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DATA-VALIDATION PROGRAM PLAN
DOT&PF Statewide PFAS
VARIOUS SITES, ALASKA

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ACRONYMS

| | |
|-------|---|
| AAC | Alaska Administrative Code |
| CCV | continuing calibration verification |
| COC | chain-of-custody |
| °C | degrees Celsius |
| CSP | Contaminated Sites Program |
| DEC | Alaska Department of Environmental Conservation |
| DQO | data quality objective |
| DVPP | Data-Validation Program Plan |
| EB | equipment blank |
| EDD | electronic data deliverable |
| EPA | U.S. Environmental Protection Agency |
| FB | field blank |
| GRO | gasoline range organics |
| ICV | initial calibration verification |
| IDA | isotope dilution analyte |
| LCS | laboratory control sample |
| LCSD | laboratory control sample duplicate |
| LOD | limit of detection |
| LOQ | limit of quantitation |
| MB | method blank |
| mm | millimeter |
| MRL | method reporting limit |
| MS | matrix spike |
| MSD | matrix spike duplicate |
| %R | percent recovery |
| PFAS | per- and polyfluoroalkyl substances |
| PQL | practical quantitation limit |
| QAPP | quality assurance program plan |
| QA | quality assurance |
| QC | quality control |
| RPD | relative percent difference |
| SDG | sample delivery group |
| SGS | SGS North America, Inc. |
| SOP | standard operating procedure |
| SRF | sample receipt form |
| TB | trip blank |
| USACE | US Army Corps of Engineers |
| VOA | volatile organic analysis |
| VOC | volatile organic compound |
| WO | work order |

Exhibit 1-1: Definition of Flags

| Flag | Displayed as | Description |
|------|---|--|
| U | < [reporting limit] | The analyte was not detected; the result is listed as less than the reporting limit. |
| UJ | < [reporting limit] J* | The analyte was not detected; the listed reporting limit may not represent the true reporting limit due to sample-handling or laboratory quality-control (QC) failures (i.e., the listed reporting limit may be inaccurate or imprecise). |
| UB | < [LOQ or reported concentration] B* | The analyte is considered not detected due to sample-contamination identified in a blank; the result is listed as less than the limit of quantitation (LOQ) or the concentration originally reported in the sample (higher of the two values). |
| J | [Result] J – Flag applied by laboratory [Result] J* – Flag applied by reviewer | The result is an estimated quantity. The analyte was detected below the LOQ or was affected by QC failures. |
| JL | [Result] JL* | The result is an estimated quantity and may be biased low due to QC failures. |
| JH | [Result] JH* | The result is an estimated quantity and may be biased high due to QC failures. |
| N | [Result] JN* | The analyte was tentatively identified, and the result is an estimated quantity. |
| R | R* | The results are unusable. The sample results are rejected due to severe QC deficiencies. The analyte may or may not be present in the sample. |

NOTES:

* Flag applied by reviewer.

LOQ = limit of quantitation, QC = quality control

1 INTRODUCTION

This Data-Validation Program Plan (DVPP) was prepared to describe Shannon & Wilson’s procedures for reviewing and qualifying analytical data in an objective and consistent manner.

This DVPP describes the process for qualifying analytical data based on quality assurance/quality control (QA/QC) review of Level II laboratory reports and electronic data deliverables (EDDs). This DVPP is intended to provide guidance for conducting a U.S. Environmental Protection Agency (EPA) Stage 2a Validation (EPA 2009). A more critical level of validation is beyond the scope of this DVPP, but the DVPP does present guidance for determining whether additional review should be conducted, based on information received from the laboratory. This DVPP also assesses the quality of the analytical data using PARCCS parameters (precision, accuracy, representativeness, comparability, completeness, and sensitivity).

This DVPP provides information about references used during the data-validation process and presents data qualifiers used to “flag” analytical data. The standard set of flags used to validate analytical data along with their definitions are presented in Exhibit 1-1. Methods for applying data qualifiers are referenced primarily from the following EPA guidance documents:

- EPA National Functional Guidelines for Organic Superfund Methods Data Review, November 2020 (EPA 2020b);
- EPA National Functional Guidelines for Inorganic Superfund Methods Data Review, November 2020 (EPA 2020a); and
- EPA Data Review and Validation Guidelines for Perfluoroalkyl Substances (PFASs) Analyzed Using EPA Method 537, November 2018 (EPA 2018a)

In some cases, the following US Army Corps of Engineers (USACE) guidance document is also referenced to formulate opinions when EPA guidance documents recommend exercising professional judgment:

- USACE Engineering Manual 200-1-10, Guidance for Evaluating Performance-Based Chemical Data, June 2005 (USACE 2005).

Additional references are listed in Section 12.0 and cited throughout the text.

In general, most data review guidelines included in this DVPP are drawn from federal guidance documents. However, in some cases federal guidance is not consistent, is outdated, or does not account for specific issues addressed in this DVPP; in these cases, the guidance presented in the DVPP is based on standard industry practice or site-specific

considerations, which are based on Shannon & Wilson chemists’ years of professional experience and discussions with the Alaska Department of Environmental Conservation (DEC).

Most quality assurance program plans (QAPPs) specify data quality objectives (DQOs) for items such as laboratory control sample (LCS) recovery and target reporting limits. This document does not present such limits, but instead defers to internal laboratory control limits that are statistically derived, frequently updated, and within the requirements of the laboratory’s national certification, and thus compliant with federal requirements. A glossary of terms is included in Appendix A.

2 LABORATORY CERTIFICATION AND DELIVERABLES

2.1 Laboratory Certification

The DEC Contaminated Sites Program (CSP) has an approval process for laboratories conducting analytical testing of various analytes; other DEC programs have their own laboratory certification programs. When using a new laboratory or analytical method, the DEC website is checked to verify that the laboratory analyzing project samples is certified as “approved.” Laboratory certification is not required in cases where DEC does not list an analytical method. The websites do not appear to be updated frequently and laboratories may be certified without being listed on the website. Certifications can be requested from the laboratory.

In cases where the original laboratory subcontracts analysis to a network or referral laboratory, “ref lab”, the referral laboratory shall also be verified for DEC approval, where applicable. This information may be found in the following websites listed in Exhibit 2-1, below:

Exhibit 2-1: Links to DEC-Approved Laboratories

| DEC-Approval Authority | Website |
|--|---|
| Contaminated Sites Program | https://dec.alaska.gov/spar/csp/lab-approval/list-of-approved-labs |
| Drinking Water Program - Chemical Laboratories | https://dec.alaska.gov/eh/lab/chem-lab-cert-status.aspx |
| Drinking Water Program - Microbiological Laboratories | https://dec.alaska.gov/eh/lab/micro-lab-cert-status.aspx |

2.2 Laboratory Deliverables

Laboratory Level II reports and EDDs are obtained directly from the laboratory via e-mail or laboratory data websites. The laboratory reports and EDDs are reviewed for completeness and revised reports are requested where there is missing or incorrect information.

Laboratory reports are provided in Adobe Acrobat (.pdf) format, while EDDs are provided in extensible markup language (.xml) format, or another similar format. It may be necessary to engage with the laboratory regarding a database compatible EDD format.

Laboratory reports and EDDs are grouped by the work order (WO) number assigned when the laboratory receives the sample delivery group (SDG). SDGs are determined by the samples and analyses listed on the chain-of-custody (COC) record.

3 CHAIN-OF-CUSTODY

Evidence of sample custody from the time of collection to the time of receipt by the laboratory is documented on the COC record. A COC contains the signatures of individuals collecting, shipping, and receiving each sample. The COC is reviewed to verify it is signed and dated by the sampler, the local receiving staff (unless shipped directly), and the laboratory's receiving staff. Carriers who are only involved in the transport of sealed coolers (e.g., Lynden Transport, Inc.) are not required to sign the COC. We consider a sample to be in custody if it is:

- in a person's actual possession;
- in view, after being in physical possession;
- sealed so no one can tamper with it, after having been in physical custody; or
- in a secured area, restricted to authorized personnel.

If the COC record is not complete and accurate (e.g., signatures missing, date/time discrepancies, lack of custody seals), professional judgment must be used as to whether to qualify the data. The reviewer should consider rejecting data and recollecting the samples, if possible, if it is suspected that custody was intentionally breached, and the samples may have been tampered with. However, if there is a simple omission or minor discrepancy, the data may be usable without qualification if the source of the omission or discrepancy is known and accounted for.

The COC also provides the requested analyses for each documented sample. COCs are reviewed to verify the correct analyses were requested, and that sample names match those on the sample-collection logs. Where discrepancies are noted, the laboratory will coordinate

with the sampling team to confirm the correct sample names are used in reporting the results.

4 SAMPLE HANDLING, CONDITION, PRESERVATION, AND HOLDING TIMES

Evidence of sample condition is documented on the laboratory's sample receipt form (SRF) upon delivery. SRFs document QC non-conformance issues during sample handling, where such information exists. When samples are delivered to a local sample-receiving office prior to transport to the analytical laboratory, SRFs are completed at each location.

The following sections generally apply to soil and water. For sample-handling requirements for other media besides soil and water samples, data reviewers should reference the individual EPA sampling and analysis methods and/or laboratory sampling guides. In general, data qualification based on sample-handling failures is the same for other media as for soil and water samples; however, the sample-handling requirements may be different and must be assessed on a method-specific basis.

4.1 Acceptable Temperatures

SRFs are reviewed to verify samples were received within the acceptable temperature range. Temperature of the coolers and/or temperature blanks should be documented at each receiving location. Samples are considered to be within the acceptable temperature range if received between 0 degrees Celsius (°C) and 6 °C, where temperature preservation is required. This range is referenced in multiple guidance (e.g., EPA 2020a, 2020b, 2018b), noting that water samples received below this cutoff are acceptable in the absence of ice.

Data qualification based on temperatures outside the acceptable criteria may vary for different analyses and sample matrices. For example:

- PFAS have high chemical and biological stability. Samples with temperature exceedances submitted for PFAS analysis are unlikely to be adversely affected by elevated cooler temperatures. The data reviewer should note the discrepancy on the LDRC; however, we do not consider the PFAS results affected if the temperature is below 0 °C, or 6 °C and 10 °C.
- Some volatile organic compounds (VOCs) have low chemical stability and may be reduced in concentration by elevated cooler temperatures. The data reviewer should note the discrepancy and note that the VOC analysis results may be affected.
- Samples that are collected frozen (<-7 °C) may be maintained frozen until sub-sampled and preserved, if allowed by the project work plan (DEC 2019a).

Exhibit 4-1 provides general guidelines for qualifying results for samples received outside the acceptable temperature range; however, the individual extraction or analytical methods should be consulted, and professional judgment used.

Exhibit 4-1: Sample-Temperature Actions

| Matrix | Criteria | Action | |
|---------------------------------|-----------------------------|-------------------|-------------------------------|
| | | Detected Analytes | Analytes Not Detected |
| Water | 0 °C – 6 °C | | No qualification |
| | 0 °C – 6 °C; ice in samples | J | UJ |
| | < 0 °C; no ice in samples | | No qualification |
| | < 0 °C; ice in samples | J | UJ |
| | > 6 °C | JL | UJ ¹ |
| Soil | 0 °C – 6 °C | | No qualification |
| | < 0 °C | | No qualification ² |
| | > 6 °C | JL | UJ ¹ |
| PFAS Impacted Soil and Water | 0 °C – 10 °C ³ | | No qualification |
| | < 0 °C | | No qualification ² |
| | > 10 °C | N | UN |

NOTES:

- 1 Use professional judgment when qualifying sample results based on temperature exceedance, considering the volatility of the analyte. If temperatures are higher than 10 °C or are suspected to have been above 6 °C for an extended period (e.g., over 24 hours), reviewer should consider rejecting sample results for volatile analytes that were not detected.
- 2 Use professional judgment and refer to method-specific requirements for non-standard analyses and matrices.
- 3 Samples shall be protected from light and refrigerated at ≤ 6°C (but not frozen) from the time sample collection until receipt at the laboratory.

°C = degrees Celsius, PFAS = per- and polyfluoroalkyl substances

4.2 Sample Preservation

Some analyses require additional sample preservatives along with maintaining the samples within the acceptable temperature range. Various guidance documents (EPA 2018b; USACE 2005) and individual EPA extraction methods list sample-preservation requirements for individual methods and matrices. SGS North America, Inc. (SGS) has condensed this information into one concise table including bottle type and volume requirements; this bottle guide table is included in Appendix B. The laboratory SRF documents whether samples were received with proper preservative and within relevant pH limits.

Not all data are affected the same way by failure to properly preserve samples, therefore, individual extraction or analytical methods should be consulted, and professional judgement used. For example:

- If the pH is outside method requirements for inorganic analytes in aqueous samples and the laboratory adjusts the pH immediately upon receipt at the laboratory within the method-specified holding time, allowing time for the sample to equilibrate prior to digestion, the sample results are not affected (EPA 2020a).
- In the case where one analyte is the degradation byproduct of another analyte, the degraded species may increase in a sample following storage with inadequate preservation (USACE 2005); the same may occur if holding times are exceeded (see Section 4.3, below).
- For metals speciation (e.g., Fe²⁺ vs. Fe³⁺), acidification can result in an increase in the reduced form and a decrease in the oxidized form. Professional judgment should be used for qualifying data for any samples with preservation issues.

In most cases where sample preservation is inadequate, sample results should be considered estimated and qualified using the criteria listed in Exhibit 4-2 below.

Exhibit 4-2: Preservation Actions

| Criteria | Action | |
|--|-------------------|-----------------------|
| | Detected Analytes | Analytes Not Detected |
| Adequate Preservation ^{1,2} | No qualification | |
| Inadequate Preservation ^{1,2} | JL | UJ |

NOTES:

- 1 Per regulatory guidance and/or method specific or preservation requirements.
- 2 Use professional judgment and refer to method-specific requirements for non-standard analyses and matrices.

4.3 Holding Times

Samples are required to be extracted and/or analyzed within method-specific holding times. The holding time begins immediately following sample collection and are calculated on a per-day basis, except for short-holding-time analyses where the holding time is measured in hours (typically for analyses listed with a holding time of 72 hours or less). Holding times are included on the bottle guides in Appendix B for standard analyses.

Holding times are evaluated based on the matrix and method. Certain methods list a collection-to-analysis holding time (e.g., analysis of volatile organic compounds in soil, where extraction occurs in the field at the time of collection), while others list separate holding times for collection to extraction and for extraction to analysis.

In general, where holding times are exceeded, sample results shall be qualified using the criteria listed in Exhibit 4-3.

Exhibit 4-3: Holding-Time Actions

| Analysis | Criteria | Action | |
|-------------------------|--|-------------------|-----------------------|
| | | Detected Analytes | Analytes Not Detected |
| PFAS | $t \leq HT$ | No qualification | |
| | $t > HT$ | N | UN |
| | $t > 2x HT$ (gross exceedance) | N | UN |
| All Others ¹ | $t \leq HT$ | No qualification | |
| | $HT < t \leq 2 \times HT$ (marginal exceedance) | JL | UJ |
| | $t > 2x HT$ (gross exceedance) | JL | R |

NOTES:

1 Use professional judgment and refer to method-specific requirements for non-standard analyses and matrices.

HT = method (technical) holding time; t = actual holding time

A sample with a *marginal hold time exceedance* is described as sample that was analyzed outside of the method hold time, but within twice the hold time. A sample with a *gross hold time exceedance* is described as a sample analyzed after more than twice the hold time.

As with sample preservation, professional judgment must be used when qualifying data based on holding-time exceedance, as there can be situations where certain analytes are affected differently than others. For example:

- For analytes that are degradation byproducts of one another, the degraded species may increase if a sample is analyzed outside of the method hold time (USACE 2005).
- PFAS are stable substances and are unlikely to experience degradation within typical laboratory hold time limit exceedances. PFAS samples with marginal or gross hold time exceedances are tentatively identified and flagged with a “N”. PFAS analytical results should not be rejected for hold time failures unless professional judgement deems otherwise.
- Preservation failures coupled with a marginal holding-time exceedance may warrant rejection of results for analytes that were not detected.

