STANDARD OPERATING PROCEDURE NO. 28.0

ANALYTICAL DATA VALIDATION

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1.0 PURPOSE AND SCOPE

This standard operating procedure (SOP) describes procedures to be used to conduct an independent review of environmental analytical laboratory data so that data of known and documented quality will be used for all reporting and environmental decision making at the Powertech Mine

This SOP includes two levels of data review, evaluation of sample-specific parameters and evaluation of laboratory performance parameters. All data generated for the Powertech Mine will receive an evaluation of sample-specific parameters. In addition, 10% of the data packages received (per analysis type per sampling event/episode) containing data will also receive a review of laboratory performance parameters.

This SOP addresses the protocols that will be followed for the sample-specific parameters and laboratory performance parameters data review levels. The review of sample-specific parameters is described in Section 4.1. The review of laboratory performance parameters is discussed in Section 4.2. In addition, Section 5 discusses the associated documentation.

2.0 RESPONSIBILITIES AND QUALIFICATIONS

The Project Manager or QA Manager has the overall responsibility for implementing this SOP. They will be responsible for assigning appropriate environmental staff to implement this SOP and for ensuring that the procedures are followed.

All personnel performing these procedures are required to be familiar with environmental data, its generation, and its reporting. In addition, all personnel are required to have a complete understanding of the procedures described within this SOP. Activity-specific training regarding these procedures will be provided by the QA Manager or designee to personnel implementing this SOP, as necessary.

All environmental staff are responsible for reporting deviations from this SOP to the Project Manager or QA Manager.

3.0 LIST OF RELATED SOPs

There are no other SOPs that are directly related to this SOP.

4.0 DATA REVIEW PROCEDURES

As noted in Section 1.0, all analytical data used for reporting and environmental decision making at Powertech's Mine will receive a review independent of the laboratory to ensure that data are of known and documented quality.

The review of sample-specific parameters includes evaluating parameters that are sample related. These include: case narrative comments, chain-of-custody and sample condition upon receipt, holding times, method blank results, surrogate recoveries, matrix spike recoveries, laboratory duplicate or spike duplicate analysis, post-digestion spike recoveries, ICP serial dilution analysis agreement, internal standard performance, and results for field quality control samples (e.g. field

duplicates, rinsate blanks, field blanks, and trip blanks). The sample-specific review is described in Section 4.1. Sample-specific parameters will be reviewed and evaluated for all data.

The review of laboratory performance parameters includes evaluating operations that are in the control of the laboratory, but are independent of the field samples being analyzed. These include: initial calibration, initial and continuing calibration verification, laboratory control sample analysis, compound identification, result calculation (i.e., quantitation), data transcription (i.e.,verification), and method specific quality control requirements (e.g. thermal stability, tuning, resolution, mass calibration, interference check sample analysis). Evaluation of these parameters provides an assessment of overall system performance. The review of laboratory performance parameters is discussed in Section 4.2. Laboratory performance parameters will be reviewed for at least 10% of the data packages (per method per sampling event) received.

During the data review process, data validation qualifiers, as defined in Table 1, will be assigned to the results, as necessary, to indicate any potential limitation on the use of the data. In addition, data qualifier codes and bias codes as defined in Table 2 will be added to the results to indicate the reason(s) for qualification and the associated bias direction, if discernable. Data validation narratives will be generated which document the results of all data review activities, all data qualification assigned, and any limitations on the use of the data.

4.1 REVIEW OF SAMPLE-SPECIFIC CRITERIA

The review of sample-specific criteria includes evaluating parameters that are sample related. Each of the subsections below describes how each parameter is evaluated. While most parameters to evaluate are pertinent to all methods, some are method specific (e.g. see Section 4.1.6). In general, the hierarchy for acceptance criteria used to evaluate each parameter is as follows:

- Method specified acceptance criteria.
- Acceptance ranges based on laboratory historical data.

According to this hierarchy, a parameter is first evaluated against the requirements set forth in the quality assurance plan. If the criteria are not specified in the quality assurance plan, then the parameter is evaluated against the requirements stated in the analytical method. If the method does not specify acceptance criteria, results for the parameter are compared to acceptance ranges based on laboratory historical data.

No recalculation of results from the raw data or transcription error checking will be performed during the review of the sample-specific criteria as recalculation and transcription error checking is completed during the review of laboratory performance criteria.

4.1.1 Case Narrative Comments

The data validation process begins with an examination of the case narrative. Any analytical problems noted in the case narrative are noted in the data validation narrative along with a summary of the effect on the usability of the data.

4.1.2 Chain-of-Custody and Sample Receipt

The chain of custody (COC) documentation, sample receipt, and log-in information are reviewed. The analytical results received are compared against those requested on the COC form. Any COC problems or discrepancies and any problems noted by the laboratory with regard to sample condition upon receipt are noted in the data validation narrative along with a statement of the effect on the usability of the data.

4.1.3 Holding Times

Collection-to-analysis holding times are calculated by computing the difference between the sample collection date and the sample analysis date. The collection dates are found on the COC and analysis dates are reported on the analysis run logs. The holding times are compared to the acceptance limits contained in respective analytical methods, as applicable. Results for analyses not performed within holding time limits will be qualified as estimated ("J/UJ"). If the holding time is grossly exceeded (more than two times the holding time limit), the data reviewer should use professional judgement to evaluate the need to reject non-detectable results.

A qualifier code of "HT" will be assigned to all results qualified or rejected on the basis of holding times.

