	5,164		
4	18-Oct-06	2,76	2,96	0,867	0,0173	6,587		
5	5-Apr-07	4,13	3,105	1,185	0,034	8,42		
6	20-Aug-07	6,62	2,47	0,939	0,035	10,029		
7	10-Jan-08	2,81	1,34	0,749	0,025	4,899		
8	26-Jun-08	4,1	2,99	1,16	0,0252	8,25		
9	15-Dec-08	3,05	2,45	0,827	0,025	6,327		
10	7-Apr-09	3,82	2,53	0,815	0,025	7,165		
11	24-Jul-09	5,21	2,88	0,996	0,0306	9,086		
12	20-Jan-10	4,14	2,02	0,592	0,155	6,752		
13	14-Jul-10	4,04	2,34	0,815	0,0399	7,195		
14	18-Feb-11	3,55	2,13	0,765	0,0429	6,445		
15	12-Jul-11	6,155	2,725	1,01	0,0324	9,89		
16	28-Feb-12	4,38	1,7	0,539	0,025	6,619		
17	10-Jul-12	3,84	1,44	0,53	0,0291	5,81		
18	23-Oct-12	3,83	1,59	0,58	0,025	6		
19	31-Jan-13	4,93	1,44	0,483	0,0362	6,853		
20	25-Apr-13	5,91	2,19	0,794	0,0357	8,894		
21	19-Jul-13	6,97	2,26	0,671	0,0819	9,901		
22	14-Oct-13	3,59	1,24	0,506	0,025	5,336		
23	23-Jan-14	4,13	1,48	0,566	0,0252	6,176		
24	2-Apr-14	4	1,7	0,752	0,025	6,452		
25	31-Jul-14	3,85	1,29	0,446	0,2	5,586		
26								
27								
28								
29								
30								
Coefficient of Variation:		0,30	0,27	0,29	1,01	0,21		
Mann-Kendall Statistic (S):		91	-140	-167	79	-14		
Confidence Factor:		98,3%	>99.9%	>99.9%	96,6%	61,8%		
Concentration Trend:		Increasing	Decreasing	Decreasing	Increasing	Stable		



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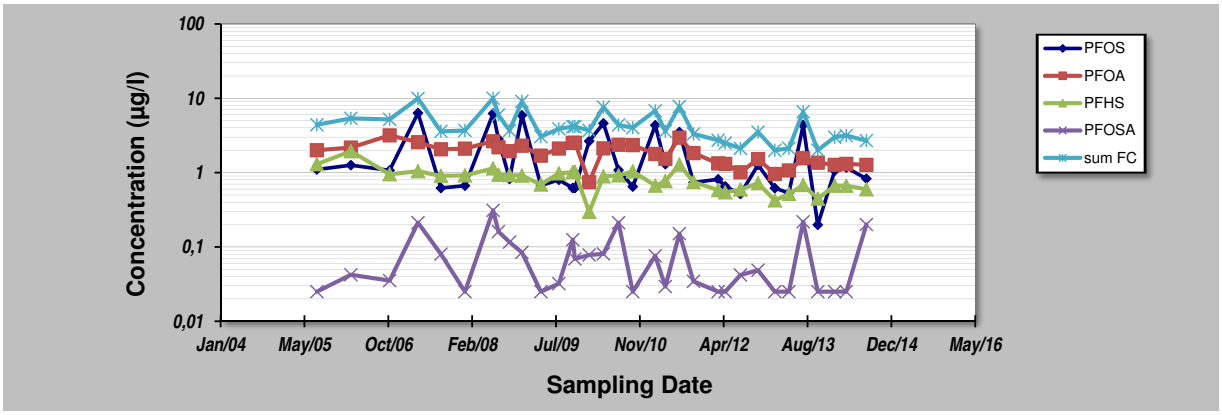
GSI MANN-KENDALL TOOLKIT
for Constituent Trend Analysis

Evaluation Date:	06-okt-14	Job ID:	0243999
Facility Name:	3M	Constituent:	Blokkersdijkvijver standaard
Conducted By:	MCO	Concentration Units:	µg/l