4.4 Sample Condition

Sample condition is documented on the laboratory’s SRFs. Professional judgment should be used to determine if qualification of analytical results is necessary for cases where sample condition is compromised. Some common circumstances that may affect sample results are listed below:

1. **Broken Container:** Sometimes 1-L bottle lids crack upon tightening, but no liquid is lost. As long as the lid is replaced prior to sample shipment, often by the laboratory sample-receiving office, results are considered not affected. Most water analyses require at least one duplicate bottle to be filled. If only one of the bottles is broken and the analysis is performed with the intact bottle, no qualification is required other than noting the broken container on the data-review checklist (DEC 2019b). However, if the sample with the broken container was used for analysis, the analytes in question could oxidize, volatilize, degrade, or react, causing the concentration to at least be considered estimated ; professional judgment should be used to determine if the analyses are affected by the addition of air. Affected sample results shall be qualified using the criteria listed in Exhibit 4-4.
2. **Leaking methanol** (soil volatile organic analysis [VOA]): When collecting soil samples for volatile analysis, 25 mL of methanol is added to the sample container to perform the sample extraction and preserve the target analytes in the sample. If the methanol leaks out, it leads to a low bias in the calculated soil mass. The overall concentration of the analyte is determined by dividing the mass of the analyte by the mass of the soil, thus imparting a high bias to the sample result (see calculation below). The results for samples with leaking shall be qualified using the criteria listed in Exhibit 4-4. Professional judgment shall be used to determine if results should be rejected due to severely compromised sample integrity (e.g. complete loss of methanol, etc.)

$$\text{Mass}_{\text{soil}} = \text{Mass}_{\text{total}} - \text{Mass}_{\text{MeOH}} - \text{Mass}_{\text{jar}}$$

$$\text{Concentration}_{\text{analyte}} = \text{Mass}_{\text{analyte}} / \text{Mass}_{\text{soil}}$$

3. **Headspace in VOA vial:** For the analysis of gasoline range organics (GRO) and VOCs in water samples, the absence of headspace is necessary to prevent volatile analytes from partitioning out of the aqueous phase. As noted in the VOC method 5021A, “it is possible for the sample to generate some headspace during storage. This headspace will appear in the form of microbubbles and should not exceed 5-6 millimeters (mm)... Studies conducted by the EPA indicate that [bubbles not exceeding 6 mm in diameter] did not adversely affect volatiles data.” This assessment is applied to the VOC analyses; bubbles larger than 6 mm in diameter are considered an unacceptable level of headspace. When unacceptable headspace is present, results shall be qualified using the criteria listed in Exhibit 4-4.
4. **Soil analysis reported using “wet weight”:** When collecting soil samples, an additional jar is provided for the laboratory to determine the percent solids. In the absence of the additional percent-solids jar, the laboratory may report soil concentrations using the “wet weight.” The overall concentration of the analyte is determined by dividing the mass of the analyte by the mass of the soil. In cases where a dry weight was not determined, the concentration may be reported using a wet weight. The results for samples reported using the wet weight shall be qualified using the criteria listed in Exhibit 4-4.

Other sample-condition anomalies than those listed above may occur. These anomalies should be addressed using available guidance, individual extraction or analytical methods, and the reviewer's professional judgement.

Exhibit 4-4: Sample Condition Actions

| Criteria | Action | |
|--------------------------------------|-------------------|-------------------------------|
| | Detected Analytes | Analytes Not Detected |
| Broken Container | JL | UJ ¹ |
| Leaking Methanol (soil VOA) | JH ² | No qualification ³ |
| Headspace in VOA Vial ≤ 6 mm | JL | UJ |
| Headspace in VOA Vial > 6 mm | JL | R |
| Soil Analysis Reporting "Wet Weight" | JL | UJ |

NOTES:

- 1 Use professional judgement and consider rejecting data depending on how much sample leaked or the volatility of the analyte.
 - 2 Use professional judgement and consider rejecting data if the sample integrity has been severely compromised (e.g. complete loss of methanol, etc.)
 - 3 Not detected analytes are not considered affected if there is sufficient methanol to run the analysis.
- mm = millimeter; VOA = volatile organic analysis

4.5 Sample Processing

Many laboratory methods require additional sample processing at the laboratory prior to analysis. Preparatory batches are groups of analytical project samples and QC samples that processed together at the laboratory, including required steps such as extraction or digestion. Laboratory methods for GRO, DRO, and VOCs require additional preparation to extract a subsample or add surrogate analytes to the samples. The laboratory reports a unique preparation batch ID and extraction date and time. Any QC failures are often applied with the batch group.

The analytical batch is a set of prepared samples (e.g., extracts for GRO or DRO), or samples not requiring preparation (e.g., PFAS or metals analysis) that are analyzed on the instrument together, without interruption. Samples within a preparatory batch may be split into multiple analytical batches.

5 ANALYTICAL SENSITIVITY

Analytical sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected or quantified (USACE 2005). Analytical sensitivity is

evaluated by comparing the appropriate laboratory reporting limit for not-detected results to the relevant cleanup level or action limit, where such standards exist.

In general, regulatory limits used to check analytical sensitivity are listed in Chapter 75 of Title 18 of the Alaska Administrative Code (18 AAC 75) for soil and water; analytes without regulatory limits are compared to the relevant, project-specific or analyte-specific action limit at the time of comparison.

In cases where the reporting limit exceeds the regulatory limit, a note will be added to the DEC data-review checklist (DEC 2019) and associated results tables noting the reporting limit is elevated. Reporting limits that exceed regulation limits should be identified using the following criteria listed in Exhibit 5-1.

Exhibit 5-1: Elevated Reporting Limit Actions

| Criteria | Action |
|---|--|
| Reporting Limit ¹ ≤ Cleanup Level / Action Level | No note |
| Reporting Limit ¹ > Cleanup Level / Action Level | Note should be added to the Checklist and Results Tables |

NOTES:

1 The reporting limit used for the analytical sensitivity comparison should be described in the DEC data-review checklist.

5.1 Reporting Limit Terminology

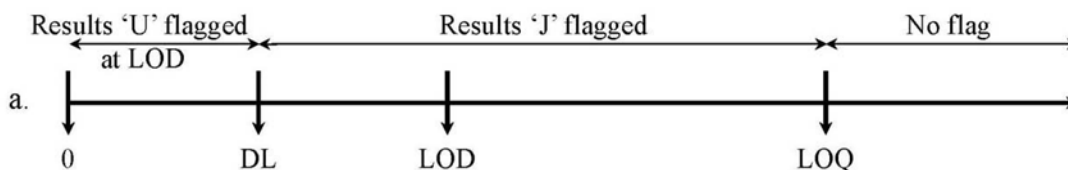
SGS typically uses reporting limits described in the Department of Defense (DoD)/ Department of Energy (DOE) Quality Systems Manual (QSM) for Environmental Laboratories Version 5.3. and reports a detection limit (DL), limit of detection (LOD), and limit of quantification (LOQ) for each analyte. These definitions are summarized below.

- **Detection limit (DL):** “the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type I error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.” Analytes not detected above the DL are reported at the LOD value.
- **Limit of detection (LOD):** “the smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.” SGS establishes the LOD as half the LOQ.
- **Limit of quantification (LOQ):** “the smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and

within the calibration range.” Results reported between the DL and LOQ are considered estimated and flagged with a ‘J’ by the laboratory.

Exhibit 5-2 illustrates the relationship between the DL, LOD, and LOQ.

Exhibit 5-2 Summary of DOD QSM Reporting Limits



Eurofins TestAmerica reporting limits are summarized below:

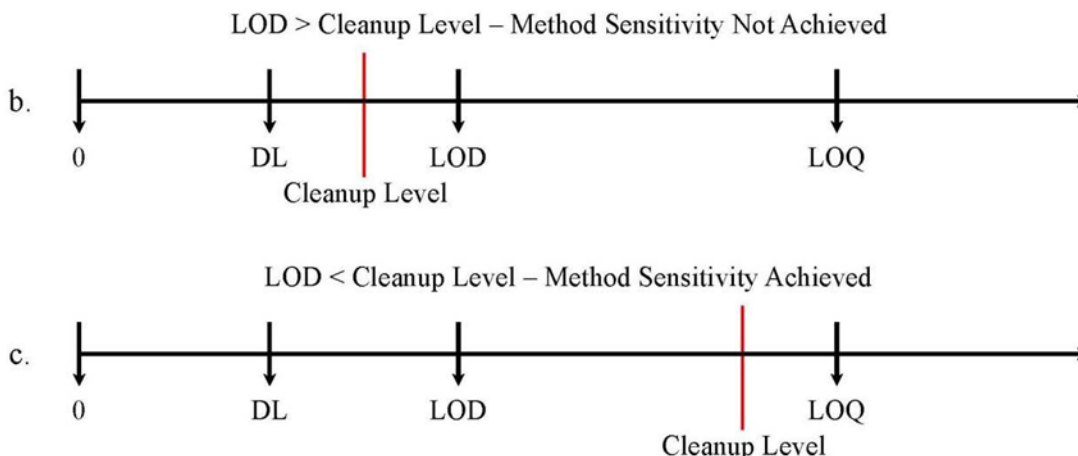
- Method detection limit (MDL): the lowest concentration of an analyte that is distinguishable from the method blank with 99% confidence. (40 CFR Part 136 Appendix B). The MDL is equivalent to the DL as defined by DoD/DOE QSM.
- Reporting limit (RL): the lowest concentration of an analyte that can be detected with known and recorded precision and bias. This value is equivalent to the LOQ as defined by DoD/DOE QSM.

Some laboratory deliverables do not report LOQs, and may report analytical sensitivity limits using the LOQ, practical quantitation limit (PQL), or method reporting limit (MRL). It is important to note the LOQ, PQL and MRL are interchangeable terms and depends on the laboratory for which term is used in reporting the results. For the purposes of this DVPP, the LOQ is referenced.

Shannon & Wilson typically requests inclusion of laboratory qualifiers for detected results reported below the LOQ to denote that the low-level results may be imprecise. Laboratory-added flags are replaced where Shannon & Wilson applies flags to denote directional bias or inaccuracy identified during the data review process.

Exhibit 5-3 provides a summary of laboratory result flags applied to each range and an example of acceptable and unacceptable (elevated) reporting limits.

Exhibit 5-3: Relationship between DL, LOD, LOQ, and Corresponding Laboratory Result Flags and Cleanup Levels.



NOTES:

- b. Unacceptable LOD-to-cleanup-level relationship.
- c. Acceptable LOD-to-cleanup-level relationship.

Note that these are example scenarios; not all data are compared using the LOD, and therefore this figure does not apply to data received from all laboratories.

DL = detection limit; LOD = limit of detection; LOQ = limit of quantitation.

6 BLANK SAMPLES

Blank samples are analyzed to check for possible contributions to the analytical results from cross-contamination between samples, or from sample-contamination from an outside source. Typically, the following blank samples are reviewed in conjunction with project samples, where appropriate:

- method blanks;
- trip blanks (volatile analytes only);
- field blanks; and
- equipment blanks.

Each of these blanks check for sample-contamination issues at various steps between sample collection and analysis. Detections in one blank can cause related detections in other blank samples. For example, a detection in a method blank can cause detections in corresponding trip blanks or equipment blanks. Therefore, it is important to investigate blank detections to determine at what step sample-contamination was first introduced; data-qualification should proceed beginning at this level.

For the purposes of this DVPP (Level II data review), blank detection evaluation should proceed using the following hierarchy:

1. method blank;
2. trip blank;
3. field blank; and
4. equipment blank

Additional details regarding these types of blanks are provided in sections 6.1 through 6.4 below. Additional blanks collected or analyzed by the lab for method-specific requirements should be evaluated on a case-by-case basis.

Data-qualification procedures are identical between blank types within a given matrix; however, the list of affected project samples vary. Exhibit 6-1 presents data-qualification criteria for samples affected by detections in a blank sample; these criteria are generally consistent with those presented in EM 200-1-10 (USACE 2005).

Exhibit 6-1: Actions for Blank Detections

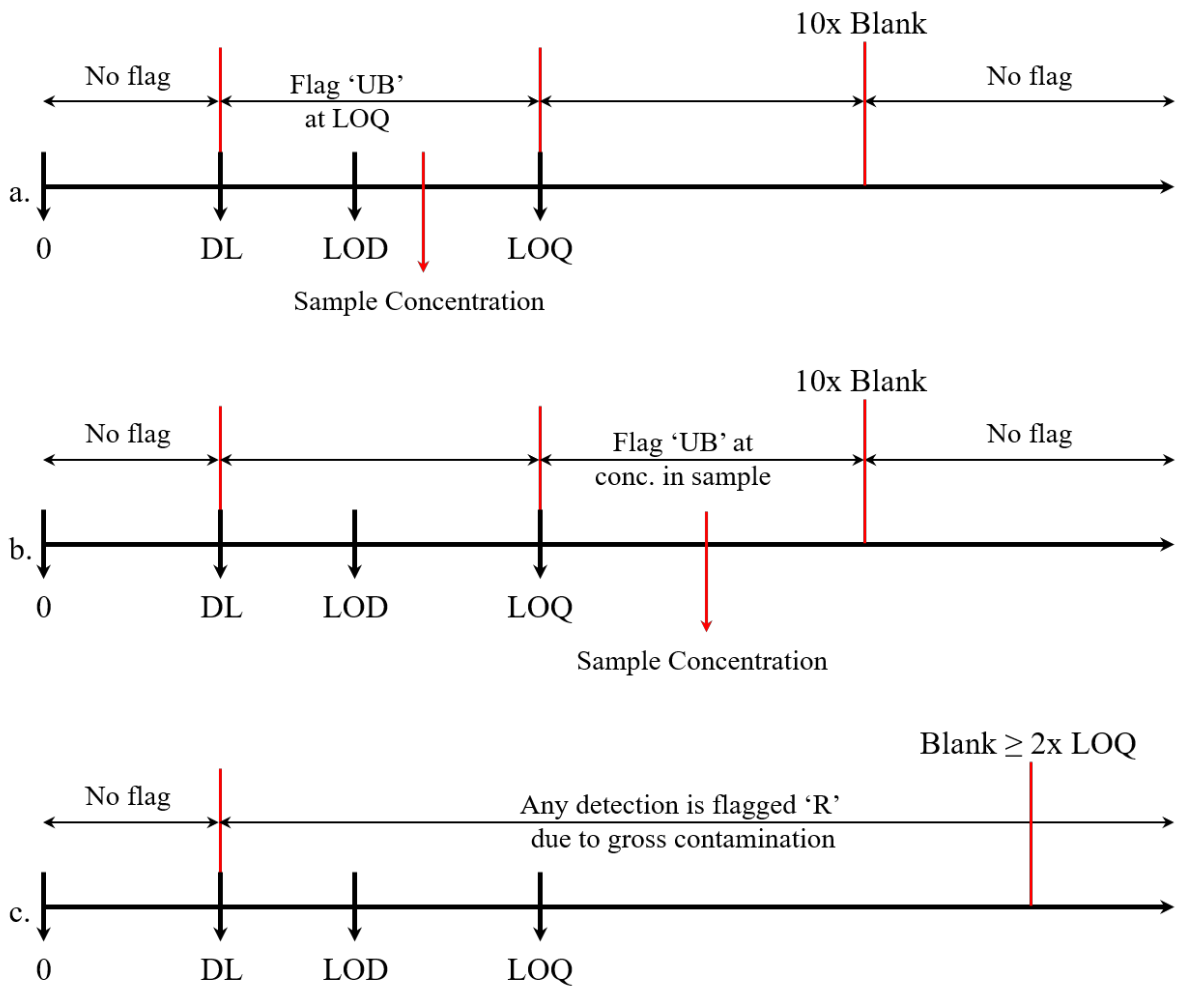
| Analysis | Concentration in corresponding project sample | Action |
|----------------------------|---|----------------------------------|
| Marginal Exceedance | | |
| | DL < blank < 2x LOQ | |
| PFAS | sample is ND | No qualification |
| | sample < LOQ < 10x blank | UB at the LOQ |
| | LOQ ≤ sample < 10x blank | UB at the detected result |
| | sample ≥ 10x blank | No qualification |
| All Others ¹ | sample is ND | No qualification |
| | sample < LOQ < 5x blank | UB at the LOQ |
| | LOQ ≤ sample < 5x blank | UB at the detected result |
| | 5x blank ≤ sample < 10x blank | JH |
| | sample ≥ 10x blank | No qualification |
| Gross Exceedance | | |
| | blank ≥ 2x LOQ² | |
| All Analytes | sample is ND | No qualification |
| | sample is detected | R |

NOTES:

- 1 Use professional judgment and refer to method-specific requirements for non-standard analyses and matrices.
- 2 Use professional judgment to assess the reported LOQ. If elevated, reference a typical LOQ for a non-detect result.
DL = detection limit, LOQ = limit of quantitation (also known as PQL or MRL), y = concentration in blank, z = concentration in corresponding sample

Exhibits 6-2 and 6-3 presents a visual example of flagging criteria for a blank detection for PFAS and all other analyses, respectively.