4.1.4 Method Blank (Preparation Blank)

The results for method blanks and calibration blanks will be reviewed. Sample results for analytes detected in an associated blank at concentrations <5x the equivalent blank concentration will be qualified as nondetect ("U"). For the common organic laboratory contaminants, acetone, methylene chloride, 2-butanone, cyclohexane, and phthalates, sample results <10x the concentration in the associated blank will be qualified as nondetect ("U"). The result will be qualified as nondetect at the reported concentration if the reported concentration is greater than the reporting limit (>RL) or as nondetect ("U") at the reporting limit if the reported concentration is <RL

If reported, negative blank concentrations will be evaluated for potential effects (low bias) on sample data when the absolute value of the negative concentration is >RL. If the negative concentration in a blank may potentially have produced more than a 25% effect on a reported sample result or sample reporting limit, the associated sample result will be qualified as estimated ("J/UJ"). For example, if the associated blank result is –2 mg/l, the RL is 1 mg/l, and the associated sample result is 5 mg/l, the sample result will be qualified as estimated because a potential low bias of 2 mg/l represents 40% of the reported concentration and the absolute value of the blank concentration is >RL.

Continuing calibration blank samples are considered to be associated with all samples back to the previously analyzed continuing calibration blank sample and up to the next continuing calibration blank sample in the analytical run.

A qualifier code of "MB" or "CCB" will be assigned to all results qualified on the basis of method blank or continuing calibration blank results, respectively. For results qualified as nondetect, the bias direction is considered to be indeterminate as the reporting limit is adjusted accordingly. For results qualified as estimated on the basis of blank results, the bias direction is low.

4.1.5 Matrix-Dependent Quality Control

Matrix dependent quality control (QC) samples are used to evaluate how the sample matrix affects the accuracy and precision of the analytical results.

In order to evaluate how the site-specific sample matrix affects the accuracy of the analysis; the laboratory will spike one or two additional aliquots of a field sample with known amounts of target analytes and prepare the spiked samples in a fashion identical to that of the field samples. The amount of each spiked analyte recovered can be used to infer the accuracy of the analysis on the site-specific sample matrix.

To assess the precision of the analysis on the site-specific sample matrix, a laboratory duplicate or spike duplicate sample is prepared. A laboratory duplicate sample is a laboratory split of a homogenized environmental sample that is prepared and analyzed in a manner identical to that of the original sample. A matrix spike duplicate is similar with the exception that both aliquots are spiked with known amounts of target analytes. The closeness of the agreement between the two results can be used to infer the precision of the analysis on the site-specific sample matrix.

For inorganic methods, one aliquot is typically spiked and for organic methods, two aliquots are typically spiked. For inorganic methods, a duplicate sample is typically used to assess precision whereas for organic methods, a spiked duplicate is typically used. These conventions were developed based on the probability of finding the target analytes in the sample matrix. However, some laboratories choose to do matrix spike and matrix spike duplicates for some of their inorganic analyses.

The subsections below describe how the results for matrix QC samples will be evaluated.

4.1.5.1 Matrix Spike (MS) Analysis

The matrix spike results, expressed as percent recovery of the spiked analytes, are used to assess effects of the general sample matrix on the accuracy of the analysis.

The matrix spike recoveries are compared to the appropriate acceptance ranges per Section 4.1 for instances in which the native sample concentration was less than four times the spike level. When sample concentrations of an analyte are ≥ four times the spiking concentration, the results are considered to be inappropriate for assessing accuracy. The reviewer should also be aware that a matrix spike recovery may be outside acceptance limits when the parent sample was quantified by method of standard additions but the matrix spike was not. In such a case, the matrix spike recovery is not an appropriate measure of accuracy. Data associated with matrix spike recoveries that are outside the acceptance range will be qualified as follows using guidance from Functional Guidelines.

- If the recovery of an inorganic matrix spike analyte exceeds the upper limit of the acceptance range, suggesting a potential high bias in sample results, positive results for that target analyte in all associated samples are qualified as estimated ("J"); whereas, nondetect results for that analyte are considered to be acceptable for use without qualification.
- If the recovery of an inorganic matrix spike analyte is below the lower limit of the acceptance range, but ≥30% (≥10% for organics), suggesting a potential low bias in sample results, both positive and nondetect results for that analyte in all associated samples for

inorganic methods or only the parent sample for organics methods are qualified as estimated ("J/UJ").

- If the matrix spike recovery for an inorganic analyte was <30% (<10% for organics), nondetect results are qualified as unusable ("R") and positive results are qualified as estimated ("J") per Functional Guidelines guidance.

If a matrix spike duplicate is also prepared, the reviewer must use professional judgement and consider the recoveries for both the matrix spike sample and the matrix spike duplicate sample prior to assigning data qualifiers for inorganic data. All instances in which professional judgement is used to assign data qualifiers will be detailed in the individual data review narratives.

The reviewer should note that for organic data, no qualification of associated samples in the batch or data package will be performed on the basis of matrix spike recoveries alone. The data reviewer should use professional judgement and consider the results of other QC measures such as surrogate recoveries in conjunction with MS/MSD results to determine the need for extending qualification for the affected analytes to the other associated samples.

A qualifier code of "MS" will be assigned to all results qualified as estimated or unusable (rejected) on the basis of matrix spike and/or matrix spike duplicate recoveries. The assigned bias code will reflect the inferred bias direction.

