Standard Operating Procedure for the Waste Management and Remediation Division

Data Validation
of Third-Party Petroleum Release Investigation, UST Closure, and Change-in-Service Data Submittals

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SOP WST-2014-10, Version 1

State of Idaho
Department of Environmental Quality

Waste Management and Remediation

May 2017

Approval Signatures

This statewide standard operating procedure (SOP) becomes effective on the date of the last approval signature.

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1 Purpose and Applicability

This standard operating procedure (SOP) was created for Idaho Department of Environmental Quality (DEQ) Waste Management and Remediation Division (WMR) staff to conduct data validation of third-party data submittals. DEQ quality assurance project plans (QAPPs) for third-party submittals will identify the level of data validation to be conducted using a graded approach based on the type of project. This SOP identifies the steps DEQ Waste Management and Remediation Division staff assigned as the Regional Office Project Quality Assurance Officer (QAO) will take in conducting the data validation. A data validation checklist is included in Appendix B of the Third-Party Petroleum Release Investigation and UST Closure and Change-in-Service QAPP (DEQ 2014). Data validation methods, including the level of data validation, are presented in Section 23 of the Third-Party Petroleum Release Investigation and UST Closure and Change-in-Service QAPP (DEQ 2014). This SOP supplements the Third-Party Petroleum Release Investigation and UST Closure and Change-in-Service QAPP.

1.1 Mission and Authority

This SOP provides a process for conducting data validation of third-party petroleum release investigation data submittals and UST closure and change-in-use data submittals.

1.2 Program Objectives

The objective is statewide consistency for conducting data validation of third-party petroleum release investigation data submittals and UST closure and change-in-service data submittals. The goal of data validation is to evaluate whether the data quality goals and requirements established in the QAPP have been achieved, and to determine the impact on data quality of those that were not met. Data validation activities will be documented on the checklist (see Section 4.4) and entered into TRIM (see Section 5).

2 Definitions

Accuracy: The closeness of agreement between an observed value and an accepted reference value. Typically, spiked sample recoveries are used to assess laboratory accuracy as well as satisfactory performance of blank analyses. Accuracy requirements are identified in the specific third-party data QAPP under which the data is being evaluated.

Analyte: The element, ion, compound, or aggregate property of a sample for which an analysis seeks to determine its quantity and/or presence.

Blank sample: Samples of known matrix free of the specific constituents selected for analysis. Blank samples are typically submitted to the laboratory blind and are used to measure data accuracy. Blank samples may also reveal contamination problems due to sample collection method or sampling conditions.

Completeness: The percentage of total measurements completed that are not qualified, thus increasing the degree of confidence in the reported result. Completeness requirements are identified in the specific third-party data QAPP under which the data is being evaluated.

Data Qualifier: A letter assigned to an analytical result during data validation which generally denotes a modified degree of confidence from the reported analytical result. Data qualifiers used by DEQ are defined as follows:

i) **J** The analyte was identified and the associated numerical value is considered an approximate or estimated concentration of the analyte in the sample.

ii) **R** The sample results are rejected due to deficiencies in achieving quality control criteria. The presence or absence of the analyte cannot be verified.

iii) **U** The analyte was analyzed for, but was not detected above the MDL or reporting limit concentration.

iv) **UJ** The analyte was analyzed for and not detected. The associated value is an estimate and may not accurately reflect the concentration in the sample.

v) **B** The analyte was identified in blank samples.

Data Package: A collection of information that includes data from analysis of all samples associated with a work request, including field and analytical samples, re-analyses, blanks, duplicates, and spikes.

Data Validation: A technical review performed to compare data with established quality criteria to ensure the data are adequate for the intended use. Data validation confirms that the verified results meet the overall quality requirements of the intended use.

Data Verification: An evaluation of the completeness, correctness, consistency and conformance/compliance of the data against pre-determined requirements, and to ensure that the records associated with the data reflect actual activities.

