

July 18, 2018

#### Ms. Karen Cahill

New York State Department of Environmental Conservation 615 Erie Boulevard Syracuse, New York 13204

> RE: Results of May 2018 Sampling Event for Emerging Contaminants Krutulis Property NYSDEC Site Code – 727009 Kirkville, New York

FILE: 2874/69452

#### Dear Ms. Cahill:

This letter report summarizes the results of the sampling and analysis of 1,4-dioxane and per- and polyfluoroalkyl substances (PFAS) for the Krutulis Property (Site) in Kirkville, New York. This sampling was requested by the New York State Department of Environmental Conservation (NYSDEC) in an April 10, 2018 letter. Activities summarized in this report were completed in accordance with the Work Plan submitted on April 27, 2018<sup>1</sup> (Attachment A).

#### **BACKGROUND**

The detection of 1,4-dioxane and PFAS in drinking water supplies in New York State has been widely publicized over the past several years. Neither NYSDEC nor the United States Environmental Protection Agency (USEPA) have issued regulatory standards for 1,4-dioxane or PFAS in groundwater or drinking water. However, USEPA issued a drinking water lifetime health advisory for two perfluoroalkyl compounds (perfluorooctanoic acid [PFOA] and perfluorooctanesulfonic acid [PFOS]) in May 2016 that is 70 nanograms per liter (ng/L), individually or combined. This advisory has been adopted by the New York State Department of Health (NYSDOH).

1,4-Dioxane is most often associated with 1,1,1-trichloroethane (TCA) in which it was used as a stabilizer. Based on review of Site data, trichloroethene (TCE) and dichloroethene (DCE) are the dominant constituents of concern at the Site, while TCA was not considered of concern during the initial Site evaluation and not included in the Site's target VOCs.

Methods PFAS have been used in many residential, commercial, and industrial products, and their occurrence is widespread. The two most widely known PFAS are perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS). PFAS are predominantly associated with the storage, training and/or use of aqueous film-forming foam (AFFF) for flammable liquids, although other uses for PFAS are noted. Some of these uses include the

<sup>&</sup>lt;sup>1</sup> As discussed under *Methods*, this work was performed in conjunction with a semi-annual groundwater sampling event on May 2, 2018 prior the NYSDEC formally approving the Work Plan. The NYSDEC did review the Work Plan and provided preliminary approval accompanied by a Site visit during sampling. Formal approval was received on June 18, 2018, with specific items discussed and addressed during a June 29, 2018 call between the NYSDEC and OBG.







manufacture of specialty/coated fabrics, paper and carpet, and in products used for waterproofing and stain resistance. None of this is known to be associated with the Krutulis Property.

#### **METHODS**

#### **GROUNDWATER SAMPLING**

The PFAS and 1,4-dioxane samples were collected on May 2, 2018 from seven monitoring wells (MW-1, MW-2, MW-3S, MW-3D, MW-4, MW-6S, and MW-6D) in conjunction with a routine semi-annual groundwater monitoring event for volatile organic compounds (VOCs). The monitoring well locations are shown on **Figure 1**.

The trace levels of PFAS and 1,4-dioxane that can be in the environment (e.g., background) combined with the very low analytical detection/reporting limits required a careful sampling protocol utilizing measures (i.e., PFAS sample collection before 1,4-dioxane and VOCs, appropriate clothing and sampling equipment, decontamination) to prevent cross contamination. Prior to the sampling event, the PFAS-related sample collection and handling precautions outlined in the NYSDEC sampling protocol provided in **Attachment A** were reviewed.

Each well was purged and sampled via conventional methods using a dedicated PVC bailer with Delrin® ball valve suspended by a nylon-coated, stainless steel wire. Prior to purging at each well location, a water level measurement was recorded using a water level probe that was thoroughly decontaminated using PFAS-free water and Alconox® detergent.

Water quality parameters including temperature, conductivity, and pH, were measured using a Hanna 991301 water quality meter. The measured water quality parameters and water levels were recorded on groundwater sampling forms (**Attachment B**) along with visual and olfactory observations noted at the start and end of purging.

The PFAS and 1,4-dioxane groundwater samples were collected directly from the dedicated bailers into the laboratory-provided bottleware prior to collection of the VOC samples. PFAS samples were collected in certified PFAS-free bottles, immediately capped, labelled, and placed into a cooler containing ice before collecting the 1,4-dioxane and VOCs samples. Additionally, the PFAS were stored separately from the 1,4-dioxane and VOCs samples. The sample identifier, location, date, time, and sample collector were recorded on the groundwater sampling log and a chain-of-custody form. Sample coolers were delivered to the TestAmerica (TA) Service Center in Syracuse, New York. The PFAS samples were shipped to the TA facility in Sacramento, California, and the 1,4-dioxane samples were shipped to the TA facility in Buffalo, New York.

Several QC samples were also collected, including a field duplicate (FD), matrix spike (MS), and matrix spike duplicate (MSD) for PFAS and 1,4-dioxane analyses. For the PFAS analyses, a field reagent blank (FRB) was also collected.<sup>2</sup> Additionally, an equipment blank (EB) sample was collected for PFAS and 1,4-dioxane analyses from a new bailer immediately prior to deployment into MW-3S using PFAS-free water.

It is important to note the potential sources of cross contamination identified as follows:

<sup>&</sup>lt;sup>2</sup> The FRB is PFAS-free water supplied by the laboratory that is transferred into an empty laboratory-supplied container in the field.



- Bailers from previous VOC sampling events were present in most the wells suspended by twine. These were removed several days before the PFAS and 1,4-dioxane sampling to help reduce potential crosscontamination.
- Many of the neighboring houses along Marsh Mill Road displayed "Mosquito Authority" signs that signified pesticide application to control mosquitos had occurred in the area due to the Site's proximity to the Cicero Swamp and the concern for insect-transmitted diseases. While not considered likely, cross-contamination was possible if PFAS or 1,4-dioxane were present in the pesticide.
- TA provided the sample bottles for both PFAS and 1,4-dioxane in one of the four coolers.
- TA provided 1-liter high density polyethylene (HDPE) bottles for the PFAS samples, which was inconsistent with the 250-milliliter HDPE bottles specified in the Work Plan. However, a PFAS specialist at TA was consulted to determine the best path forward; rather than postponing the sampling event, TA recommended filling a single 1-liter HDPE bottle for the groundwater, FD, MS/MSD, and EB samples, with subsequent transfer into two 250-milliliter HDPE bottles at the TA Service Center before shipping to the TA Sacramento, California facility.

The last two specific items were also identified in the NYSDEC's June 18, 2018 email approving the Work Plan and were discussed and addressed in our June 29, 2018 conference call. Based on the below *Results and Discussion*, the potential sources for cross-contamination identified above did not influence the analytical results.

#### LABORATORY ANALYSES

For the PFAS analyses, TA analyzed the groundwater samples and associated QC samples in accordance with USEPA Method 537 (modified)<sup>3</sup> for the 21 PFAS analytes (**Attachment A**). For the 1,4-dioxane samples, TA used USEPA Method 8270D with selected ion monitoring (SIM). The laboratory provided a full category B deliverable per the NYSDEC analysis and reporting guidelines included in **Attachment A**. The data were validated by a data validator, and a Data Usability Summary Report (DUSR) was prepared (**Attachment C**).

#### **RESULTS AND DISCUSSION**

The analytical results from the May 2, 2018 sampling event are presented in **Table 1**. The following observations are based on a review of the analytical results.

- 1,4-Dioxane was not detected in any of the groundwater samples or the QC samples.
- PFOS and PFOA, which are the two PFAS with a drinking water lifetime health advisory, were not detected in any of the groundwater samples or the QC samples.
- Only two of the remaining 19 PFAS were detected in the groundwater samples and were quantitated as follows:

<sup>&</sup>lt;sup>3</sup> USEPA Method 537 was promulgated for drinking water not groundwater. In addition, it was promulgated for 14 PFAS. Laboratories must report use of a "modified" method for use on groundwater samples to quantify additional PFAS constituents (*e.g.*, the 21 listed by NYSDEC in **Appendix C**) and to get the desired detection/reporting limits.



- » Perfluorobutanoic acid (PFBA) was detected in the sample from MW-6S at a concentration of 5.3 JN<sup>4</sup> ng/L (the qualifier is based on matrix interference).
- » Perfuorohexanesulfonic acid (PFH<sub>x</sub>S) was detected in the sample from MW-2 at a concentration of 0.29 JN ng/L (the qualifier is based on peak data interpretation).
- » PFBA and PFH $_x$ S were also detected in the FRB (0.89 J ng/L and 0.32 J ng/L, respectively) and EB (0.38 J ng/L and 0.31 J ng/L, respectively). However, the laboratory acknowledged the detections may have been attributed to the handling/transfer of the samples at TA's Service Center. Additionally, the laboratory method blank contained PFH $_x$ S (0.279 J ng/L), which suggests that the detections noted in the FRB and EB could be possibly due to laboratory artifact.

Additionally, the potential sources for cross-contamination identified above did not influence the analytical results.

#### **CONCLUSIONS**

The following conclusions were developed based on this effort.

- No PFOS, PFOA, or 1,4-dioxane were detected in any of the groundwater samples or the associated QC samples.
- The lack of PFOS, PFOA, and 1,4-dioxane and minimal detections of PFBA and PFHxS are consistent with expected results based on the Site history.
- Based on the results of the sampling event, no further evaluation for 1,4-dioxane or PFAS is considered necessary for the Krutulis Site.

The sampling event establishes that there is no environmental concern from PFAS and 1,4-dioxane on-site, and no further action is warranted. If you should have any questions, please feel free to contact Mr. Douglas Morrison of Bristol-Myers Squibb Company at (315) 432-4851 or me at (315) 956-6665.

Very truly yours,

O'BRIEN & GERE ENGINEERS, INC.

is lu po

Charles W. Sharpe, P.E.

**Project Manager** 

<sup>&</sup>lt;sup>4</sup> "JN" indicates that the target analyte was "tentatively identified" as present and the associated numerical value is the estimated concentration in the sample.