4.1.5.2 Laboratory Duplicate (LD) Sample Analysis

The duplicate and spike duplicate sample analysis results are used to evaluate the precision of the laboratory analyses. Laboratory duplicate or spike duplicate results are evaluated using concentration dependent evaluation criteria.

- When both results are > 5x RL, compare the relative percent difference (RPD) between the sample results to a criterion of ≤20% for aqueous samples, ≤35% for soil and sediment samples, and ≤50% for biota samples.
- If either sample concentration is ≤ 5x RL, compare the absolute difference between the results to a criterion of ≤1x the greater RL for aqueous samples, ≤2x the greater RL for soil and sediment samples, and ≤4x the greater RL for biota samples.

All evaluations are done using the higher RL and the RL is used in calculating the absolute difference for a nondetect result. If the applicable duplicate evaluation criterion is not met for an analyte, all associated sample data for that analyte will be qualified as estimated ("J/UJ").

A qualifier code of "D" will be assigned to all results qualified on the basis of laboratory duplicate or spike duplicate results. A bias direction of indeterminate will be assigned to results qualified on the basis of duplicate results.

4.1.6 Method-Specific Quality Control Measures

The individual methods include method-specific QC measures. The procedures used to evaluate the results obtained for method specific quality control measures are described below. Section 4.1.6.1 describes method specific QC measure for inorganic methods and Section 4.1.6.2 describes methods specific QC measures for organic methods.

4.1.6.1 Inorganic Method Specific QC Measures

For inorganic methods, method specific QC measures may include post-digestion spikes, serial dilution tests, internal standard performance, and cation/anion balance calculation. Evaluation procedures for each of these QC measures are described below.

4.1.6.1.1 Post Digestion Spike Recovery

The analyte recoveries obtained for post-digestion spike analyses will be compared to the appropriate acceptance ranges per Section 4.1. Under some circumstances, laboratories will quantify results by the method of standard additions to compensate for low post-digestion spike recovery. In such a case, the low post-digestion spike recovery would not indicate poor accuracy. However, if the result for the sample on which the post-digestion spike analysis was performed was not obtained by the method of standard additions and the post-digestion spike recovery is outside of the acceptance limits, qualify the result for the sample on which the post-digestion spike was run based on the following guidance:

- If the recovery is > the upper acceptance limit, detectable results are qualified as estimated ("J"). No action needs to be taken for non-detects.
- If the recovery is < the lower acceptance limit, but ≥ 30%, detectable and non-detectable results are qualified as estimated ("J/UJ").
- If the recovery is <30%, detectable results are qualified as estimated ("J") and non-detectable results are qualified as unusable ("R").

The data reviewer should use professional judgement in conjunction with other QC sample results, such as matrix spike recoveries, to determine the need for qualification of results for other samples (if any) associated with the post-digestion spike analysis.

A qualifier code of "PDS" will be assigned to all results qualified or rejected on the basis of post-digestion recoveries and the assigned bias code will reflect the inferred bias direction.

4.1.6.1.2 Serial Dilution Test

ICP serial dilutions are run to help evaluate whether or not significant physical or chemical interferences exist due to sample matrix. Serial dilution analyses are typically conducted at a frequency of 1/20 samples (one analysis per metals data package). When analyte concentrations are sufficiently high (the concentration in the original sample is minimally a factor of 50 above the instrument detection limit [IDL] or method detection limit [MDL]), the results obtained for a five fold-dilution of the original sample are compared to the original results by means of a percent difference (%D). The %D is compared to a precision acceptance limit of ±10%. If the absolute value of the %D between the diluted and original result is >10%, all results for that analyte in that sample batch are qualified as estimated ("J/UJ").

Generally, the diluted result can be considered to be the more accurate result, as long as the diluted concentration is well above the detection limit. Therefore, the data reviewer can generally discern a potential bias direction from a comparison of the diluted and undiluted results. For example, if the diluted result is higher than the original result, the bias direction (associated with the original result) is considered to be potentially low.

A qualifier code of "DL" will be assigned to all results qualified on the basis of serial dilution results along with an appropriate bias code.

4.1.6.1.3 Internal Standards

Internal standards are used routinely in the analysis for metals by ICP-MS; however, internal standards may be used in the analysis of metals by ICP-ES. Internal standard recoveries for every sample and standard (as the requested level of reporting permits evaluation) will be compared to an acceptance range of 30-00%. Results associated with internal standard recoveries outside the acceptance range where the sample was not diluted and reanalyzed will be qualified as estimated (J/UJ). If upon reanalysis the internal standard recoveries are still outside the acceptance range, the results will be qualified as estimated (J/UJ).

4.1.6.1.4 Anion/Cation Balance

Because water is generally electrically neutral, the sum of the dissolved cation concentrations (expressed in milli-equivalents per liter) should equal the sum of the dissolved anion concentrations. For projects in which the major cations and anions are being analyzed, the data reviewer may evaluate whether there is an acceptable balance between anion concentrations and cation concentrations. It should be noted that both major cations and anions must be analyzed to complete the anion/cation balance. In accordance with Standard Methods #1030F, the equation used to calculate anion-cation balances is:

$$percent\ difference = 100\% \times (\Sigma cations - \Sigma anions) / (\Sigma cations + \Sigma anions)$$

Laboratory accuracy control limits for these types of analytes are typically ±30%. This level of accuracy is considered to be fully acceptable in meeting the end use objectives of groundwater monitoring. A 30% bias in the metals analysis corresponds to an anion-cation balance percent difference of approximately 13%. Therefore, since a 30% bias is considered not to adversely affect the usability of the data, an evaluation criterion of a percent difference less than ± 13% will be utilized for anion-cation balance evaluation. If the anion/cation balance is greater than ±13% the data reviewer should use professional judgement to discern likely causes of the imbalance and need for qualification of data.