Sampling Point ID:	PFOS	PFOA	PFHS	PFOSA	sum FC		
Sampling Event	Sampling Date	BLOKKERSDIJKVIJVER STANDAARD CONCENTRATION (µg/l)						
1	10-Aug-05	1,1	2	1,3	0,025	4,4		
2	1-Mar-06	1,26	2,17	1,941	0,0422	5,371		
3	18-Oct-06	1,07	3,17	0,949	0,0352	5,189		
4	5-Apr-07	6,33	2,565	1,045	0,212	9,94		
5	20-Aug-07	0,62	2,06	0,904	0,08	3,584		
6	10-Jan-08	0,666	2,1	0,916	0,025	3,682		
7	26-Jun-08	6,23	2,64	1,14	0,31	10,01		
8	28-Jul-08	2,83	2,19	0,936	0,161	5,956		
9	2-Oct-08	0,82	1,94	0,924	0,116	3,684		
10	15-Dec-08	5,89785	2,29905	0,9113	0,0843	9,1082		
11	7-Apr-09	0,67	1,69	0,691	0,025	3,051		
12	23-Jul-09	0,799	2,105	0,976	0,0321	3,88		
13	14-Oct-09	0,621	2,515	1,015	0,125	4,151		
14	27-Oct-09	0,621	2,52	1,02	0,069	4,161		
15	20-Jan-10	2,65	0,742	0,296	0,078	3,688		
16	15-Apr-10	4,6	2,12	0,8915	0,0806	7,6115		
17	14-Jul-10	1,08	2,37	0,916	0,212	4,366		
18	7-Oct-10	0,647	2,34	1,04	0,025	4,027		
19	18-Feb-11	4,34	1,78	0,668	0,076	6,788		
20	19-Apr-11	1,3	1,53	0,768	0,0293	3,598		
21	12-Jul-11	3,505	2,95	1,285	0,151	7,74		
22	4-Oct-11	0,7355	1,8286	0,74725	0,0344	3,31135		
23	28-Feb-12	0,813	1,33	0,581	0,025	2,724		
24	10-Apr-12	0,652	1,31	0,542	0,025	2,504		
25	10-Jul-12	0,516	1,01	0,594	0,0419	2,12		
26	23-Oct-12	1,27	1,52	0,719	0,0485	3,509		
27	31-Jan-13	0,617	0,958	0,423	0,025	1,998		
28	25-Apr-13	0,533	1,07	0,516	0,025	2,119		
29	19-Jul-13	4,35	1,56	0,681	0,218	6,591		
30	14-Oct-13	0,198	1,36	0,447	0,025	2,005		
31	23-Jan-14	1,06	1,27	0,666	0,025	2,996		
32	2-Apr-14	1,17	1,31	0,666	0,025	3,146		
33	31-Jul-14	0,828	1,27	0,596	0,2	2,694		
34								
35								
Coefficient of Variation:		1,00	0,32	0,37	0,92	0,50		
Mann-Kendall Statistic (S):		-99	-246	-276	-96	-222		
Confidence Factor:		93,5%	>99.9%	>99.9%	92,9%	>99.9%		
Concentration Trend:		Prob. Decreasing	Decreasing	Decreasing	Prob. Decreasing	Decreasing		



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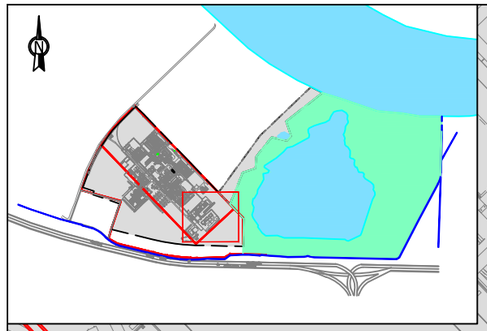
Bijlage 9

Onttrekkingsfilters grondwateronttrekking



PP05
PP05-still
PP01
PP04-still
016
017
029
026
028
034
0804
0801
0803
PP07
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PP06-still
PP06
011
2
3
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4
6
8
9
10
PP10
PP02
PP08
PP08-still
PP09
RREIN 3M

Legende:

Pompput

3M Terrein

Ondergrondse Dijk

0 10 20 30 40 50 (m)

Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
NVE	NHE	DNU	v1.0	☒ FINAL ☐ DRAFT

Environmental Resources Management n.v.