Letter Attachments:

Table 1 – Groundwater Analytical Data – Summary Table

Figure 1 – Well Location Map

Attachment A – Emerging Contaminants Sampling and Analysis Work Plan

Attachment B - Groundwater Sampling Logs

Attachment C – Data Usability Summary Report

cc: R. Jones, New York State Department of Health (electronic copy [ec])

M. Schuck, New York State Department of Health (ec)

J.R. Pooler, Bristol-Myers Squibb Company (w/Attachments)

D. Morrison, Bristol-Myers Squibb Company (w/Attachments)

Richard and Pamela Mellor (Site owners) (w/Attachments)

C. Calkins, O'Brien & Gere (ec)

 $I:\ \ Bms. 2874 \land 9452. Krutulis-Ground \setminus Docs \setminus Emerg\_Contam\_Lrt\_Rpt \setminus 01-Letter\_Rpt\_Rev\_6. docx$ 



#### TABLE 1

#### **Krutulis Property**

#### Kirkville, New York Site

#### Groundwater Analytical Data - May 2018 - PFAS & 1,4-dioxane

PARAMETERS	USEPA Drinking Water Standards (ppt)	MW-1	MW-2	MW-3S	MW-3D	MW-4	Field Duplicate (MW-4)	MW-6S	MW-6D	Field Reagent Blank	Equipment Blank
USEPA Method 8270D SIM											
1,4-Dioxane	NC	<0.21	<0.21	<0.19	<0.19	<0.20	<0.20	<0.20	<0.20	N/C	<0.20
USEPA Method 537 (modified)											
Perfluorobutanoic acid (PFBA)	NC	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	5.3 JN	<2.0	0.89 J	0.38 J
Perfluoropentanoic acid (PFPeA)	NC	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	<2.0	<1.9
Perfluorohexanoic acid (PFH <sub>x</sub> A)	NC	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	<2.0	<1.9
Perfluoroheptanoic acid (PFHpA)	NC	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	<2.0	<1.9
Perfluorooctanoic acid (PFOA)	70	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	<2.0	<1.9
Perfluorononanoic acid (PFNA)	NC	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	<2.0	<1.9
Perfluorodecanoic acid (PFDA)	NC	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	<2.0	<1.9
Perfluoroundecanoic acid (PFUnA)	NC	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	<2.0	<1.9
Perfluorododecanoic acid (PFDoA)	NC	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	<2.0	<1.9
Perfluorotridecanoic acid (PFTriA)	NC	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	<2.0	<1.9
Perfluorotetradecanoic acid (PFTeA)	NC	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	<2.0	<1.9
Perfluorobutanesulfonic acid (PFBS)	NC	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	<2.0	<1.9
Perfluorohexanesulfonic acid (PFHxS)	NC	<1.9	0.29 JN	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	0.32 J	0.31 J
Perfluoroheptanesulfonic acid (PFHpS)	NC	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	<2.0	<1.9
Perfluorooctanesulfonic acid (PFOS)	70	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	<2.0	<1.9
Perfluorodecanesulfonic acid (PFDS)	NC	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	<2.0	<1.9
Perfluorooctane Sulfonamide (FOSA)	NC	<1.9	<2.0	<2.0	<2.0	<1.9	<2.0	<1.9	<2.0	<2.0	<1.9
N-methyl perfluorooctane sulfonamidoacetic acid (NMeFOSAA)	NC	<19.0	<2.0	<2.0	<2.0	<19.0	<20.0	<19.0	<20.0	<20.0	<19.0
N-ethyl perfluorooctane sulfonamidoacetic acid (NEtFOSAA)	NC	<19.0	<2.0	<2.0	<2.0	<19.0	<20.0	<19.0	<20.0	<20.0	<19.0
6:2FTS	NC	<19.0	<2.0	<2.0	<2.0	<19.0	<20.0	<19.0	<20.0	<20.0	<19.0
8:2FTS	NC	<19.0	<2.0	<2.0	<2.0	<19.0	<20.0	<19.0	<20.0	<20.0	<19.0

#### Notes:

- 1) All values are in ng/L (ppt). Detected values shown in **bold** text.
- 2) J Result is less than the reporting limit but greater than or equal to the MDL, and the concentration is an estimated value.
- 3) JN Indicates that the target analyte has been "tentatively identified" as present and the associated numerical value is the estimated concentration in the sample.
- 4) NC No Criteria.
- 5) PFHxS was detected in the laboratory method blank at 0.279 J ng/L.





#### **LEGEND**

→ MW-1

MONITORING WELL LOCATION

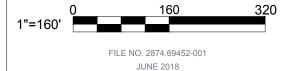
\_\_\_\_

PROPERTY LINE

LIMITS OF KRUTULIS
PROPERTY -REGISTRY
SITE #727009

KRUTULIS PROPERTY KIRKVILLE, NEW YORK

MONITORING WELL LOCATIONS





O'BRIEN & GERE ENGINEERS, INC.

#### **Attachments**

#### **Attachment A**

Emerging Contaminants
Sampling and Analysis
Work Plan

Included after Work Plan

- 1) NYSDEC's PFAS
  Monitoring Wells
  Sample Protocol
- 2) NYSDEC's Groundwater Sampling for Emerging Contaminants Analysis and Reporting Guidelines



OBG | There's a way

April 27, 2018

#### Ms. Karen Cahill

New York State Department of Environmental Conservation 615 Erie Boulevard Syracuse. New York 13204

RE: Site #727009 Krutulis Property
NYSDEC Request for Sampling of Emerging Contaminants

FILE: 2874/69452

#### Dear Ms. Cahill:

This letter provides the draft work plan required in the April 10, 2018 letter to Douglas Morrison of Bristol-Myers Squibb Company (B-MS) requesting groundwater sampling for per- and polyfluoroalkyl substances (PFAS) and 1,4-dioxane at the Krutulis Property. This work plan presents the Investigation Approach, Data Evaluation and Reporting, and Schedule below.

The PFAS and 1,4-dioxane sampling will be performed concurrent with the April semi-annual volatile organic compound (VOC) sample event on May 2, 2018 required under the Krutulis Property Groundwater Monitoring Plan (GWMP). The NYSDEC's review and approval is requested before this sample event to allow B-MS to meet the GWMP.

#### **INVESTIGATION APPROACH**

The proposed scope and methods associated with sampling groundwater at the Krutulis Property for PFAS and 1,4-dioxane are presented below. The health and safety considerations associated with this work are also presented below.

#### **GROUNDWATER SAMPLING**

*Objective*: Groundwater sampling will be conducted at the Krutulis Property to evaluate if PFAS and 1,4-dioxane are present on-site.

Approach: One round of samples will be collected from seven of the eight Krutulis Property groundwater monitoring wells in conjunction with the semi-annual groundwater volatile organic compound (VOC) sampling event. These target locations (MW-1, MW-2, MW-3S, MW-3D, MW-4, MW-6S, and MW-6D) are presented on **Figure 1**. One downgradient well (MW-4) was selected for this sampling since MW-4 and MW-5 are close, and MW-4 can be considered representative of the downgradient conditions. Additionally, MW-4 requires less sampling equipment due to seasonal access issues (*i.e.*, chest waders) and selected to help minimize the risk of cross-contamination. MW-5 could be sampled at a later date if PFAS and/or 1,4-dioxane are detected at appreciable levels at MW-4 and/or upon NYSDEC request.

The monitoring wells will be purged and sampled using a PVC bailer with Delrin® ball valve suspended by a nylon-coated stainless steel wire. A minimum of three well volumes of groundwater will be purged from each







monitoring well prior to sample collection. Field measurements for temperature, pH, and conductivity will be recorded subsequent to removal of each well volume during the purging process.

Once three well volumes have been purged from the well, the groundwater samples will be collected in accordance with sampling methodology provided in Collection of Groundwater Samples for Perfluorooctanoic Acid (PFOA) and Perfluorinated Compounds (PFCs) from Monitoring Wells Sample Protocol (NYSDEC, June 2016). Samples will be collected by filling the appropriate laboratory containers with water from the dedicated bailer. The required 1,4-dioxane and VOC samples for each location will be collected only after the sample bottles for PFAS have been filled and placed in the appropriate cooler(s).

Non-dedicated sampling equipment (i.e., water level probe) will be decontaminated between locations by washing with detergent (Alconox®) and PFAS-free distilled water and rinsed twice with PFAS-free distilled water to remove detergent residue. If a separate phase/sheen is noted during sampling activities, the decontamination process will be repeated, as needed, to ensure that the decontamination is effective.

Quality assurance and quality control samples (QA/QC) will be collected during this event including at least one equipment blank (i.e., new PVC bailer), one field duplicate, and one matrix spike/matrix spike duplicate. Additionally, an equipment blank will be collected from a new, unused bailer. Field notes including a sampling checklist will be used to document and record field conditions that could potentially result in crosscontamination.

The samples from each location will be submitted to TestAmerica Laboratories (TAL) for analysis via EPA Method 8270D SIM for 1,4-dioxane and Modified EPA Method 537 for the 21 PFAS analytes identified in Groundwater Sampling for Emerging Contaminants (NYSDEC, April 2018). Analytical data from the laboratory will be received in a full category B deliverable with an electronic data deliverable (EDD) for upload into an EQuIS database. Additionally, a Data Usability Summary Report (DUSR) will be prepared by a data validator and submitted along with the analytical results.

#### **HEALTH AND SAFETY**

Health and safety procedures will be in accordance with the OBG Corporate Health and Safety Program. The Job Safety Analysis (ISA) for this semi-annual sampling program has been updated for 2018 and will be available onsite. All workers will be required to don a minimum of Level D personal protective equipment (PPE), including: hard hat, safety glasses, safety shoes, high visibility vest, and spun-poly coveralls (where appropriate). The level of PPE required will be evaluated based on potential exposure to hazards (i.e., ticks) as outlined in the ISA and PFAS cross-contamination concerns (i.e., Ansell TNT® Powder-Free Nitrile Gloves gloves).

#### **DATA EVALUATION AND REPORTING**

A letter report will be submitted to NYSDEC upon completion of the requirements in *Groundwater Sampling for* Emerging Contaminants (NYSDEC, April 2018). The letter report will include a summary of the field activities and analytical results, with a figure presenting sampling locations and tabulated analytical results. A DUSR prepared by a data validator will be included as an attachment, and the required electronic data submission will accompany the letter report.

#### **SCHEDULE**

The sampling will occur in conjunction with the April (spring) groundwater semi-annual sampling event. This is currently planned for May 2, 2018 to facilitate procuring the appropriate sampling equipment (e.g., PVC bailers, Ansell TNT® Powder-Free Nitrile Gloves gloves). The May sampling instead of April was approved by the NYSDEC during an April 16, 2018 phone call between Karen Cahill (NYSDEC) and Chuck Sharpe (OBG).



Please feel free to contact me at (315) 956-6665 or charles.sharpe@obg.com or Mr. Douglas Morrison at (315) 432-4851 if you have any questions or comments.

Very truly yours,

O'BRIEN & GERE ENGINEERS, INC.

Charles W. Sharpe, P.E.