4.1.6.2 Organic Method Specific QC Measures

For organic methods, method specific QC measures may include surrogate compound recovery and internal standard performance. Evaluation procedures for each of these QC measures are described below.

4.1.6.2.1 Surrogate Spike Compound Recovery

The surrogate recoveries obtained for each sample analysis for which surrogates were analyzed will be compared to the acceptance range specified in the QUALITY ASSURANCE PLAN, method, or that provided by the laboratory, as appropriate (per Section 4.1). Results for analytes in the sample associated with surrogate recoveries outside the acceptance range will be qualified as follows:

- If the surrogate recovery is greater than the upper acceptance limit for any surrogate (for semivolatile organics by GC/MS, two or more surrogates in either fraction must be high), suggesting a potential high bias in reported results, all positive results for associated analytes in that sample are qualified as estimated ("J") whereas non-detect results are considered to be acceptable for use without qualification.
- If the surrogate recovery is < the lower acceptance limit but ≥10% (for semivolatile organics by GC/MS, two or more surrogates in either fraction are out with at least one of them being less than the lower limit but ≥10%), suggesting a potential low bias in reported results, positive and nondetect results for associated analytes in that sample are qualified as estimated ("J" or "UJ").
- If any surrogate recovery is <10%, positive results for associated analytes in that sample are qualified as estimated ("J") whereas associated non-detect results are qualified as unusable ("R").

It is important to note that professional judgement may be utilized in assigning data qualification especially for methods in which more that one surrogate compound is used or in which there may have been multiple reasons for qualification on an individual result, or there may have been multiple analyses of the same sample. The data review narrative will detail any instance in which professional judgement was used.

A qualifier code of "SUR" will be assigned to all results qualified or rejected on the basis of surrogate recoveries. An appropriate bias code will be assigned.

4.1.6.2.2 Internal Standards

The site wide QUALITY ASSURANCE PLAN and/or analytical method, as appropriate (per Section 4.1) will be used to determine the QC acceptance criteria for internal standard area counts for GC/MS organic analysis. Internal standard area counts are not a direct measure of the accuracy of the analysis. Low internal standard area counts for sample analysis relative to those observed in the associated continuing calibration analysis may be indicative of low extraction or purging efficiency which decreases the analysis sensitivity (raises the detection limit). High internal standard area counts may be indicative of co-eluting interferences at the retention time of the internal standard in the sample, may be caused by a drift in detector sensitivity, or may be caused by injection of a different amount of sample extract. Co-eluting interferences to the internal standard may result in a low bias in reported results quantified by the given internal standard. Injection of a larger volume of extract would result in increased sensitivity of the analysis (lowered detection limit).

- If data validation indicates that internal standard area counts are below the lower acceptance limit, then results reported as not-detected shall be qualified as estimated ("UJ") and results

reported as detected will not require qualification since the calculation corrects for reduced extraction efficiency.

- If data validation indicates that internal standard area counts are above the upper acceptance limit, then results reported as detected or as not-detected shall be qualified as estimated ("J/UJ").

A qualifier code of "IS" will be assigned to all results qualified on the basis of internal standard area counts.

4.1.6.3 Balance of Total versus Partial Analyses

Results for the total analysis of a particular analyte should be greater than the results for a partial analysis of that analyte. For example, the results for total metals should be greater than or equal to the results for dissolved metals and ammonia concentrations should not be greater than Total Kjeldahl Nitrogen (TKN) concentrations. Because all results are limited by the accuracy of the analysis, the criteria for accuracy of the analysis are used as the basis for criteria to evaluate the agreement between the results for the partial analysis and the total portion.

- In instances where the value for a partial analysis exceeds that for a total analysis and both of the results are >5xRL, the criterion utilized is that the two values should agree within ±30%. For example, the partial analysis result should not be more than 30% higher than the total analysis result.
- In instances where the value for a partial analysis exceeds that for a total analysis and either of the results is ≤5xRL, the absolute difference between the results is compared against an evaluation criterion of ± 2x RL.

All evaluations are done using the higher RL and the RL is used for calculating the absolute difference for nondetect results. If the results for the partial versus total analyses do not satisfy the appropriate evaluation criterion, when the result for partial analysis was greater than that for the total analysis, the reviewer should use professional judgment to discern the probable cause and need for qualification of the data.

A qualifier code of "TvP" will be assigned to results qualified as estimated based on the comparison of the results for a total analysis and its corresponding partial analysis.

4.1.7 Field Quality Control Samples

Field QC samples include field duplicate samples, rinsate blank samples, field blank samples, and trip blank samples.

4.1.7.1 Field Duplicate Result Agreement

Field duplicate samples may be collected in order to assess the overall precision of the analyses (analytical and sampling precision) and/or the representativeness of the samples to the medium sampled. Criteria for evaluating field duplicate results are not provided in the Functional Guidelines. Therefore, analytical results obtained for field duplicate sample pairs are compared to each other using the concentration dependent criteria described below.

- When both the sample and duplicate values are >5xRL, acceptable sampling and analytical precision is indicated by an RPD between the results of ≤30% (≤50% for soil samples).
- Where the result for one or both analytes of the field duplicate pair is <5xRL, satisfactory precision is indicated if the absolute difference between the field duplicate results is <2xRL (<3.5xRL for soil samples).