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1000 Brussel
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Tel.: 03/287.36.50
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Place de l'Université 16
1348 Louvain-la-Neuve
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Fax.: 010/48.35.36

Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.:	Titel:
0243999	5de Tussentijds verslag Bodemsaneringswerken

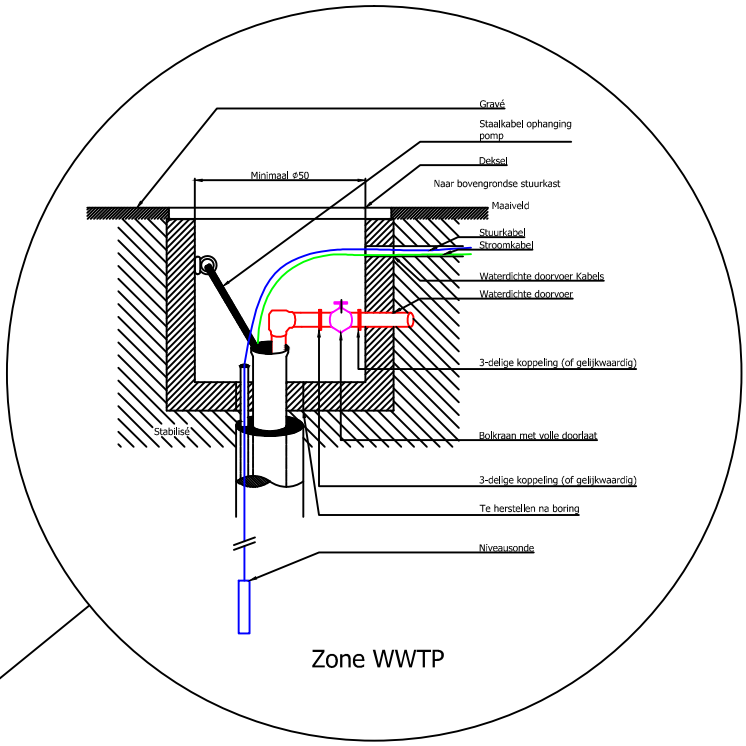
Figuur:	Beschrijving:
9	Onttrekkingsfilters grondwateronttrekking

Fase: TTV-5

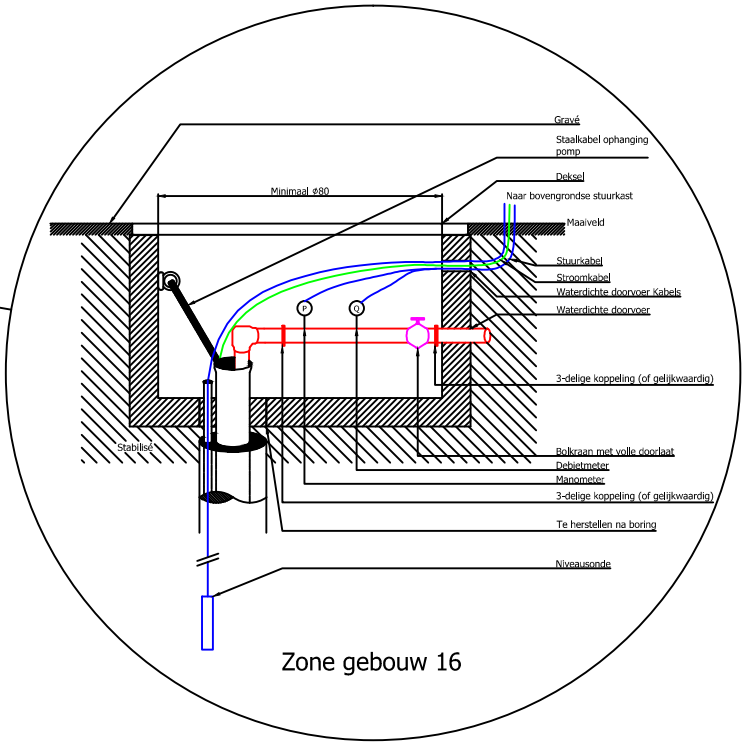
Schaal:	Formaat:	Bestandsnaam:	Datum:
1/1500	A3	3M Zwijndrecht.dwg	02/02/2015