Duplicate samples: Two samples collected from the same location and representing the same sampling event which are carried through all assessment and analytical procedures in an identical manner. Duplicate samples are collected sequentially, or nearly so, from the same sample location or split from the same container and analyzed for the same analytes. Duplicate samples may be “replicates” (samples taken one immediately after the other, separated only by the actual time required to fill the sample container), or “splits” (subsamples drawn from the same initial volume of sample matrix). Duplicate samples are analyzed to verify sampling and analytical reproducibility and sample repeatability, i.e. precision.

Equipment blank: A sample of matrix of known constituent quantity that has passed through or over non-dedicated sampling equipment to verify the cleaning procedure (decontamination) between samples.

Field blank: A clean sample of known matrix that is placed into a sampling container and otherwise treated the same as other samples collected to verify general sampling and handling procedures.

Holding Time: The time period from sample collection to laboratory analysis. For some analyses, the time from sample collection to sample preparation or extraction must also be considered.

Laboratory blank: A laboratory blank is a sample of an uncontaminated reference matrix. The laboratory blank is analyzed to evaluate the accuracy of the analysis.

Laboratory Duplicate Sample: A laboratory duplicate sample is a sample that is split by the laboratory into two separate and identical samples. The two samples are analyzed and a comparison of the results relative percent difference (RPD) is used to assess laboratory precision.

Laboratory Quality Control Sample: Laboratory control samples (LCSs) are samples that contain a known concentration of analytes and are analyzed to assess the overall method performance. They undergo the same preparatory and determinative procedures as the project environmental samples and are the primary indicator of laboratory performance. Laboratory control sample blank recoveries are used to measure accuracy. The RPD for duplicate LCS recoveries is used to measure precision.

Matrix: The dominant material of which the sample to be analyzed is composed. Matrix is not synonymous with phase (solid, vapor, or liquid).

Matrix Spike and Matrix Spike Duplicate (MS/MSD) Samples: Introduction of a known concentration of an analyte into a blank matrix sample to provide information about the bias of the measurement methodology. Samples may be submitted without identification as a spiked sample revealed to the analyzing laboratory. Percent recoveries (%R) on MS samples will be compared to %R of LCS samples. An MS, MSD pair can be used to assess precision.

MDL: Method detection limit (MDL) is the lowest concentration of a substance that can be measured with 99% confidence that the substance is present in the sample.

Precision: The agreement among a set of replicate measurements without assumption of knowledge of the true value. Precision is calculated by means of duplicate/replicate analyses. These samples will contain concentrations of analyte above the MDL, and may involve the use of matrix spikes. The most commonly used measures of precision are the relative percent difference (RPD) when comparing duplicate and standard samples. Precision requirements are identified in the specific third-party data QAPP under which the data is being evaluated.

Professional Judgment: Discernment that is a cumulative result of scientific and technical training, experience in analytical testing and reporting, and good understanding of specific method-required quality assurance and quality control (QA/QC) procedures.

Spike sample: A sample to which known concentrations of analytes have been added in such a manner as to minimize the change in the matrix of the original sample.

Trip blank: Generally pertain to volatile organic compound (VOC) samples. A trip blank is a clean sample prepared by the laboratory prior to the sampling event and transported with the sample containers to the site and back to the laboratory with the samples collected in the field (i.e., trip blanks accompany sample containers throughout the sampling event). Trip blanks are

analyzed for VOCs or dissolved gasses to verify that the sample containers are clean and free of contamination through outside influences.

Usability: The percentage of the total measurements requested that are not rejected and deemed usable.

3 Personnel Qualifications

DEQ staff conducting data validation of third-party data submittals under this SOP must have experience in petroleum release investigations, and/or UST closure and change-in-use requirements typical of an Analyst 3 or 4, as well as a working knowledge of QA/QC requirements.