All evaluations are done using the higher RL and the RL is used for calculating the absolute difference for nondetect results. If the above criteria are not met for an analyte, all associated sample data for that analyte should be qualified as estimated ("J/UJ").

A qualifier code of "FD" will be assigned to results qualified as estimated on the basis of field duplicate agreement.

4.1.7.2 Rinsate Blank Results

The results for rinsate blanks reported in the data package will be reviewed. Sample results for analytes detected in an associated rinsate blank at concentrations <5x the equivalent blank concentration (<10x for common laboratory contaminants) will be qualified as nondetect ("U"). The result will be qualified as nondetect at the reported concentration if the reported concentration is >RL or as nondetect ("U") at the RL if the reported concentration is <RL. For aqueous blanks applied to soil/sediment samples, qualification is assigned based on comparison of the sample result to the equivalent concentration in the blank. The equivalent concentration is determined by assuming that all of the analyte present in the blank aliquot analyzed is present in the soil sample aliquot analyzed. The reviewer should note that the blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the 5x or 10x criterion, such that a comparison of the total contamination is actually made.

A qualifier code of "RB" will be assigned to all results qualified on the basis of rinsate blank results. A bias code of indeterminate will be assigned.

4.1.7.3 Field Blank Results

The results for field blanks reported in the data package will be reviewed. Sample results for analytes detected in an associated field blank at concentrations <5x the equivalent blank concentration (<10x for common laboratory contaminants) will be qualified as nondetect ("U"). The result will be qualified as nondetect at the reported concentration if the reported concentration is >RL or as nondetect ("U") at the RL if the reported concentration is <RL. For aqueous blanks applied to soil/sediment samples, qualification is assigned based on comparison of the sample result to the equivalent concentration in the blank. The equivalent concentration is determined by assuming that all of the analyte present in the blank aliquot analyzed is present in the soil sample aliquot analyzed. The reviewer should note that the blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the 5x or 10x criterion, such that a comparison of the total contamination is actually made.

A qualifier code of "FB" will be assigned to all results qualified on the basis of rinsate blank results. A bias code of indeterminate will be assigned.

4.1.7.4 Trip Blank Results

The results for trip blanks reported in the data package will be reviewed. Sample results for analytes detected in an associated trip blank at concentrations <5x the equivalent blank concentration (<10x for common laboratory contaminants) will be qualified as nondetect ("U"). The result will be qualified as nondetect at the reported concentration if the reported concentration is >RL or as nondetect at the RL if the reported concentration is <RL.

For aqueous blanks applied to soil/sediment samples, qualification is assigned based on comparison of the sample result to the equivalent concentration in the blank. The equivalent concentration is determined by assuming that all of the analyte present in the blank aliquot analyzed is present in the soil sample aliquot analyzed. The reviewer should note that the blank analyses may not involve the same weights, volumes, or dilution factors as the associated samples. These factors must be taken into consideration when applying the 5x or 10x criterion, such that a comparison of the total contamination is actually made.

A qualifier code of "TB" will be assigned to all results qualified on the basis of rinsate blank results. A bias code of indeterminate will be assigned.

4.1.8 Reporting Limits

For the contracted laboratories are reporting positive results below their standard reporting limits (RLs) when the values are greater than the instrument detection limit (IDL) or method detection limit (MDL). These detection and/or reporting levels and associated degree of uncertainty are discussed below.

The MDL is defined in 40CFR136, Appendix B as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The MDL is determined from the analysis of spiked samples containing the analyte in a given matrix. MDLs are preparation- and method-specific. The MDL is calculated by multiplying the standard deviation of the measurements by the student t-value for seven replicate analyses (i.e., 3.14).

Inorganic results are reported down to the instrument detection limit (IDL). An IDL determined by analyzing seven replicates of an undigested, spiked, clean sample matrix on 3 non-consecutive days. For each day, the standard deviation of the readings is calculated. The standard deviations are then averaged and multiplied by 3. Thus, the IDL represents the concentration level necessary to produce a signal greater than 3 times the average standard deviations of the mean noise level for the 21 sample analyses.

At the MDL or IDL, results may have a high degree of uncertainty in the actual concentration (often more than 100%). Results reported as detected at the IDL may also have about a 50% chance of being nondetects (i.e. false positives meaning that the true sample concentrations are less than the IDL or MDL).

RLs or Practical Quantitation Limits (PQLs) are typically set at some factor above the IDL or MDL to ensure greater confidence in the accuracy of the associated quantitative value. Thus, at the RL (or PQL), a value typically set at 3-10 times the IDL or MDL, the degree of uncertainty would be more like +/- 25%. Thus, the PQL is the smallest concentration of the analyte that can be reported with a specific degree of confidence (i.e., the low concentration point of the calibration curve is less than or

equal to the RL/PQL). When the RL/PQL is adjusted for sample weight, percent moisture, and dilution factor for individual samples, the result is a sample-specific quantitation limit or SQL.

To reflect the higher degree of uncertainty associated with values reported between the IDL/MDL and RL/PQL, these results are qualified as estimated ("J"). A qualifier code of SQL, denoting sample quantitation limit, is assigned to results qualified for this reason. A bias direction of indeterminate is assigned.

4.1.9 Other Items Identified in the Case Narrative

If an issue identified in the case narrative is not covered by the subsections above and is found to potentially adversely affect data quality, the data reviewer shall evaluate the problem based on the quality assurance plan and/or method requirements, as applicable. If the quality assurance plan and/or analytical method does not specify requirements related to the criterion under evaluation, the data reviewer should utilize professional judgement to evaluate the effect of the reported item or condition on the associated analytical data. All uses of professional judgement shall be described in the report of the data validation process.