Bijlage 10

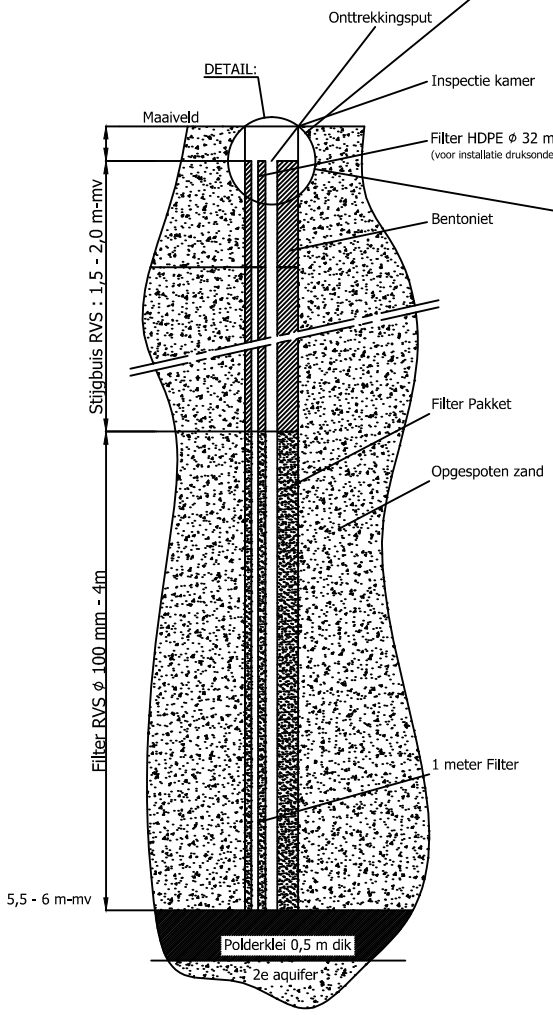
Schematische weergave pompput en inspectiekamer



Zone WWTP



Zone gebouw 16



Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
NVE	NHE	DNU	v1.0	☒ FINAL ☐ DRAFT

Environmental Resources Management n.v.

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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:
0243999	5de Tussentijds verslag Bodemsaneringswerken

Figuur:	Beschrijving:
10	Doorsnede Pompput en inspectiekamer
Fase: TTV-5	

Schaal:	Formaat:	Bestandsnaam:	Datum:
-/-	A4	3M-Doorsnedes.dwg	02/02/2015

Bijlage 11

Nota stabiliteitsdeskundige

Grontmij

Notitie

Referentienummer	Datum	Kenmerk
0188502	3 april 2014	0108-0304

Betreft
Opvolging zettingsmetingen 3M

Op verzoek van 3M werden de stabiliteitsstudie en de opvolging van de zettingen doorgenomen. De doelstelling was een second opinion te vormen in verband met de vraag tot stopzetting van de opvolging van de zettingen.

Voor de conclusie hebben we ons gebaseerd op volgende documenten:

- Nota stabiliteit opgesteld door Arcadis op datum van 9/10/2008
- Settlement analysis opgesteld door Alpha studieburo op datum van 23/03/2010
- Opvolging zettingen opgesteld door Alpha studieburo op datum van 13/09/2010, 19/09/2011, 06/12/2012
- Overzicht met zettingen genomen op punt 1 t.e.m. 20, startend vanaf 15/03/2010 t.e.m. 02/07/2013

In de studie van Arcadis wordt een maximale zetting van 16 mm berekend. Dit is toelaatbaar.

In de documenten i.v.m. de opvolging van de zettingen wordt een zetting van 3mm opgegeven als een eerste toetsingscriterium.
Gedurende de volledige opvolgingsperiode die gerapporteerd is door Alpha studieburo, tot 6/12/2012, is de toetsingswaarde niet bereikt.
Er werd een maximale zetting van 2,3 mm bereikt volgens de rapportage. Nazicht van de waardes doet me echter vaststellen dat er op 15/02/2011 een grotere zetting van 2,9 mm werd opgemeten t.p.v. punt 9.
Dit blijft nog steeds onder de toetsingswaarde.

Na de laatste rapportage zijn er nog twee zettingsmetingen uitgevoerd. De zettingen zijn in bijna elk punt verwaarloosbaar klein.
Enkel punt 9 en 16 vertonen nog zettingen > 1mm.
Voor punt 9 wordt nu een zetting van 2,7 mm bereikt.

Aangezien dit de toetsingswaarde benadert en deze meting dateert van 02/07/2013 en de zetting de laatste metingen toegenomen is, wordt geadviseerd op zijn minst voor dit punt nog een recente meting uit te voeren. Op basis daarvan kan overgegaan worden tot beslissing tot stopzetting van de opvolging van de zettingen.

Belangrijke voorwaarde blijft alleszins dat de opgepompte debieten in de toekomst, nooit de tot op heden opgepompt debieten mogen overschrijden.

