

Data Validation Summary Report

(DVSR ID: TetraTech-M16Supplemental-2018rev1)

Vacuum Enhanced Recovery Treatability Study Supplemental Sampling Nevada Environmental Response Trust Site Henderson, Nevada

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LIST OF ACRONYMS/ABBREVIATIONS

Acronyms/Abbreviations	Definition
BW	blank water
CCB	continuing calibration blank
CCV	continuing calibration verification
DL	detection limit
DMC	deuterated monitoring compound
DQO	data quality objectives
DUP	duplicate
DVSR	data validation summary report
EB	equipment blank
EDD	electronic data delivery
FB	field blank
FD	field duplicate
GC-MS	gas chromatography-mass spectroscopy
ICAL	initial calibration
ICB	initial calibration blank
ICS	interference check samples
ICV	initial calibration verification
LCS	laboratory control sample
MDL	method detection limit
MS/MSD	matrix spike/matrix spike duplicate
NORM	normal field sample
NDEP	Nevada Division of Environmental Protection
NERT	Nevada Environmental Response Trust
NFG	National Functional Guidelines
%C	percent completeness
%D	percent difference or drift
%R	percent recovery
%RSD	percent relative standard deviation
PARCCS	precision, accuracy, representativeness, comparability, completeness, sensitivity
PQL	practical quantitation limit
QA	quality assurance
QAPP	quality assurance project plan

Acronyms/Abbreviations	Definition
QC	quality control
RL	reporting limit
RPD	relative percent difference
RRF	relative response factor
SDG	sample delivery group
SQL	sample quantitation limit
TB	trip blank
Tetra Tech	Tetra Tech, Inc.
Treatability Study	Vacuum Enhanced Recovery Treatability Study
USEPA	United States Environmental Protection Agency
µg/L	micrograms per liter
VOC	volatile organic compound
WG	groundwater
WQ	water quality assurance sample

CERTIFICATION

**Data Validation Summary Report
(DVSR ID: TetraTech-M16Supplemental-2018rev1)
Vacuum Enhanced Recovery Treatability Study Supplemental Sampling**

**Nevada Environmental Response Trust Site
(Former Tronox LLC Site)
Henderson, Nevada**

Nevada Environmental Response Trust (NERT) Representative Certification

I certify that this document and all attachments submitted to the Division were prepared at the request of, or under the direction or supervision of NERT. Based on my own involvement and/or my inquiry of the person or persons who manage the systems(s) or those directly responsible for gathering the information or preparing the document, or the immediate supervisor of such person(s), the information submitted and provided herein is, to the best of my knowledge and belief, true, accurate, and complete in all material respects.

Office of the Nevada Environmental Response Trust

Le Petomane XXVII, not individually, but solely in its representative capacity as the Nevada Environmental Response Trust Trustee

Signature: Jay A. Steinberg, not individually, but solely in his representative capacity as President of the Nevada Environmental Response Trust Trustee

*not individually, but solely
as President*

Name: Jay A. Steinberg, not individually, but solely in his representative capacity as President of the Nevada Environmental Response Trust Trustee

Title: Solely as President and not individually

Company: Le Petomane XXVII, Inc., not individually, but solely in its representative capacity as the Nevada Environmental Response Trust Trustee

Date: 2/11/19

CERTIFICATION

I hereby certify that I am responsible for the services described in this document and for the preparation of this document. The services described in this document have been prepared in a manner consistent with the current standards of the profession, and to the best of my knowledge, comply with all applicable federal, state, and local statutes, regulations, and ordinances. I hereby certify that all laboratory analytical data was generated by a laboratory certified by the NDEP for each constituent and media presented herein.

Description of Services Provided: Prepared Data Validation Summary Report (DVSR ID: TetraTech-M16Supplemental-2018rev1) Vacuum Enhanced Recovery Treatability Study Supplemental Sampling



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February 11, 2019

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Nevada CEM Certificate Number: 2167
Nevada CEM Expiration Date: September 18, 2020

1.0 INTRODUCTION

On behalf of the Nevada Environmental Response Trust (NERT), Tetra Tech, Inc. (Tetra Tech) has prepared this Data Validation Summary Report (DVSR) to assess the validity and usability of laboratory analytical data from the supplemental samples associated with the Vacuum Enhanced Recovery Treatability Study (Treatability Study) for the NERT site, located in Clark County, Nevada. Sampling protocol can be found in Vacuum Enhanced Recovery Treatability Study Work Plan (Tetra Tech, 2017). Tetra Tech collected supplemental groundwater and quality assurance and quality control (QA/QC) samples to aid in assessing data quality.

TestAmerica, Inc. provided laboratory analytical services. The analyses were performed by the methods shown in Table 1.

The laboratory assigns job numbers, also called sample delivery groups (SDGs), to all samples. The samples associated with QA/QC are designed to document the data quality of the samples in each sampling round or within an SDG. Table 2 cross-references each sample with its analysis, SDG, collection date, client sample number, laboratory sample number, QC type, matrix, and stage of validation. Samples included in Table 2 are project samples submitted in the DVSR electronic data deliverable (EDD). Field readings for the samples in Table 2 are submitted in a separate EDD table because they are not validated. The laboratory data package may be found in Appendix B.

The laboratory analytical data were verified and validated in accordance with procedures described in the *Quality Assurance Project Plan, Revision 2* (Ramboll Environ, 2017), *NDEP Data Verification and Validation Requirements* (NDEP, 2017), and the references contained therein. All samples were validated to Stage 4. The review process uses professional judgment and National Functional Guidelines (NFG) guidance to determine the final qualifiers, which are added to the database and presented in the DVSR tables. The Stage 4 data validation checklist is found in Appendix A.