4 Procedures

4.1 Review Applicable Documents

The data validator (Regional Office Project QAO) will be familiar with the Third-Party Petroleum Release Investigation and UST Closure and Change-in-Service QAPP (DEQ 2014) under which the data validation is conducted. Data validation applies to activities conducted in the field as well as in the analytical laboratory. Therefore, the data validator must review the available information and reports regarding the field activities conducted and the laboratory analysis of samples collected by the third-party. The data validator will also review the data review and verification documentation for the project. The data review and verification is typically conducted by the Regional Office Project Manager, or other staff member, assigned to the project.

4.2 Data Validation of Field Activities

The data validator will conduct the following analysis, as applicable, based on the information provided by the third-party:

4.2.1 Field Records.

Evaluate submitted field records for consistency. Field records will include field instrument calibration data, if used. For ground water sampling events, field records will also include appropriate field parameter data collected prior to the collection of groundwater samples, unless passive ground water sampling techniques are employed. Indicators for potentially improper field records may include:

- Unexpected field conditions (e.g., adverse terrain or inclement weather may prompt ‘cutting of corners’ to collect data and samples).
- Absence of field instrument calibration data or unusual calibration data for photoionization detector (or other field instrument) results in potential improper

screening of soil and soil vapor borings and collection of soil and soil vapor samples.

Qualification. Any field record inconsistencies, discrepancies, or missing information must be documented with an explanation provided in a validation narrative.

4.2.2 Sample Collection and Handling.

Review submitted sample collection and handling information, including specific sample collection procedures. If DEQ staff were on-site during all or part of the field activities, review DEQ field records and third-party submitted records to identify potential indicators of sampling problems. Indicators for improper sampling procedures may include:

- Homogenized or composite samples for VOC analysis that have not used EPA Method 5035 sampling procedures.
- Sample locations in close proximity to potential sources of contaminant interference (e.g., soil and soil vapor samples near (six inches to one foot) asphalt when polycyclic aromatic hydrocarbon (PAH) analysis is to be performed).
- Biased sampling locations (e.g., collecting samples to bias the result away from contaminated areas).
- Sample dates and times that do not match other information.
- Inconsistencies between chain-of-custody (COC) and other information.

Qualification. Any sample collection and handling inconsistencies, discrepancies, or missing information must be documented with an explanation provided in a validation narrative.

4.3 Data Validation of Analytical Laboratory Activities

The data validator will conduct the following analysis, as applicable, based on the information provided by the third-party:

4.3.1 Chain of Custody.

1. Chain of custody must include:
    a. Each sample must have an assigned unique number.
    b. The date and time of sample collection.
    c. The requested analytical method or analyte for each sample.
    d. Sample preservative or preservation method (e.g. HCl, ice, etc.).
    e. Sample matrix (e.g., soil or soil vapor).

f. Sample numbers assigned by the laboratory must correspond to the assigned unique sample number throughout the analysis.

g. Chain-of-custody forms must have appropriate signatures identifying possession transfers throughout the process.

2. Qualification. Any COC discrepancies must be documented with an explanation provided in a validation narrative.

4.3.2 Holding Times.

1. The holding time requirements are listed in the analytical method used by the laboratory. Holding times for typical analytical methods are provided in Appendix B of DEQ SOP WST-2014-11 (2014a). Sample holding times are calculated by comparing the sample collection date and time on the COC form with the dates and times of analysis, including extraction dates, reported in the laboratory data sheets. For some analyses, the time from sample collection to sample preparation (e.g., extraction) must also be considered.

2. Qualification.

a. If the holding time was greater than twice the method-required holding time, non-detected analytes will be qualified unusable ("R") and positive results will be considered approximate and qualified with a "J."

b. If the holding time was equal to or less than twice the requested holding time, qualify the results as approximate ("J").

4.3.3 Sample Preservation.

1. The preservation requirements are listed in the analytical method used by the laboratory. Preservation for typical analytical methods is provided in Appendix B of DEQ SOP WST-2014-11 (2014a). Examine the digestion and/or distillation logs to determine if samples were preserved at the proper pH.