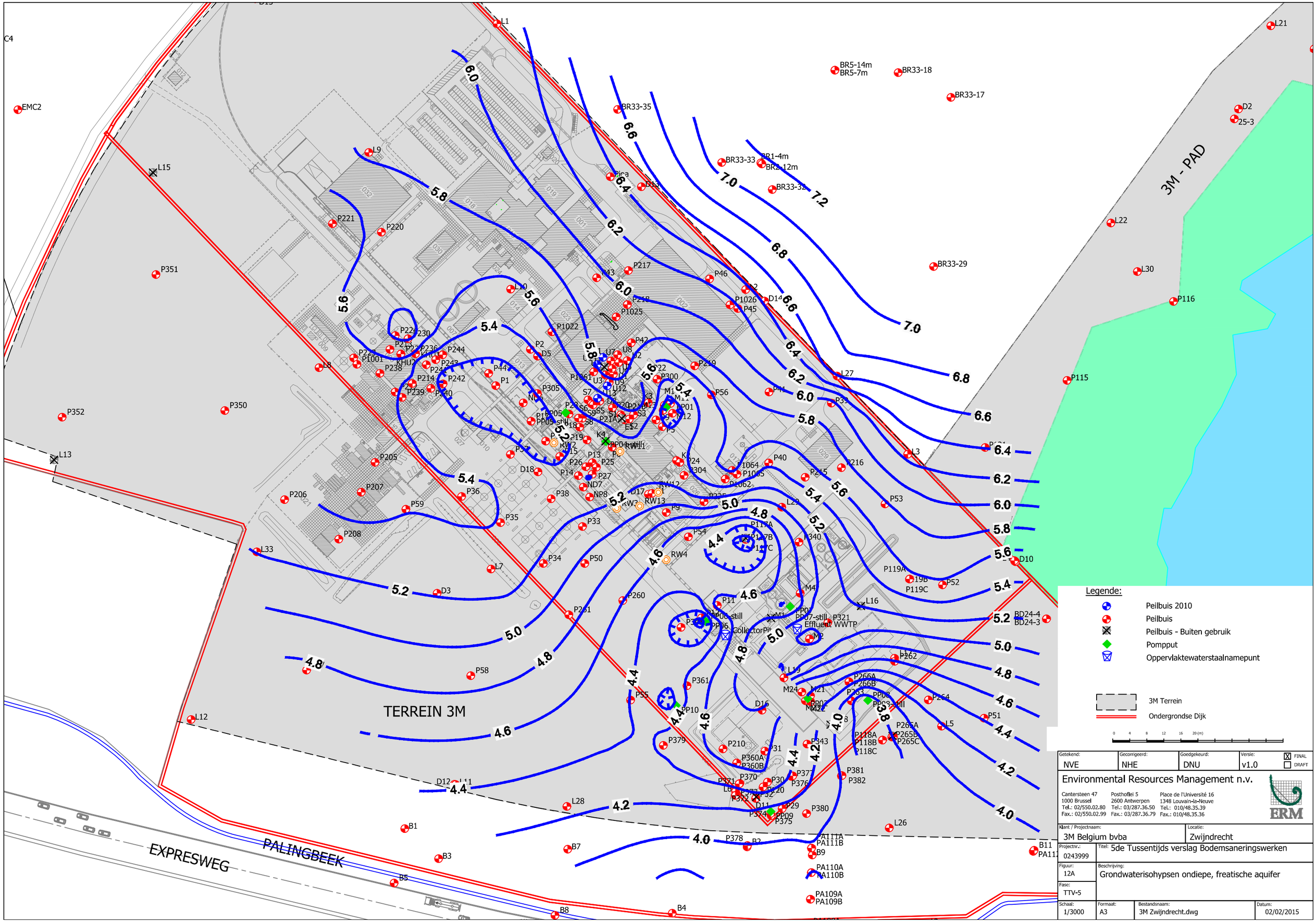
Namens Grontmij nv,
dd. 03/04/2014

Mario Vernyns
Sr. Projectleider

Jan De Kinderen
Teamleider Bodem

Bijlage 12

Stijghoogtes en grondwaterisohypsen eerste en tweede aquifer



Getekend:	Gecorrigeerd:	Goedgekeurd:	Versie:	
NVE	NHE	DNU	v1.0	☒ FINAL ☐ DRAFT

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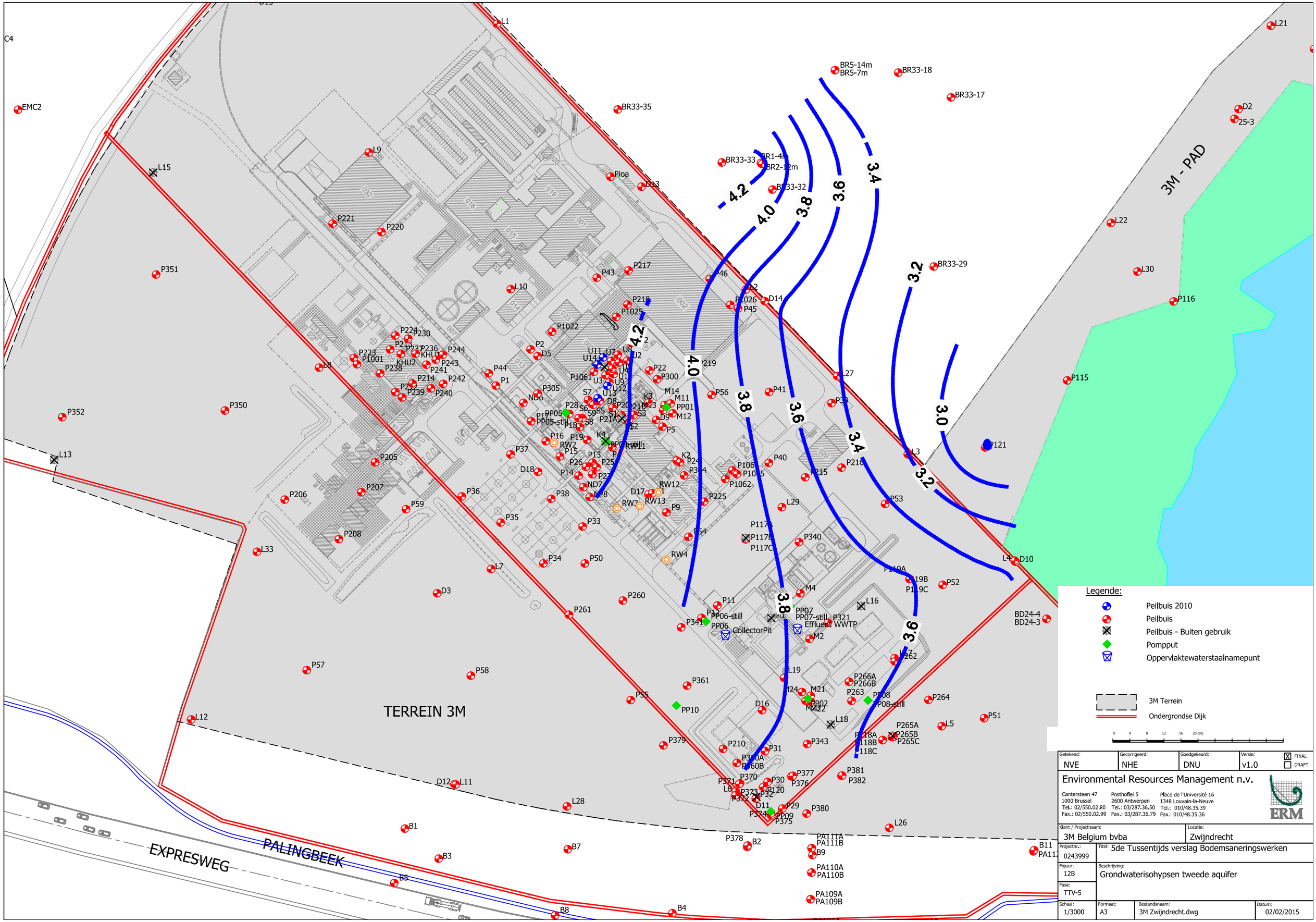
Klant / Projectnaam:	Locatie:
3M Belgium bvba	Zwijndrecht

Projectnr.:	Titel:
0243999	5de Tussentijds verslag Bodemsaneringswerken

Figuur:	Beschrijving:
12A	Grondwaterisohypsen ondiepe, freatische aquifer

Fase:
TTV-5

Schaal:	Formaat:	Bestandsnaam:	Datum:
1/3000	A3	3M Zwijndrecht.dwg	02/02/2015



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Klant / Projectnaam: 3M Belgium bvba	Locatie: Zwijndrecht
Projectnr.: 0243999	Titel: 5de Tussentijds verslag Bodemsaneringswerken
Figuur: 12B	Beschrijving: Grondwaterisohypsen tweede aquifer
Fase: TTV-5	
Schaal: 1/3000	Formaat: A3
Bestandsnaam: 3M Zwijndrecht.dwg	Datum: 02/02/2015