This report summarizes the QA/QC evaluation of the data using precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS) relative to the project data quality objectives (DQOs). This report provides a quantitative and qualitative assessment of the data and identifies potential sources of error, uncertainty, and bias that may affect the overall usability of the data.

2.0 PRECISION AND ACCURACY OF ENVIRONMENTAL DATA

Environmental data quality depends on sample collection procedures, analytical methods and instrumentation, documentation, and sample matrix properties. Both sampling procedures and laboratory analyses contain potential sources of uncertainty, error, and/or bias, which may affect the overall quality of a measurement. Errors for sample data may result from incomplete equipment decontamination, inappropriate sampling techniques, sample heterogeneity, improper filtering, and improper preservation. The accuracy of analytical results is dependent on selecting appropriate analytical methods, maintaining equipment properly, and complying with QC requirements. The sample matrix also is an important factor in the ability to obtain precise and accurate results within a given medium.

Environmental and laboratory QA/QC samples provide information on the effects of sampling procedures and evaluate laboratory contamination, laboratory performance, and matrix effects. Field QA/QC samples include equipment blanks (EBs), field blanks (FBs), field duplicates (FDs), matrix spike/matrix spike duplicates (MS/MSDs), and trip blanks (TBs). Laboratory QA/QC samples include method blanks, laboratory control samples (LCSs), laboratory duplicates (DUP), and additional MS/MSDs needed to meet method requirements.

2.1 PRECISION

Precision is a measure of the agreement of analytical results under a given set of conditions. It is a quantity that is not measured directly but is calculated from concentrations. Precision can be expressed as the relative percent difference (RPD) between two measurements:

$$RPD = \frac{(C1 - C2) * 100}{(C1 + C2) / 2}$$

where:

C1 = reported concentration for the sample

C2 = reported concentration for the duplicate

Precision can be expressed as the percent relative standard deviation (%RSD) between three or more measurements:

$$\%RSD = (s/\bar{a}) * 100$$

where:

%RSD = percent relative standard deviation

s = standard deviation

\bar{a} = mean of replicate analyses

Precision is assessed by calculating %RSD during an initial calibration (ICAL) and RPD from the percent recoveries of the spiked compounds for each sample in the MS/MSD pair. In the absence of an MS/MSD pair, a laboratory duplicate can be analyzed as an alternative means of assessing precision. An additional measure of sampling precision is obtained by collecting and analyzing field duplicate samples, which are compared using the RPD results as the evaluation criteria.

MS and MSD samples are field samples which have been spiked by the laboratory with target analytes prior to preparation and analysis. These samples measure the appropriateness of the analytical method and effectiveness in recovering target analytes from a specific environmental matrix. The LCS sample is spiked with the same target analytes as the MS/MSD using an interference-free matrix instead of a field sample aliquot. The LCS measures laboratory efficiency in recovering target analytes in the absence of matrix interferences. It is used to verify that the analyses are being performed in control.

The laboratory analyzes laboratory replicates. A field sample is analyzed and an unspiked duplicate of that sample is also analyzed. The data reviewer compares the reported results of the primary analysis and the laboratory duplicate and calculates RPDs to assess laboratory precision.

Calibration precision is determined by calculating %RSD. Laboratory and field sampling precision are evaluated by calculating RPDs for field sample duplicate pairs, if collected. The sampler collects two field samples at the same location and under identical conditions. The laboratory then analyzes the samples under identical conditions.

An RPD outside the allowed limit between MS/MSD samples or DUP samples indicates imprecision. Imprecision is the variance in the consistency with which the laboratory arrives at a reported result. The actual analyte concentration may be higher or lower than the reported result.

Possible causes of poor precision include sample heterogeneity, sample matrix interference, improper sample collection or handling, inconsistent sample preparation, instrument column fouling, and poor instrument stability. In duplicate pairs, results may be reported in either the primary or duplicate samples at levels below the practical quantitation limit (PQL) or non-detected. Since these values are estimated, RPD exceedances from these duplicate pairs do not suggest a significant impact to data quality.

2.2 ACCURACY

Accuracy is a measure of the closeness of agreement between a measured value and the true value of an analytical parameter. It may be used to identify bias in each measurement system. Recoveries outside acceptable QC limits may be caused by factors such as instrumentation, analyst error, or matrix interference. Accuracy is assessed through the analysis of continuing calibrations, MS, MSD, LCS, and surrogates. In some cases, samples from multiple SDGs were within one QC batch and therefore are associated with the same laboratory QC samples. Accuracy is determined using the percent recovery (%R) of MS and LCS analyses.

Percent recovery is calculated using the following equation:

$$\%R = (A-B)/C \times 100$$

where:

A = measured concentration in the spiked sample

B = measured native concentration in the unspiked sample

C = concentration of the spike

The percent recovery of each analyte spiked in MS/MSD samples and LCS is evaluated with the acceptance criteria specified by the QAPPs and laboratory limits. Spike recoveries outside the acceptable QC accuracy limits provide an indication of bias, where the reported data may overestimate or underestimate the actual concentration of compounds detected or quantitation limits reported for environmental samples.

2.3 REPRESENTATIVENESS

Representativeness is a qualitative parameter that expresses the degree to which the sample data are characteristic of a population. It is evaluated by reviewing the QC results of blanks, samples, and holding times. Positive detects of compounds in the blank samples identify compounds that may have been introduced into the samples during sample collection, transport, preparation, or analysis. The QA/QC blanks collected and analyzed are method blanks, calibration blanks, EBs, FBs, and TBs.