Exhibit 6-2: Example- Qualification Criteria for PFAS Blank Detections



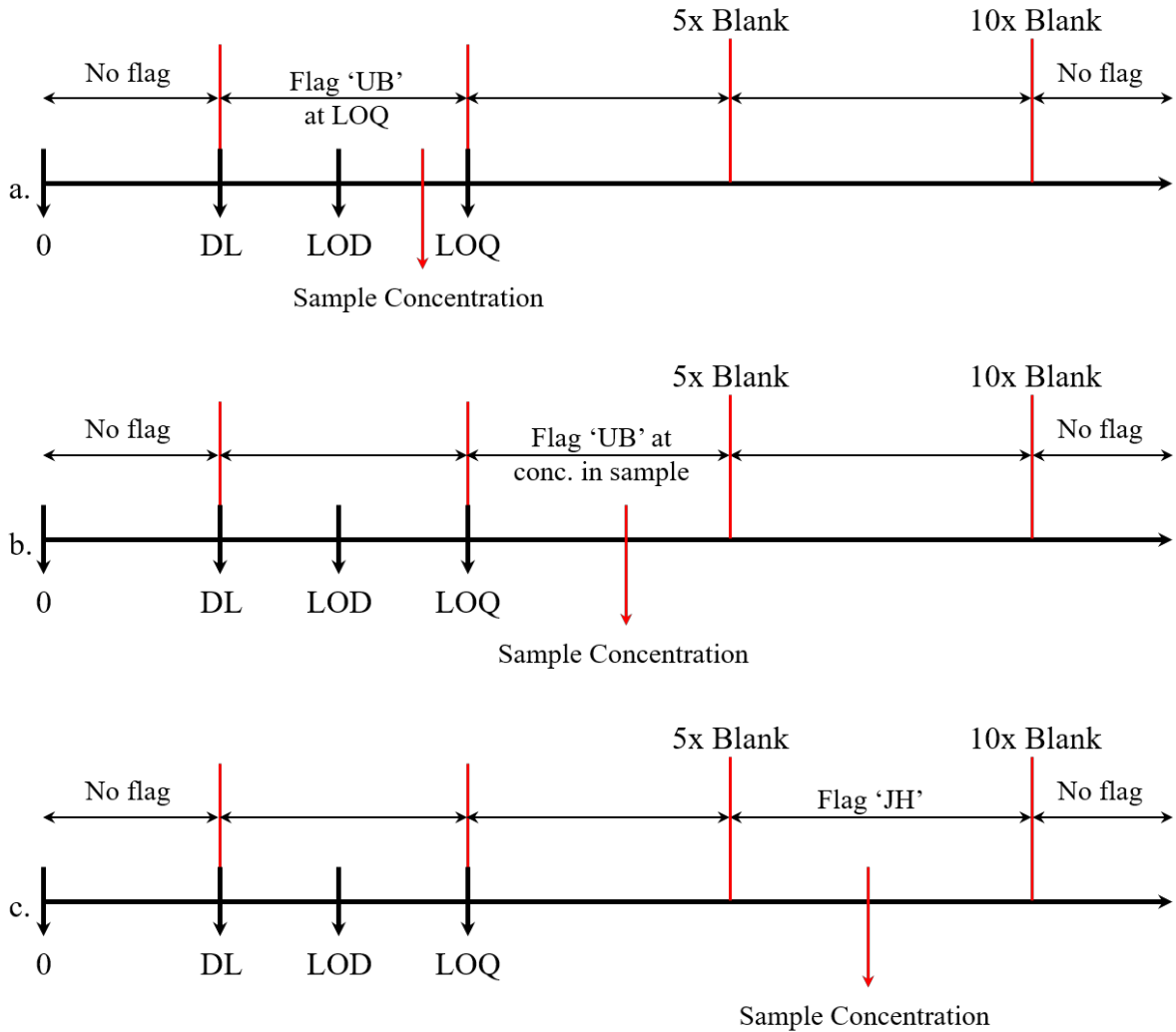
NOTES:

Project-sample results would be qualified as follows:

- a) Flag 'UB' at the LOQ.
- b) Flag 'UB' at the concentration detected in the sample.
- c) Flag 'R' for any detection in the sample.

DL = detection limit; LOD = limit of detection; LOQ = limit of quantitation (also known as PQL or MRL).

Exhibit 6-3: Example Qualification Criteria for Non-PFAS Blank Detections



NOTES:

Project-sample results would be qualified as follows:

- a) Flag 'UB' at the LOQ.
- b) Flag 'UB' at the concentration detected in the sample.
- c) Flag 'JH' at the concentration detected in the sample.

DL = detection limit; LOD = limit of detection; LOQ = limit of quantitation (also known as PQL or MRL).

6.1 Method Blanks

Method blank (MB) samples are prepared by the laboratory with every preparatory batch, at a minimum rate of one MB per 20 samples. MBs are samples of clean media (soil, water, etc.) that are subjected to the same procedures as project samples to extract a given analyte(s). MBs are evaluated to determine if the method of extraction, cleanup, or analysis introduces any contamination during the process.

The reviewer will check that MBs were prepared and analyzed by the laboratory at the required frequency, and that no analytes were reported in the MBs. If an analyte is reported in an MB, all samples in the corresponding preparatory batch should be evaluated for that analyte. Data qualifiers should be applied according to Exhibit 6-1, above.

6.2 Trip Blanks

Trip blank (TB) samples are prepared by the laboratory and one TB should always accompany each cooler containing samples for volatile analysis and stay with the samples. A TB is not required for semi-volatile or non-volatile analytes. TBs serve to check for cross-contamination or contamination from an outside source during sample collection, storage, transportation, and processing by the laboratory.

The reviewer will check that TBs were prepared, transported, and analyzed with any samples analyzed for volatile analyses (i.e., VOCs and GRO), and that no analytes were reported in the TB. A minimum of one TB per cooler is required; the cooler containing the TB and samples for VOC analysis should be clearly identified on the COC. If an analyte is reported in a TB, all samples in the corresponding cooler should be evaluated for the detected analyte and, if necessary, qualified based on the criteria presented in Exhibit 6-1, above. If the sampler did not document which cooler contained the TB, and there is more than one cooler containing samples for VOC analysis, all VOC samples in the work order should be considered potentially affected.

6.3 Field Blanks

Field blank (FB) samples are collected in the field by sample personnel. The sampler opens a sample bottle in the same air space as the corresponding project sample and collects the field blank by filling the bottle with laboratory provided deionized water. The FB is used to assess for possible contamination from the sampling site. If an analyte is reported in the FB, the corresponding sample should be evaluated for the detected analytes and, if necessary, qualified based on the criteria presented in Exhibit 6-1, above.

6.4 Equipment Blanks

Equipment blank (EB) samples are collected in the field by the sampling personnel. The EB is used to determine if decontamination of reusable sampling equipment between sampling locations is sufficient. The reviewer will check that EBs were collected at the required frequency, and that no analytes were reported in the EBs. If an analyte is reported in an EB, all samples collected using the same sampling equipment on the same day will be evaluated (determined based on field sampling logs, and if necessary, qualify based on the criteria presented in Exhibit 6-1, above).

7 ACCURACY

Accuracy is evaluated at multiple levels throughout the analytical process, using a variety of techniques. It is assessed at the preparatory batch level using recovery information from LCS and laboratory control sample duplicates (LCSDs), matrix spike samples (MSs) and matrix spike duplicates (MSDs), and surrogates or isotope dilution analytes (IDAs). MS/MSD and surrogate or IDA recovery information are used to determine whether there is interference from the sample matrix that affects the accuracy of the reported results. The following sections discuss these QC samples in association with the preparatory batch. However, note that there are some analytical methods for inorganics that do not require a preparatory batch and the LCS, LCSD, MS, and MSD QC sample are assessed at the analytical-batch level. Accuracy is also assessed at the analytical-batch level using recovery information from initial calibration verification (ICV) and continuing calibration verification (CCV) samples, where information is available in the Level II data deliverable.

7.1 Laboratory Control Samples

LCSs (also referred to as blank spikes) are prepared by the laboratory with every preparatory batch, at a minimum of one LCS per 20 samples, where required. In some cases, analytical protocol requires the laboratory also analyze an LCSD to assess laboratory precision (see Section 8.1 for assessment of laboratory precision). LCSs and LCSDs are prepared using the same extraction method that is applied to the project samples using laboratory-grade, blank-matrix samples spiked with a known concentration of analyte(s). The laboratory reports a percent recovery (%R) of the spiked amount for each analyte added to the blank sample. The laboratory maintains acceptance limits for LCS/LCSD recovery; these limits are reported in the Level II laboratory report for comparison.

The reviewer will check that LCSs were reported at the required frequency, and that LCS/LCSD recoveries are within laboratory control limits. An LCS or LCSD recovery failure affects all corresponding samples in the same preparatory batch for the affected analyte(s).

The following guidelines in Exhibit 7-1 will be used for qualifying sample results associated with LCS/LCSD-recovery failures.

Exhibit 7-1: Actions for LCS/LCSD and MS/MSD Recovery Failures

| Analysis | LCS/LCSD or MS/MSD Results | Action | |
|-------------------------|----------------------------------|-------------------|-----------------------|
| | | Detected Analytes | Analytes Not Detected |
| PFAS | %R < 10% | JL | R |
| | 10% ≤ %R < LCL | JL | UJ |
| | %R > UCL ² | JH | No qualification |
| All Others ¹ | %R < Control Limits ² | JL | UJ |
| | %R within Control Limits | No qualification | |
| | %R > Control Limits ² | JH | No qualification |

NOTES:

- 1 Use professional judgment and refer to method-specific requirements for non-standard analyses and matrices.
- 2 If LCS/LCSD recovery is grossly outside control limits (recoveries less than 10% or greater than 250%) the reviewer should use professional judgment when qualifying the data. The reviewer should consider rejecting results for analytes not detected where the recovery was below 10% (USACE 2005).

LCL = lower control limit, %R = percent recovery, UCL = upper control limit

7.2 Matrix Spike Samples

For certain methods, the laboratory analyzes an MS/MSD in addition to the LCS. MS/MSDs are prepared and analyzed on a preparatory batch basis and are analyzed with every 20 samples when used. They consist of project (native) samples spiked with a known concentration of analyte(s) and prepared using the same method that is applied to project samples to extract the analyte(s). The MS and MSD are used to determine the presence of matrix interferences and evaluate the analytical accuracy for a given method and matrix, expressed as a %R of the spiked amount added to the field sample.

The reviewer will check to make sure that MS/MSDs were analyzed at the frequency required by analytical methods or project-specific requirements. Some methods may require the analysis of an MS/MSD pair, but insufficient sample volume may prevent the laboratory from providing these QC samples. The laboratory’s standard operating procedures (SOPs) may allow for an LCSD instead of an MS/MSD for these cases.

The reviewer will check that %R for each analyte is within laboratory control limits. If there is a recovery failure, only the field sample utilized for the MS/MSD (the parent sample) is typically considered affected; however, the reviewer should use professional judgment whether other samples in the same preparatory batch have sufficiently similar matrices to be considered affected as well. For example, if an MS/MSD recovery failure is reported for one of two field duplicate samples, it should be assumed there were similar matrix effects in the duplicate, and corresponding results should also be qualified.

Before MS/MSD recovery is evaluated, two important factors must be considered:

1. Verify that the field sample chosen for the MS/MSD is part of the project-sample set currently being reviewed. The laboratory may run samples from other projects in the same preparatory batch and it is possible that the original sample selected for the MS/MSD may not be from the work order reviewed. In this case, it cannot be confirmed that the parent sample matrix is similar to the matrix in the project samples and the recovery failures do not affect data quality for the project-sample set.
2. Verify that the spiking concentration is high relative to the native concentration of the analyte. In accordance with EM 200-1-10 (USACE 2005):

If the native concentration of a target analyte is high relative to the spiking concentration, then this may contribute a significant uncertainty to the recovery calculations; the MS recovery may not be representative of actual method performance for the matrix. In the absence of other guidance, evaluate the MS recovery when the spiking concentration is at least two times greater than the native analyte concentration (USACE 2005).

If the above criteria are met, then results associated with the failures in the original project sample should be qualified using the criteria listed in Exhibit 7-1.

For metals analysis where MS/MSD recovery failures occur, different criteria are used. For metals analysis using most analytical methods, if a matrix spike recovery failure occurs and the sample concentration is greater than the spike concentration, the laboratory is required to conduct a post-digestion spike. A post-digestion spike is where the original sample is spiked at twice the native concentration so that recovery can be evaluated. In this case, refer to the data-qualification criteria in the spiked sample analysis section in the National Functional Guidelines for Inorganic Methods Data Review (EPA 2017a) under the relevant analytical technique.

7.3 Surrogates and Isotope Dilution Analytes

Surrogates are organic compounds that are similar to the analytes being evaluated by a given method (often a deuterated version of the one of the analytes). They are used to identify matrix interferences and inefficiencies in sample extraction for organic analyses. The surrogates are introduced into a field- or laboratory-QC sample prior to sample preparation and analysis. Accuracy is expressed as a %R of the spiked amount added to the sample.

Some methods require analysis using an isotope-dilution method, which uses IDAs instead of a surrogate, and corrects raw data of the associated analyte concentration based on the recovery of the IDA.

The reviewer will check that surrogates and/or IDAs were analyzed for each sample for each organic analysis (including laboratory QC samples), and that recoveries were reported within laboratory-control limits. If there is a reported recovery failure, it is considered to affect only the analytes associated with the surrogate/IDA (see Appendix C for a surrogate/IDA association list) for the corresponding project with the reported failure. However, there are a few special considerations when qualifying data based on surrogate-recovery failures:

1. Matrix interference: Recovery failures due to matrix interference (coelution of an interfering analyte or other matrix interactions) are considered to affect data quality, and results should be qualified as described in Exhibit 7-2. The laboratory typically documents in the case narrative whether a surrogate/IDA recovery failure was due to matrix interference.
2. Dilution: Recovery failures may be observed due to dilution of the surrogates and are not considered to affect the data (USACE 2005). The laboratory typically documents surrogate failures due to dilution in the case narrative. Refer to number 4 for IDA recovery failure assessments.
3. Surrogate/IDA recovery failures in laboratory QC samples: Surrogate/IDA failures in an LCS, LCSD, MS, or MSD are not considered to affect the project sample data as long as the recovery of individual analytes associated with that surrogate/IDA are within the laboratory control limits for the LCS/LCSD/MS/MSD sample. However, gross or systematic surrogate/IDA recovery failures should be considered along with all other QC information for the preparatory batch and the results evaluated according to professional judgment.
4. IDA recovery in project samples: As part of the analytical procedure for isotope-dilution methods, a given analyte concentration is corrected based on the recovery of the associated IDA. Therefore, recovery inefficiencies are somewhat self-correcting, and one would expect less inaccuracy due to slight matrix effects. However, recovery outside the recovery limits may indicate there are significant matrix effects that the method is unable to adequately correct for. Results should be qualified as described in Exhibit 7-2.