Program Manager

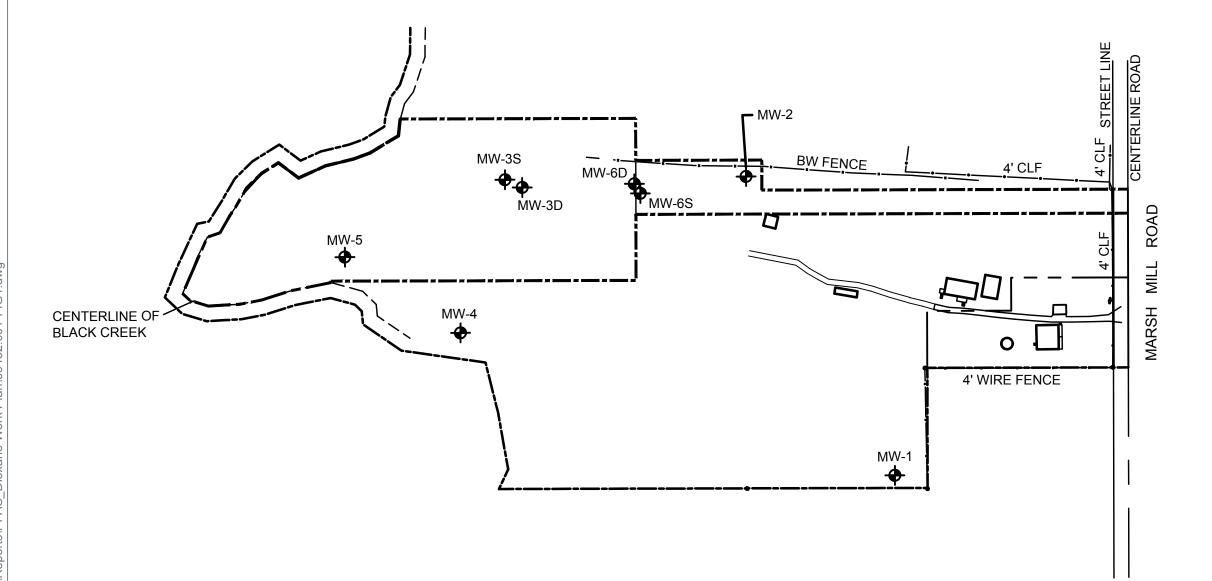
#### Attachment

cc: Douglas Morrison, Bristol-Myers Squibb Company (ec)

J.R. Pooler, Bristol-Myers Squibb Company (ec)

Harry Warner, NYSDEC (ec) Maureen Schuck, NYSDOH (ec) Richard and Pamela Mellor (1 copy)

I:\Bms.2874\69452.Krutulis-Ground\Corres\PFAS WP Ltr\_04-2018.docx





#### **LEGEND**

**→** MW-1

MONITORING WELL LOCATION

\_\_\_\_

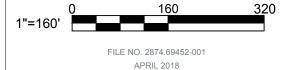
PROPERTY LINE

\_----

LIMITS OF KRUTULIS PROPERTY -REGISTRY SITE #727009

KRUTULIS PROPERTY KIRKVILLE, NEW YORK

MONITORING WELL LOCATIONS





O'BRIEN & GERE ENGINEERS, INC.

# NYSDEC's PFAS Monitoring Wells Sample Protocol

# Collection of Groundwater Samples for Perfluorooctanoic Acid (PFOA) and Perfluorinated Compounds (PFCs) from Monitoring Wells Sample Protocol

Samples collected using this protocol are intended to be analyzed for perfluorooctanoic acid (PFOA) and other perfluorinated compounds by Modified (Low Level) Test Method 537.

The procedure used must be consistent with the NYSDEC March 1991 Sampling Guidelines and Protocols <a href="http://www.dec.ny.gov/docs/remediation\_hudson\_pdf/sgpsect5.pdf">http://www.dec.ny.gov/docs/remediation\_hudson\_pdf/sgpsect5.pdf</a> with the following materials limitations.

At this time acceptable materials for sampling include: stainless steel, high density polyethylene (HDPE), PVC, silicone, acetate and polypropylene. Equipment blanks should be generated at least daily. Additional materials may be acceptable if preapproved by NYSDEC. Requests to use alternate equipment should include clean equipment blanks. NOTE: Grunfos pumps and bladder pumps are known to contain PFC materials (e.g. Teflon<sup>TM</sup> washers for Grunfos pumps and LDPE bladders for bladder pumps). All sampling equipment components and sample containers should not come in contact with aluminum foil, low density polyethylene (LDPE), glass or polytetrafluoroethylene (PTFE, Teflon<sup>TM</sup>) materials including sample bottle cap liners with a PTFE layer. Standard two step decontamination using detergent and clean water rinse will be performed for equipment that does come in contact with PFC materials. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFC materials must be avoided. Many food and drink packaging materials and "plumbers thread seal tape" contain PFCs.

All clothing worn by sampling personnel must have been laundered multiple times. The sampler must wear nitrile gloves while filling and sealing the sample bottles.

Pre-cleaned sample bottles with closures, coolers, ice, sample labels and a chain of custody form will be provided by the laboratory.

- 1. Fill two pre-cleaned 500 mL HDPE or polypropylene bottle with the sample.
- 2. Cap the bottles with an acceptable cap and liner closure system.
- 3. Label the sample bottles.
- 4. Fill out the chain of custody.
- 5. Place in a cooler maintained at 4 ± 20 Celsius.

Collect one equipment blank for every sample batch, not to exceed 20 samples.

Collect one field duplicate for every sample batch, not to exceed 20 samples.

Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, not to exceed 20 samples.

Request appropriate data deliverable (Category A or B) and an electronic data deliverable.

NYSDEC's Groundwater Sampling for Emerging Contaminants Analysis and Reporting Guidelines

#### **Groundwater Sampling for Emerging Contaminants**

February 2018

<u>Issue:</u> NYSDEC has committed to analyzing representative groundwater samples at remediation sites for emerging contaminants (1,4-dioxane and PFAS) as described in the below quidance.

#### **Implementation**

NYSDEC project managers will be contacting site owners to schedule sampling for these chemicals. Only groundwater sampling is required. The number of samples required will be similar to the number of samples where "full TAL/TCL sampling" would typically be required in a remedial investigation. If sampling is not feasible (e.g., the site no longer has any monitoring wells in place), sampling may be waived on a site-specific basis after first considering potential sources of these chemicals and whether there are water supplies nearby.

Upon a new site being brought into any program (i.e., SSF, BCP), PFAS and 1,4-dioxane will be incorporated into the investigation of groundwater as part of the standard "full TAL/TCL" sampling. Until an SCO is established for PFAS, soil samples do not need to be analyzed for PFAS unless groundwater contamination is detected. Separate guidance will be developed to address sites where emerging contaminants are found in the groundwater. The analysis currently performed for SVOCs in soil is adequate for evaluation of 1,4-dioxane, which already has an established SCO.

#### **Analysis and Reporting**

Labs should provide a full category B deliverable, and a DUSR should be prepared by a data validator.

The work plan should explicitly describe analysis and reporting requirements.

<u>PFAS sample analysis</u>: Samples should be analyzed by an environmental laboratory certified by ELAP to use EPA method 537 or ISO 25101. ELAP does not currently offer certification for PFAS analysis of non-drinking water samples (including groundwater, soil and sediment), so there is no requirement to use an ELAP certified method. The preferred method is the modified EPA Method 537. Labs have been able to achieve reporting limits for PFOA and PFOS of 2 ng/l (part per trillion). If labs are not able to achieve similar reporting limits, the NYSDEC project manager will make case-by-case decisions as to whether the analysis can meet the needs for the specific site.

<u>PFAS sample reporting:</u> DER has developed a PFAS target analyte list (below) with the intent of achieving reporting consistency between labs for commonly reportable analytes. It is expected that reported results for PFAS will include, at a minimum, all the compounds listed. This list may be updated in the future as new information is learned and as labs develop new capabilities. If lab and/or matrix specific issues are encountered for any particular compounds, the NYSDEC project manager will make case-by-case decisions as to whether particular analytes may be temporarily or permanently discontinued from analysis for each site. Any technical lab issues should be brought to the attention of a NYSDEC chemist.

Some sampling using this full PFAS target analyte list is needed to understand the nature of contamination. It may also be critical to differentiate PFAS compounds associated with a site from other sources of these chemicals. Like routine refinements to parameter lists based on investigative findings, the full PFAS target analyte list may not be needed for all sampling intended to define the extent of

contamination. Project managers may approve a shorter analyte list (e.g., just the UCMR3 list) for some reporting on a case by case basis.

<u>1,4-Dioxane Analysis and Reporting:</u> The method detection limit (MDL) for 1,4-dioxane should be no higher than 0.28  $\mu$ g/l (ppb). ELAP offers certification for both EPA Methods 8260 and 8270. In order to get the appropriate detection limits, the lab would need to run either of these methods in "selective ion monitoring" (SIM) mode. DER is advising PMS to use 8270, since this method provides a more robust extraction procedure, uses a larger sample volume, and is less vulnerable to interference from chlorinated solvents (we acknowledge that 8260 has been shown to have a higher recovery in some studies).

#### **Full PFAS Target Analyte List**

Group	Chemical Name	Abbreviation	CAS Number
	Perfluorobutanesulfonic acid	PFBS	375-73-5
	Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluoroalkyl sulfonates	Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Sanonates	Perfluorooctanessulfonic acid	PFOS	1763-23-1
	Perfluorodecanesulfonic acid	PFDS	335-77-3
	Perfluorobutanoic acid	PFBA	375-22-4
	Perfluoropentanoic acid	PFPeA	2706-90-3
	Perfluorohexanoic acid	PFHxA	307-24-4
	Perfluoroheptanoic acid	PFHpA	375-85-9
Dorfluoroollad	Perfluorooctanoic acid	PFOA	335-67-1
Perfluoroalkyl carboxylates	Perfluorononanoic acid	PFNA	375-95-1
	Perfluorodecanoic acid	PFDA	335-76-2
	Perfluoroundecanoic acid	PFUA/PFUdA	2058-94-8
	Perfluorododecanoic acid	PFDoA	307-55-1
	Perfluorotridecanoic acid	PFTriA/PFTrDA	72629-94-8
	Perfluorotetradecanoic acid	PFTA/PFTeDA	376-06-7
Fluorinated Telomer	6:2 Fluorotelomer sulfonate	6:2 FTS	27619-97-2
Sulfonates	8:2 Fluorotelomer sulfonate	8:2 FTS	39108-34-4
Perfluorooctane- sulfonamides	Perfluroroctanesulfonamide	FOSA	754-91-6
Perfluorooctane-	N-methyl perfluorooctanesulfonamidoacetic acid	N-MeFOSAA	2355-31-9
sulfonamidoacetic acids	N-ethyl perfluorooctanesulfonamidoacetic acid	N-EtFOSAA	2991-50-6