A method blank is a laboratory grade water or solid matrix that contains the method reagents and has undergone the same preparation and analysis as the environmental samples. The method blank provides a measure of the combined contamination derived from the laboratory source water, glassware, instruments, reagents, and sample preparation steps. Method blanks are prepared for each sample of a similar matrix extracted by the same method at a similar concentration level.

Several methods require the use of initial calibration blanks (ICBs) and continuing calibration blanks (CCBs). ICBs and CCBs are laboratory-grade water samples that are analyzed at the beginning, during, and at the end of sample analysis runs. The frequency is dependent on the analytical method. These blanks estimate residual contaminants from the previous sample or standards analysis and measure baseline shifts that commonly occur in emission and absorption spectroscopy.

EBs consist of analyte-free water poured over or through the sample collection equipment. The water is collected in a sample container for laboratory analysis. These blanks are collected after the sampling equipment is decontaminated; they are used to measure effectiveness of the decontamination procedure. Equipment blanks are collected and analyzed for all target analytes.

FBs consist of analyte-free source water stored at the sample collection site. The water is collected from each source water used during each sampling event. Field blanks were collected and analyzed for all target analytes.

TBs consist of analyte-free water prepared at the laboratory, shipped to the field with sample containers, and returned to the laboratory with the samples receiving volatile organic compound (VOC) analysis. The trip blank is analyzed for VOCs using the same sample preparation and analysis procedures used for the actual field samples.

Contaminants found in both the environmental sample and the blank sample are assumed to be laboratory artifacts if both values are less than the PQL or if a sample result and blank contaminant value are greater than the PQL and the sample result is less than 10 times the blank contaminant value.

Holding times are evaluated to assure that the sample integrity is intact for accurate sample preparation and analysis. Holding times are specific for each method and matrix analyzed. Holding time exceedance can cause loss of sample constituents due to biodegradation, precipitation, volatilization, and chemical degradation. Sample results for analyses that were performed after the method holding time are qualified according to NDEP requirements. The qualifiers and bias recommendations are taken from USEPA National Functional Guidelines (NFGs), per NDEP guidance.

2.4 COMPARABILITY

Comparability is a qualitative characteristic that defines the extent to which the data for a chemical parameter measurement are consistent with, and may be compared with, data from other sampling events. Comparability is dependent upon the design of the sampling plans and execution of activities consistent with approved plans. Factors affecting comparability include sample collection and handling techniques, matrix type, and analytical method. Comparability is achieved through the use of standard techniques to collect representative samples, consistent application of analytical method protocols, and use of appropriate units in reporting analytical results. Comparability is also dependent upon other PARCCS criteria, because only when precision, accuracy, and representativeness are known can datasets be compared with confidence.

2.5 COMPLETENESS

Completeness is defined as the percentage of acceptable sample results compared to the total number of sample results. Completeness is evaluated to determine if an acceptable amount of usable data were obtained so that a valid scientific site assessment can be completed. Completeness equals the total number of sample results for each fraction minus the total number of rejected sample results divided by the total number of sample results multiplied by 100. As specified in the project DQOs, the goal for completeness for target analytes in each analytical fraction is 90 percent.

Percent completeness is calculated using the following equation:

$$\%C = (T - R)/T \times 100$$

where:

%C = percent completeness

T = total number of sample results
R = total number of rejected sample results

Completeness is also determined by comparing the planned number of samples per method and matrix as specified in the QAPPs, with the number determined above.

2.6 SENSITIVITY

Sensitivity is the ability of an analytical method or instrument to discriminate between measurement responses representing different concentrations. It is generally used to describe the instrument detection limits (DLs) or PQLs established to meet project DQOs. The method detection limit (MDL) represents the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. Sample quantitation limits (SQLs) are adjusted MDL values that reflect sample-specific actions, such as dilutions or varying aliquot sizes. The laboratory data reports show MDL in place of the SQL. The MDL was adjusted to reflect the sample analysis conditions. The PQL is the minimum concentration that can be reported based on the analysis of a specific matrix. The PQL is often the lowest acceptable calibration point for the analyte.

For this project, the laboratory data reports show reporting limit (RL) in place of the PQL. The laboratory reported detected analytes down to the adjusted MDL/SQL. All results reported between the SQL and PQL were qualified "J" by the laboratory. Sample results are compared to method and field quality blank results to identify possible effects of laboratory background and field procedures on sensitivity.

3.0 VALIDATION RESULTS AND PARCCS

This section discusses the validation results and the associated PARCCS criteria. Before conducting the PARCCS evaluation, the analytical data were validated.

Samples not meeting the acceptance criteria were denoted with a validation qualifier that indicates a deficiency with the data. Table 3 contains validation qualifiers used in data validation.

When more than one validation qualifier is applicable to a data point, the final validation qualifier applied is based on the following hierarchy:

R > J	R takes precedence over the J qualifier.
J+	The high bias (J+) qualifier is applied to detected results only.
J > J+ or J-	The unbiased (J) qualifier supersedes biased (J+ or J-) qualifiers since it is not possible to assess the direction of the potential bias.
J = J+ plus J-	Adding biased (J+ or J-) qualifiers with opposite signs results in an unbiased qualifier (J).
UJ = U plus J	The UJ qualifier is used when a non-detected (U) flag is added to a (J) flag.

Table 4 identifies the QC elements reviewed for each validation level. The actual elements are method-dependent.

Table 5 lists the reason codes used. Reason codes explain why data were qualified and identify possible limitations of data use. Reason codes are cumulative except when one of the flags is R. In that case, only the reason code associated with the R flag is used.

Table 6 presents the overall qualified results after the validation qualifiers and associated reason codes were applied.

