SECTION FIFTEEN

# PRELIMINARY SITE CHARACTERIZATION QUALITY ASSURANCE SUMMARY TECHNICAL MEMORANDUM

# MOLYCORP MINE RI/FS

REVISION 0

*Prepared for*
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# SECTIONFIFTEEN Quality Assurance Summary

Data collection efforts for the Molycorp RI/FS were governed by the Draft Final Molycorp RI/FS Work Plan, which included the QAPP as Volume III and the associated Final Field Sampling Plan, Standard Operating Procedures, and Health and Safety Work Plan as Volume IV (Revision 1.0, July 11, 2002).

The Molycorp RI/FS Work Plan was developed by applying the Data Quality Objectives (DQO) process, which is a systematic planning tool based on the Scientific Method that is used for establishing criteria for data quality and for developing data collection designs. Establishing formal data quality objectives during the Work Plan development allows a clear and unambiguous definition of project objectives, decisions, and decision criteria so that data of sufficient type, quality, and quantity are generated to meet project objectives. The formal implementation of a DQO process brings structure to the planning process, thereby resulting in defensible decision making.

The U.S. Environmental Protection Agency's Guidance for the Data Quality Objectives Process (EPA QA/G-4, Final, August 2000) was utilized during the planning process. The QAPP provides general guidance on developing data quality criteria and performance specifications for decision-making and addresses application of the EPA's seven step DQO process for site investigations.

The QAPP stated the objective for the RI/FS and specified the data quality requirements necessary to meet those objectives. The methods and procedures used to implement and accomplish the project objectives are described throughout the QAPP. In order to assure the consistency and thoroughness of data generation, SOPs for field sampling, sample custody, equipment operation and calibration, laboratory sample analysis, data reduction, and data reporting were utilized. Additionally, the quality of data generated was assessed to assure that all data are scientifically valid and of known and documented quality. This was largely accomplished by establishing acceptance limits for the data quality indicators of precision, accuracy, completeness, representativeness, and comparability, and by testing generated data against acceptance criteria established for these indicators during the data validation process.

Precision, accuracy, completeness, representativeness, and comparability are the criteria used to evaluate data quality. A description of each measure is provided in Section A.7.4 of the QAPP. In order to meet the intended uses of the data, specific numeric acceptance limits were established for precision, accuracy, and completeness. The established precision and accuracy limits are those limits specified in Table B.4.4-1a of the QAPP. These limits will ensure that routinely generated data are valid and defensible and are of known and acceptable precision and accuracy.

This section discusses the overall data quality of the RI/FS data set. In Section 15.1, the data validation procedures are summarized. In Section 15.2, significant matrix effects are discussed. In Section 15.3, an overview summary of the validation results is presented. In Section 15.4, field and laboratory chemical constituents are discussed. And finally, in Section 15.5, a general overall assessment of the data quality with respect to the data quality indicators is provided.

# SECTIONFIFTEEN Quality Assurance Summary

## 15.1 VALIDATION PROCEDURES

In order to assess the overall usability of the data, all chemical data packages received were validated and the results were presented in two types of reports. Section 15.1.1 discusses the data validation procedures implemented. Section 15.1.2 discusses the data validation qualifiers, reason codes, and bias codes that were assigned to chemical analytical results during the validation process, as necessary. Data validation narrative reports were generated to document the results of all data validation activities, all data qualification assigned, and any limitations on the use of the data. Section 15.1.3 discusses the types and content of the resultant data validation reports.

### 15.1.1 Data Validation Process

All analytical data used for RI/FS reporting and environmental decision making at the Molycorp mine received a review independent of the laboratory to ensure that data are of known and documented quality. The non-air data validation process consisted of evaluation of laboratory performance criteria and sample-specific criteria in accordance with SOP 12.1, Analytical Data Validation for RI/FS data. The air monitoring data were collected in a program outside the RI/FS, but are considered pertinent to RI work. The quality and usability of the air data validated is presented in Section 3.1.1.1 of the final report entitled, *Air Quality Assessment of Molycorp, Inc. Questa Division Tailings Facility*, prepared by Applied Measurement Science (May 19, 2004). Air quality is discussed in Section 14.

The review of sample-specific parameters for non-air data includes evaluating parameters that are field 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 analyses, post-digestion spike recoveries, ICP Spectroscopy serial dilution analysis agreement, internal standard performance, and results for field quality control samples (e.g., field duplicates, rinsate blanks). All data packages received a review of sample-specific parameters.