Bold entries depict the 6 original UCMR3 chemicals

Attachment B –
Groundwater Sampling
Logs

G OB	G	Standard	d Groundv	vater Sampling Log	· ėli ID:	<u>MW-1</u>
Project No.:	69452	Evacuation	n Method:	PVC Bailer	Field Personnel:	SGM/MM
Site Name:	BMS - Krutulis	Samplin	ig Method: _	Conventional	Date:	5/2/2018
Site Loc.:	Kirkville, NY	Equipm	nent Used: _	PVC Bailer - Dedica	ted Weather:	86°F, SUNN
Vell Informatio						<del> </del>
å	Depth of Well: _	18.59 ft. bmp*		. / ft. of casing by diamete	•	
	Depth to Water: _	2.36 ft. bmp*		" Well = 0.041 X LWC	X Well C	
Length of W	/ater Column (LWC):	16-23 ft.		" Well = 0.163 X LWC	· □ Protec	tive Casing
	Well Volume: _	<b>Ĵ, </b> ∕₀gal.	□ 4	" Well = 0.653 X LWC	☐ Other:	
	3X Well Volume: _	nSD gal.	□ 6	" Well = 1.469 X LWC		
Purc	ge Start Time:	13/15	<u> </u>	Volume removed b	efore sampling:	v8.0 gal.
	rge End Time:	13:24			he well go dry?: yes /	
Groundwater I	Parameters:					
	Volume Purged:	Temperature:		pH:	Conductivity:	Other:
	(gallons)	(Celcius)		(S.I. Units)	mS/cm	TORBIOTY,
	(gunono)	1) A		(c cc)	۸ ۱۱۱	< 1.an
Initial:		14.4		8.10	0.71	- 100
_4	22.6	9.00		8.02	0.42	-100
	~5.0	9.1	,	7.96	0.44	< 100
_	~0.0	97		8.06	0.46	<100
_	3,0				<u> </u>	- (0)
_				<del></del>	<del></del>	
					·	
		•				
				······································		
					·	
					<u> </u>	
Groundwater	Sample:	20		•		
	ime Collected: 131	<u>30</u>			1	
Physical Appea	arance at Start:			Physical Appearan	ce Upon Sampling:	
	Color:	l or	a		Color: Cuar	
	Odor: 🔥	ONE	•		Odor: None	· .
Turbidity	(> 100 NTU): NO			Turbidity (> 10	0 NTU): <b>V</b>	
Sneen/r Samples colle	Free Product:	Ne		Sheen/Free	Todace. Todace	
·	ntainer Size	Container Type	# Collected	Field Filtered	Preservative	Container p
COI	T L	AMBER CHIS	3	Lieig Lilieieg	NONE	N/A
	12	PURSTIC	3	No	None	NA
	40 mc	VOA	2	NO	HCL	N/A
<u> </u>						
2- 6 1						

6	BG	Standard	d Groun	dwater Sampling Log	Well	ID: <u>MW-2</u>
Project No.	: 69452	Evacuatio	n Method:	PVC Bailer	Field Person	nel:SGM/MM
Site Name	: BMS - Krutulis	Samplin	ng Method:	Conventional		ate: 5/2/2018
Site Loc.	: Kirkville, NY	Equipm	nent Used:	PVC Bailer - Dedica	ted Weath	ner: 80°F, Sunny
Well Inform						<u> </u>
	Depth of Well:	19.21 ft. bmp*	Well V	ol. / ft. of casing by diame		ement Point:
	Depth to Water:	7.14 ft. bmp*		1" Well = 0.041 X LWC	X We	ell Casing
Length o	of Water Column (LWC):	12.07 ft.	Х	2" Well = 0.163 X LWC	. $\square$ Pro	otective Casing
	Well Volume:	~2.() gal.		4" Well = 0.653 X LWC	□ Oth	ner:
	3X Well Volume:	gal.		6" Well = 1.469 X LWC	·	
F	Purge Start Time:	14:4()		Volume removed	before sampling:	~(0.0) gal.
Purge End Time:		14:55		Did	the well go dry?: yes	s / (no)
Groundwat	er Parameters:					
,	Volume Purged:	Temperature:	•	pH:	Conductivity:	Other:
	(gallons)	(Celcius)		(S.I. Units)	mS/cm	(TURBIDITY)
Initial:		12.2		10,57	0.09	2100
,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	22.0	8.9		6.70	0.18	<100
		8,9	<del></del>	6.97	0.21	<u>~100</u>
	24.0	011				
	~ (e. 0	8.1		7.00	0.22	<u> &lt; 100</u>
			<u></u>			
	•					
			<del></del>		·	
44	<del> </del>	<del></del>		<u> </u>		
			-			
Groundwat	er Sample:					
	Time Collected: 15;	00				
Physical Ap	pearance at Start:			Physical Appearar	nce Upon Sampling:	
	Color:C@	av			Color: Clear	
	Odor: No	V€:			Odor: None	- <del>-</del>
Turbid	lity (> 100 NTU):			Turbidity (> 10	0 NTU):	
1	en/Free Product: Ne			Sheen/Free		
Samples co						
-	Container Size	Container Type	# Collecte	ed Field Filtered	Preservative	Container pH
	16	OLASS.	1	NO	NONC	NA
	16	PLASTIC		<u> </u>	Nowe	NA NA
	4() mL	VOA		NO	HCL.	NA.
Notes:						

G OB(	G	Standa	Standard Groundwater Sampling Log				Well ID: M	
Project No.:	69452	Evacuati	ion Method:	PVC	Bailer	Field	Field Personnel:	
Site Name:	BMS - Krutulis	Sampli	ing Method:	Conv	entional		Date:	5/2/2018
Site Loc.:	Kirkville, NY	Equip	ment Used:	PVC Bailer	- Dedicated		Weather:	75°F, SUNN
/ell Information	n:		···.					
	Depth of Well:	18.36 ft. bmp*	Well V	ol. / ft. of casing b	y diameter:	* N	/leasurement	Point:
	Depth to Water:	Above ft. bmp*		1" Well = 0.041	X LWC		X Well Cas	sing
Length of Wa	ı :(ater Column (LWC	3	Х	2" Well = 0.163	X LWC		□ Protectiv	e Casing
				4" Well = 0.653	X LWC		□ Other:	
	3X Well Volume:			6" Well = 1.469	X LWC		<del></del>	
		0955		Volume	emoved hefo	ore sampling:	<del>\tag{\tau}</del>	9.0 gal
_	e Start Time:			voluitie i		well go dry?:		<u> </u>
	ge End Time:	1030						
Groundwater P	arameters:							
	Volume Purged:	Temperature	<b>)</b> :	pH:		Conductivity:		Other:
	(gallons)	(Celcius)		(S.I. Units)		mS/cm		(TURBIDITY)
Initial:	0	17.3 7.99		0.39		<100		
ai	3	10.5		8.14		0.39		2100
_		10.7		8.23		0.42		< 100
_	6					0.41		< 100
	9	10.6	·	7.69		0,11		2 100
				•			_	
. —								
					<del></del>			
	<u> </u>							
								· · · · · · · · · · · · · · · · · · ·
<del>-</del>						1	<del></del>	
_								
_					<del></del>	<u></u>		
Groundwater	Sample:				- · · · · · · · · · · · · · · · · · · ·			
	ime Collected: 10	235_		Physica	i Annagrance	e Upon Samplin	a.	
Physical Appe	arance at Start:			PHYSICA				
	Color:	lear			•	Color:		
	Odor: 🔥	one				Odor:		
Turbidity		10		Turi	bidity (> 100 l	NTU):	0	
	· · · · · · · · · · · · · · · · · · ·	line			neen/Free Pro		u	
Samples coll						<del></del>		
	ntainer Size	Container Type	# Colle		ld Filtered	Preserv		Container
	1 1-	AMBERGLASC			NO		NE	12/4
	12	PLASTIC	1		<u> </u>		NE NEHCL	W/A W/A
	40 mc	VOA	2		<i>.</i>	700	10 // CC	· _ · · // 0
	•		<del> </del>					
l <del></del>	<u> </u>		<del>                                     </del>					
		<del></del>						
<del></del>								

<b>G</b> o	BG	Standa	rd Ground	dwater Sampling	Log	We	ell ID:	MW-3D
Project No.	.: 69452	Evacuat	ion Method:	PVC Ba	iler	Field Pers	sonnel: _	SGM/MM
Site Name	: BMS - Krutulis	Sampl	ing Method:	Convention	onal	Date:		5/2/2018
Site Loc.	.: Kirkville, NY	Equip	ment Used:	PVC Bailer - D	Dedicated	Weather:		75°F, SUNN
Well Inform		·	<del></del>					
	Depth of Well:	<del></del>		ol. / ft. of casing by d			suremen	
	Depth to Water:			1" Well = 0.041 X L\		X	Well Ca	_
Length o	of Water Column (LWC):	1) 561		2" Well = 0.163 X L\				ive Casing
	Well Volume:			4" Well = 0.653 X LV			Other:_	
	3X Well Volume:	13,75 gal.	. 0	6" Well = 1.469 X LV	NC			
F	Purge Start Time:	0955		Volume remo	oved before	sampling:		-15-0 gal.
٠.	Purge End Time:	1040			Did the we	•		no
Groundwate	er Parameters:				·=		·	
	Volume Purged:	Temperature:	<b>!</b>	pH:		Conductivity:		Other:
	(gallons)	(Celcius)		(S.I. Units)		mS/cm		(TURBIDAY)
Initial:	0	10.4		8.14		1 27		<100
	~5.0	10.4		8 18		0.34	· –	
,	210.	10.8		7 9/2		<u> </u>	. <b>-</b>	< (00)
	~15	- 10 6		7.18		$\frac{0.00}{4.00}$	-	< 100
		10.0		8.00		0.56	. <u>-</u>	<u> </u>
	- June 1	•				·	. <u>-</u>	
		<u> </u>					. <u>-</u>	
		- <u> </u>						
					<u></u>		_	
a.							_	
		·					_	
•							_	
		,	· -		<u> </u>			·
		· · · · · · · · · · · · · · · · · · ·	· _		- —		_	
Groundwate	er Sample: Time Collected: [O	1		<u> </u>		· · · · · · · · · · · · · · · · · · ·		
	pearance at Start:	<u>12_</u>		Physical App	naranca lin	on Compling:		
	Color:	~ V		<u>i Hyşidai Fippi</u>				
					Colo		<del></del>	
= 4.1 m	Odor: <u>Net</u>		<del></del>		Odo		<u> </u>	
	ty (> 100 NTU): No		<del></del>	Turbidity	(> 100 NTU	): <u>VO</u>		
	n/Free Product:	( <u></u>	<del></del>	Sheen/F	Free Produc	t: _/\0		·
Samples col						``		
Co	ontainer Size	Container Type	# Collected			Preservative		Container pH
	1	AMBERGUSS PUNSTIC	<del></del>	100		NONE		N/A
· · _ · _	40 ML	VOA		NO		-New 1	400	N/A N/A
						10017	7	
		-		<del>-  </del>				
lotes:						·		