2. Qualification. If samples were not preserved properly, such as not maintained at a temperature specified by the analytical method (e.g., 4^{o} C $\pm 2^{o}$ C), use professional judgment based on the constituents of concern (e.g., Improper preservation for analysis of metals in soil may not impact results, but proper preservation of water for analysis of metals is more critical. Proper preservation for VOC samples is critical.). Qualify potentially compromised data $\geq$ MDL as estimated (J). Qualify data < MDL as unusable (R).

4.3.4 Sample Containers.

1. Typical sample container information is provided in Appendix B of DEQ SOP WST-2014-11 (2014a). Make note of any laboratory reported problems, such as sample leakage, broken containers, inadequate sample volume, inappropriate

sample containers, air pockets or bubbles for VOC samples, or other similar information.

2. Qualification. Qualify data as estimated (J) or as unusable (R) based on professional judgment of sample container type and condition on the impact to data quality and usability (e.g., VOC water sample with unexplained air bubbles or VOC soil sample with voids/air pockets is problematic). Qualify uncertain data ≥ MDL as estimated (J) and qualify data < MDL as unusable (R).

4.3.5 Sample Analytical Methods.

1. Verify the appropriate analytical method was requested by the third-party on the COC and utilized by the laboratory. Typical analytical method information is provided in Appendix B of DEQ SOP WST-2014-11 (2014a). Verify the laboratory properly accounted for dilution, if utilized, in the sample analysis and reported result.

2. Qualification. Any analytical method discrepancies must be documented with an explanation provided in a validation narrative.

4.3.6 Laboratory Practices.

1. Review all available laboratory information and data submitted to evaluate potential for improper laboratory practices. For most third-party data submittals, detailed laboratory information will likely not be provided or submitted to DEQ. See Section 4.2 of the EPA QA/G-8 (Guidance on Environmental Data Verification and Data Validation for the following:

   a. Failure to analyze samples.

   b. Failure to conduct method-required analytical process.

   c. Manipulation of samples prior to analysis.

   d. Manipulation of results during analysis.

   e. Post-analysis alteration of results.

2. Qualification. Any laboratory process discrepancies must be documented with an explanation provided in a validation narrative. Qualify data affected by improper laboratory practices as unusable (R).

4.3.7 Method Detection Limits.

1. Ensure correct MDLs are used as indicated below for petroleum projects:

   a. DEQ residential use screening levels from the Standards and Procedures for Application of Risk Based Corrective Action at Petroleum Release Sites (IDAPA 58.01.24); *http://adminrules.idaho.gov/rules/current/58/0124.pdf*, and the Petroleum Risk Evaluation Manual (2012 or more recent version);

http://www.deq.idaho.gov/waste-mgmt-remediation/remediation-activities/risk-evaluation-manuals.aspx.

b. For used oil constituents, see DEQ Used Oil UST Closure and Release Sampling Standard Operating Procedures (TRIM 2016BAF24).

2. Qualification. Elevated data (i.e., sample result is greater than the screening level) will be qualified as estimated (J) when the MDL > screening levels. Data rejection (R) would be appropriate if the sample data were low (i.e., near the screening level or non-detect) when the MDL > screening level, or when the MDL is near the screening level.

4.3.8 Comparability

1. Comparability is satisfied by the third-party conducting sample collection and handling processes that are consistent with "standard practice" or "industry accepted practices", and the laboratory performing sample analysis follows standard preparation and analysis procedures.

2. Qualification. Any deviations from "standard practice" sample collection, handling, preparation and analysis must be documented with an explanation provided in a validation narrative.