The review of laboratory performance parameters for non-air data 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. Laboratory performance parameters were reviewed for at least 10 percent of RI/FS data packages (per method per sampling event) received. Problems identified during the laboratory performance parameter review as potentially being systematic laboratory performance issues were then also evaluated for all data packages for the specific sampling event.

The hierarchy for acceptance criteria used to evaluate each parameter, as specified in SOP 12.1, was to follow the criteria specified in the RI/FS QAPP (SOP 12.1), then method-specified

criteria, and finally (if prior references did not specify the criteria in question), laboratory historically determined acceptance ranges.

During the data review process, data validation qualifiers were 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 were also added to the results and to the database to indicate the reason(s) for qualification and the associated bias direction, if discernable. The following section provides the definition of all validation, reason, and bias codes used.

### 15.1.2 Data Validation Qualifiers, Reason Codes, and Bias Codes

In accordance with SOP 12.1, data validation qualifiers were assigned to results associated with quality control results not meeting project objectives (i.e., acceptance criteria) as defined in the QAPP. In addition, reason codes and bias direction codes were assigned to all qualified data. Table 15-1 summarizes the data validation qualifiers used and the associated definitions. Table 15-2 summarizes the qualifier reason codes and bias direction codes.

During the data review process, the data reviewer recorded all data validation qualifiers and associated qualification reason codes and bias codes onto the laboratory data reporting forms (also known as "Form 1s"). Copies of the data sheets were given to the database administrator so that the data qualifiers, reason codes, and bias codes could be entered into the database. The original qualified data sheets were returned to the data packages, which are retained in the URS project files. Additionally, the qualified data sheets and laboratory chain-of-custody (COC) records were scanned so that the resultant portable document files (pdf) could be retained in the administrative record in a condensed and electronic format.

### 15.1.3 Data Validation Reports

The data validation reports consisted of two types of reviews for RI/FS data. The first type of review encompasses the data validation review narrative. For each data package, a data validation review narrative report was prepared. In all cases in which professional judgment was exercised in evaluating the need for qualification, the basis for the professional judgment is provided in the data review narrative report. The second type of review encompasses all of the data from a distinct sampling event. As discussed below, for each sampling event, several quality control measures were assessed by matrix in an overall collective sense for the sampling event. For each event, this evaluation is described in the DVR and the associated data validation review narrative reports are included in the DVR as attachments.

After completing the review of sample-specific and laboratory performance parameters in accordance with SOP 12.1, the site-specific matrix spike results, laboratory duplicate results, serial dilution results, blanks (field and rinsate), and field duplicate results were assessed collectively by matrix and sampling event to determine the need for additional qualification of sample results of similar matrix. The reason for this is that site-specific quality control (QC) samples are considered to be much more representative of the site sample matrix and are a good indication of whether there is a matrix effect present with a similar matrix. Therefore, samples were designated on the COC that were to be run as site-specific QC samples to meet the

frequency of site-specific QC specified in the RI/FS QAPP. These QC samples were spread across data packages and not all packages contained a set of site-specific QC samples. A collective evaluation of all of the site-specific QC samples of a similar matrix was performed to determine whether or not problems identified in a given QC sample are generally true for the site-specific matrix or are likely limited to the specific sample being used for the QC measure. If the matrix effect was judged to be generally present for a given matrix, then qualification of all results for samples of that matrix was performed. If the matrix effect was judged to be limited to the specific sample used for the QC measure, then qualification of only this parent sample was considered warranted.

For each discrete sampling event, a DVR was prepared. Each DVR includes the following information:

- The field and QC samples collected in the sampling event along with frequency of QC sample collection.
- The data packages the results for the sampling event were reported in.
- The data package(s) used to assess laboratory performance parameters.
- The collective assessment of the matrix QC results for the sampling event (matrix spike, lab duplicate, and serial dilution results) and any associated sample qualification.
- The collective assessment of the field QC results for the sampling event (field and rinsate blanks, where applicable, and field duplicate results) and any associated sample qualification.
- An overall assessment of data, with respect to the data quality indicator parameters of Precision, Accuracy, Completeness, Representativeness, and Comparability (PACRC) and sensitivity.
- Additionally, all data review narratives for the pertinent data packages are included as attachments to the DVR for each event.

A total of 52 DVRs were prepared for work conducted under the RI/FS. Table 15-3 summarizes the distinct sampling events, the associated data packages, the matrices, and the DVR number assigned to the event.