4.1.10 Completeness of the Data Package

The analytical data packages are evaluated for completeness of deliverables against the following criteria:

- Presence of tabulated results for all specified compounds identified and quantified and RLs for all analytes.
- Presence of results for all methods requested on the COC forms for each sample.
- Presence of a case narrative, COC forms, and the sample receiving forms.
- Presence of: QC summary forms for blank results; QC summary forms for MS results with calculated percent recoveries; QC summary forms for post-digestion spike recoveries (as required) with calculated percent recoveries; QC summary forms for laboratory duplicates and/or spike duplicate results and calculated RPDs; QC summary forms for serial dilution test with calculated %Ds; and QC summary forms for LCS sample results with calculated percent recoveries.
- When full data packages are requested, the package will also be reviewed for QC summary forms for initial and continuing calibration verification as well as supporting raw data for all of the aforementioned items and any pertinent review parameter discussed in Section 4.2.

Data package deliverables that do not meet the above criteria are documented, and the missing deliverables will be requested from the contracted laboratory. Any documents not obtainable from the laboratory are noted in the data review narrative.

4.2 REVIEW OF LABORATORY PERFORMANCE PARAMETERS

The review of laboratory performance parameters includes evaluating operations that are in the control of the laboratory, but are independent of the field samples being analyzed. Evaluation of these parameters provides an overall representation of the analytical system at the time of analysis. For laboratory performance parameters will be reviewed for 10% of the data packages

received per analysis type per sampling event/episode. If review of any of the laboratory performance parameters indicates a systematic problem may exist, that review parameter will be evaluated for all data packages from that laboratory for that sampling event/episode.

Each of the subsections below describes in general how each laboratory performance parameter is evaluated. As noted in the introduction to Section 4, the hierarchy for criteria used to evaluate each parameter is as follows. A parameter is first evaluated against the requirements set forth in the quality assurance plan. If the quality assurance plan addresses that parameter, the parameter is evaluated against the requirements stated in the analytical method. If the method does not specify acceptance criteria, results for the parameter are compared to acceptance ranges based on laboratory historical data.

While conducting the review described below, the data reviewer will evaluate whether the case narrative adequately summarizes all issues potentially affecting data quality (i.e., is the case narrative a reliable indicator of potential problems within the entire data package?). This assessment will be used to determine the need to evaluate specific laboratory performance parameters for the entire data set rather than just the predetermined portion of the data set (i.e., 10%).

4.2.1 Initial Calibration

The requirements set forth in the quality assurance method, as applicable, will be used to evaluate whether:

- The initial calibration was performed at the required frequency using the proper number of standards at the proper concentrations,
- Whether the RL or CRQL is supported by the low point standard,
- Whether adequate response was obtained for each analyte for each standard,
- Whether the applicable linearity criteria were met, and
- Whether the initial calibration was verified properly.

If the initial calibration evaluation criteria for any analyte are not satisfied, then all results for that analyte associated with the initial calibration will be qualified as estimated ("J/UJ"). A qualifier code of "ICAL" or "ICV" will be used depending on whether the condition was due to the initial calibration or verification of the initial calibration. If the data reviewer can discern a probable magnitude and/or direction of bias to the associated sample results based on the information provided, then appropriate qualifier bias codes will be assigned.

4.2.2 Continuing Calibration Verification

The requirements set forth in the quality assurance plan and/or method, as applicable, will be used to evaluate whether:

- The continuing calibration verification was performed at the required frequency using the proper standard at the proper concentration,
- Whether adequate response was obtained for each analyte, and

- Whether the responses obtained indicate that the instrumentation is still operating within an acceptable range (drift).

If the continuing calibration evaluation criteria for any analyte are not satisfied, then all results for that analyte associated with the continuing calibration will be qualified as estimated ("J/UJ"). A qualifier code of "CCV" or "CCAL" will be used for inorganic and organic methods, respectively. If the data reviewer can discern a probable magnitude and/or direction of bias to the associated sample results based on the information provided, then appropriate qualifier bias codes will be assigned.

4.2.3 Laboratory Control Sample Analysis

Laboratory control samples (LCSs) are "clean" well-characterized samples used to monitor the laboratory's day-to-day performance of routine analytical methods. LCSs are prepared by spiking samples of a "clean" matrix with known amounts of target analytes and then processing the sample in the same fashion as all other samples. LCSs are used to monitor the accuracy and precision of the analytical process independent of matrix effects. The accuracy of the analytical process is evaluated using the calculated percent recoveries (%Rs) of the spiked analytes.

The reviewer will verify that all target analytes were spiked into the LCS sample. The LCS percent recoveries will then be compared to the acceptance limits in the quality assurance plan, method, or laboratory historical limits (if the laboratory acceptance limits are considered to be comparable to those specified in the methods), as applicable.