3.1 PRECISION

3.1.1 Instrument Calibration

The objective of the ICAL is to ensure that an instrument can produce acceptable qualitative and quantitative data by determining the ratio of instrument response to analyte concentration. %RSD is used to evaluate ICAL results in method SW-8260B and provides a means of evaluating precision within an analytical system. All %RSDs were acceptable. No data were qualified for imprecision in the ICAL.

3.1.2 MS/MSD Samples

MS/MSD RPDs were within the acceptance criteria as stated in the QAPP.

3.2 ACCURACY

3.2.1 Calibration and Continuing Calibration

As stated previously, the objective of initial calibration is to ensure that an instrument is capable of producing acceptable qualitative and quantitative data by determining the ratio of instrument response to analyte concentrations. Typically, inorganic methods use regression models for initial calibration. Regression may also be used in organic analyses. The correlation coefficient indicates the linearity of the calibration curve. The coefficient of determination is an overall measure of the accuracy of the regression calibration curve. The objective of continuing calibration is to ensure that the instrument continues to meet the sensitivity and linearity

criteria throughout each analytical sequence. Initial and continuing calibration verification (CCV) results provide a means of evaluating accuracy. Percent difference or drift (%D), percent recovery (%R), correlation coefficient, and coefficient of determination are the parameters used to measure the effectiveness of instrument calibration. %R and %D are used to verify the ongoing calibration acceptability of the analytical system.

Calibration %D and %R criteria were met.

3.2.2 MS/MSD Samples

No data were qualified for MS/MSD %Rs. Analytes that were present in the parent sample in concentrations greater than 4 times the amount spiked were not qualified.

3.2.3 LCS Samples

No data were qualified for LCS %R outliers.

3.2.4 Serial Dilutions

The serial dilution is used to determine whether physical or chemical interferences exist due to matrix. Serial dilution %Ds were less than 10 percent as required in the inorganic NFG.

3.2.5 Interference Check Samples

Interference check samples (ICS) are analyzed in the following methods: EPA 314.0 and SW-6010B. All interference check %Rs met acceptance criteria of 80 to 120 percent.

3.2.6 Surrogates

Surrogates are added to all samples analyzed by EPA 300.1B and SW-8260B to measure the efficiency of the analytical method. No data were qualified for surrogate recovery outliers.

3.2.7 Analyte Quantitation and Target Identification

Raw data were evaluated in Stage 4 validation. All analyte quantitation and target identifications reviewed matched the reported values.

3.3 REPRESENTATIVENESS

3.3.1 Sample Preservation and Holding Times

Holding times and sample preservation were evaluated to verify compliance with the analytical methods. The samples met the preservation and holding time criteria shown in the QAPP.

3.3.2 Blanks

Method blanks, ICBs, CCBs, EBs, FBS, and TBs were analyzed to evaluate representativeness. No analytes were detected in any blanks.

3.4 COMPARABILITY

The laboratory used standard analytical methods for all analyses. In all cases, the SQLs attained were at or below the PQLs. Target compounds detected below the PQLs were flagged "J" by the laboratory and should be considered estimated. One qualified result is shown with reason code "sp" in Table 6. The comparability of the data is acceptable.

3.5 COMPLETENESS

The overall completeness level attained for the field samples, EBs, FBs, and TBs is 100 percent and meets the project goal of 90 percent. The percentage was calculated as the total number of accepted (non-rejected) sample results divided by the total number of sample results multiplied by 100. Completeness by method is depicted in Table 8.

3.6 SENSITIVITY

The calibrations were evaluated for instrument sensitivity and were determined to be technically acceptable. Due to high analyte concentrations, many analytical runs were analyzed at dilutions. For diluted analyses, SQLs and PQLs were elevated.

3.6.1 Initial and Continuing Calibration

For method SW-8260B, the relative response factors (RRFs) for 1,1,2-trichloroethane and 1,2-dichloropropane in the ICAL and continuing calibration verification were less than the organic NFG requirement. The organic NFG requires an RRF of 0.200 for these compounds. It recommends rejecting the data point for RRFs < 0.200. Since method SW-8260B and the lab's operating procedure do not require a minimum RRF for 1,1,2-trichloroethane or 1,2-dichloropropane, the validator using professional judgment qualified the results "UJ." The PQLs may be inaccurate or imprecise. Ten results were qualified "UJ" and are found in Table 6 with reason code "c." The calibration outliers are found in Table 7.

3.6.2 Internal Standards

Internal standards were added to samples analyzed by methods SW-6010B and SW-8260B. In SW-6010B, internal standards were used to determine the existence and magnitude of instrument drift and physical interferences. In SW-8260B, internal standard areas and retention times were evaluated to ensure that instrument sensitivity and response remained stable during analysis. No analytes were qualified for internal standard anomalies.

4.0 CONCLUSIONS AND RECOMMENDATIONS

The analytical data quality assessment for the analytical results generated during the Vacuum Enhanced Recovery Treatability Study at the NERT site in Henderson, Nevada, established that the overall project requirements and completeness levels were met. Sample results were qualified for RRF outliers and detection between the SQL and PQL. Sample results that were qualified as estimated are usable for limited purposes only.

5.0 REFERENCES

Nevada Division of Environmental Protection (NDEP). (2018). *NDEP Data Verification and Validation Requirements*.

Ramboll Environ. (2017). *Quality Assurance Project Plan, Revision 2, Nevada Environmental Response Trust Site, Henderson, Nevada*.

Tetra Tech. (2017). *Vacuum Enhanced Recovery Treatability Study Work Plan*.