69452  BMS - Krutulis  Kirkville, NY  Depth of Well:  Depth to Water: er Column (LWC): Well Volume: 3X Well Volume: Start Time: e End Time:	18.63 ft. bmp  0.62 ft. bmp  17.81 ft.  2.90 gal.	*	Convention PVC Bailer - De  Yol. / ft. of casing by di 1" Well = 0.041 X LW 2" Well = 0.163 X LW 4" Well = 0.653 X LW 6" Well = 1.469 X LW	ameter: * N	Personnel: SGM/I  Date: 5/2/20  Weather: 75° F  Measurement Point:  X Well Casing  Protective Casing  Other: 4	)18
Depth of Well: Depth to Water: er Column (LWC): Well Volume: 3X Well Volume: Start Time:	18.63 ft. bmp 0.62 ft. bmp 17.81 ft. 2.90 gal. 8.71 gal.	* Well \ * \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \	PVC Bailer - December 201. / ft. of casing by discussion of the case of the casing by discussion of the case of th	ameter: * M	Weather: 75° F / .  Measurement Point:  X Well Casing  ☐ Protective Casing	
Depth of Well: _ Depth to Water: _ er Column (LWC): _ Well Volume: _ 3X Well Volume: _ Start Time: _ End Time: _	18.63 ft. bmp 0.62 ft. bmp 17.81 ft. 2.90 gal. 8.71 gal.	* Well \ *	/ol. / ft. of casing by di 1" Well = 0.041 X LW 2" Well = 0.163 X LW 4" Well = 0.653 X LW 6" Well = 1.469 X LW	ameter: * N /C : /C [	// // // // // // // // // // // // //	Supry
Depth of Well: _ Depth to Water: _ er Column (LWC): _ Well Volume: _ 3X Well Volume: _ Start Time: _ End Time: _	0.62 ft. bmp ft. 2.90 gal. 8.71 gal.	*	1" Well = 0.041 X LW 2" Well = 0.163 X LW 4" Well = 0.653 X LW 6" Well = 1.469 X LW	IC I	X Well Casing  Protective Casing	
Depth to Water: er Column (LWC): Well Volume: 3X Well Volume: Start Time:	0.62 ft. bmp ft. 2.90 gal. 8.71 gal.	*	1" Well = 0.041 X LW 2" Well = 0.163 X LW 4" Well = 0.653 X LW 6" Well = 1.469 X LW	IC I	X Well Casing  Protective Casing	
er Column (LWC):  Well Volume:  3X Well Volume:  Start Time:	$\begin{array}{c c} \hline 17.81 & \text{ft.} \\ \hline 2.90 & \text{gal.} \\ \hline 8.71 & \text{gal.} \\ \hline \hline -0900 & 08 \\ \hline \end{array}$	<b>X</b>	2" Well = 0.163 X LW 4" Well = 0.653 X LW 6" Well = 1.469 X LW	/C [	☐ Protective Casing	
Well Volume: _ 3X Well Volume: _ Start Time: _ End Time: _	2.90 gal. 8.71 gal.		4" Well = 0.653 X LW 6" Well = 1.469 X LW	/C		
3X Well Volume: Start Time:	8.71 gal.		6" Well = 1.469 X LV		□ Other: <u>~</u>	
Start Time:	<del>-9900</del> 08			/C		
End Time:		ss				
End Time:			Volume remo	ved before sampling:	29.0	gal.
rameters:				Did the well go dry?:	yes /no	
				***		
Volume Purged:	Temperatu	re:	pH:	Conductivity:	Othe	er:
(gallons)	(Celcius	· )	(S.I. Units)	mS/cm	(_TURI	BIDITY)
0	+1 6		7.96	0.47		ŕ
					Turkidity	
						<u> </u>
9	<del></del>	<u>-</u>				
		<del></del>				
••						
					<u> </u>	
					· ·	
	· .				<del></del>	
	••••				<del></del>	
			· · · · · · · · · · · · · · · · · · ·			
		·				
mple:	0					
	<u>o</u>		Physical App	earance Upon Sampling:		
	_			Color: Cile	٠. حـ	•
						_
			Turbidity			_
, <del></del>				· · · · ———		
	<u> </u>	<u> </u>	Sneen/i	-ree Product: 70014		
		I		, Dunnamus	this Conta	
		# Collect				
L		2				7
OML	VOA	2	N			4
	mple: General Start: Color: Clean Odor: None 100 NTU): > 1 Product: None Color: Size L L Cone Size L C	(gallons) (Celcius)  O 11.6  3 10.5  6 10.5  9 10.6  mple: e Collected: 0910  nce at Start:  Color: Clear Odor: None 100 NTU): > 10 NTU  e Product: MML  ed: her Size Container Type  L Ambel GASS  L Pow	(gallons)  (Celcius)  (D 1).6  3 10.5  6 10.5  9 10.6  POLICE  Collected: O9 10  nce at Start:  Color: Clear Odor: None  100 NTU): > 10 NTV  e Product: None  Amber GASS  L Pour 2  Swrt VOA 2  Swrt VOA 2	(gallons)   (Celcius)   (S.I. Units)	(gallons)   (Celclus)   (S.I. Units)   ms/cm	(gallons) (Celcius) (S.I. Units) ms/cm (IDE)  0

G OF	3 <b>G</b>	Standa	rd Ground	lwater Sampling Lo	g	Well ID: <u>MW</u> -	
Project No.:	69452	Evacuat	ion Method:	PVC Bailer	Field	Field Personnel: SGN	
Site Name:	BMS - Krutulis	Sampl	ing Method:	Conventional		Date: 5/2/2018	
Site Loc.:	Kirkville, NY	Equip	ment Used:	PVC Bailer - Ded	icated	Weather:	30° F. SUNNY
Well Informati	Alaa.			• •		<del> </del>	
weii intorma	Depth of Well:	22.12 ft. bmp*	Well Ve	ol. / ft. of casing by diam	neter: * N	/leasurement	Point:
	Depth to Water:	(q.5) ft. bmp*		1" Well = 0.041 X LWC		X Well Ca	
I enath of	Water Column (LWC):	15 jal ft.		2" Well = 0.163 X LWC			re Casing
Longaro	Well Volume:	2.54 gal.		4" Well = 0.653 X LWC		□ Other:	o caemy
	3X Well Volume:	7. 63 gal.		6" Well = 1.469 X LWC			· · · · · · · · · · · · · · · · · · ·
Pı	urge Start Time:	11:50		Volume remove	d before sampling:	م (	9.0 gal.
	urge End Time:	12:10			id the well go dry?:	yes / (r	
	<u> </u>	_12.10_	·	<i>-</i>	u the well go dry?.	yes / (	10/
Groundwate	r Parameters:						
	Volume Purged:	Temperature	:	pH:	Conductivity:		Other:
	(gallons)	(Celcius)		(S.I. Units)	mS/cm		(TOBBIDITY
Initial:	0	in.S		c 19	033		<100
_	23	8 9	<del></del>	8 24	0.35		< (60
-	N6	8. 30	<del></del> .	8.28	0.34	<del></del>	Z 100
-	~ 9	- G 2	<u> </u>		0.34	<del></del>	(100)
-		<u> </u>		8.25	- 0.74	<del></del> _	-100
-							
		· -					
_			<u>.                                    </u>				
		•					
•				-		<del></del>	
·			<del></del> .				
-			<del>·                                      </del>		-		
<u> </u>							
Groundwate	r Sample:	, i C					
	Time Collected: 12 earance at Start:	<u>`1.2</u>		Physical Annea	rance Upon Sampling:	ı	
1 11yoloul 1 lpp		llar		rryolograpped			
	· <del>-</del>	)oine	<del></del>	_	Odor: Non	L	
		Jo		Turbidity (>			
Sheen	/Free Product:/	10		Sheen/Fre	e Product: Vo		
Samples col	lected:						
Co	ontainer Size	Container Type	# Collecte				Container pH
	16	PLASTIC	}_	No No	Now		NA NA
	40mL	VOA	Ž	NO	Non		N/A
<u> </u>							
Notes							
Notes:							

	BG	Standa	d Groundy	vater Sampling Lo	y V	Well ID:	<u>MW-6D</u>
Project No.:	69452	Evacuati	on Method: _	PVC Bailer	Field Pe	ersonnel:	SGM/MM
Site Name:	BMS - Krutulis	Sampli	ng Method: _	Conventional		Date:_	5/2/2018
Site Loc.:	Kirkville, NY	Equip	ment Used:	PVC Bailer - Dedic	ated \	Weather:_	80°F, SVNNV
Well Inform							
	Depth of Well:	35.93 ft. bmp*		. / ft. of casing by diame		asurement	
	Depth to Water:	6.54 ft. bmp*		" Well = 0.041 X LWC	X	Well Ca	-
Length o	f Water Column (LWC):	29.39 ft.		" Well = 0.163 X LWC	. 🗆		ve Casing
	Well Volume:	4.8 gal.		" Well = 0.653 X LWC		Other:	
	3X Well Volume:	~15 <u>O</u> gal.	□ 6	" Well = 1.469 X LWC			
F	Purge Start Time:	11:40		Volume removed	before sampling:	21	15.0 gal.
I	Purge End Time:	12:23		Dic	the well go dry?:	yes /C	no
Groundwate	er Parameters:						
	Volume Purged:	Temperature:		pH:	Conductivity:		Other:
	(gallons)	(Celcius)	·	(S.I. Units)	mS/cm		(TURBIDIT
Initial:	$\mathcal{O}$	14.2		7.89	0.37		<100
	4.8	10.7		7.97	0.36	<del>-</del> -	4100
	9.6	10.2		8.06	0.36		4100
	N 15	10.5		7,90	0.36		**
	_ ~ 13	10.2		7, 10	<u> </u>		2100
			<del></del> -				· · · · · · · · · · · · · · · · · · ·
			<del>-</del> -	· · · · · · · · · · · · · · · · · · ·			
		· · · · · · · · · · · · · · · · · · ·	<del>-</del> -	***			
		-					
Groundwate	er Sample: Time Collected: 12	24					
Physical App	pearance at Start:			Physical Appeara	nce Upon Sampling:		
	Color: CU	ear			Color: Cua	-	
		ONE	<del></del> -		Odor: None		· · · · · · · · · · · · · · · · · · ·
Turhidi	ty (> 100 NTU):			Touchtable / A			· · · · · · · · · · · · · · · · · · ·
		ĵ		Turbidity (> 10 Sheen/Free			
Samples co				0.1001,/1.100	7-0		
С	ontainer Size	Container Type	# Collected	Field Filtered	Preservative	e	Container pH
	11_	AMBEZGUSS		100	IVONE		N/A
	40mi	PURCTIC	<u> </u>	NO	NONE		~ //A
				,,,,,	TILL		/+\
	<u> </u>	٦	· · ·	<del></del>			·

Attachment C – Data Usability Summary Report

TO: C. Sharpe Scott Mosher cc: file

FROM: KA Storne

**B-MS Krutulis Site Data Usability** RE:

**Summary Report** 

FILE: 2874/69452 DATE: June 29, 2018

This data usability summary report (DUSR) presents the results of data validation performed for samples collected at the Bristol-Myers Squibb (B-MS) Krutulis Site. Sample collection activities were conducted on May 2. 2018.

TestAmerica Laboratories, Inc. of Sacramento, California (TA Sacramento) and TA Buffalo of Amherst, New York performed the laboratory analyses for the sampling event. The laboratory package contains summary forms for quality control analysis and supportive raw data. Table 1 below summarizes the sample analysis submitted for data validation.

**Table 1. Analytical Methods and References** 

Analysis and Laboratory	Method	Reference
Pre- and Polyfluorinated Substances (PFAS) performed by TA Sacramento	Laboratory SOP based on Modified USEPA Method 537, Version 1.1 (TestAmerica Sacramento. 2017. Per- and Polyfluorinated Substances (PFAS) in Water, Soils, Sediments and Tissue (Method 537 Modified), SOP No. WS-LC-0025, Rev 2.2, 02/02/2017)	1
1,4-Dioxane performed by TA Buffalo	USEPA Methods 3510C/8000C/8270D	2

#### Notes:

- 1. USEPA. 2009. Modified Determination of Selected Perfluorinated Alkyl Acids in Drinking Water by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS), Version 1.1, Cincinnati, Ohio.
- 2. USEPA. 2007. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, Update IV. Washington D.C.

The samples submitted for data validation are summarized in the attached **Appendix A**. **Appendix B** presents the specific data validation approach applied to data generated for this investigation. **Appendix C** presents the laboratory quality assurance/quality control (QA/QC) analyses definitions.