- If the LCS recovery for an analyte is greater than the upper acceptance limit, suggesting a potential high bias in reported results, all positive results for that analyte in all associated samples will be qualified as estimated ("J") whereas nondetect results will be considered acceptable for use without qualification because the high bias does not affect nondetect results.
- If the LCS recovery for an **inorganic** analyte is less than the lower acceptance limit but ≥30%, suggesting a potential low bias in reported results, positive and nondetect results for that analyte in all associated samples will be qualified as estimated ("J" or "UJ").
- If the LCS recovery for an **inorganic** analyte is <30%, positive sample results will be qualified as estimated ("J"), whereas nondetect results will be qualified as unusable ("R") for all associated sample results.
- If the LCS recovery for an **organic** analyte is less than the lower acceptance limit but ≥10%, positive and nondetect results for that analyte in all associated samples will be qualified as estimated ("J" or "UJ").
- If the LCS recovery for an **organic** analyte is <10%, positive sample results will be qualified as estimated ("J") whereas nondetect results will be qualified as unusable ("R") for all associated sample results.

In the case of unacceptably low LCS recoveries, the reviewer will verify that the laboratory re-prepared and re-analyzed all associated samples; including the LCS and that acceptable results were obtained for the new LCS.

A qualifier code of "LCS" will be assigned to all results qualified as estimated or rejected on the basis of LCS recoveries.

4.2.4 Compound Identification

For 10% of the results reported in the data packages under going an evaluation of laboratory performance parameters, the reviewer will verify that results positively identified meet all identification acceptance criteria as specified in the analytical method. In addition, the reviewer will examine the data for false negative results.

For organics, this may encompass comparing retention times against retention time windows, evaluating the agreement between dual column confirmation results, comparing relative retention times (RRTs) for samples to RRTs for standards, and comparison of mass spectral data to reference spectra, depending on the analytical technique employed (note: this listing is not all inclusive).

For inorganic methods, compound identification is generally not reviewable from the data packages. However, for some methods, there are items the reviewer can check such as comparing the %RSDs for replicate measurements to a method specific criterion and that target analytes elute in the proper order and expected retention time.

4.2.5 Target Analyte Quantification

The reviewer will verify that reported sample concentrations can be recalculated from the raw data for 10% of the reported sample results in the data packages under going an evaluation of laboratory performance parameters. The reviewer will verify that reported results were calculated using the proper signal response for the sample, calibration factor or relative response factor, internal standard response, dilution factor, internal standard concentration or mass, percent solids, sample weights or volumes, final extract volume, etc. as applicable to the analytical method.

If errors are found in the reported sample results, the laboratory will be contacted and corrected results will be requested. The data review narrative will detail any such instances and the resultant resolution. The reviewer will collate the revised data into the data package and mark the all revised and all superseded data accordingly.

In some cases, multiple analyses for the same sample may be reported. The multiple analyses may be due to high target analyte concentrations that necessitate dilutions, interferences, or QC failures (e.g. low surrogate recoveries). When there is more than one set of data reported for a sample, the reviewer will need to select the best set of data to report based on all of the supporting QC information. This may involve selecting results from each of the multiple analyses. The data review narrative will detail the results selected for reporting and the supporting rationale. The data sheets will be marked to indicate which results were selected for reporting and which were not.

4.2.6 Verification

The reviewer will verify that information reported on the summary forms was calculated properly and that the results are traceable back to the raw data. In addition, the reviewer may also verify that all spike solutions and standards were used within their recommended shelf lives.

If errors are found in the reported sample results, the laboratory will be contacted and corrected results will be requested. The data review narrative will detail any such instances and the resultant resolution. The reviewer will collate the revised data into the data package and mark all revised and all superseded data accordingly.

4.2.7 Method Specific Quality Control Checks

The supporting QC data will be reviewed to evaluate if the method-specific QC checks were conducted and whether the method-specified acceptance criteria were met. The table below summarizes the method specific QC checks typical of each analytical technique. The reviewer will consult the quality assurance plan and analytical method for evaluation criteria used to evaluate method-specific QC checks.

QC Check	ICP-ES	ICP-MS	Wet Chemistry	GC	GC/MS	HPLC
Tuning		✓			✓	
Interference Check Sample	✓	✓				
Thermal Stability		✓				
Spectral Resolution		✓			✓	
Mass Calibration		✓			✓	
Chromatography			✓	✓	✓	✓

ICP-ES = Inductively coupled plasma atomic emission spectroscopy.
ICP-MS = Inductively coupled plasma mass spectroscopy.
GC = Gas chromatography.
GC/MS = Gas chromatography mass spectroscopy.
HPLC = High pressure liquid chromatography.

5.0 DOCUMENTATION

This section describes the documentation that will be generated as part of the data review procedure. Section 5.1 describes data review worksheets which are generic tools the validator may elect to use to facilitate the review. All data validation results will be documented in a narrative report. Section 5.2 describes the contents of the resultant data validation reports.

5.1 DATA REVIEW WORKSHEETS

Figures 1 and 2 provide generic data review worksheets for the sample-specific criteria and laboratory performance criteria reviews, respectively, which may be used to facilitate the data review process. These forms are intended to be used as general guides for each of the parameters requiring evaluation under each type of review; use of these forms is not mandatory. Due to space limitations and the number of analytical methods, the specific evaluation criteria are not included in the tables. The analytical methods should be consulted for specifications of all pertinent evaluation criteria. The data reviewer may choose to jot these criteria on the forms in the column titled "criteria." A separate form may be completed for each method. Additional pages may be added as necessary to detail all aspects of the data review.

5.2 DATA REVIEW NARRATIVE REPORTS

All data review activities will be detailed in a data validation narrative report. At a minimum, the report will include an introduction (Section 1), a summary of the data review process (Section 2), data review narratives for the review of laboratory performance parameters (Section 3), data review narratives for the review of sample-specific parameters conducted on each package (Section 4), and an overall assessment of the data (Section 5). The overall assessment will state any limitations to the usability of the data as well as address the quantitative and qualitative data quality indicators of sensitivity, accuracy, precision, completeness, representativeness, and comparability. All data review reports will be peer reviewed by a qualified person to assure compliance with the procedures described in this SOP.