Tables

Table 1 Analytical Methods

Method	Parameters	Number of Aqueous Samples
EPA 300.1B	Chlorate and Chlorite	4
EPA 314.0	Perchlorate	4
SW-6010B	Chromium	4
SW-7199	Chromium [VI]	4
SW-8260B	Volatile Organic Compounds (VOCs)	5

Table 2 Sample Cross-Reference

SDG	Client Sample ID	Lab Sample ID	Matrix	Sample Date	Type	Validation Stage	EPA 300.1B	EPA 314.0	SW-6010B	SW-7199	SW-8260B
440-222284-1	VER-01D-20181015	440-222284-1	WG	10/15/2018	NORM	Stage 4	X	X	X	X	X
440-222284-1	VER-01I-20181015	440-222284-2	WG	10/15/2018	NORM	Stage 4	X	X	X	X	X
440-222284-1	VER-20181015-TB	440-222284-3	BW	10/15/2018	TB	Stage 4					X
440-222284-1	VER-20181015-FB	440-222284-4	BW	10/15/2018	FB	Stage 4	X	X	X	X	X
440-222284-1	VER-20181015-EB	440-222284-5	BW	10/15/2018	EB	Stage 4	X	X	X	X	X

Table 3 Validation Qualifiers and Definitions

Validation Qualifier	Definition
J-	The result is an estimated quantity, but the result may be biased low.
J	The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
J+	The result is an estimated quantity, but the result may be biased high.
NJ	The analyte has been “tentatively identified” or “presumptively” as present and the associated numerical value is the estimated concentration in the sample.
U	The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
UJ	The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
R	The data are unusable. The sample results are rejected due to serious deficiencies in meeting QC criteria. The analyte may or may not be present in the sample.

Table 4 Validation Checks and Stages

Verification and Validation Checks	Stage 2A	Stage 2B	Stage 4
Documentation identifies the laboratory receiving and conducting analyses, and includes documentation for all samples submitted by the project or requester for analyses.	X	X	X
Requested analytical methods were performed and the analysis dates are present.	X	X	X
Requested target analyte results are reported along with the original laboratory data qualifiers and data qualifier definitions for each reported result (and the uncertainty of each result and clear indication of the type of uncertainty reported if required, e.g., for radiochemical analyses).	X	X	X
Requested target analyte result units are reported (along with their associated uncertainty units if required, e.g., for radiochemical analyses).	X	X	X
Requested reporting limits for all samples are present and results at and below the requested (required) reporting limits are clearly identified (including sample detection limits if required).	X	X	X
Sampling dates (including times if needed), date and time of laboratory receipt of samples, and sample conditions upon receipt at the laboratory (including preservation, pH, and temperature) are documented.	X	X	X
For radiochemical analyses, the sample-specific critical values (sometimes called "critical level," "decision level," or "detection threshold") and sample-specific minimum detectable value, activity, or concentration for all samples are reported, and results at and below the requested (required) critical values are clearly identified.	X	X	X
For radiochemical analyses, the chemical yield (if applicable to the method) and reference date and time (especially for short lived isotopes) are reported for all samples (as appropriate).	X	X	X
Sample results are evaluated by comparing sample conditions upon receipt at the laboratory (e.g., preservation checks) and sample characteristics (e.g., percent moisture) to the requirements and guidelines present in national or regional data validation documents, analytical method(s), or contract.	X	X	X
Requested methods (handling, preparation, cleanup, and analytical) are performed.	X	X	X
Method dates (including dates, times and duration of analysis for radiation counting measurements and other methods, if needed) for handling (e.g., Toxicity Characteristic Leaching Procedure), preparation, cleanup and analysis are present, as appropriate.	X	X	X
Sample-related QC data and QC acceptance criteria (e.g., method blanks, surrogate recoveries, deuterated monitoring compound (DMC) recoveries, laboratory control sample (LCS) recoveries, duplicate analyses, matrix spike and matrix spike duplicate recoveries, serial dilutions, post digestion spikes, standard reference materials) are provided and linked to the reported field samples (including the field quality control samples such as trip and equipment blanks).	X	X	X
Requested spike analytes or compounds (e.g., surrogate, DMCs, LCS spikes, post digestion spikes) have been added, as appropriate.	X	X	X
Sample holding times (from sampling date to preparation and preparation to analysis) are evaluated.	X	X	X

Table 4 Validation Checks and Stages

Verification and Validation Checks	Stage 2A	Stage 2B	Stage 4
Frequency of QC samples is checked for appropriateness (e.g., one LCS per 20 samples in a preparation batch).	X	X	X
Sample results are evaluated by comparing holding times and sample-related QC data to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract.	X	X	X
Initial calibration data (e.g., initial calibration standards, initial calibration verification [ICV] standards, initial calibration blanks [ICBs]) are provided for all requested analytes and linked to field samples reported. For each initial calibration, the calibration type used is present along with the initial calibration equation used including any weighting factor(s) applied and the associated correlation coefficients, as appropriate. Recalculations of the standard concentrations using the initial calibration curve are present, along with their associated percent recoveries, as appropriate (e.g., if required by the project, method, or contract). For the ICV standard, the associated percent recovery (or percent difference, as appropriate) is present.		X	X
Appropriate number and concentration of initial calibration standards are present.		X	X
Continuing calibration data (e.g., continuing calibration verification [CCV] standards and continuing calibration blanks [CCBs]) are provided for all requested analytes and linked to field samples reported, as appropriate. For the CCV standard(s), the associated percent recoveries (or percent differences, as appropriate) are present.		X	X
Reported samples are bracketed by CCV standards and CCB standards as appropriate.		X	X
Method specific instrument performance checks are present as appropriate (e.g., tunes for mass spectrometry methods, DDT/Endrin breakdown checks for pesticides and aroclors, instrument blanks and interference checks for ICP methods).		X	X
Frequency of instrument QC samples is checked for appropriateness (e.g., gas chromatography-mass spectroscopy [GC-MS] tunes have been run every 12 hours).		X	X
Sample results are evaluated by comparing instrument-related QC data to the requirements and guidelines present in national or regional data validation documents, analytical method(s), or contract.		X	X
Instrument response data (e.g., GC peak areas, ICP corrected intensities) are reported for requested analytes, surrogates, internal standards, and DMCs for all requested field samples, matrix spikes, matrix spike duplicates, LCS, and method blanks, as well as calibration data and instrument QC checks (e.g., tunes, DDT/Endrin breakdowns, interelement correction factors, and Florisil cartridge checks).			X
Reported target analyte instrument responses are associated with appropriate internal standard analyte(s) for each (or selected) analyte(s) (for methods using internal standard for calibration).			X