4.3.9 Review QC Data (Precision, Accuracy).

1. Ensure precision and accuracy calculations conducted by third parties are valid and correct if LCS, matrix spikes, or surrogate spikes are conducted and recoveries are reported by the laboratory and submitted by the third-party for accuracy, and/or if duplicate samples are collected by the third-party or internal laboratory duplicate samples are analyzed with the samples and the information is submitted by the third-party. For petroleum release investigations and UST closure or change-in-service activities, field quality control sample results, except for trip blanks, are considered to be supplemental data. However, laboratories routinely conduct internal quality control analyses. Therefore, laboratory quality control data is considered to be minimum acceptance criteria. third

2. Qualification. Apply appropriate data qualifier based on the precision and accuracy requirements identified in the specific QAPP utilized for data evaluation.

    a. Accuracy is to be within the ranges of acceptability for percent recovery identified by the specific laboratory conducting the analysis for each method and analyte; if LCS, matrix spikes, or surrogate spikes are conducted and recoveries are reported by the laboratory and submitted by the third-party for the analysis. Accuracy is minimum acceptance criteria.

    b. Precision for laboratory duplicate data (for laboratory control samples or matrix spike samples) is to be within the ranges of acceptability,

based on RPD, identified by the specific laboratory conducting the analysis for each method and analyte and reported by the third-party. Precision is minimum acceptance criteria.

c. Precision for field duplicate samples, if collected by the third-party, is to be within ± 50%, based on RPD for soil samples. Precision for field duplicate ground water samples, if collected by the third-party, is to be within ± 30% based on RPD. Precision for field duplicate soil vapor samples, if collected by the third-party, is to be within ± 25% based on RPD. Precision is supplemental information and not considered to be minimum acceptance criteria.

In general, data generated with accuracy and precision exceeding the criteria will be rejected and not used in decision making, unless sufficient supplemental information is available to support use of the data.

4.3.10 Review Blank Sample Results.

1. No contaminants will be present in blank samples. Examine results and identify samples with constituents reported in the blanks at a concentration equal to or greater than the MDL. If problems with any blanks exist, all data associated with the sample must be carefully evaluated to determine whether or not there is an inherent variability in the data, or if the problem is an isolated occurrence not affecting other data. For most third-party data submittals, blank sample information may not be available or submitted to DEQ. Field blank and equipment blank sample information is considered to be supplemental and is not included as minimum acceptance criteria. Trip blank sample information is considered minimum acceptance criteria when VOC analyses occur. Blank samples may consist of one or more of the following:

    a. Field blank – a field blank is a clean matrix sample placed into a sampling container and otherwise treated the same as other samples taken from the field to check general sampling and handling procedures.

    b. Trip blank – a trip blank is a laboratory supplied sample (typically distilled or deionized water) that accompanies each shipment of samples for VOC analysis and is analyzed to assess potential contamination during sample shipment.

    c. Equipment blank – equipment blanks consist clean matrix that has passed through or over non-dedicated sampling equipment to verify the decontamination procedure between samples. If no special equipment is used that requires decontamination, such as dedicated monitoring well tubing, then equipment blanks are not necessary.

2. Qualification. When blank sample results demonstrate that contamination has been detected, the Regional Office Project QAO will discuss the situation with the Regional Office Project Manager to consider on a case-by-case basis if the contamination is significant enough to reject the data (R) or use the B flag.

4.3.11 Representativeness.

1. Representativeness is satisfied by confirming that sampling locations are properly selected, sample collection procedures are appropriate and consistently followed, a sufficient number of samples are collected, MDLs are less than screening criteria, and analytical results are useable (see Section 4.3, 4.4.1-5 and 4.4.12 of this SOP).

    a. Field data is likely Level I (e.g. PID) and laboratory data is likely Level III/Stage 1 or Stage 2A (see Appendix A of DEQ SOP WST-2014-11 [2014a]). Analytical results must be current (within the last 12 months) to be considered representative of site conditions and status. Historical, peer-reviewed published data of equivalent quality may be used, but do not represent current site conditions if more than 12 months old.