Excluding the exceptions listed above, data affected by surrogate/IDA recovery failures should be qualified using the following criteria listed in Exhibit 7-2.

Exhibit 7-2: Actions for Surrogate or Isotope Dilution Analyte Recovery Failures

| Type | Criteria | Action | |
|-----------|---------------------------|----------------------------|-----------------------|
| | | Detected Analytes | Analytes Not Detected |
| IDA | %R < 10% | J | R |
| | 10% ≤ %R < LCL | J | UJ |
| | %R < LCL (diluted sample) | Use professional judgement | N/A ¹ |
| | %R > UCL | J | No qualification |
| | %R within range | No qualification | |
| Surrogate | %R < range | JL ² | UJ ³ |
| | %R within range | No qualification | |
| | %R > range | JH ² | No qualification |

NOTES:

- 1 Non-detects should be reported from the undiluted analysis.
- 2 Use professional judgment when the bias is poorly defined. Only impart a bias to the qualified data if the bias is well defined (i.e., if there is more than one surrogate in the analysis, where recovery failures are in the same direction). Otherwise, it may be more conservative to simply qualify the results as estimated ('J'; USACE 2005).
- 3 Use professional judgment when evaluating gross recovery failures. The reviewer should consider rejecting the results where analytes are not detected if the associated surrogate recovery is below 20% (USACE 2005).

LCL = lower control limit, %R = percent recovery, UCL = upper control limit

7.4 Calibration Verification Samples

Calibration verification samples are not typically reported in the Level II data reports provided by the laboratory (aside from appearing in the EDD), and review of such samples is outside the scope of this DVPP. The laboratory may have requirements to re-calibrate the instrument if calibration verification fails or other corrective action. However, this is not always possible, and occasionally calibration verification failures occur and are reported in the case narrative of the Level II laboratory report. Calibration verification samples are described briefly below.

ICV samples are clean extraction solvent spiked with a known analyte concentration, using a different source than that of the primary calibration standards, and analyzed immediately following instrument calibration. Similarly, CCV samples are calibration standards that are analyzed at the beginning of each analytical batch and periodically throughout the run.

The laboratory evaluates ICV and CCV recovery information based on their internal acceptance criteria; in some cases, they also evaluate relative percent difference between CCVs to determine if drift is occurring. As stated above, calibration-level data review is beyond the scope of this DVPP and may be conducted as part of a Level IV data-validation, if calibration issues are identified in the case narrative. Professional judgment should dictate whether any samples in an analytical batch with unresolved CCV failures should be

considered preliminary pending further investigation. For these circumstances, contact the laboratory for more direction and ask the Senior Laboratory Analyst to provide justification for using the data and any bias resulting from these QC failures. Request that the laboratory report be revised to include the justification.

8 PRECISION

Precision refers to the repeatability of measurements (USACE 2005). Precision is evaluated using laboratory QA/QC and field-duplicate samples. The following sections describe the duplicate-sample information that is commonly used to assess precision. However, this is not an exhaustive list, and the laboratory may occasionally analyze other duplicate samples that should also be considered. For most analyses, at least one laboratory QC-sample duplicate must be analyzed; this can include a LCSD, MSD, or a laboratory duplicate.

Each type of duplicate is evaluated in the same manner (LCS/LCSD, MS/MSD, laboratory duplicate and field duplicates). A relative percent difference (RPD) is calculated between the duplicate results for a given analyte using the following equation presented in Exhibit 8-1.

Exhibit 8-1: RPD Calculation

| Equation | Variable and Definition | |
|--|-------------------------|-----------------------------|
| $RPD = \frac{ R_1 - R_2 }{(R_1 + R_2)/2} \times 100\%$ | RPD | Relative Percent Difference |
| | R1 | Primary Result |
| | R2 | Duplicate Result |

The resulting RPD is compared to laboratory control limits (for laboratory QC samples), or project or regulatory DQOs for field duplicates. For purposes of this DVPP, the DEC-recommended water-sample DQO of 30% and soil-sample DQO of 50% are used to assess precision of field-duplicate samples.

The guidelines presented in Exhibit 8-2 will be used for qualifying sample results associated with duplicate-sample RPD failures. The treatment of a failure is the same across types of duplicate samples, but the samples that are affected vary. Refer to the following sections for details.

Exhibit 8-2: Actions for Duplicate-Sample RPD Failures

| Criteria | Action | |
|----------------------------|-------------------|-----------------------|
| | Detected Analytes | Analytes Not Detected |
| RPD ≤ Control Limit or DQO | No qualification | |
| RPD > Control Limit or DQO | J | UJ |

DQO = data quality objective, RPD = relative percent difference

8.1 Laboratory Control Sample Duplicates

Precision can be evaluated between LCS and LCSD results for a given analyte. The laboratory calculates the RPD using the equation presented in Exhibit 8-1 for each analyte. The reviewer will check that each RPD is within the laboratory control limits. RPD failures for specific analytes in the LCS/LCSD are considered to affect the precision of that analyte in each corresponding project sample in the same preparatory batch. Affected results should be flagged according to the criteria presented in Exhibit 8-2.

8.2 Matrix Spike Duplicates

Precision can be evaluated between the MS and the MSD results for a given analyte. The laboratory calculates the RPD for each analyte. The reviewer will check that each RPD is within the laboratory control limits. RPD failures for specific analytes in the MS/MSD are considered to affect the precision of that analyte in the parent sample spiked for the MS/MSD. Professional judgment should be used to determine whether additional samples should be qualified (based on similarity of sample matrix).

RPD failures should be considered to affect the data regardless of the concentration spiked, as long as the laboratory calculates the RPD based on the total analyte concentration quantified in the MS/MSD. If the laboratory calculates the RPD based only on what was recovered of the spike, it should be treated as for MS/MSD recovery, with failures only considered to affect data quality if the spiking concentration is at least double the native concentration of the analyte. Affected results should be flagged according to the criteria presented in Exhibit 8-2.

8.3 Laboratory Duplicates

For select analyses, or when insufficient volume is submitted for analysis of an MS and MSD, the laboratory may analyze a project sample twice (referred to as a laboratory duplicate). The laboratory calculates an RPD between the original result and the duplicate-sample result for each analyte. The reviewer will check that each RPD is within the laboratory control limits. As with MS/MSDs, laboratory duplicate RPD failures are considered to affect the precision of the affected analyte only in the parent sample used for the duplicate analysis. Affected results should be flagged according to the criteria presented in Exhibit 8-2.

8.4 Field-Duplicate Samples

Field-duplicate samples are duplicate samples collected from the same location and submitted to the laboratory performing the requested analysis. The duplicate sample will

have a “dummy” sample number and submitted to the laboratory as a regular sample (i.e., the duplicate is submitted “blind”). These field duplicates are used to determine the reproducibility of the sampling technique, as well as the subsequent laboratory analysis. Sample homogeneity is necessary to obtain acceptable values for the RPD and any heterogeneity should be noted during sampling.

For field-duplicate pairs, the reviewer will calculate an RPD using the equation presented in Exhibit 8-1. An RPD will only be calculated if both sample results are detected above the detection limit. The calculated RPD will be compared to the standard DQOs of 30% for water or 50% for soil. Field-duplicate RPD failures are considered to affect only the results of the duplicate pair; affected data will be qualified based on the criteria in Exhibit 8-2.

In the event that one of the results is above the LOQ but the other result is below the detection limit (not detected) and J-flag detections are reported for the project, the reviewer should use professional judgment and consider qualifying the detected and non-detect result as estimated even though an RPD cannot be calculated. This may be evidence of samples having been mislabeled (in the field or the laboratory), sample heterogeneity, or some other issue; further investigation may be warranted.

9 REPRESENTATIVENESS

Representativeness is defined in Chapter One of the EPA SW-846 Update V Revision 2 (EPA 2014) as the degree to which data accurately and precisely represents a characteristic of a population for a sampling point. Representativeness is dependent on proper execution of the approved sampling program, which is agreed upon by the DEC, DOT&PF, and Shannon & Wilson. To assess sample representativeness, sample-log sheets will be reviewed to ensure the samples were collected according to the approved sampling program and the results therefore represent the location and depth sampled. In addition, where possible, the analytical result for each sample will be compared to the historical results to check that the result is consistent with the broader data set for that location.

There are instances where sample collection procedures deviate from the sampling program and may affect the sample representativeness. Professional judgement is used to assess the data usability based on these deviations. Some of these infrequent instances are presented in Exhibit 9-1 along with qualifications to the data.

Exhibit 9-1: Actions for Deviations from Sampling Program

| Sampling Type | Description of Deviation | Action | |
|--|--|-------------------|-----------------------|
| | | Detected Analytes | Analytes Not Detected |
| Monitoring Well/ Residential Sampling | Purging/stabilization criteria not met | J | UJ |
| Residential Sampling – Organic Analyses | Sample collected post treatment (especially for collection post carbon filter) | JL | UJ ¹ |
| Residential Sampling – Inorganic Analyses | Sample collected post treatment (especially iron analyses collected post sediment filter) | JL | UJ |

NOTES:

- 1 Use professional judgment. The reviewer should consider rejecting the results where organic analytes are not detected and samples were collected post carbon filter. At minimum, the non-detect results should be considered estimated and flagged 'UJ' to identify the sample collection discrepancy.

10 LABORATORY APPLIED FLAGS

The laboratory is required to qualify data that does not meet laboratory QC standards. The data qualifiers, flagging criteria, and flagging procedures are detailed in the laboratory’s SOPs. The lab does not interpret the impact of an applied flag on the data, rather the flags are meant to draw the attention of the reviewer to an area where laboratory QC criteria is not met. When data is reviewed and validated, the information the laboratory reported is taken and evaluated to determine the effect of the QC deficiency on the data and apply appropriate flags as defined in this document.

In some cases, laboratory applied flags are not needed and may be removed for reporting. For example:

When an MS and/or MSD sample has a %R failure, but the spiking concentration is not high relative to the native parent sample concentration, then the %R failure is not applicable. The flag the lab applies to the data is therefore not necessary and is removed the analytical reporting table.

In some cases, laboratory applied flags are overwritten by flags applied by Shannon & Wilson. For example:

When a sample result exceeds the calibration range, the lab may flag the affected data with an ‘E’. Calibration exceedances are flagged with a ‘J’ in the analytical reporting table overwriting the ‘E’ flag.

In either case listed above, laboratory applied flags are maintained in the laboratory report for reference.

See Exhibit 10-1 for common laboratory applied flags that are either overwritten by a S&W applied flag or are removed from the analytical reporting tables because they are deemed unnecessary after the data-validation process. The flags remain in the laboratory report for reference.

Exhibit 10-1: Actions for Common Laboratory Applied Flags

| Laboratory Applied Flag ¹ | Flag Description | Shannon & Wilson Applied Flag |
|--------------------------------------|--|---------------------------------------|
| I | Value is the estimated maximum possible concentration. Case Narrative flag description: The "I" qualifier means the transition mass ratio for the indicated analyte was outside of the established ratio limits. The qualitative identification of the analyte has some degree of uncertainty. However, analyst judgement was used to positively identify the analyte. | J |
| E | Result exceeded calibration range. | J |
| B | Compound was found in the blank sample | See Exhibit 6-1 for flagging criteria |
| * | LCS or LCSD is outside acceptance limits. | See Exhibit 7-1 for flagging criteria |
| * | Isotope dilution analyte is outside acceptance limits | See Exhibit 7-2 for flagging criteria |
| 4 | MS, MSD: The analyte present in the original sample is greater than 4 times the matrix spike concentration; therefore, control limits are not applicable. | See Exhibit 7-2 for flagging criteria |
| F1 | MS and/or MSD recovery is outside acceptance limits. | See Exhibit 7-2 for flagging criteria |
| F2 | MS/MSD RPD exceeds control limits | See Exhibit 8-2 for flagging criteria |

NOTES:

1 This is not meant to be a comprehensive list of flags applied by the laboratory, but rather a list of the most encountered laboratory flags that are often not applicable after data-validation. Labs do not always use identical flags for the same QC failure; therefore, this information will be extrapolated to address the specific flags used by each laboratory and applied to each data set on a case-by-case basis.

LCS = laboratory control sample, LCSD = laboratory control sample duplicate, MS = matrix spike, MSD = matrix spike duplicate, RPD = relative percent difference.

11 COMPARABILITY

Chapter One of the EPA SW-846 Update V Revision 2 (EPA 2014) defines comparability as the expression of the degree of confidence with which one data set can be compared to another. Per the EPA SW-846 Update V Revision 2, a measurement is considered to be valid if they are unqualified or qualified as estimated data during validation. The reviewer and data users should qualitatively assess the comparability between historical and current data sets and use caution in combining data sets if the quality of the data is uncertain. For

example, current analytical methods may not be comparable to historical methods where the MRL was elevated.

12 COMPLETENESS

Chapter One of the EPA SW-846 Update V Revision 2 (EPA 2014) defines completeness as the measure of valid data collected compared to the amount planned. The SW-846 defines a valid datum as a measurement that is “unqualified or qualified as estimated [biased high, low, or no direction] during (data) validation.” The overall data set from a sampling event will be evaluated to determine if the completeness goal of 85-percent useable data was achieved. Completeness is calculated by comparing the amount of useable (valid) data to the overall number of samples planned. A completeness value below 85- percent may be cause for collecting additional analytical samples.

13 DATA-VALIDATION PLAN UPDATES

This DVPP will be reviewed upon request of DOT&PF or as needed based on changes in the laboratory reporting process.

Exhibit 13-1: Summary of DVPP Updates

| Document Version | Date | Personnel |
|------------------|------------|--------------------|
| V.0 | May 2020 | AMJ, MXJ, KRF |
| V.1 | March 2022 | RLW, AMJ, MXJ, KRF |

14 REFERENCES

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- U.S. Department of Defense (DoD), 2019, Quality Systems Manual for Environmental Laboratories v5.3, DoD, May.
- U.S. Army Corps of Engineers (USACE), 2005, Engineering Manual (EM) 200-1-10, Guidance for Evaluating Performance-Based Chemical Data, USACE, June.
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- U.S. Environmental Protection Agency (EPA), 2018b, Chapter Four - Organic Analytes. In SW-846 Update VI – Revision 6 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA, December

Appendix A

Glossary

APPENDIX A: GLOSSARY

GLOSSARY

The glossary of terms is adapted from Chapter 1 of EPA's Project Quality Assurance and Quality Control EPA SW-846 Update V Revision 2 (EPA 2014)

- Accuracy: a measure of how close a value is to the true value and is measured by percent recovery.
- Analytical batch: a group samples that does not require processing (e.g., digestion or extraction) or has been prepared and analyzed with the same reagents, calibration curve and quality control samples.
- Analytical sensitivity: the amount of analyte necessary to produce a detector response that can be reliably detected or quantified.
- Bias: a distortion in the sampling, measuring, or data evaluation process that results in error.
- Chain of custody: a record of individuals collecting, shipping, and receiving each sample.
- Comparability: the expression of the degree of confidence with which one data set can be compared to another.
- Completeness: the measure of valid data collected compared to the amount planned.
- Continuing calibration verification (CCV) sample: a quality control sample analyzed at the beginning of each analytical batch and periodically throughout the run made from calibration standards.
- Data review: the initial data quality assessment completed upon receipt of the laboratory electronic data deliverables that discuss the sample handling, condition, preservation, hold times, accuracy and precision of quality control samples and project samples, representativeness, comparability, and completeness.
- Data validation: the final review completed by staff members to validate the initial data review process.
- Detection limit (DL): the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type I error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.*
- Electronic Data Deliverable (EDD): a document provided by the lab to summarize project and quality control sample results.
- Equipment blank (EB): a sample collected in the field used to determine if decontamination of reusable sampling equipment between sampling locations is sufficient.