Full validation was performed on the samples collected for this investigation. The analytical data generated for this investigation were evaluated using the QA/QC criteria established in the laboratory standard operating procedures (SOPs), the methods, and professional judgement. Since data validation guidelines for USEPA Method 537 are not available, application of qualifiers for excursions from the laboratory SOP and the method was based on the general approach used to qualify samples described in the following document.

USEPA. 2016. National Functional Guidelines for High Resolution Superfund Methods Data Review. EPA-542-B-16-001. Washington, D.C.

Application of qualifiers to the 1,4-dioxane data affected by excursions from the method criteria was based on guidance provided in the following document and professional judgment.

USEPA. 2008. USEPA Region II Validating Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry SW-846 Method 8270D, SOP HW-22 Revision 4. Reviewed 2009.

The following qualifiers are used in this type of data validation:

- "R" Indicates that the reporting limit (RL) or sample result has been identified as unusable due to a major deficiency in the data generation process. The data were rejected and should not be used for any qualitative or quantitative purposes.
- "U" Indicates that the analyte was not detected and the sample RL is presented. This qualifier is also used to signify blank excursions.
- "J" Indicates that the concentration should be considered approximate. The target analyte was positively identified and the associated numerical value is the approximate concentration; either the data quality criteria were not met or the concentration of the target analyte was greater than the method detection limit (MDL) and below the RL.
- "UJ" Indicates that the analyte was analyzed for and was not detected; however, the RL is presented and should be considered approximate. This qualifier is used when data quality criteria were not met.
- "JN" Indicates that the target analyte has been "tentatively identified" as present and the associated numerical value is the estimated concentration in the sample. This qualifier may be applied due to data interpretation issues.

The data quality evaluation results in only one type of qualifier for each analyte; in a case when several qualifiers are applicable to the same analyte, the cumulative effect of the various QA/QC excursions is employed in assigning the final data qualifiers. For example, the final data qualifier is the "R" qualifier if a sample result is affected by low LCS recovery, for which the "UJ" qualifier is applied, but low MS/MSD recoveries result in the rejection of the sample result (application of the "R" qualifier). QA/QC excursions that do not result in the qualification of an analyte are not discussed, except for those excursions that provide useful information to the data user.

The data validation included an evaluation of the following parameters, where applicable:

- Chain-of-custody records
- Sample collection and sample preservation
- Holding times
- Instrument performance
- Calibration
- Blank analysis
- Surrogate recovery
- Matrix spike (MS)/matrix spike duplicate (MSD) analysis
- Laboratory control sample (LCS)/Laboratory control sample duplicate (LCSD) analysis
- Field duplicate samples analysis
- Isotopic dilution analyte (IDA) performance
- Internal standards performance
- Target compound identification, quantitation, and reporting limits (RLs)
- Documentation completeness

The following sections of this memorandum present the results of the comparison of the analytical data to the QA/QC criteria specified above.

#### CHAIN OF CUSTODY RECORD AND SAMPLE COLLECTION

Samples submitted for PFAS analysis were collected at the sampling location using one 1-L plastic containers that were provided by the TA Service Center in Syracuse, New York. After receiving the samples, the TA Service Center representative decanted the samples into 250-mL containers as requested by TA Sacramento. This was done at the recommendation of a TA PFAS specialist.

Sample collection using two 250-mL container is approved by TA Sacramento for PFAS analysis. TA Sacramento indicated that decanting samples from a 1-L container to a 250-mL container, which adds to sample handling time, is not "the ideal approach" to PFAS sampling.

During sample preparation in the laboratory, samples submitted for PFAS analysis were decanted prior to extraction due to the presence of excess sediment in the samples. The laboratory did not contact OBG for these samples although laboratory policy includes contacting the client to discuss the actions to be taken when particulates are found in samples. Decanting is a normal procedure due to the occurrence of sediment in non-potable water samples, such as groundwater monitoring well samples, and the impact the sediment has on the analytical equipment.

Samples submitted for PFAS analysis were relinquished and received by the TA Service Center in Syracuse, New York on May 2, 2018. During the second sample transfer on May 2, 2018, samples were relinquished by a different TA service center representative to a courier. The samples were received at TA Sacramento on May 3, 2018. The courier name and tracking numbers were not listed on the record but were provided separately by the laboratory.

Samples submitted for 1,4-dioxane analysis were relinquished from the field collection representative and received by the TA Service Center in Syracuse, New York on May 2, 2018. The courier and courier number were not recorded on the chain-of-custody record. Also, the custody seal numbers were not documented on the record.

The sample collection times listed on the sample labels for sample submitted for PFAS analysis were inconsistent with the chain-of-custody record information for the following samples: MW-3S-050218 and MW-4-050218. The correct samples times were communicated to the laboratory. Although listed on the chain-of-custody record, sample MW-5-050218 was removed from the sample list based on communications to the laboratory.

#### PFAS DATA EVALUATION SUMMARY

The following QA/QC parameters were found to meet validation criteria or did not result in additional qualification of sample results:

- Sample preservation
- Holding times
- Instrument performance
- Calibration
- Surrogate recovery
- MS/MSD analysis
- LCS /LCSD analysis
- Field duplicate samples analysis



- IDA performance
- Internal standards performance
- Target compound identification, quantitation and RLs
- Documentation completeness

Deviations from QA/QC criteria that resulted in qualified data and additional observations are summarized below.

#### I. Blank Analysis

Target analytes were detected in the field reagent blank (FRB-050218), equipment blank (EB-050218), and method blanks for PFAS. The following samples were qualified as non-detected (U) for the minor representativeness blank excursions:

- Perfluorohexanesulfonic acid (PFHxS) in samples MW-1-050218, MW-2-050218, MW-3S-050218, MW-3D-050218, MW-4-050218, MW-6S-050218, MW-6D-050218, and FD-050218 [MW-4-050218].
- Perfluorobutanoic acid (PFBA) in samples MW-1-050218, MW-6D-050218, and FD-050218 [MW-4-050218].

TA Sacramento indicated that detection of the two PFAS target analytes (PFBS and PFHxS) in the field reagent blank and equipment blank may have been due to the handling and transfer of the samples at the TA Service Center as described above. In addition, the laboratory method blank also contained PFHxS that is possibly due to laboratory artifact. Based on input from the laboratory, PFAS analytes have been detected in a variety of clean samples due to their ubiquitous nature.

#### II. Target Analyte Quantitation, Identification and RLs

A data validation qualifier was applied to the following sample result:

- The result for perfluorohexanesulfonic acid (PFHxS) in sample MW-2-050218 was qualified as approximate (IN) based on peak data interpretation.
- The result for perfluorobutanoic acid (PFBA) in sample MW-6S-050218 was qualified as approximate (JN) based on matrix interference.

The validation approach utilized for this data set is presented in **Appendix B**. The following observations pertain to the laboratory target analyte quantitation and identification process applied to data.

- The laboratory analyst's experience and judgement are used to evaluate and report target analyte identification and concentrations, based on interpretation of target analyte peak shape, the chromatography baseline, target analyte retention time, and signal strength.
- During data validation, peak integration and identification interpretation performed by the laboratory analysts was reviewed. Validation qualifiers were applied to sample results when chromatography, retention times, or peak shapes may have impacted sample identifications and/or concentrations.

#### 1,4-DIOXANE DATA EVALUATION SUMMARY

The following QA/QC parameters were found to meet validation criteria or did not require additional comments:

- Sample preservation
- Holding times
- Instrument performance
- Calibration



- Blank analysis
- Surrogate recovery
- MS/MSD analysis
- LCS /LCSD analysis
- Field duplicate samples analysis
- Internal standards performance
- Target compound identification, quantitation and RLs
- Documentation completeness

Deviations from QA/QC criteria were not identified during data validation.

#### **DATA USABILITY**

The data from the samples presented in **Appendix A** were evaluated based on QA/QC criteria established by the laboratory SOP and methods. Data validation qualifiers were applied to data based on the general approach presented in **Appendix B**.

Major deficiencies which would have resulted in rejected data were not identified for data from this sampling event. Minor deficiencies in the data generation process resulted in approximation of some sample data and data qualified as non-detected.

This section summarizes the adherence of the analytical data to precision, accuracy, representativeness, comparability, completeness, and sensitivity data quality objectives. Data quality was evaluated using percent usability, defined as the percentage of sample results that are usable for qualitative and quantitative purposes.

**Precision:** Data usability with respect to precision is 100%

**Sensitivity:** Sensitivity is established by RLs, which represent measurable concentrations of analytes that can be quantified with a designated level of confidence and are less than the project action limits established for the project. Dilutions were not performed for sample analyses. Data usability with respect to sensitivity is 100%.

**Accuracy:** Data usability with respect to accuracy is 100%.

**Representativeness:** Data usability with respect to representativeness is 100%.

**Comparability:** Comparability is not compromised provided that the analytical method approach did not change over time. A major component of comparability is the use of standard reference materials for calibration and QC. Since the modified analytical method approach and reporting procedures were consistently used by the laboratory, the comparability criteria for the analytical data were met.

**Completeness:** Overall, considering the complete data set, 100% of the data were usable for quantitative and quantitative purposes based on the data validation performed.



		Su	ımmary Table of S	Samples Va	lidated
Laboratory Performing Analysis	Date Collected	Field Chain-of-Custody Sample Identification	Laboratory Identification	Matrix	Analysis Requested
TA Sacramento	5/2/2018	MW-1-050218, MS/MSD	480-135293-1	Aqueous	PFAS by Laboratory SOP Based on Modified USEPA Method
TA Sacramento	5/2/2018	MW-2-050218	480-135293-2	Aqueous	PFAS by Laboratory SOP Based on Modified USEPA Method
TA Sacramento	5/2/2018	MW-3S-050218	480-135293-3	Aqueous	PFAS by Laboratory SOP Based on Modified USEPA Method
TA Sacramento	5/2/2018	MW-3D-050218	480-135293-4	Aqueous	PFAS by Laboratory SOP Based on Modified USEPA Method
TA Sacramento	5/2/2018	MW-4-050218	480-135293-5	Aqueous	PFAS by Laboratory SOP Based on Modified USEPA Method
TA Sacramento	5/2/2018	MW-6S-050218	480-135293-7	Aqueous	PFAS by Laboratory SOP Based on Modified USEPA Method
TA Sacramento	5/2/2018	MW-6D-050218	480-135293-8	Aqueous	PFAS by Laboratory SOP Based on Modified USEPA Method
TA Sacramento	5/2/2018	FRB-050218	480-135293-9	Aqueous	PFAS by Laboratory SOP Based on Modified USEPA Method
TA Sacramento	5/2/2018	EB-050218	480-135293-10	Aqueous	PFAS by Laboratory SOP Based on Modified USEPA Method
TA Sacramento	5/2/2018	FD-050218 [MW-4-050218]	480-135293-11	Aqueous	PFAS by Laboratory SOP Based on Modified USEPA Method
TA-Buffalo	5/2/2018	MW-1-050218, MS/MSD	480-135293-1	Aqueous	1,4-Dioxane
TA-Buffalo	5/2/2018	MW-2-050218	480-135293-2	Aqueous	1,4-Dioxane
TA-Buffalo	5/2/2018	MW-3S-050218	480-135293-3	Aqueous	1,4-Dioxane
TA-Buffalo	5/2/2018	MW-3D-050218	480-135293-4	Aqueous	1,4-Dioxane
TA-Buffalo	5/2/2018	MW-4-050218	480-135293-5	Aqueous	1,4-Dioxane
TA-Buffalo	5/2/2018	MW-6S-050218	480-135293-7	Aqueous	1,4-Dioxane
TA-Buffalo	5/2/2018	MW-6D-050218	480-135293-8	Aqueous	1,4-Dioxane
TA-Buffalo	5/2/2018	FRB-050218	480-135293-9	Aqueous	1,4-Dioxane
TA-Buffalo	5/2/2018	EB-050218	480-135293-10	Aqueous	1,4-Dioxane
TA-Buffalo	5/2/2018	FD-050218 [MW-4-050218]	480-135293-11	Aqueous	1,4-Dioxane

#### Notes:

TA Sacramento indicates TestAmerica Laboratories, Inc. located in Sacramento, Californica.