Table 4 Validation Checks and Stages

Verification and Validation Checks	Stage 2A	Stage 2B	Stage 4
Fit and appropriateness of the initial calibration curve used or required (e.g., mean calibration factor, regression analysis [linear or non-linear, with or without weighting factors, with or without forcing]) is checked with recalculation of the initial calibration curve for each (or selected) analyte(s) from the instrument response.			X
Comparison of instrument response to the minimum response requirements for each (or selected) analyte(s)			X
Recalculation of each (or selected) opening and closing CCV (and CCB) response from the peak data reported for each (or selected) analyte(s) from the instrument response, as appropriate			X
Compliance check of recalculated opening and/or closing CCV (and CCB) response to recalculated initial calibration response for each (or selected) analyte(s)			X
Recalculation of percent ratios for each (or selected) tune from the instrument response, as appropriate			X
Compliance check of recalculated percent ratio for each (or selected) tune from the instrument response.			X
Recalculation of each (or selected) instrument performance check (e.g., DDT/Endrin breakdown for pesticide analysis, instrument blanks, interference checks) from the instrument response			X
Recalculation and compliance check of retention time windows (for chromatographic methods) for each (or selected) analyte(s) from the laboratory reported retention times			X
Recalculation of reported results for each reported (or selected) target analyte(s) from the instrument response			X
Recalculation of each (or selected) reported spike recovery (surrogate recoveries, DMC recoveries, LCS recoveries, duplicate analyses, matrix spike and matrix spike duplicate recoveries, serial dilutions, post digestion spikes, standard reference materials, etc.) from the instrument response			X
Each (or selected) sample result(s) and spike recovery(ies) are evaluated by comparing the recalculated numbers to the laboratory reported numbers according to the requirements and guidelines present in national or regional data validation documents, analytical method(s) or contract.			X
All required instrument outputs (e.g., chromatograms, mass spectra, atomic emission spectra, instrument background corrections, and interference corrections) for evaluating sample and instrument performance are present.			X
Sample results are evaluated by checking each (or selected) instrument output (e.g., chromatograms, mass spectra, atomic emission spectra data, instrument background corrections, interference corrections) for correct identification and quantitation of analytes (e.g., peak integrations, use of appropriate internal standards for quantitation, elution order of analytes, and interferences).			X
Each (or selected) instrument's output(s) is evaluated for confirmation of non-detected or tentatively identified analytes.			X

Table 5 Reason Codes

Reason Code	Description of Qualification
a	Qualified due to low abundance (radiochemical activity)
be	Qualified due to equipment blank contamination
bf	Qualified due to field blank contamination
bl	Qualified due to lab blank contamination
bt	Qualified due to trip blank contamination
bp	Qualified due to pump blank contamination (for wells without dedicated pumps)
br	Qualified due to filter blank contamination (aqueous hexavalent chromium and dissolved sample fractions)
c	Qualified due to calibration problems
cp	Qualified due to insufficient ingrowth (radiochemical only)
dc	Dual column confirmation % difference exceeded
e	Sample concentration exceeded the calibration range
fd	Qualified due to field duplicate imprecision
h	Qualified due to holding time exceedance
i	Qualified due to internal standard areas or retention times
k	Qualified as Estimated Maximum Possible Concentrations (dioxins and PCB congeners)
l	Qualified due to LCS recoveries
ld	Qualified due to lab duplicate imprecision (matrix duplicate, MSD, LCSD)
m	Qualified due to matrix spike recoveries
nb	Qualified due to negative lab blank contamination (nondetect results only)
nd	Qualified due to non-detected target analyte
o	Other
p	Qualified as a false positive due to contamination during shipping
pH	Sample preservation not within acceptance range
q	Qualified due to quantitation problem
s	Qualified due to surrogate recoveries
sd	Serial dilution did not meet control criteria
sp	Detected value reported between MDL/SQL and RL/PQL
st	Sample receipt temperature exceeded
t	Qualified due to elevated helium tracer concentrations
vh	Headspace detected in aqueous sample containers submitted for volatile analysis
x	Qualified due to low % solids
z	Qualified due to interference check sample results