- Field blank (FB): a sample of laboratory-provided deionized water collected in the same air space as the corresponding project sample used to assess for possible contamination from the sampling site.
- Hold Time: a specified amount of time in which project samples should be extracted and analyzed, as determined by the laboratory or analytical method.
- Initial Calibration Verification (ICV) sample: a quality control sample analyzed immediately following instrument calibration that consist of clean extraction solvent spiked with a known analyte concentration, using a different source than that of the primary calibration standards.
- Isotope dilution Analyte (IDA): Isotopically labeled analogs of analytes of interest added to all project and quality control samples for isotope dilution analyses.* The isotopically labeled analog is used to assess method performance (recoverability) and quantification based on signal ratios.
- Laboratory Control Sample: a sample included in the preparatory batch that is prepared using the same extraction method that is applied to the project samples with laboratory-grade, blank-matrix samples spiked with a known concentration of analyte(s).
- Laboratory Level II Report: a summary of laboratory results that includes a case narrative, surrogate recoveries, chain of custody, method blank, laboratory control samples and matrix spike samples summary, and additional duplicate samples.
- Laboratory Level IV Report: a summary of laboratory results that includes elements of a Level II Report in addition to the following: GC/MS tune, initial calibration, continuing calibration verification (CCV), and raw data logs (i.e., instruments logs, data sheets, spectra, extraction logs, etc.)
- Limit of detection (LOD): the smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.*
- Limit of quantification (LOQ): the smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.*
- Matrix Spike Sample: a representative, randomly chose project sample that is “spiked” with laboratory-provided concentrations of analytes.
- Method blank (MB): analyte-free water used to assess background interference or contamination in the laboratory that may result in a false positive.
- Precision: a measure of analytical reproducibility and is measured by relative percent differences or relative standard deviation.

- Preparatory batch: a group of samples processed as an entire group with the same reagents, equipment, and laboratory personnel within a 24-hour period.
- Quality assurance (QA): the process to show relevant parties that quality control standards are being met throughout the review and validation process.
- Quality control (QC): a process to verify the acceptability and accuracy of analytical results.
- Qualifiers: a denotation to a data result to call out specific QC issues. Qualifiers are also known as flags and can be applied to the laboratory or during the data review process.
- Reporting limit (RL): a general term used to describe the lowest concentration of an analyte that produces result with known precision and bias.
- Representativeness: as the degree to which data accurately and precisely represents a characteristic of a population for a sampling point or environmental condition.
- Sample receipt form (SRF): the laboratory's documentation of sample condition and QC non-conformance issues during sample handling.
- Surrogate: an organic compound that is similar to the target analytes added to quality control samples and project samples to assess matrix effects and instrument performance for project samples.
- Trip blank: a sample free of volatile analytes that accompanies volatile between the laboratory and field sampling site, to account for contamination related to shipping and field handling.

* denotes the definition is provided by DoD/DOE QSM 5.3

Appendix B
Bottle Guide

APPENDIX B: BOTTLE GUIDE

EUROFINS TESTAMERICA BOTTLES

| Method | Media | Container and Sample Volume | Preservation | Holding Time |
|-----------|--|-----------------------------|--------------|--------------|
| EPA 537.1 | drinking water | 2 x 250-ml HDPE bottle | Trizma® | 14 days |
| | Groundwater, surface water, wastewater | 2 x 250-ml HDPE bottle | None | 14 days |
| | soil | 1 x 4-oz HDPE soil jar | none | 14 days |



| Parameter | Method | Matrix | Recommended Container/Size | Preservative | Holding Time * | Other Notes |
|--|---------------------------------|---------------|---|--|---|---|
| 1,4-Dioxane | SW 8270 | water | 2x250 ml amber glass | 0-6° C | 7 days | (Ref Lab) |
| 1,4-Dioxane | EPA 522 | DW | ? | ? | 28 days | (Ref Lab) |
| 1,4-Dioxane | SW 8260C SIM | water | 3x40 ml VOA vials | HCl; 0-6° C | 14 days | |
| 1,4-Dioxane | SW 8260C SIM | soil | 1x4 oz prewt'd amber (2nd 4 oz unpreserve % solids jar if no other analyses) | MeOH+BFB; 0-6° C | 14 days | |
| Acidity as CaCO3 | SM 2310B | water | 1x250 ml HDPE | 0-6° C | 14 days | should be analyzed in the field |
| Acute Whole Effluent Toxicity (AWET) | <i>(depends on permit)</i> | water | 1x2-8 gallon plastic (see permit) | 0-6° C | 24 hrs | (Ref Lab) need permit #/etc. |
| Alcohols: see Glycols or Alcohols | | | | | | |
| Alkalinity as CaCO3 (Total or Full) | SM 2320B | water | 1x250 ml HDPE | 0-6° C | 14 days | should be analyzed in the field |
| Ammonia | SM 4500-NH3-G modified | soil | 1x4 oz glass | 4° C | 28 days | |
| Ammonia | SM 4500NH3-G | water | 1x125 ml HDPE | H2SO4; 0-6° C | 28 days | |
| Anion/Cation Balance | SM 1030E | water | 1x60 ml Nalgene for NO2+NO3 1x250 ml HDPE for metals 1x500 ml HDPE for other analyses | H2SO4 HNO3 unpreserved | ASAP | field-filter for dissolved metals; other container unpreserved for alkalinity and anion analyses. |
| Asbestos | PCM or TEM | air | cartridge | none | n/s | (Ref Lab) |
| Asbestos | PLM or TEM | solids | any | none | n/s | (Ref Lab) |
| Asbestos | TEM | DW | 2x1 L amber glass | 0-6° C | 48 hrs or ozonate | (Ref Lab) leave 20% headspace |
| Biochemical Oxygen Demand (BOD) | SM 5210B | water | 1x1 L HDPE (depending on matrix) | 0-6° C | 48 hrs | |
| Bromate | EPA 300.1 | water | 125 ml HDPE (special order) | 1.25 ml 5% EDA 0-6° C | 28 days | (Ref Lab) |
| Bromide | EPA 300.0/SW 9056A | soil | 1x4 oz glass | 0-6° C | 28 days | |
| Bromide | EPA 300.0/SW 9056A | water | 1x60 ml Nalgene | 0-6° C | 28 days | |
| BTEX | SW 8021B/8260C | soil | 1x4 oz prewt'd amber (2nd 4 oz unpreserve % solids jar if no other analyses) | MeOH+BFB; 0-6° C | 28 days for AK101 (14 days for BTEX) | field-preservation required; use 50 g soil & 25 ml MeOH (can combo with GRO) TB required |
| BTEX | SW 8021B/8260C | water | 3x40 ml amber VOA vials w/ septa | HCl; 0-6° C | 14 days | (can combo with GRO) allow no headspace; TB required |
| CAN (Total Coliform, Arsenic, Nitrate) | SM 9223B, EPA 200.8, SM 4500NO3 | DW | sterile 120 ml container for coli 1x120 mL Nalgene for metals 60 ml Nalgene for NO2+NO3 | Na2S2O3 for coli; HNO3 for metals; H2SO4 for NOx; chill recommended | 30 hrs for coli | |
| CAN (Total Coliform, Arsenic, Nitrate) | SM 9223B, EPA 200.8, SM 4500NO4 | DW with PWSID | sterile 120 ml container for coli 1x120 mL Nalgene for metals 60 ml Nalgene for NO2+NO4 | Na2S2O3 for coli; HNO3 for metals; H2SO4 for NOx; 2-6° C | 30 hrs for coli | |
| Carbamates | EPA 531.1 | DW | 3x40 ml amber VOA vials w/ septa (special order) | Na2S2O3; Monochloroacetic Acid; 0-6° C | 7 days | (Ref Lab) |
| Carbamates | EPA 531.1 | DW with PWSID | 3x40 ml amber VOA vials w/ septa (special order) | Na2S2O3; Monochloroacetic Acid; 2-6° C | 7 days | (Ref Lab) |
| Chemical Oxygen Demand (COD) | EPA 410.4 | water | 1x125 ml HDPE | H2SO4; 0-6° C | 28 days | |
| Chlorate | EPA 300.1 | water | 1x125 ml HDPE (special order) | 1.25 ml 5% EDA 0-6° C | 28 days | (Ref Lab) |
| Chloride | EPA 300.0/SW 9056A | soil | 1x4 oz glass | 0-6° C | 28 days | |
| Chloride | EPA 300.0/SW 9056A | water | 1x60 ml Nalgene | 0-6° C | 28 days | |

| Parameter | Method | Matrix | Recommended Container/Size | Preservative | Holding Time * | Other Notes |
|---|----------------------|--------------------------------|--|---|-------------------|---|
| Chlorite | EPA 300.1 | water | 1x125 ml HDPE (special order) | 1.25 ml 5% EDA 0-6° C | 14 days | (Ref Lab) |
| Chlorophyll a | SM 10200H | water | 1x1 L amber glass (special order filters) | freeze filter ASAP | 21 days | (Ref Lab) use 4.25 cm GF-B filter; field-filter & freeze |
| Chromium, Hexavalent | SM 3500Cr or SW 7196 | water | 1x125 ml HDPE | 0-6° C | 24 hrs | |
| Chromium, Hexavalent | SW 7196 | soil | 1x4 oz amber glass | 0-6° C | 28 days | (Ref Lab) |
| Chronic Whole Effluent Toxicity (CWET) | (depends on permit) | water | 1x2-8 gallon plastic (see permit) | 0-6° C | 24 hrs | (Ref Lab) need permit specs |
| Coliform, Fecal (MF) | SM 9222D | water | sterile 120 ml container filled to 100 ml mark | Na2S2O3; 0-8° C | 8 hrs | |
| Coliform, Total (MF) | SM 9222B | water | sterile 120 ml container filled to 100 ml mark | Na2S2O3; chill recommended | 30 hrs | (Ref Lab) for quantification of Total coliform colonies, use method 9223B Quantitray |
| Coliform, Total (P/A or Quantitray) | SM 9223B | DW, DW with PWSID, water | sterile 120 ml container filled to 100 ml mark | Na2SO3; chill recommended | 30 hrs | (Contact SGS PM to make arrangements if hold time is other than 30 hours.) |
| E. coli (LT2 Quantitray) | SM 9223B | DW, DW with PWSID | sterile 120 ml container filled to 100 ml mark | Na2S2O3; <10° C | 30 hrs | (Contact SGS PM to make arrangements if hold time is other than 30 hours.) |
| Color, True or Apparent | SM 2120B | water | 1x250 ml HDPE | 0-6° C | 48 hrs | |
| Conductivity | SM 2510B | water | 1x250 ml HDPE | 0-6° C | 28 days | |
| Corrosivity (see pH) | | | | | | |
| Cryptosporidia | EPA 1623 | water | 1x10 L cubitainer | 0-6° C | 24 hrs | (Ref Lab) (can combo with Giardia) |
| Cyanide, Total | SM 4500CN-C,E | DW/W | 1x125ml amber HDPE | (Sodium Arsenite if chlorinated) NaOH; 0-6° C | 14 days | |
| Cyanide, Total | SM 4500CN-C,E | DW with PWSID | 1x125ml amber HDPE | (Sodium Arsenite if chlorinated) NaOH; 2-6° C | 14 days | |
| Cyanide, Weak Acid Dissociable | SM 4500CN-I | water | 1x125ml amber HDPE | NaOH; 0-6° C | 14 days | |
| Diesel Range Organics (DRO) | AK102 | oil | 1x20 ml scintillation vial | none | n/s | can combo with RRO |
| Diesel Range Organics (DRO) | AK102/8015C | soil | 1x4 oz amber glass | 0-6° C | 14/40 days (*) | can combo with RRO |
| Diesel Range Organics (DRO) | AK102/8015C | water | 2x1 L amber glass | HCl; 0-6° C | 14/40 days (*) | can combo with RRO |
| Diesel Range Organics (DRO)-Low Vol. | AK102/8015C | water | 2x250 ml amber glass | HCl; 0-6° C | 14/40 days (*) | |
| Dioxins | EPA 1613 | DW | 2x1 L amber glass | Na2S2O3; 0-6° C | 28 days | (Ref Lab) |
| Dioxins | EPA 1613 | DW with PWSID | 2x1 L amber glass | Na2S2O3; 2-6° C | 28 days | (Ref Lab) |
| Dioxins | SW 8280B or 8290A | soil | 1x4 oz amber | 0-6° C | n/s | (Ref Lab) |
| Dioxins | SW 8280B or 8290A | water | 2x1 L amber glass | 0-6° C | n/s | (Ref Lab) |
| Diquat/Paraquat | EPA 549.2 | DW | 1x1 Liter amber poly | Na2S2O3; 0-6° C | 7 days | (Ref Lab) |
| Diquat/Paraquat | EPA 549.2 | DW with PWSID | 1x1 Liter amber poly | Na2S2O3; 2-6° C | 7 days | (Ref Lab) |
| Dissolved Metals (see Metals, Dissolved) | | | | | | |
| Dissolved Organic Carbon (DOC) | SM 5310B | water | 1x125 ml amber glass | HCl; 0-6° C | 28 days | field-filter; unpres. if lab-filtered (should be field-filtered) |
| Dissolved Oxygen | SM 4500O2-G | water | BOD bottle w/ stopper | 0-6° C | 15 minutes (ASAP) | should be analyzed in the field; allow no headspace |
| EDB/DBCP/1,2,3-TCP | SW 8260C SIM | water | 3x40 ml amber VOA vials w/ septa | HCl; 0-6° C | 14 days | TB required, allow no headspace |
| EDB/DBCP/1,2,3-TCP | SW 8260C SIM | soil | 1x4 oz prewt'd amber (2nd 4 oz unpreserve % solids jar if no other analyses) | MeOH+BFB; 0-6° C | 14 days | TB required |
| EDB/DBCP/1,2,3-TCP | EPA 504.1 | DW | 3x40 ml amber VOA vials w/ septa | 0-6° C | 14 days | (Ref Lab) TB required allow no headspace |
| EDB/DBCP/1,2,3-TCP | EPA 504.1 | DW with PWSID | 3x40 ml amber VOA vials w/ septa | 2-6° C | 14 days | (Ref Lab) TB required allow no headspace |