TA Buffalo indicates TestAmerica Buffalo located in Amherest, New York.

PFAS indicates perfluorinated substances.

SOP indicates standard operating procedure.

MS/MSD indicates matrix spike/matrix spike duplicate.

TB indicates trip blank.

FRB indicates field reagent blank.

FD indicates field duplicate.

The sample identification utilized for field duplicate is shown in brackets.





	Dioxane via USEPA SW-846 8270D SIM						
General Validation Approach	indicate both major and minor excursions so that data associated with any type of excursion are identified to the data user. Major excursions result in data being rejected (R), indicating that the data are considered unusable for either quantitative or qualitative purposes. Minor excursions result in sample data being qualified as approximate (J, UJ, JN) or non-detected (U) that is otherwise usable for quantitative or qualitative purposes.  Excursions are subdivided into excursions that are within and out of the laboratory's control. Excursions involving laboratory control sample recovery, calibration response, method blank excursions, low or high spike recovery due to inaccurate spiking solutions or poor instrument response, holding times, interpretation errors, and quantitation errors are within the control of the laboratory. Excursions resulting from poor spike recovery due to interference from the sample matrix is an example of an excursion that is not within the laboratory's control if the laboratory has followed proper method procedures, including applying appropriate sample preparation techniques.						
Applying Professional Judgment	USEPA data validation directs professional judgment to be used when applying qualifiers in some cases, considering the laboratory analysis approach and method requirements.						
Validation Guidelines – PFAS	TestAmerica Sacramento's laboratory standard operating procedure (SOP) reflects a modified version of USEPA Method 537, since Method 537 applies to only drinking water sample matrices. Therefore, evaluation of other aqueous and solid data is based on the laboratory SOP's requirements. Since data validation guidelines for Method 537 are not currently available, application of qualifiers for excursions from the laboratory SOP is based on the general approach used to qualify data described in the National Functional Guidelines for High Resolution Superfund Methods Data Review, April 2016.						
Validation Guidelines – 1,4-Dioxane	OBG data validation approach for the remaining analyses is based on current Region II guidelines for SW-846 methods.						
Validation Qualifiers	<ul> <li>"R" Indicates that the reporting limit (RL) or sample result has been identified as unusable due to a major deficiency in the data generation process. The data were rejected and should not be used for any qualitative or quantitative purposes.</li> <li>"U" Indicates that the analyte was not detected and the sample RL is presented. This qualifier is also used to signify blank excursions.</li> <li>"J" Indicates that the concentration should be considered approximate. The target analyte was positively identified and the associated numerical value is the approximate concentration; either the data quality criteria were not met or the concentration of the target analyte was greater than the method detection limit (MDL) and below the RL.</li> <li>"J+" Indicates that the concentration should be considered approximate and biased high. This qualifier identifies a deficiency in the data generation process.</li> <li>"J-" Indicates that the concentration should be considered approximate and biased low. This qualifier identifies a deficiency in the data generation process.</li> <li>"UJ" Indicates that the analyte was analyzed for and was not detected; however, the RL is presented and should be considered approximate. This qualifier is used when data quality criteria were not met.</li> <li>"JN" Indicates that the target analyte has been "tentatively identified" as present and the associated numerical value is the estimated concentration in the sample. This qualifier may be applied due to data interpretation issues.</li> </ul>						

Method 537 and 1,4-Dioxane via USEPA SW-846 8270D SIM		
Overall PFAS Method Summary	The TA Sacramento SOP, based on a modified Method 537, utilizes liquid chromatography/ tandem mass spectrometry (LC/MS/MS) and isotope dilution technique to analyze environmental samples for per- and polyfluorinated substances (PFAS). This includes the utilization of Isotope Dilution Analytes (IDAs) that are added to samples in the sample extraction process. Since IDAs respond in the same manner as target analytes, the IDA is used for target analyte identification and for calculating sample concentrations.	
Cooler Temperature	Results for samples submitted analyses that are impacted by coolers that did not contain ice, or if the ice melted upon receipt and the cooler temperatures are greater than 10°C are qualified as approximate (UJ, J).  If samples are delivered to the laboratory the same day as sample collection and samples did not have sufficient time to reach 10°C, samples are not qualified unless proper preservation was not provided for samples between sample collection and sample receipt at the laboratory.  Results for samples received at ambient temperature involved in extended shipment-day issues may be rejected applying professional judgment.	
Holding Time for PFAS	issues may be rejected applying professional judgment.  Samples are stored at 4±2°C.  Aqueous samples must be extracted within 7 days of collection and extracts analyzed within 40 days of extraction.  Results for samples properly preserved and analyzed outside of but less than two times the holding time window for preparation and/or analysis are qualified as approximate (UJ, J). Non-detected results for samples properly preserved and analyzed greater than two times the holding time window for preparation and/or analysis are rejected (R).  Detected results for samples properly preserved and analyzed greater than two times the holding time window for preparation and/or analysis are qualified as approximate (J).	
Holding Time for Organics (1,4- Dioxane)	Results for samples properly preserved and analyzed outside of but less than two times the holding time window established in the QAPP for preparation and/or analysis are qualified as approximate (UJ, J).  Non-detected results for samples properly preserved and analyzed greater than two times the holding time window for preparation and/or analysis are rejected (R).  Detected results for samples properly preserved and analyzed greater than two times the holding time window for preparation and/or analysis are qualified as approximate (J).  The entire sample target list for a VOC sample impacted by a holding time excursion is qualified.	
Calibration Evaluation for PFAS	A minimum of five to six calibration standards are analyzed to generate average response factors, linear or quadratic fit calibration curves. The calibrations are evaluated using the criteria of less than 35 percent relative standard deviation (%RSD) for target analytes quantitated by IDAs, less than 50 %RSD for target analytes quantitated by internal standards (without IDAs) or a correlation coefficient (r) greater than 0.995 for a linear fit.  Initial calibration verification (ICV) mid-range standard (from a separate source) must be within 60 to 140 percent recovery (%R) for target analytes associated with IDAs and within 50 to 150%R for target analytes without IDAs. IDAs in the ICV must be greater than or equal to 50%R and less than or equal to 150%R.	

OBG Data validation approach for PFAS in Aqueous Samples by TestAmerica Sacramento Laboratory - Modified USEPA Method 537 and 1,4-Dioxane via USEPA SW-846 8270D SIM			
	Mid-level continuing calibration verifications (CCVs) are analyzed at the beginning of the analysis sequence, at the end, and after every 10 samples. The CCV recovery must be within 60 to 140 percent recovery (%R) for target analytes associated with IDAs and within 50 to 150%R for target analytes without IDAs. IDAs must be greater than or equal to 50%R and less than or equal to 150%R.  Due to relative standard deviation (RSD) calibration excursions, detected results for analytes		
Calibration Actions for PFAS	in samples associated with the calibration are qualified as approximate (J). Non-detected results associated with RSD excursions may be qualified as approximate (UJ) based on professional judgment.		
	If the RSD calibration excursion is greater than 90, detected results for analytes in samples associated with the calibration are qualified as approximate (J) and non-detected results may be rejected (R) applying professional judgment.		
	For ICV excursions, detected and non-detected results for analytes in samples associated with the calibration are qualified as approximate (J, UJ).		
	Due to %D CCV excursions, detected and non-detected results for analytes in samples associated with the calibration are qualified as approximate (J, UJ).		
	SVOC target analytes are evaluated using the criteria of $<$ 20% RSD or correlation coefficient of 0.990 for initial calibration curves.		
Calibration Evaluation for SVOCs	Calibration verifications are evaluated using a criterion of 20%D for the target analytes. Initial calibrations and calibration verifications are also evaluated using the criterion for RFs listed in the method.		
	ICV recoveries are evaluated using laboratory control limits if available or 70 to 130%.		
Calibration Actions for Organics	Due to relative standard deviation (RSD) calibration excursions, detected results for analytes in samples associated with the calibration are qualified as approximate (J). Non-detected results associated with RSD excursions may be qualified as approximate (UJ) based on professional judgment.		
	If the RSD calibration excursion is greater than 90, detected results for analytes in samples associated with the calibration are qualified as approximate (J) and non-detected results may be rejected (R), applying professional judgment.		
	Due to %D calibration verification excursions, detected and non-detected results for analytes in samples associated with the calibration are qualified as approximate (J, UJ). The response direction and detection of target analytes in associated sample may be considered in applying qualifiers.		
	For response factor excursions, detected results are qualified as approximate (J) and non-detected results are rejected (R).		
	For initial calibration verifications (ICV) excursions, detected and non-detected results for analytes in samples associated with the calibration are qualified as approximate (J, UJ). The response direction and detection of target analytes in associated sample may be considered in applying qualifiers.		

Equipment blanks (rinsate blanks) are associated with samples collected in the same day (or sampling event) using the same sample collection equipment and decontamination solutions. When sampling equipment or decontamination solutions are changed, a new equipment blank should be collected. Each sample should be associated with one equipment blank, which is collected as close to the sample collection date/time as possible. Field blanks are associated with the sample containers used to collect samples. When

Field blanks are associated with the sample containers used to collect samples. When sampling container lots are changed, a new field blank should be collected.

Method blanks are associated with samples prepared at the same time as the samples. Method blanks should reflect the sample matrix type.

#### Associating Samples with Field and Laboratory QC Samples

Laboratory Control Samples (LCSs) are solutions containing known amounts of target analytes, analyzed within the laboratory to evaluate recovery of target analytes without sample matrix impacts. The LCSs are associated with samples prepared at the same time as the samples.

MS/MSD samples are collected in the field and spiked with known amounts of target analytes, analyzed in the laboratory to evaluate recovery and precision of target analytes. MS/MSDs measure the impact of matrix interference on target analytes. The MS/MSDs must be prepared using project samples and are associated with samples prepared at the same time with the same matrix type.

Field duplicates and collocated samples are duplicate samples collected in the field to measure field precision and are associated with samples of the same matrix type.

In the case that insufficient QC samples are provided due to field or laboratory problems, professional judgment is used to associate each sample with a QC sample that reflects the sample matrix and analysis conditions.