TABLE 1
DATA VALIDATION QUALIFIER DEFINITIONS

QUALIFIER	DEFINITIONS [1,2]
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
J	The analyte was positively identified; the associated numeric value is the approximate concentration of the analyte in the sample (i.e., estimated value).
UJ	The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associate numerical value represents its approximate concentration.
R	The data are unusable and have been rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte can not be verified.

[1] USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, February 1994.

[2] USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, October 1999.

TABLE 2
DATA VALIDATION QUALIFIER CODES AND BIAS DIRECTION CODES

Qualifier Code	Data Quality Condition Resulting In Assigned Qualification
general use	
HT	Holding time requirement was not met
P	Preservation requirement(s) not met
MB	Method blank or preparation blank contamination
LCS	Laboratory control sample evaluation criteria not met
MS	Matrix spike and/or matrix spike duplicate accuracy evaluation criteria not met
D	Duplicate or spike duplicate precision evaluation criteria not met
FB	Field blank contamination
RB	Rinsate blank contamination
FD	Field duplicate evaluation criteria not met
TvP	Partial analysis results greater than total analysis results; difference is greater than accuracy limitations of the method
ID	Target compound identification criteria not met
IS	Internal standard evaluation criteria not met
CO	Suspected carry-over
SQL	Reported sample concentration is between the method detection limit and the sample quantitation limit.
RL	Reporting limit exceeds decision criterion (for nondetects)
LR	Over linear range without re-analysis
inorganic methods	
ICV	Initial calibration verification evaluation criteria not met
CCV	Continuing calibration verification evaluation criteria not met
CCB	Continuing calibration blank contamination
ICS	Interference Check Sample evaluation criteria not met
PDS	Post-digestion spike recovery outside acceptance range
MSA	Method of standard additions correlation coefficient < 0.995
DL	Serial dilution results did not met evaluation criteria
organic methods	
TUNE	Instrument performance (tuning) criteria not met
ICAL	Initial calibration evaluation criteria not met
CCAL	Continuing calibration evaluation criteria not met
SUR	Surrogate recovery outside acceptance range
Bias Codes	**Bias Direction**
H	Bias in sample result likely to be high
L	Bias in sample result likely to be low
I	Bias in sample result is indeterminate

Figure 1: Data Review Worksheet for Sample-Specific Parameters

Data Package _____ **Lab** _____

Date _____ **Matrix** _____ **Sampling Event** _____

Case Narrative Comments: _____

Parameter	Criteria	Criteria Satisfied?	Details	Actions (qualified data)
COC and Sample Receipt		Y N NA		
Holding Times		Y N NA		
Method Blank		Y N NA		
Matrix QC*	(Field ID or Batch QC?)			
• MS		Y N NA		
• MS/MSD		Y N NA		
• LD		Y N NA		
Method QC*				
• Surrogates		Y N NA		
• PDS/GFAA QC		Y N NA		
• Serial Dilution		Y N NA		
• Internal Standards		Y N NA		
• Total vs. Partial		Y N NA		
• Cation/Anion Balance		Y N NA		
Field QC*	(Field ID)			
• Field Duplicate		Y N NA		
• Rinsate Blank		Y N NA		
• Field Blank		Y N NA		
• Trip Blank		Y N NA		
• Other (e.g., splits)		Y N NA		

Parameter	Criteria	Criteria Satisfied?	Details	Actions (qualified data)
Other review parameters evaluated based on case narrative comments or review of laboratory performance parameters		Y N NA		

* As applicable to the method.

Completeness of the package:_____

_____

_____

Additional Comments/Concerns:_____

_____

_____

_____

_____

_____

General Overall Assessment:

_____Data are usable without qualification.

_____Data are usable as qualified (detailed in narrative).

_____Some or all data are unusable for any purpose (detailed in narrative).

Figure 2: Data Review Worksheet for Laboratory Performance Parameters

Data Package _____ **Lab** _____

Date _____ **Matrix** _____ **Sampling Event** _____

Parameter	Criteria	Criteria Satisfied?	Details	Actions (qualified data)
Initial Calibration • Number/Conc. of points • Low standard vs. RL • Goodness of Fit • Analytical sequence		Y N NA Y N NA Y N NA Y N NA		
Initial/Continuing Calibration Verification • Adequate frequency? • Adequate recovery? • Stability of CFs/RRFs? • Replicate agreement?		Y N NA Y N NA Y N NA Y N NA		
Laboratory Control Sample • Second source? • Adequate recovery? • Replicate agreement?		Y N NA Y N NA Y N NA		
Compound Identification • RTs or RRTs • Second Column Conf. • Mass Spectrum		Y N NA Y N NA Y N NA		
Quantification Were the proper internal standards and response factors used, as applicable? Are reported sample results adjusted for? • DFs • Sample Size • Dry Weight Agreement between replicate instrument measurements?		Y N NA Y N NA Y N NA Y N NA Y N NA		
Verification • CFs/RRFs calculated properly? • %Rs calculated properly? • %Ds calculated properly? • Transcription errors?		Y N NA Y N NA Y N NA Y N NA		
Method Specific QC • Thermal Stability • Tuning • Resolution • Mass Calibration • ICS		Y N NA Y N NA Y N NA Y N NA Y N NA		