| Parameter | Method | Matrix | Recommended Container/Size | Preservative | Holding Time * | Other Notes |
|--|---|-----------------|--|--|---|--|
| EDB/DBCP/1,2,3-TCP | SW 8011 | soil | 1x4 oz amber | 0-6° C | 14 days | (Ref Lab) allow no headspace |
| EDB/DBCP/1,2,3-TCP | SW 8011 | water | 3x40 ml amber VOA vials w/ septa | 0-6° C | 14 days | (Ref Lab) TB required allow no headspace |
| Endothall | EPA 548.1 | DW | 1x125 ml amber glass | Na2S2O3; 0-6° C | 7 days | (Ref Lab) |
| Endothall | EPA 548.1 | DW with PWSID | 1x125 ml amber glass | Na2S2O3; 2-6° C | 7 days | (Ref Lab) |
| Enterococci | Enterolert | water | sterile 120 ml container filled to 100 ml mark | Na2S2O3; 0-6° C | 8 hrs | |
| EPH | NW-EPH | soil | 1x4 oz amber glass | 0-6° C | 14/40 days (*) | (Ref Lab) |
| EPH | NW-EPH | water | 2x500 ml amber (special order) | HCl; 0-6° C | 7/40 days (*) | (Ref Lab) |
| Explosives | SW 8330A | soil | 1x4 oz amber glass | 0-6° C | 7 days | (Ref Lab) |
| Explosives | SW 8330A | water | 2x1 L amber glass | 0-6° C | 7 days | (Ref Lab) |
| Fluoride | EPA 300.0/SW 9056A | water | 1x60 ml Nalgene | 0-6° C | 28 days | |
| Fluoride | EPA 300.0/SW 9056A | soil | 1x4 oz glass | 0-6° C | 28 days | |
| Gasoline Range Organics (GRO) | AK101/8015C | oil | 1x20 ml scintillation vial | none | n/s | (can combo with BTEX) |
| Gasoline Range Organics (GRO) | AK101/8015C | soil | 1x4 oz prew't'd amber (2nd 4 oz unpreserve % solids jar if no other analyses) | MeOH+BFB; chill recommended | 28 days for AK101 (14 days for BTEX) | field-preservation required; use 50 g soil & 25 ml MeOH (can combo with BTEX) TB required |
| Gasoline Range Organics (GRO) | AK101/8015C | water | 3x40 ml amber VOA vials w/ septa | HCl; 0-6° C | 14 days | (can combo with BTEX) allow no headspace; TB required |
| Giardia | EPA 1623 | water | 1x10 L cubitainer | 0-6° C | 24 hrs | (Ref Lab) (can combo with Crypto) |
| Glycols or Alcohols | SW 8015 modified | water | 3x40 ml VOA vials | HCl; 0-6° C | 14 days | (Ref Lab) specify each compound |
| Glycols or Alcohols | SW 8015 modified | liquid | 1x120 ml amber glass | HCl; 0-6° C | 14 days | (Ref Lab) specify each compound |
| Glycols or Alcohols | SW 8015 modified | solid | 1x4 oz glass | HCl; 0-6° C | 14 days | (Ref Lab) specify each compound |
| Glyphosate | EPA 547 | DW | 1x125 ml amber glass | Na2S2O3; 0-6° C | 7 days | (Ref Lab) |
| Glyphosate | EPA 547 | DW with PWSID | 1x125 ml amber glass | Na2S2O3; 2-6° C | 7 days | (Ref Lab) |
| Gross Alpha &/or Gross Beta | EPA 900 | water | 1x1 L HDPE | HNO3 (preserved at lab) | none | (Ref Lab) |
| Gross Heating Value | ASTM D 240 | oil | 1x20 ml scintillation vial | none | n/s | |
| Haloacetic Acids Formation Potential | SM 5710/6251B | DW/W | 2x1 Liter | 0-6° C | ASAP/14 days | (Ref Lab) |
| Haloacetic Acids Formation Potential | SM 5710/6251B | DW/W with PWSID | 2x1 Liter | 2-6° C | ASAP/14 days | (Ref Lab) |
| Haloacetic Acids | EPA 552.3 | DW/W | 1 x 250 ml narrow mouth amber glass | NH4Cl; 0-6° C | 14 days | (Ref Lab) |
| Haloacetic Acids | EPA 552.3 | DW/W with PWSID | 1 x 250 ml narrow mouth amber glass | NH4Cl; 2-6° C | 14 days | (Ref Lab) |
| Hardness | SM 2340B | water | 1x250 ml HDPE | HNO3 | 180 days | |
| Herbicides | EPA 515.4 | DW | 2x125 ml amber glass | Sodium Sulfite; 0-6° C | 14 days | (Ref Lab) |
| Herbicides | EPA 515.4 | DW with PWSID | 2x125 ml amber glass | Sodium Sulfite; 2-6° C | 14 days | (Ref Lab) |
| Herbicides | EPA 555 | DW | 2x1 L amber glass | Na2S2O3; 0-6° C | 7/40 days (*) | (Ref Lab) |
| Herbicides | EPA 555 | DW with PWSID | 2x1 L amber glass | Na2S2O3; 2-6° C | 7/40 days (*) | (Ref Lab) |
| Herbicides | SW 8151A | soil | 1x4 oz amber | 0-6° C | 14/40 days (*) | (Ref Lab) |
| Herbicides | SW 8151A | water | 2x250 ml amber glass | 0-6° C | 7/40 days (*) | (Ref Lab) |
| Heterotrophic Plate Count (Pour Plate) | SM 9215B | water | sterile 120 ml container filled to 100 ml mark | Na2S2O3; chill recommended | 30 hrs for Pool/Spa 8 hrs for Drinking & Reagent Water | (Contact SGS PM to make arrangements if hold time is other than 30 hours.) |
| Ignitability, Seta Flash | SW 1020B | oil | 1x4 oz glass | none | n/s | |
| Inorganic Contaminants, Primary | EPA 200.8 and 300.0, SM 4500CN-C,E, 4500NO3-F | DW | 1x250 ml HDPE for metals; 1x120 ml Nalgene for cyanide; 1x60 ml Nalgene for NO2+NO3; 1x60 ml Nalgene for anions | HNO3 for metals; NaOH for CN; H2SO4 for NOx; none for F; 0-6° C | 28/180 days; 14 days; 28 days; 28 days | If samples for metals are not acid preserved they must be received by the lab within 14 days of sampling |

| Parameter | Method | Matrix | Recommended Container/Size | Preservative | Holding Time * | Other Notes |
|---|---|---------------|--|--|---|---|
| Inorganic Contaminants, Primary | EPA 200.8 and 300.0, SM 4500CN-C,E, 4500NO3-F | DW with PWSID | 1x250 ml HDPE for metals; 1x120 ml Nalgene for cyanide; 1x60 ml Nalgene for NO2+NO3; 1x60 ml Nalgene for anions | HNO3 for metals; NaOH for CN; H2SO4 for NOx; none for F; 2-6° C | 28/180 days; 14 days; 28 days; 28 days | If samples for metals are not acid preserved they must be received by the lab within 14 days of sampling |
| Karl Fisher Water Content | ASTM D 1744 | oil | 1x20 ml scintillation vial | none | n/s | |
| Kjeldahl Nitrogen: see Total Kjeldahl N | | | | | | |
| Langlier Index | SM 2330B | DW | 1x250 ml HDPE for metals 1x500 ml HDPE for other analyses | HNO3 for metals; 0-6° C for others | ASAP | (req's pH, TDS, Alkalinity & Hardness) |
| Langlier Index | SM 2330B | DW with PWSID | 1x250 ml HDPE for metals 1x500 ml HDPE for other analyses | HNO3 for metals; 2-6° C for others | ASAP | (req's pH, TDS, Alkalinity & Hardness) |
| Lead in Paint | SW 6020A | solid | any | none | 6 months | |
| Lead/Copper Rule | EPA 200.8 | DW | 1x1 L HDPE (No substitution) | HNO3 | 6 months | "First Draw" collection required If samples for metals are not acid preserved they must be received by the lab within 14 days of sampling |
| MBAS: see Surfactants | | | | | | |
| Mercury, Dissolved | EPA 200.8/245.1 or SW 6020A/7470A | Water | 1x250 mL HDPE | HNO3 | 28 days | field-filter; unpres. if lab-filtered (should be field-filtered) If samples for metals are not acid preserved they must be received by the lab within 14 days of sampling |
| Mercury, Methyl- | EPA 1630 | Water | 1x250 ml Teflon (special order) | HCl | 90 days | (Ref Lab) |
| Mercury, Total | EPA 200.8/245.1 or SW 6020A/7470A | Water | 1x250 mL HDPE | HNO3 | 28 days | If samples for metals are not acid preserved they must be received by the lab within 14 days of sampling |
| Mercury, Total | SW 6020A/7470A/7471B | soil | 1x4 oz glass | none; 0-6° C | 28 days | |
| Mercury, Trace by CVAF (Low Level) | EPA 1631E | water | 1x500 ml FLPE, Teflon or amber glass | HCl | 90 days | TB recommended |
| Metals, Dissolved (other than Hex.Cr) | EPA 200.8 | water | 1x250 ml HDPE | HNO3 | 28 days for Hg 180 days for metals | field-filter; unpres. if lab-filtered (should be field-filtered) If samples for metals are not acid preserved they must be received by the lab within 14 days of sampling |
| Metals, Dissolved (other than Hex.Cr) | SW 6020A | water | 1x250 ml HDPE | HNO3 | 28 days for Hg 180 days for metals | field-filter; unpres. if lab-filtered(should be field-filtered) If samples for metals are not acid preserved they must be received by the lab within 14 days of sampling |
| Metals, Total (other than Hex.Cr) | EPA 200.8 | water | 1x250 ml HDPE | HNO3 | 28 days for Hg 180 days for metals | |
| Metals, Total (other than Hex.Cr) | SW 6020A | soil | 1x4 oz glass | 0-6° C | 28 days for Hg 180 days for metals | |
| Metals, Total (other than Hex.Cr) | SW 6020A | oil | 1x20 ml scintillation vial | n/a | 28 days for Hg 180 days for metals | |
| Metals, Wipes | SW 6020A | wipes | premoistened "Ghost Wipe" | n/a | 28 days for Hg 180 days for metals | wipe 10x10 cm area |
| Methane/Light Gases | RSK 175 | water | 3x40 ml amber VOA vials w/ septa | HCl; 0-6° C | 14 days | (Ref Lab) allow no headspace |
| Nitrate+Nitrite, Total | SM 4500NO3-F | DW/W | 1x60 ml Nalgene | H2SO4; chill recommended | 28 days | |
| Nitrate+Nitrite, Total | SM 4500NO3-F | DW with PWSID | 1x60 ml Nalgene | H2SO4;0-6°C | 28 days | Samples received < 24 hours from collection need to be in the process of cooling. |
| Nitrate | EPA 300.0/SW 9056A | DW | 1x60 ml Nalgene | 0-6° C | 48 hrs | Samples received < 24 hours from collection need to be in the process of cooling. |

| Parameter | Method | Matrix | Recommended Container/Size | Preservative | Holding Time * | Other Notes |
|-----------------------------------|---|---------------|--|-----------------------------|----------------|---|
| Nitrate | EPA 300.0/SW 9056A | DW with PWSID | 1x60 ml Nalgene | 0-6°C | 48 hrs | Samples received < 24 hours from collection need to be in the process of cooling. |
| Nitrate | EPA 300.0/SW 9056A | soil | 1x4 oz glass | 0-6°C | 28 days | |
| Nitrite | EPA 300.0/SW 9056A | DW | 1x60 ml Nalgene | 0-6°C | 48 hrs | Samples received < 24 hours from collection need to be in the process of cooling. |
| Nitrite | EPA 300.0/SW 9056A | DW with PWSID | 1x60 ml Nalgene | 0-6°C | 48 hrs | Samples received < 24 hours from collection need to be in the process of cooling. |
| Nitrite | EPA 300.0/SW 9056A | soil | 1x4 oz glass | 0-6°C | 28 days | |
| Odor | SM 2150B | DW | 1x1L amber glass | 0-6°C | 48 hrs | (Ref Lab) |
| Odor | SM 2150B | DW with PWSID | 1x1L amber glass | 0-6°C | 24 hrs | (Ref Lab) |
| Oil & Grease, HEM | EPA 1664A | water | 2x1L amber glass | HCl; 0-6°C | 28 days | |
| Oil Burn Specs (OBS) | 40 CFR 279.11 (PCBs, As, Cd, Cr, Pb, Total Halogens & Ignitability) | oil | 1x4 oz glass | none | n/s | |
| Ortho-Phosphate | SM4500P-E | water | 1x60 ml Nalgene | 0-6° C | 48 hrs | |
| PAH | EPA 525.2 | DW | 2x1 L amber glass | Sodium Sulfite; HCl; 0-6° C | 14 days | (Ref Lab * verify cmpd list *) |
| PAH | EPA 525.2 | DW with PWSID | 2x1 L amber glass | Sodium Sulfite; HCl; 2-6° C | 14 days | (Ref Lab * verify cmpd list *) |
| PAH | EPA 625M-SIM; SW 8270D-SIM | soil | 1x4 oz amber glass | 0-6° C | 14/40 days (*) | |
| PAH | EPA 625M-SIM; SW 8270D-SIM | water | 2x250 ml amber glass | 0-6° C | 7/40 days (*) | |
| PAH Trace | EPA 625M-SIM; SW 8270D-SIM | water | 2x1 L amber glass | 0-6° C | 7/40 days (*) | |
| PCB Wipes | SW 8082A | wipes | 1 gauze wipe w/ 4 oz glass (septa lid) | Hexane | n/s | wipe 10x10 cm area |
| PCBs | EPA 508 | DW | 2x1 L amber glass | Na2S203; 0-6° C | 1 year (*) | (Ref Lab; can combo with Pest) |
| PCBs | EPA 508 | DW with PWSID | 2x1 L amber glass | Na2S203; 2-6° C | 1 year (*) | (Ref Lab; can combo with Pest) |
| PCBs | EPA 608 | water | 2x1 L amber glass | 0-6° C | 1 year (*) | (Ref Lab; can combo with Pest) |
| PCBs | SW 8082A | oil | 1x20 ml scintillation vial | none | n/s | |
| PCBs | SW 8082A | soil | 1x4 oz glass | 0-6° C | n/s | |
| PCBs | SW 8082A | water | 2x1 L amber glass | 0-6° C | n/s | |
| PCBs in Transformer Oil | SW 8082A | oil | 1x20 ml scintillation vial | none | n/s | |
| Percent Solids (Moisture Content) | SM 2540G (modified) | soil | 1x4 oz amber glass | 0-6° C | 14 days | |
| Pesticides | EPA 508 | DW | 2x1 L amber glass | Na2S203; 0-6° C | 7/40 days (*) | (Ref Lab; can combo with PCBs) |
| Pesticides | EPA 508 | DW with PWSID | 2x1 L amber glass | Na2S203; 2-6° C | 7/40 days (*) | (Ref Lab; can combo with PCBs) |
| Pesticides | EPA 608 | water | 2x1 L amber glass | 0-6° C | 7/40 days (*) | (Ref Lab; can combo with PCBs) |
| Pesticides | SW 8270D-SIM | oil | 1x20 ml scintillation vial | none | n/s | |
| Pesticides | SW 8270D-SIM | soil | 1x4 oz amber glass | 0-6° C | 14/40 days (*) | |
| Pesticides | SW 8270D-SIM | water | 2x1 L amber glass | 0-6° C | 7/40 days (*) | |
| PFAs (Polyfluorochemicals) | PFAs | water | 1x1 L polycarbonate (special order) | 0-6° C w/Trizma | 28 days | (Ref Lab) should include temp blank in same type bottle |
| PFAs (Polyfluorochemicals) | 537 | DW | 2x250 ml polycarbonate (special order) | 0-6° C w/Trizma | 14 days | (Ref Lab) should include temp blank in same type bottle |
| PFAs (Polyfluorochemicals) | PFAs | Soil | 1 x 4 oz polycarbonate (special order) | 0-6° C | 28 days | (Ref Lab) should include temp blank in same type bottle |
| PFAs (Polyfluorochemicals) | PFAs | Product | 2x250 ml polycarbonate (special order) | 0-6° C | N/A | (Ref Lab) should include temp blank in same type bottle |
| pH | SM 4500H-B | water | 1x250 ml Nalgene | 0-6° C | ASAP/7 days | should be field analyzed |
| pH Corrosivity | SW 9040C | liquid | 1x4 oz glass | none | ASAP/7 days | |
| pH Corrosivity | SW 9045D | solid | 1x4 oz glass | none | ASAP/7 days | |