The laboratory control limit (CL) provided in the laboratory SOP is used to assess MS/MSD, LCS, surrogate and IDA data.

In the case that excursions are identified in more than one quality control sample of the same matrix within one sample delivery group, samples are batched according to sample preparation or analysis date and qualified accordingly.

In general, if percent recoveries are less than laboratory CLs but greater than 10%, non-detected and detected results are qualified as approximate (UJ, J).

If percent recoveries are greater than laboratory CLs, detected results are qualified as approximate (J).

# Evaluation and Actions for MS/MSD, LCS, Surrogate and Field Duplicate for Organic Data

If percent recoveries are less than 10%, detected results are qualified as approximate (J) and non-detected results may be rejected (R), applying professional judgement.

If RPDs for MSDs are outside of laboratory CLs, detected results are qualified as approximate (J). Non-detected results may not be qualified, applying professional judgment.

Qualification is performed only when both MS and MSD recoveries are outside of laboratory CLs.

Qualification is not performed for MS/MSD results if the sample concentration is greater than 4 times the MS or MSD spike concentration.

Non-detected data are rejected (R) in the case that both MS/MSD recoveries are less than 10%.

Qualification is not performed if MS/MSD are outside of laboratory CLs if the analysis was performed using a dilution factor of 10 times or more, applying professional judgment.

Qualification of data associated with MS/MSD or field duplicate excursions is limited to the un-spiked sample or the field duplicate pair, respectively.

Field duplicate data are evaluated against relative percent difference (RPD) criteria of less than 50% for aqueous samples and less than 100% for soils when results are greater than or equal to five times the RL. When a field duplicate result is less than five times the RL, a control limit of plus or minus two times the RL (difference criterion) is applied. If RPDs or differences are outside of criterion, detected and non-detected results are qualified as approximate (UJ, J).

#### Evaluation Field Duplicate Data for PFAS

**Evaluation and** 

Results (Trip,

Equipment)

Method, Field,

**Actions for Blank** 

Field duplicate data are evaluated against RPD criteria of less than 30% when results are greater than or equal to five times the RL. When a field duplicate result is less than two times the RL, a control limit of 50 RPD is applied. If RPDs are outside of criterion, detected and non-detected results are qualified as approximate (UJ, J).

Blanks are analyzed to evaluate laboratory and/or field contamination of project samples. Method blanks evaluate potential laboratory contamination and field and equipment blanks evaluate potential field contamination.

Blanks are not qualified due to contamination of another blank.

Sample results qualified as non-detected (U) are treated as detected results when qualifying for other excursions.

- For blank results less than the RL, samples with concentrations less than the RL are reported at the RL and qualified as non-detected (U). Samples with concentrations greater than or equal to the RL are not qualified or the Blank Rule Option may be applied.
- 2. For blank results greater than the RL, samples with concentrations less than the RL are reported at the RL and qualified as non-detected (U). Samples with concentrations greater than or equal to the RL and less than the blank contamination level are reported and qualified as non-detected (U). Samples with concentrations greater than or equal to the RL and greater than or equal to the blank contamination level are not qualified or the Blank Rule Option may be applied.
- 3. For blank results equal to the RL, sample concentrations less than the RL are reported at the RL value and qualified as non-detected (U). Samples greater than or equal to the RL are not qualified or the Blank Rule Option may be applied.
- 4. For gross contamination in blanks (interference peaks, poor baselines), all associated sample detected results may be rejected (R) or qualified as non-detected (U), applying professional judgment.

Blank Rule Option - If a target analyte is detected in a blank at a concentration greater than the MDL, for samples with concentrations less than five times the blank concentration, the sample is qualified as non-detected (U) and reported at the RL.

If methylene chloride, acetone, 2-butanone, or phthalates are detected in the sample at a concentration that is less than ten times the concentration in the associated blank, the sample result is qualified as "U". If other target analytes are detected in the sample at a concentration that is less than five times the concentration detected in the associated blank, the sample result is qualified as "U".

#### Evaluation of Isotope Dilution Analytes for PFAS

The isotope dilution technique includes the utilization of IDAs, which are carbon-13 labeled analogs, oxygen-18 labeled analogs or deuterated analogs of target analytes which are added to samples in the sample extraction process. Since IDAs respond in the same manner as target analytes, the IDA is used for target analyte identification and for calculating sample concentrations.

The IDAs are evaluated in sample results using control limits of 25% to 150%. Based on the laboratory SOP, low recoveries of IDAs are acceptable as long as the signal-to-noise (S/N) ratio is greater than 10:1.

Results for target analytes associated with IDAs with recoveries greater than or less than 10% with a S/N of greater than 10:1 are qualified as approximate (J, UJ).

Results for target analytes associated with IDA with recoveries less than 10% with a S/N of less than 10:1 are rejected (R) using professional judgment.

#### Evaluation of Internal Standards for Organics

Internal standard recoveries are evaluated using control limits of from 50% of the lower standard area to 100% of the upper standard area of the associated calibration verification standard.

The results associated with internal standard area recoveries 25% or greater but less than 50% are qualified as approximate (J, UJ).

Non-detected results associated with internal standard area recoveries less than 25% are rejected (R), using professional judgment.

## Target Analyte Identifications

If incorrect target analyte identifications were made due to laboratory errors, the associated result will be corrected or rejected (R), applying professional judgment.

- 1. The laboratory is performing identification and quantitation for sample data using the guidance presented in the USEPA Technical Advisory Laboratory Analysis of Drinking Water Samples for PFOA using EPA Method 537 Rev. 1.1.
- 2. The laboratory analyst's experience and judgement are used to report both target analyte identification and concentrations, which are based on interpretation of peak shape, chromatography baseline, retention time and signal strength. The laboratory did not utilize ion ratios for target analyte identification for this method.
- 3. During data validation, peak integration and identification interpretation performed by the laboratory analysts is reviewed. Validation qualifiers are applied to sample results when overall chromatography, retention times or peak shapes may have impacted sample identifications and/or concentrations.

# Target Analyte Identification and Quantitation for PFAS (TA Sacramento SOP)

- 4. IDAs are utilized to identify target analytes (TAs), where certified quantitation/calibration- quality IDA standards are available for inclusion in the calibration. For target analytes without IDAs, an IDA is assigned for retention time identification and quantitation and are presented in the laboratory SOP.
- 5. Branched-chain isomers may be identified for perfluorooctanoic acid (PFOA), perfluorohexanesulfonic acid (PFHxS) and perfluorooctane sulfonic acid (PFOS). The laboratory only processes and evaluates PFOA, PFOS, and PFHxS for both linear and branched isomers based on current literature. Other target analytes are quantitated based on a single linear isomer peak, defined by the calibration solutions in the initial calibration.
- 6. The laboratory may utilize a qualitative/technical mixture for the PFOA standard to determine retention times only for the PFOA branch-chain isomers. A separate, certified quantitation/calibration-quality standard source, which only includes the linear isomer, may be used for calibration.
- 7. Samples results are qualified as approximate (J) but bias is not assigned to sample results with branch-chain isomers when quantitated using linear calibration standards or standards that do not include the branches present in the samples.

- Target analytes are identified using TA and IDA comparisons of retention time (RT) and relative retention time (RRT - defined as the RT of the target analyte/RT of the IDA), as applicable based on laboratory experience.
- 9. According to the laboratory, the data system's integration parameters are optimized for minimal manual intervention. Laboratory analyst's experience and judgement are used to evaluate TA peaks. Should there be any issue with the data system's integration the analyst will overwrite the data report with a manual integration. This manual integration will take into account TA signal strength, overall chromatography and baseline determinations, which can vary from sample to sample, especially for low level detections.

# Target Analyte Identifications for Organics

If incorrect target analyte identifications were made due to data interpretation or laboratory transcription errors, the associated result will be corrected or rejected (R), applying professional judgment.

Source: OBG



	Laboratory QA/QC analyses definitions
QA/QC Term	Definition
Reporting limit (RL) or Quantitation limit (QL)	The level above which numerical results may be obtained with a specified degree of confidence; the minimum concentration of an analyte in a specific matrix that can be identified and quantified above the MDL and within specified limits of precision and bias during routine analytical operating conditions.
Method detection limit (MDL)	The minimum concentration of an analyte that undergoes preparation similar to the environmental samples and can be reported with a stated level of confidence that the analyte concentration is greater than zero.
Calibration	Compliance requirements for satisfactory instrument calibration are established to verify that the instrument is capable of producing acceptable quantitative data. Initial calibration demonstrates that the instrument is capable of acceptable performance at the beginning of analysis and calibration verifications document satisfactory maintenance and adjustment of the instrument on a day-to-day basis.
Relative standard deviation (RSD)	The standard deviation divided by the mean; a unit-free measure of variability.
Correlation coefficient	A measure of the strength of the relationship between two variables.
Relative percent difference (RPD)	Used to compare two values; the relative percent difference is based on the mean of the two values, and is reported as an absolute value (e.g., always expressed as a positive number or zero).
Percent difference (%D)	Used to compare two values; the %D indicates both the direction and the magnitude of the comparison; the %D may be either negative, positive, or zero.
Percent recovery (%R)	The act of determining whether the methodology measures all the target analytes contained in a sample.
Calibration blank	Consists of acids and reagent water used to prepare samples for analysis. This type of blank is analyzed to evaluate whether contamination is occurring during the preparation and analysis of the sample.
Method blank	A water or soil blank that undergoes the preparation procedures applied to a sample ( <i>i.e.</i> , extraction, digestion, clean-up). These samples are analyzed to examine whether sample preparation, clean-up, and analysis techniques result in sample contamination.
Field/equipment blank	Collected and submitted for laboratory analysis, where appropriate. Field/equipment blanks are handled in the same manner as environmental samples. Field/equipment blanks are analyzed to assess contamination introduced during field sampling procedures.
Internal standards performance	Compounds not found in environmental samples which are spiked into samples and quality control samples at the time of sample preparation for organic analyses. Internal standards must meet retention time and recovery criteria specified in the analytical method. Internal standards are used as the basis for quantitation of the target analytes.
Surrogate recovery	Compounds similar in nature to the target analytes but not expected to be detected in the environmental media which are spiked into environmental samples, blanks, and quality control samples prior to sample preparation for organic analyses. Surrogates are used to evaluate analytical efficiency by measuring recovery.
Laboratory control sample (LCS)	Standard solutions that consist of known concentrations of the target analytes spiked into laboratory analyte-free water or sand. They are prepared or purchased from a certified manufacturer from a source independent from the calibration standards to provide an independent verification of the calibration procedure. They are prepared

Laboratory QA/QC analyses definitions		
QA/QC Term	Definition	
	and analyzed following the same procedures employed for environmental sample analysis to assess method accuracy independently of sample matrix effects.	
Matrix	The material of which the sample is composed or the substrate containing the analyte of interest, such as drinking water, waste water, air, soil/sediment, or biological material.	
Retention time (RT)	The time a target analyte is retained on a chromatography column before elution. The identification of a target analyte is dependent on a target compound's RT falling within the specified retention time window established for that compound.	
Relative retention time (RRT)	The ratio of the retention time of a compound to that of a standard.	
Source: OBG		