2. Qualification. Document representativeness in a validation narrative.

4.3.12 Completeness (90% verified data related to minimum acceptance criteria).

1. Summarize the total number of analyses requested for each analyzing laboratory, noting the number of analyses flagged with a data qualifier which limits the data's usability (e.g. laboratory-applied qualifier or DEQ J or R qualifier). The percent completeness (%C) is calculated using:

    %C = ((Total Data Obtained – Flagged Data (J or R))/(Total Data Requested))*100

2. Qualification. If data completeness falls below 90%, the Regional Project QAO will discuss the situation with the Regional Project Manager to consider, on a case-by-case basis, if the data submittal is to be rejected or partially accepted.

4.4 Data Validation Report

For most projects, only a representative effort will be made under data validation (i.e., not all projects undergo data validation). The completed data validation checklist (see Appendix B of the Third-Party Petroleum Release Investigation and UST Closure and Change-in-Service QAPP [DEQ 2014b]) will be the Data Validation Report. The data validation checklist will summarize the data validation process conducted by the Regional Office Project QAO for the project and identify qualified data and narratively qualified data. The data validation checklist will also summarize any deviations identified and provide a determination of those deviations on data quality and usability. The data validation checklist will be submitted to the Regional Office Project Manager.

In the event that significant problems are discovered through the application of this validation process, additional action may be taken to ensure minimum data quality is achieved. These may include, but are not limited to, a more thorough validation process following EPA (2002) guidance, and the development of Corrective Action Report and Corrective Action Plan in conformance to DEQ's Quality Management Plan (2012).

5 Records

The validation report (from Section 4.4 of this SOP) will be entered into TRIM following applicable program SOPs:

- UST documents will be entered into TRIM per the TRIM SOP (TRIM 2011BAQ8).
- LUST documents will be entered into TRIM per the TRIM SOP (TRIM 2012BAQ6).
- General remediation documents will be entered into TRIM per the TRIM SOP (TRIM 2011BAQ3).

6 References

DEQ (Idaho Department of Environmental Quality). 2012 or more recent version. Risk Evaluation Manual. Boise, ID: DEQ. *http://www.deq.idaho.gov/waste-mgmt-remediation/remediation-activities/risk-evaluation-manuals.aspx*.

DEQ (Idaho Department of Environmental Quality). 2012. Quality Management Plan. Boise, ID: DEQ. TRIM record number 2012AEC1.

DEQ (Idaho Department of Environmental Quality). 2014b. Standard Operating Procedure for Data Review and Verification of Third-party Petroleum Release Investigation and Underground Storage Tank Closure and Change-in-Service Data Submittals. Boise, ID: DEQ. TRIM record number 2016BAF17.

DEQ (Idaho Department of Environmental Quality). 2014c. Third Party Petroleum Storage Tank Release Investigation and UST Closure and Change-in-Service Quality Assurance Project Plan. Boise, ID: DEQ. TRIM record number 2016BAF15.

DEQ (Idaho Department of Environmental Quality). Used Oil UST Closure and Release Sampling Standard Operating Procedures. Boise, ID: DEQ. TRIM record number 2016BAF24.

EPA (US Environmental Protection Agency). 2002. *Guidance on Environmental Data Verification and Data Validation* (EPA QA/G-8). Washington DC: EPA, Office of Environmental Information. EPA/240/R-02/004. Available at *http://www.epa.gov/quality/qs-docs/g8-final.pdf*

EPA (US Environmental Protection Agency). 2014 or more recent version. Regional Screening Levels. *http://www.epa.gov/reg3hwmd/risk/human/rb-concentration_table/Generic_Tables/index.htm*.

IDAPA 58.01.24. Standards and Procedures for Application of Risk Based Corrective Action at Petroleum Release Sites *http://adminrules.idaho.gov/rules/current/58/0124.pdf*

IDAPA 58.01.02. Water Quality Standards. *http://adminrules.idaho.gov/rules/current/58/0102.pdf*