| Parameter | Method | Matrix | Recommended Container/Size | Preservative | Holding Time * | Other Notes |
|---|--|------------------|---|--|--|--|
| Phase II Inorganics | EPA 200.8; EPA 300.0 | DW | 1x250 ml HDPE for metals; 1x60 ml Nalgene for anions | HNO3 for metals, unpreserved for fluoride; 0-6° C | 6 months; 28 days | If samples for metals are not acid preserved they must be received by the lab within 14 days of sampling |
| Phase II Inorganics | EPA 200.8; EPA 300.0 | DW with PWSID | 1x250 ml HDPE for metals; 1x60 ml Nalgene for anions | HNO3 for metals, unpreserved for fluoride; 2-6° C | 6 months; 28 days | If samples for metals are not acid preserved they must be received by the lab within 14 days of sampling |
| Phase V Inorganics | EPA 200.8; SM 4500CN-C,E | DW | 1x250 ml HDPE for metals; 1x125 ml Nalgene for cyanide | HNO3 for metals, NaOH for CN; 0-6° C | 6 months; 14 days | (dechlorinate before collecting for cyanide if applicable) If samples for metals are not acid preserved they must be received by the lab within 14 days of sampling |
| Phase V Inorganics | EPA 200.8; SM 4500CN-C,E | DW with PWSID | 1x250 ml HDPE for metals; 1x125 ml Nalgene for cyanide | HNO3 for metals, NaOH for CN; 2-6° C | 6 months; 14 days | (dechlorinate before collecting for cyanide if applicable) If samples for metals are not acid preserved they must be received by the lab within 14 days of sampling |
| Phenols | EPA 420.1 or SW9065 | water | 1 x 500 ml HDPE | H2SO4; 0-6° C | 28 days | (Ref Lab) |
| Phosphorus, Total | SM4500P-B,E | water | 1x125 ml HDPE | H2SO4; 0-6° C | 28 days | |
| PIWA (Private Individual Water Analysis) | SM 9223B, 2320B, 2510B, 2540C, 4500-H B, EPA 200.8, 300.0 | water | sterile 120 ml container for coli 60 ml Nalgene for NO2+NO3 1x120 mL Nalgene for metals 1x500 ml HDPE for other analyses | Na2S2O3 for coli; HNO3 for metals; H2SO4 for NOx; chill recommended | 30 hrs for coli | |
| Radiological Test Bank (i.e., Gross Alpha, Radium 226/228, Uranium) | EPA 900 EPA 903.1/904 EPA 200.8 | DW | 8x1 L HDPE (Note: Collect 2x1-L each quarter, then composite at the end of the year.) | HNO3 (preserved at lab) | 180 days | (Ref Lab) |
| Radium 226/228 | EPA 903.1/904 | water | 3x1 L HDPE | HNO3 (preserved at lab) | 6 months | (Ref Lab) |
| Radon in DW | EPA 913 or SM 7500 | water | 3x40 ml amber VOA with septa | 0-6° C | 72 hrs | (Ref Lab) |
| Residual Chlorine, Free | SM 4500CL-F | water | 1x60 ml Nalgene | 0-6° C | 15 minutes | should be field analyzed |
| Residual Chlorine, Total | SM 4500CL-G | water | 1x60 ml Nalgene | 0-6° C | 15 minutes | should be field analyzed |
| Residual Range Organics (RRO) | AK103 | oil | 1x20 ml scintillation vial | none | n/s | (can combo with DRO) |
| Residual Range Organics (RRO) | AK103 | soil | 1x4 oz amber glass | 0-6° C | 14/40 days (*) | (can combo with DRO) |
| Residual Range Organics (RRO) | AK103 | water | 2x1 L amber glass | HCl; 0-6° C | 14/40 days (*) | (can combo with DRO) |
| Residue, Filterable (TDS) | SM 2540C | water | 1x125 mL HDPE | 0-6° C | 7 days | |
| Residue, Non-Filterable (TSS) | SM 2540D | water | 1x1 L HDPE (entire volume required) | 0-6° C | 7 days | requires 1 full Liter |
| Residue, Settleable (SS or SM) | SM 2540F | water | 2x1 L HDPE (entire volume required) | 0-6° C | 48 hrs | requires 2 full Liter |
| Residue, Suspended Volatile (SVS) | SM 2540E | water | 1x1 L HDPE (entire volume required) | 0-6° C | 7 days | requires 1 full Liter |
| Residue, Total (TS) | SM 2540B | water | 1x125 ml HDPE | 0-6° C | 7 days | |
| Residue, Total Volatile (TVS) | SM 2540E | water | 1x125 ml HDPE | 0-6° C | 7 days | |
| Resistivity | SM 2510B | water | 1x125 ml HDPE | 0-6° C | 28 days | |
| Salinity by Chloride | EPA 300.0 | water | 1x60 ml Nalgene | 0-6° C | 28 days | |
| Secondary Inorganic Contaminants | EPA 200.8, 300, SM 4500H-B, 2120B, 2330B, 2320B, 2540C | DW | 1x250 mL HDPE for metals; 1x1 L HDPE for other analyses | HNO3 for metals; none for others; 0-6° C | 48 hrs for anions, pH, Alkalinity, etc. | If samples for metals are not acid preserved they must be received by the lab within 14 days of sampling |
| Secondary Inorganic Contaminants | EPA 200.8, 300, SM 4500H-B, 2120B, 2330B, 2320B, 2540C | DW with PWSID | 1x250 mL HDPE for metals; 1x1 L HDPE for other analyses | HNO3 for metals; none for others; 2-6° C | 48 hrs for anions, pH, Alkalinity, etc. | If samples for metals are not acid preserved they must be received by the lab within 14 days of sampling |
| Semivolatile Organic Cmpds (SVOC) | EPA 525.2 | DW | 2x1 L amber glass | Sodium Sulfite; HCl; 0-6° C | 14/40 days (*) | (Ref Lab * verify cmpd list *) |
| Semivolatile Organic Cmpds (SVOC) | EPA 525.2 | DW with PWSID | 2x1 L amber glass | Sodium Sulfite; HCl; 2-6° C | 14/40 days (*) | (Ref Lab * verify cmpd list *) |
| Semivolatile Organic Cmpds (SVOC) | EPA 625 | water | 2x1 L amber glass | 0-6° C | 7/40 days (*) | |

| Parameter | Method | Matrix | Recommended Container/Size | Preservative | Holding Time * | Other Notes |
|--|-------------------------------|--------|----------------------------------|----------------------|--------------------------------------|-------------------------------------|
| Semivolatile Organic Cmpds (SVOC) | SW 8270D | soil | 1x4 oz amber glass | 0-6° C | 14/40 days (*) | |
| Semivolatile Organic Cmpds (SVOC) | SW 8270D | water | 2x1 L amber glass | 0-6° C | 7/40 days (*) | |
| Settleable Matter (SS or SM): see Residue, Settleable | | | | | | |
| Solids, Total (TS): see Residue, Total | | | | | | |
| Solids, Volatile (VS): see Residue, Volatile | | | | | | |
| Specific Gravity | Lab SOP | liquid | 1x125 ml amber glass | none | n/s | |
| SPLP ... (see TCLP methods) | SW 1312... | | | | | |
| Sulfate | EPA 300.0/SW 9056A | soil | 1x4 oz glass | 0-6° C | 28 days | |
| Sulfate | EPA 300.0/SW 9056A | water | 1x60 ml Nalgene | 0-6° C | 28 days | |
| Sulfide, Total | SM 4500S-D | water | 1x125 mL HDPE | NaOH+ZnAc; 0-6° C | 7 days | |
| Sulfite | EPA 377.1 | water | 1x500 ml HDPE | 5ml 2.5% EDTA | 15 minutes | (Ref Lab) |
| Sulfolane | EPA 1625/SW8270D | soil | 1x8 oz amber glass | 0-6° C | 14/40 days (*) | |
| Sulfolane | EPA 1625/SW8270D | water | 2x1 L amber glass | 0-6° C | 7/40 days (*) | |
| Sulfur, Total | ASTM D 2622 | oil | 1x120 ml amber glass | none | n/s | (Ref Lab) |
| Surfactants (MBAS) | SM 5540C | water | 1x500 mL amber glass | 0-6° C | 48 hrs | (Ref Lab) |
| Suspended Solids (SS or SM): see Residue, Settleable | | | | | | |
| TAH | EPA 602 by 624/SW 8260B | water | 3x40 ml amber VOA vials w/ septa | HCl; 0-6° C | 14 days | allow no headspace |
| TAqH | EPA 625M-SIM; SW 8270D-SIM | water | 2x250 ml amber glass | 0-6° C | 7/40 days (*) | |
| TAqH Trace | EPA 625M-SIM; SW 8270D-SIM | water | 2x1 L amber glass | 0-6° C | 7/40 days (*) | |
| Tannin/Lignin | HACH | water | 1x250 ml amber glass | 0-6° C | 28 days | (Ref Lab) |
| TCLP Herbicides | SW 1311/8151A | water | 1x1 L amber glass | none | 14/7/40 days | (Ref Lab) |
| TCLP Herbicides | SW 1311/8151A | oil | 1x20 ml scintillation vial | none | 14/7/40 days | (Ref Lab) |
| TCLP Herbicides | SW 1311/8151A | solid | 1x8 oz amber glass | none | 14/7/40 days | (Ref Lab) |
| TCLP Metals | SW 1311/6000/7000 | water | 1x500 mL or 1Liter HDPE | none | 28 days (for Hg) 180 days (other) | |
| TCLP Metals | SW 1311/6000/7000 | oil | 1x20 ml scintillation vial | none | 28 days (for Hg) 180 days (other) | |
| TCLP Metals | SW 1311/6000/7000 | solid | 1x8 oz amber glass | none | 28 days (for Hg) 180 days (other) | |
| TCLP Pesticides | SW 1311/8270D-SIM | water | 1x1 L amber glass | none | 14/7/40 days | |
| TCLP Pesticides | SW 1311/8270D-SIM | oil | 1x20 ml scintillation vial | none | 14/7/40 days | |
| TCLP Pesticides | SW 1311/8270D-SIM | solid | 1x8 oz amber glass | none | 14/7/40 days | |
| TCLP Semivolatiles | SW 1311/8270D | water | 1x1 L amber glass | none | 14/7/40 days | |
| TCLP Semivolatiles | SW 1311/8270D | oil | 1x20 ml scintillation vial | none | 14/7/40 days | |
| TCLP Semivolatiles | SW 1311/8270D | solid | 1x8 oz amber glass | none | 14/7/40 days | |
| TCLP Volatiles | SW 1311/8260C | water | 3x40 ml amber VOA vial w/ septa | none | 14/14 days | |
| TCLP Volatiles | SW 1311/8260C | oil | 1x20 ml scintillation vial | none | 14/14 days | |
| TCLP Volatiles | SW 1311/8260C | solid | 1x4 oz amber glass | none | 14/14 days | |
| Thiocyanate | SM4500CN-M | water | 1x125ml HDPE | HNO3; 0-6° C | 28 days | (Ref Lab) Clean aqueous matrix only |
| Total Dissolved Solids (TDS): see Residue, Filterable | | | | | | |
| Total Halogens | SW 5050/9056A | oil | 1x60 ml amber glass | none | n/s | |
| Total Kjeldahl Nitrogen (TKN) | EPA 4500N-D | water | 1x125 mL HDPE | H2SO4; 0-6° C | 28 days | |
| Total Nitrogen (see: NO2/NO3, TKN and Ammonia) | | | | | | |

| Parameter | Method | Matrix | Recommended Container/Size | Preservative | Holding Time * | Other Notes |
|--|--------------------|---------------|--|---|----------------|--|
| Total Organic Carbon (TOC) | TOC-SGS SOP | soil | 1x4 oz amber | 0-6° C | 28 days | HT extended if frozen |
| Total Organic Carbon (TOC) | SM 5310B/SW 9060A | water | 1x125 ml amber glass | HCl; 0-6° C | 28 days | |
| Total Organic Halides (TOX) | SW 9020 | soil | 1x4 oz amber | 0-6° C | 28 days | (Ref Lab) |
| Total Organic Halides (TOX) | SW 9020 | water | 2x40 ml VOA or larger bottle | 0-6° C | 28 days | (Ref Lab) |
| Total Petroleum Hydrocarbons, HEM-SG | EPA 1664 SG | water | 2x1 L amber glass | HCl; 0-6° C | 28 days | |
| Total Solids: see Residue, Total | | | | | | |
| Total Suspended Solids: see Residue, Non-Filterable | | | | | | |
| Toxicity, SPP (for drilling mud) | 40 CFR ... | solid | 1 Liter | 0-6° C | 90 days | (Ref Lab) |
| TPH by 8015B: See GRO or DRO | | | | | | |
| Trihalomethane Formation Potential | SM 5710/EPA 551.1 | DW/W | 1 Liter | 0-6° C | ASAP/14 days | (Ref Lab) |
| Trihalomethane Formation Potential | SM 5710/EPA 551.1 | DW with PWSID | 1 Liter | 2-6° C | ASAP/14 days | (Ref Lab) |
| Trihalomethanes (TTHM) | EPA 524.2 | DW/W | 3x40 ml amber VOA vials w/ septa | Ascorbic Acid/ HCl; 0-6° C | 14 days | allow no headspace; TB required |
| Trihalomethanes (TTHM) | EPA 524.2 | DW with PWSID | 3x40 ml amber VOA vials w/ septa | Ascorbic Acid/ HCl; 2-6° C | 14 days | allow no headspace; TB required |
| Turbidity | SM 2130B | water | 1x60 ml Nalgene | 0-6° C | 48 hrs | |
| Turbidity | SM 2130B | DW with PWSID | 1x60 ml Nalgene | 2-6° C | 48 hrs | |
| Uranium, Total | EPA 200.8 | DW | 1x250 ml HDPE | 0-6° C | 6 months | If samples for metals are not acid preserved they must be received by the lab within 14 days of sampling |
| Uranium, Total | EPA 200.8 | DW with PWSID | 1x250 ml HDPE | 2-6° C | 6 months | If samples for metals are not acid preserved they must be received by the lab within 14 days of sampling |
| UV 254 | SM 5910B | DW | 1x250 mL amber glass | 0-6° C | 48 hrs | (Ref Lab) |
| UV 254 | SM 5910B | DW with PWSID | 1x250 mL amber glass | 2-6° C | 48 hrs | (Ref Lab) |
| VOC: Volatile Organic Compounds | EPA 524.2 | DW | 3x40 ml amber VOA vials w/ septa | (Ascorbic Acid if chlorinated) HCl; 0-6° C | 14 days | allow no headspace; TB required |
| VOC: Volatile Organic Compounds | EPA 524.2 | DW with PWSID | 3x40 ml amber VOA vials w/ septa | (Ascorbic Acid if chlorinated) HCl; 2-6° C | 14 days | allow no headspace; TB required |
| VOC: Volatile Organic Compounds | EPA 624 | water | 3x40 ml amber VOA vials w/ septa | HCl; 0-6° C | 14 days | allow no headspace; TB required |
| VOC: Volatile Organic Compounds | SW 8260C | oil | 1x20 vial or 1x40 ml VOA w/ septa | 0-6° C | 14 days | allow no headspace |
| VOC: Volatile Organic Compounds - Low Level Halogens | SW 8260C | soil | 1x4 oz prewt'd amber (2nd 4 oz unpreserve % solids jar if no other analyses) | MeOH+BFB; 0-6° C | 14 days | field-preservation required; use 50 g soil & 25 ml MeOH (can combo with BTEX) TB required |
| VOC: Volatile Organic Compounds | SW 8260C | soil | 1x4 oz prewt'd amber (2nd 4 oz unpreserve % solids jar if no other analyses) | MeOH+BFB; 0-6° C | 14 days | field-preservation required; use 50 g soil & 25 ml MeOH (can combo with BTEX) TB required |
| VOC: Volatile Organic Compounds | SW 8260C | water | 3x40 ml amber VOA vials w/ septa | HCl; 0-6° C | 14 days | allow no headspace; TB required |
| VOC: Volatile Organic Compounds Low Level (5035A FROZEN) | SW 8260C Low Level | soil | 2x40 ml VOA w/ septa; 5-ml DI water & stir bar (also provide jars for medium level VOC and % solids) | freeze w/in 48 hrs: -7 to -20° C | 14 days | field-preservation required; 5 g soil in 5 ml DI water & freeze on side immediately. TB required |

| Parameter | Method | Matrix | Recommended Container/Size | Preservative | Holding Time * | Other Notes |
|-----------|--------|--------|--|---------------------|----------------|--|
| VPH | NW-VPH | soil | 1x4 oz prewt'd amber (2nd 4 oz unpreserve % solids jar if no other analyses) | MeOH+BFB; 0-6° C | 14 days | (Ref Lab) TB required; field-preservation required; use 50 g soil & 25 ml MeOH |
| VPH | NW-VPH | water | 3x40 ml amber VOA vials w/ septa | HCl; 0-6° C | 14 days | (Ref Lab) TB required; allow no headspace |

* - Methods requiring semivolatile extraction by SW 3520/3550 have a hold time for extraction followed by a hold time for analysis of the extract.