Table 6 Results Qualified During Validation

SDG	Sample ID	Sample Date	Method	Total or Dissolved	Analyte	Result	Units	Lab Qualifier	SQL	PQL	Validator Qualifier	Reason Code	Reason Code Definition
440-222284-1	VER-01D-20181015	10/15/2018	SW-8260B	Total	1,1,2-Trichloroethane	0.25	ug/L	U	0.25	0.5	UJ	c	Calibration
440-222284-1	VER-01D-20181015	10/15/2018	SW-8260B	Total	1,2-Dichloropropane	0.25	ug/L	U	0.25	0.5	UJ	c	Calibration
440-222284-1	VER-01I-20181015	10/15/2018	SW-8260B	Total	1,1,2-Trichloroethane	0.25	ug/L	U	0.25	0.5	UJ	c	Calibration
440-222284-1	VER-01I-20181015	10/15/2018	SW-8260B	Total	1,2-Dichloropropane	0.25	ug/L	U	0.25	0.5	UJ	c	Calibration
440-222284-1	VER-01I-20181015	10/15/2018	SW-8260B	Total	Carbon tetrachloride	0.29	ug/L	J	0.25	0.5	J	sp	Detect < PQL
440-222284-1	VER-20181015-EB	10/15/2018	SW-8260B	Total	1,1,2-Trichloroethane	0.25	ug/L	U	0.25	0.5	UJ	c	Calibration
440-222284-1	VER-20181015-EB	10/15/2018	SW-8260B	Total	1,2-Dichloropropane	0.25	ug/L	U	0.25	0.5	UJ	c	Calibration
440-222284-1	VER-20181015-FB	10/15/2018	SW-8260B	Total	1,1,2-Trichloroethane	0.25	ug/L	U	0.25	0.5	UJ	c	Calibration
440-222284-1	VER-20181015-FB	10/15/2018	SW-8260B	Total	1,2-Dichloropropane	0.25	ug/L	U	0.25	0.5	UJ	c	Calibration
440-222284-1	VER-20181015-TB	10/15/2018	SW-8260B	Total	1,1,2-Trichloroethane	0.25	ug/L	U	0.25	0.5	UJ	c	Calibration
440-222284-1	VER-20181015-TB	10/15/2018	SW-8260B	Total	1,2-Dichloropropane	0.25	ug/L	U	0.25	0.5	UJ	c	Calibration

Table 7 Calibration Exceedances

SDG	Method	Calibration	Calibration ID	Parameter	Outlier	Value	Allowed
440-222284-1	SW-8260B	ICAL	20180	1,2-Dichloropropane	Relative Response Factor	0.1929	≥ 0.200
440-222284-1	SW-8260B	ICAL	20180	1,1,2-Trichloroethane	Relative Response Factor	0.1623	≥ 0.200
440-222284-1	SW-8260B	ICV	ICV 440-505728/18	1,1,2-Trichloroethane	Relative Response Factor	0.1835	≥ 0.200
440-222284-1	SW-8260B	CCV	CCVIS 440-506588/4	1,2-Dichloropropane	Relative Response Factor	0.1727	≥ 0.200
440-222284-1	SW-8260B	CCV	CCVIS 440-506588/4	1,1,2-Trichloroethane	Relative Response Factor	0.1366	≥ 0.200

Table 8 Completeness Summary

Method	Total Number of Validated Results	Number of Rejected Results	Percent Completeness
EPA 300.1B	8	0	100.0%
EPA 314.0	4	0	100.0%
SW-6010B	4	0	100.0%
SW-7199	4	0	100.0%
SW-8260B	345	0	100.0%

Appendix A

Validation Checklist

Data Verification and Validation Summary

Project Name: VER Treatability Study
 Task No.: M16
 No. of Samples: 7 with MS/MSD

SDG/Report No.: 440-222284-1
 Lab ID: Test America
 Matrix: Water

Area Reviewed	Anomalies		Qualification Required	Action Required
	Yes	No	Yes or No	
1. Sample Preservation, Handling, and Transport		X	No	None
2. Chain-of-Custody		X	No	None
3. Holding Times		X	No	None
4. Instrument Performance		X	No	None
5. Initial Calibration	X		Yes	All: Qualify 1,2-dichloropropane and 1,1,2-trichloroethane "UJ".
6. Continuing Calibration Verification	X		Yes	All: Qualify 1,2-dichloropropane and 1,1,2-trichloroethane "UJ".
7. Blanks		X	No	None
8. Surrogates/Monitoring Compounds		X	No	None
9. Matrix Spike/Matrix Spike Duplicate/MSI	X		No	None
10. Serial Dilution		X	No	None
11. Laboratory Control Samples		X	No	None
12. Interference Check Samples	X		No	None
13. Internal Standards		X	No	None
14. Duplicates	---	---	---	---
15. Compound Quantitation and Reporting Limits		X	Yes	Qualify all results detected between the SQL and PQL "J".
16. Calculations and Raw Data		X	No	None
17. Data Package/EDD comparison (10%)		X	No	None
Verification and Validation Label	Stage_4_Validation_Manual			
Verification and Validation Label Code	S4VM			
<p>Overall Assessment: Acceptable as qualified. Usability: Sample results qualified as estimated (UJ, J) are useable for limited purposes only. All other results are considered valid and useable for all purposes.</p>				

Data Verification and Validation Summary

Sample Information:

Field Sample Number	Lab Sample ID	Date Collected	Cooler Temperature
VER-01D-20181015	440-222284-1	10/15/2018	2.3 °C
VER-01I-20181015	440-222284-2	10/15/2018	2.3 °C
VER-01I-20181015-MS	440-222284-2 MS	10/15/2018	2.3 °C
VER-01I-20181015-MSD	440-222284-2-MSD	10/15/2018	2.3 °C
VER-20181015-TB	440-222284-3	10/15/2018	2.3 °C
VER-20181015-FB	440-222284-4	10/15/2018	2.3 °C
VER-20181015-EB	440-222284-5	10/15/2018	2.3 °C

The following section is intended to specify areas evaluated and issues encountered. Only applicable methods are listed.