Appendix C

Surrogate and Isotope Dilution Analyte Associations

Table 1 - Surrogate and Isotope Dilution Analyte Associations

| Analytical Method | Surrogate/ Isotope Dilution Analyte | Analyte | CAS | |
|-----------------------------|-------------------------------------|-----------------------------|---------------------------|---------|
| AK101 | 4-Bromofluorobenzene <surr> | Gasoline Range Organics | GRO | |
| AK102 | 5a Androstane <surr> | Diesel Range Organics | DRO | |
| AK103 | n-Triacontane-d62 <surr> | Residual Range Organics | RRO | |
| SW8260D (VOC) | 1,2-Dichloroethane-D4 <surr> | 1,1,1-Trichloroethane | 71-55-6 | |
| | | 1,1-Dichloroethane | 75-34-3 | |
| | | 1,1-Dichloroethene | 75-35-4 | |
| | | 1,1-Dichloropropene | 563-58-6 | |
| | | 1,2-Dichloroethane | 107-06-2 | |
| | | 1,2-Dichloropropane | 78-87-5 | |
| | | 2,2-Dichloropropane | 594-20-7 | |
| | | 2-Butanone (MEK) | 78-93-3 | |
| | | 4-Methyl-2-pentanone (MIBK) | 108-10-1 | |
| | | Benzene | 71-43-2 | |
| | | Bromochloromethane | 74-97-5 | |
| | | Bromodichloromethane | 75-27-4 | |
| | | Bromomethane | 74-83-9 | |
| | | Carbon disulfide | 75-15-0 | |
| | | Carbon tetrachloride | 56-23-5 | |
| | | Chloroethane | 75-00-3 | |
| | | Chloroform | 67-66-3 | |
| | | Chloromethane | 74-87-3 | |
| | | cis-1,2-Dichloroethene | 156-59-2 | |
| | | cis-1,3-Dichloropropene | 10061-01-5 | |
| | | Dibromomethane | 74-95-3 | |
| | | Dichlorodifluoromethane | 75-71-8 | |
| | | Methylene chloride | 75-09-2 | |
| | | Methyl-t-butyl ether | 1634-04-4 | |
| | | trans-1,2-Dichloroethene | 156-60-5 | |
| | | Trichloroethene | 79-01-6 | |
| | | Trichlorofluoromethane | 75-69-4 | |
| | | Vinyl chloride | 75-01-4 | |
| | | 4-Bromofluorobenzene <surr> | 1,1,2,2-Tetrachloroethane | 79-34-5 |
| | | | 1,2,3-Trichlorobenzene | 87-61-6 |
| | | | 1,2,3-Trichloropropane | 96-18-4 |
| | | | 1,2,4-Trimethylbenzene | 95-63-6 |
| 1,2-Dibromo-3-chloropropane | 96-12-8 | | | |
| 1,2-Dichlorobenzene | 95-50-1 | | | |
| 1,3,5-Trimethylbenzene | 108-67-8 | | | |
| 1,3-Dichlorobenzene | 541-73-1 | | | |
| 1,4-Dichlorobenzene | 106-46-7 | | | |
| 2-Chlorotoluene | 95-49-8 | | | |
| 4-Chlorotoluene | 106-43-4 | | | |

Table 1 - Surrogate and Isotope Dilution Analyte Associations

| Analytical Method | Surrogate/ Isotope Dilution Analyte | Analyte | CAS | |
|-------------------------|-------------------------------------|--------------------------------|-----------------------------|----------|
| SW8260D (VOC) | 4-Bromofluorobenzene <surr> | 4-Isopropyltoluene | 99-87-6 | |
| | | Bromobenzene | 108-86-1 | |
| | | Hexachlorobutadiene | 87-68-3 | |
| | | Naphthalene | 91-20-3 | |
| | | n-Butylbenzene | 104-51-8 | |
| | | n-Propylbenzene | 103-65-1 | |
| | | sec-Butylbenzene | 135-98-8 | |
| | | tert-Butylbenzene | 98-06-6 | |
| | Toluene-d8 <surr> | 1,1,1,2-Tetrachloroethane | 630-20-6 | |
| | | 1,1,2-Trichloroethane | 79-00-5 | |
| | | 1,2-Dibromoethane | 106-93-4 | |
| | | 1,3-Dichloropropane | 142-28-9 | |
| | | 2-Hexanone | 591-78-6 | |
| | | Bromoform | 75-25-2 | |
| | | Chlorobenzene | 108-90-7 | |
| | | Dibromochloromethane | 124-48-1 | |
| | | Ethylbenzene | 100-41-4 | |
| | | Isopropylbenzene (Cumene) | 98-82-8 | |
| | | o-Xylene | 95-47-6 | |
| | | P & M -Xylene | 95-47-6 | |
| | | Styrene | 100-42-5 | |
| | | Tetrachloroethene | 127-18-4 | |
| | | Toluene | 108-88-3 | |
| | | trans-1,3-Dichloropropene | 10061-02-6 | |
| | | Toluene | 108-88-3 | |
| | | Xylenes (total) | 1330-20-7 | |
| | SW8260D SIM (LL VOC) | 4-Bromofluorobenzene <surr> | 1,2,3-Trichloropropane | 96-18-4 |
| | | | 1,2-Dibromo-3-chloropropane | 96-12-8 |
| | | Toluene-d8 <surr> | 1,2-Dibromoethane | 106-93-4 |
| | 8270D SIMS (PAH) | 2-Methylnaphthalene-d10 <surr> | 1-Methylnaphthalene | 90-12-0 |
| 2-Methylnaphthalene | | | 91-57-6 | |
| Acenaphthene | | | 83-32-9 | |
| Acenaphthylene | | | 208-96-8 | |
| Anthracene | | | 120-12-7 | |
| Fluorene | | | 86-73-7 | |
| Fluoranthene-d10 <surr> | | Naphthalene | 91-20-3 | |
| | | Phenanthrene | 85-01-8 | |
| | | Benzo(a)Anthracene | 56-55-3 | |
| | | Benzo[a]pyrene | 50-32-8 | |
| | | Benzo[b]Fluoranthene | 205-99-2 | |
| | | Benzo[g,h,i]perylene | 191-24-2 | |
| | Benzo[k]fluoranthene | 207-08-9 | | |

Table 1 - Surrogate and Isotope Dilution Analyte Associations

| Analytical Method | Surrogate/ Isotope Dilution Analyte | Analyte | CAS |
|-------------------------|---|---|-------------|
| 8270D SIMS (PAH) | Fluoranthene-d10 <surr> | Chrysene | 218-01-9 |
| | | Dibenzo[a,h]anthracene | 53-70-3 |
| | | Fluoranthene | 206-44-0 |
| | | Indeno[1,2,3-c,d] pyrene | 193-39-5 |
| | | Pyrene | 129-00-0 |
| EPA 537.1 Mod (PFAS) | 18O2-PFHxS | Perfluorohexansulfonic acid (PFHxS) | 355-46-4 |
| | 13C2-PFHxA | Perfluorohexanoic acid (PFHxA) | 307-24-4 |
| | 13C4-PFHpA | Perfluoroheptanoic acid (PFHpA) | 375-85-9 |
| | 13C5-PFNA | Perfluorononanoic acid (PFNA) | 375-95-1 |
| | 13C3-PFBS | Perfluorobutanesulfonic acid (PFBS) | 375-73-5 |
| | 13C2-PFDA | Perfluorodecanoic acid (PFDA) | 335-76-2 |
| | 13C2-PFUnA | Perfluoroundecanoic acid (PFUnA) | 2058-94-8 |
| | 13C2-PFDoA | Perfluorododecanoic acid (PFDoA) | 307-55-1 |
| | 13C2-PFTrDA | Perfluorotridecanoic acid (PFTrDA) | 72629-94-8 |
| | 13C2-PFTeDA | Perfluorotetradecanoic acid (PFTeA) | 376-06-7 |
| | 13C3-HFPO-DA | Hexafluoropropylene oxide dimer acid (HFPO-DA) | 13252-13-6 |
| | 13C4-PFOS | Perfluorooctanesulfonic acid (PFOS) | 1763-23-1 |
| | | 4,8-Dioxa-3H-perfluorononanoic acid (DONA) | 919005-14-4 |
| | | 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS) | 756426-58-1 |
| | | 11-Chloroeicosanfluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3O11S) | 83329-89-9 |
| d3-MeFOSAA | N-Methyl perfluorooctane sulfonamidoacetic acid (N-MeFOSAA) | 2355-31-9 | |
| d5-EtFOSAA | N-Ethyl perfluorooctane sulfonamidoacetic acid (N-EtFOSAA) | 2991-50-6 | |
| 13C4-PFOA | Perfluorooctanoic acid (PFOA) | 335-67-1 | |

NOTES:

Surrogate associations for GRO, DRO, RRO, VOCs, and PAHs are based on information received February 2022 from SGS North America, Inc. and may not be representative of all laboratories.

Surrogate associations for PFAS are based on information received February 2022 from Eurofins TestAmerica, Inc. and may not be representative of all laborat PFAS analytes are associated with isotope dilution standards.

CAS No. = Chemical Abstract Service Number; DRO = diesel range organics; GRO = gasoline range organics; PAH = polynuclear aromatic hydrocarbons; PFAS = per- and poly-fluorinated alkyl substances; RRO = residual range organics; VOC = volatile organic compounds

Important Information

About Your Data-Validation Program Plan

IMPORTANT INFORMATION

CONSULTING SERVICES ARE PERFORMED FOR SPECIFIC PURPOSES AND FOR SPECIFIC CLIENTS.

Consultants prepare reports to meet the specific needs of specific individuals. A report prepared for a civil engineer may not be adequate for a construction contractor or even another civil engineer. Unless indicated otherwise, your consultant prepared your report expressly for you and expressly for the purposes you indicated. No one other than you should apply this report for its intended purpose without first conferring with the consultant. No party should apply this report for any purpose other than that originally contemplated without first conferring with the consultant.

THE CONSULTANT'S REPORT IS BASED ON PROJECT-SPECIFIC FACTORS.

A geotechnical/environmental report is based on a subsurface exploration plan designed to consider a unique set of project-specific factors. Depending on the project, these may include the general nature of the structure and property involved; its size and configuration; its historical use and practice; the location of the structure on the site and its orientation; other improvements such as access roads, parking lots, and underground utilities; and the additional risk created by scope-of-service limitations imposed by the client. To help avoid costly problems, ask the consultant to evaluate how any factors that change subsequent to the date of the report may affect the recommendations. Unless your consultant indicates otherwise, your report should not be used (1) when the nature of the proposed project is changed (for example, if an office building will be erected instead of a parking garage, or if a refrigerated warehouse will be built instead of an unrefrigerated one, or chemicals are discovered on or near the site); (2) when the size, elevation, or configuration of the proposed project is altered; (3) when the location or orientation of the proposed project is modified; (4) when there is a change of ownership; or (5) for application to an adjacent site. Consultants cannot accept responsibility for problems that may occur if they are not consulted after factors that were considered in the development of the report have changed.

SUBSURFACE CONDITIONS CAN CHANGE.

Subsurface conditions may be affected as a result of natural processes or human activity. Because a geotechnical/environmental report is based on conditions that existed at the time of subsurface exploration, construction decisions should not be based on a report whose adequacy may have been affected by time. Ask the consultant to advise if additional tests are desirable before construction starts; for example, groundwater conditions commonly vary seasonally.

Construction operations at or adjacent to the site and natural events such as floods, earthquakes, or groundwater fluctuations may also affect subsurface conditions and, thus, the continuing adequacy of a geotechnical/environmental report. The consultant should be kept apprised of any such events and should be consulted to determine if additional tests are necessary.

MOST RECOMMENDATIONS ARE PROFESSIONAL JUDGMENTS.

Site exploration and testing identifies actual surface and subsurface conditions only at those points where samples are taken. The data were extrapolated by your consultant, who then applied judgment to render an opinion about overall subsurface conditions. The actual interface between materials may be far more gradual or abrupt than your report indicates. Actual conditions in areas not sampled may differ from those predicted in your report. While nothing can be done to prevent such situations, you and your consultant can work together to help reduce their impacts. Retaining your consultant to observe subsurface construction operations can be particularly beneficial in this respect.

A REPORT'S CONCLUSIONS ARE PRELIMINARY.

The conclusions contained in your consultant's report are preliminary, because they must be based on the assumption that conditions revealed through selective exploratory sampling are indicative of actual conditions throughout a site. Actual subsurface conditions can be discerned only during earthwork; therefore, you should retain your consultant to observe actual conditions and to provide conclusions. Only the consultant who prepared the report is fully familiar with the background information needed to determine whether or not the report's recommendations based on those conclusions are valid and whether or not the contractor is abiding by applicable recommendations. The consultant who developed your report cannot assume responsibility or liability for the adequacy of the report's recommendations if another party is retained to observe construction.

THE CONSULTANT'S REPORT IS SUBJECT TO MISINTERPRETATION.

Costly problems can occur when other design professionals develop their plans based on misinterpretation of a geotechnical/environmental report. To help avoid these problems, the consultant should be retained to work with other project design professionals to explain relevant geotechnical, geological, hydrogeological, and environmental findings, and to review the adequacy of their plans and specifications relative to these issues.

BORING LOGS AND/OR MONITORING WELL DATA SHOULD NOT BE SEPARATED FROM THE REPORT.

Final boring logs developed by the consultant are based upon interpretation of field logs (assembled by site personnel), field test results, and laboratory and/or office evaluation of field samples and data. Only final boring logs and data are customarily included in geotechnical/environmental reports. These final logs should not, under any circumstances, be redrawn for inclusion in architectural or other design drawings, because drafters may commit errors or omissions in the transfer process.

To reduce the likelihood of boring log or monitoring well misinterpretation, contractors should be given ready access to the complete geotechnical engineering/environmental report prepared or authorized for their use. If access is provided only to the report prepared for you, you should advise contractors of the report's limitations, assuming that a contractor was not one of the specific persons for whom the report was prepared, and that developing construction cost estimates was not one of the specific purposes for which it was prepared. While a contractor may gain important knowledge

from a report prepared for another party, the contractor should discuss the report with your consultant and perform the additional or alternative work believed necessary to obtain the data specifically appropriate for construction cost estimating purposes. Some clients hold the mistaken impression that simply disclaiming responsibility for the accuracy of subsurface information always insulates them from attendant liability. Providing the best available information to contractors helps prevent costly construction problems and the adversarial attitudes that aggravate them to a disproportionate scale.

READ RESPONSIBILITY CLAUSES CLOSELY.

Because geotechnical/environmental engineering is based extensively on judgment and opinion, it is far less exact than other design disciplines. This situation has resulted in wholly unwarranted claims being lodged against consultants. To help prevent this problem, consultants have developed a number of clauses for use in their contracts, reports, and other documents. These responsibility clauses are not exculpatory clauses designed to transfer the consultant's liabilities to other parties; rather, they are definitive clauses that identify where the consultant's responsibilities begin and end. Their use helps all parties involved recognize their individual responsibilities and take appropriate action. Some of these definitive clauses are likely to appear in your report, and you are encouraged to read them closely. Your consultant will be pleased to give full and frank answers to your questions.

The preceding paragraphs are based on information provided by the ASFE/Association of Engineering Firms Practicing in the Geosciences, Silver Spring, Maryland