1. Sample Preservation, Handling, and Transport	
Were all samples preserved correctly? Were sample temperatures kept at 4°C (+ or – 2°C)? Were samples received in proper condition?	Yes/Yes/Yes

2. Chain-of-Custody (COC)	
Were samples recorded on the COCs? Were correct analyses performed on the samples?	Yes/Yes

3. Holding Times	
Were samples analyzed within acceptable holding times?	Yes

4. Instrument Performance	
Was BFB analyzed before and within 12 hours of sample analysis? Were mass assignments correct and normalized to m/z 95? Were ion abundance criteria met?	Yes/Yes/Yes

5. Initial Calibration (ICAL)	
Were the correct number of standards analyzed to establish the calibration curve for each analyte? Were Percent Relative Standard Deviations (%RSDs) of the Response Factors (RFs) ≤ method or national functional guideline (NFG) requirements or Coefficient of Correlation or Coefficient of Determination ≥ method or NFG requirements? Were Relative Response Factors (RRFs) and average RRFs ≥ method or NFG requirements?	Yes/Yes/No
8260B: ICAL 20180: 1,2-Dichloropropane RRF = 0.1929. NFG requires ≥ 0.200; 1,1,2-Trichloroethane RRF = 0.1623. NFG requires ≥ 0.200. ICV 440-505728/18: 1,1,2-Trichloroethane RRF = 0.1835. NFG requires ≥ 0.200. Neither method 8260B nor the lab's SOP require a minimum RRF for these compounds, so data will not be rejected.	

Data Verification and Validation Summary

6. Continuing Calibration Verification (CCV)	
Were CCVs analyzed at the beginning and end of sample analysis, if applicable? Were calibrations compared to the correct initial calibrations? Were Percent Differences (%D) \leq method or NFG requirements? Did RRFs and average RRFs meet method or NFG requirements?	Yes/Yes/Yes/No
8260B: <i>CCVIS 440-506588/4:</i> 1,2-Dichloropropane RRF = 0.1727. NFG requires \geq 0.200; 1,1,2-Trichloroethane RRF = 0.1366. NFG requires \geq 0.200. Neither method 8260B nor the lab's SOP require a minimum RRF for these compounds, so data will not be rejected.	

7. Blanks	
Does data package include a summary of blank results? Was a method blank extracted and/or analyzed for each batch? Were calibration blanks analyzed at appropriate intervals? Were analytes detected in any blanks?	Yes/Yes/Yes/No

8. Surrogates/Monitoring Compounds	
Were samples spiked with the correct surrogate compounds? Were surrogate recoveries reported on data forms? Were recoveries within laboratory limits?	Yes/Yes/Yes

9. Matrix Spike/Matrix Spike Duplicate/MSI	
Was a MS/MSD pair or MSI extracted and/or analyzed with each batch? Were recoveries/RPDs reported correctly on data forms? Were recoveries/RPDs within laboratory established limits?	Yes/Yes/No
300.1B: Chlorate recoveries were high in the MS/MSD of VER-01I-20181015. The concentration in the parent sample is $>4x$ the amount spiked, so recovery criteria do not apply. No qualification.	
314.0: Perchlorate recoveries were low in the MS/MSD of VER-01I-20181015. The concentration in the parent sample is $>4x$ the amount spiked, so recovery criteria do not apply. No qualification.	
8260B: Chloroform recovery was low in the MSD of VER-01I-20181015. The concentration in the parent sample is $>4x$ the amount spiked, so recovery criteria do not apply. No qualification.	

10. Serial Dilution	
Were serial dilutions analyzed at appropriate intervals? For results $> 50x$ the MDL, were %Ds within acceptable limits of the true value?	Yes/Yes

11. Laboratory Control Samples (LCS)	
Was a LCS analyzed with each analytical batch? Were LCS recoveries reported correctly on data forms? Were LCS recoveries within laboratory established limits?	Yes/Yes/Yes

12. Interference Check Sample (ICS)	
Were interference check samples (ICS) analyzed at appropriate intervals? Were ICS recoveries within acceptable limits of the true value? Were ICSA samples non-detect for analytes not in the solution?	Yes/Yes/No
6010B: Chromium was detected above the MDL. Interferents concentrations in the samples (from raw data) are not comparable to the concentrations in the ICSA. They are too low. No qualification.	

Data Verification and Validation Summary

13. Internal Standards (IS)	
Were ISs added to each sample in the run including calibrations, samples, and QC samples? Were area counts or Percent Relative Intensities within the acceptance range for the method? If applicable, was the Retention Time of the IS within ± 30 seconds from the RT of the IS in the associated CCV or mid-point standard from ICAL?	Yes/Yes/Yes

14. Duplicates	
Were any duplicate pairs analyzed in this SDG? For results $> 5x$ the RL, were RPDs between parent sample and duplicates \leq lab limits or $\leq 30\%$ for field duplicates? For REG/FD results $< 5x$ the RL, were differences between the two values $< RL$.	No/N/A/N/A

15. Compound Quantitation and Reporting Limits	
Were sample quantitation limits and practical quantitation limits adjusted to reflect dilutions, cleanup, and other factors? If applicable, were reporting limit check recoveries within acceptable limits?	Yes/Yes

16. Calculations and Raw Data	
Did calculated results and raw data match the reported data?	Yes
There were slight differences due to rounding.	

17. Data Package/EDD comparison (10%)	
Were 10% of the data package results compared to the electronic data? Did results match?	Yes/Yes

Validated by: Maureen McMyler 11/1/18

Appendix B

Laboratory Data Package

Due to the quantity and size of the files, the laboratory data package is being sent in a separate file.

Appendix C

DVSR Electronic Data Deliverables

Per the requirements provided by NDEP for Unified Chemical Electronic Data Deliverable Format (July 13, 2018), databases are provided in Microsoft Access format and include location, analytical and groundwater gauging data supporting the DVSR and for upload of the Companies' electronic data into the regional database maintained by NDEP. These databases are being sent in a separate file for electronic download.