## 15.2 SUMMARY OF SIGNIFICANT MATRIX EFFECTS

During the earlier stages of the RI/FS, some matrix-related analysis problems were identified. Section 15.2.1 discusses the matrix-related analysis issues for aqueous samples and Section 15.2.2 discusses the matrix-related analysis issues for soil samples.

### 15.2.1 Matrix Effects Affecting Aqueous Samples

During review of the fall 2002 groundwater and surface water data, it became apparent that there were matrix-related analysis problems. The serial dilution results, comparisons with historical

results, and charge balances suggested that matrix-related issues existed for the metals analysis, sulfate analysis, and fluoride analysis. The laboratory conducted several studies in order to determine analysis solutions to the matrix-related analysis problems. Each analysis problem, investigation, and solution is summarized below.

### *Sulfate Analyses*

The sulfate analyses were originally conducted by Ion Chromatography by Method 300.0 for the fall 2002 groundwater data, charge balances were often out of SOP 12.1 limits for samples with low pHs. For most of these, the sulfate results were greater than the reported TDS results. Additionally, many sulfate results were higher than historic results. In order to investigate the analysis issues, the laboratory conducted several re-analyses on a selected variety of samples encompassing a range of pHs and dilutions. After noting reproducibility and comparability problems despite analyzing filtered and unfiltered samples, homogenized and non-homogenized (un-mixed) samples, the same sample over five days, and using an elluent dilution technique, a different analytical method was examined.

The selected group of samples were analyzed by a turbidimetric technique using EPA Method 375.4. The charge balances using the turbidimetric method were within acceptance limits. In addition, for a five-day reproducibility study, the turbidimetric analysis method demonstrated acceptable analytical precision. Thus, all fall 2002 and December 2002 groundwater samples for which the charge balances were outside of acceptance limits were re-analyzed for sulfate using EPA Method 375.4. In addition, all subsequent sulfate analyses were conducted using EPA Method 375.4.

### *Fluoride Analyses*

When comparing results for the fall 2002 groundwater data with historic results, it was noted that many fluoride results were lower than historic results. The problem was traced to the concentration of aluminum present in the samples which complexes with the fluoride and results in suppressed measurements. Fluoride was determined using EPA Method 340.2 which includes the addition of a chelating buffer to alleviate interferences from polyvalent cations such as aluminum. However, the method can only compensate for aluminum concentrations up to 3 mg/L. Approximately 50 samples contained aluminum at concentrations greater than 3 mg/L.

To correct the problem, the laboratory re-analyzed the samples by performing the dilutions necessary to reduce the aluminum concentration to 3 mg/L or less prior to the addition of the chelating buffer. Results obtained using this procedure were comparable with historic data. Thus, all Fall 2002 and December 2002 groundwater samples for which the aluminum concentrations were greater than 3 mg/L were re-analyzed. In addition, for all subsequent fluoride analyses, the laboratory was instructed to dilute the samples based on the aluminum concentration, prior to addition of the chelating buffer.

### *Metals Analyses*

Some metals results were found to be inconsistent with historic results. Many serial dilution results differed by more than 10 percent for a 5-fold dilution. Additionally, for many samples in which the charge balance was outside the acceptance limits, the cation results appeared to be low. It was observed that the number of metals failing to meet the serial dilution acceptance criterion tended to increase as the pH of the samples decreased. These problems were most notable for samples with lower pHs (<6.70). To study these observations, 7 samples encompassing a range of pHs were selected for analysis at multiple dilutions (generally four different dilutions). Post-digestion spikes were conducted at two of the dilution levels to evaluate at what dilution levels the interference problems appeared to be minimized.

The results of this study indicated that there was a consistent and significant bias in metals results for samples with a pH less than 5.7. For samples with pH ranging from 5.7 to 6.7, there appeared to be a bias, although both the magnitude and the existence of the effect were variable. The observed biases could result in reported results more than an order of magnitude lower than true values. As such, a set of standard dilution schemes were developed to be applied to all future RI/FS analyses in order to provide assurance that the RI data would be of sufficient accuracy to meet project objectives.

Tables 15-4 and 15-5 present the dilution schemes for low pH and moderate pH samples that resulted from the dilution studies. Challenges in implementing the dilution schemes were achieving low detection limits for non-detects and complication of field logistics due to the need to collect and submit samples arranged by pH group.

The fall 2002 groundwater and surface water samples for which the cation/anion balance was out of limits or the metals concentrations did not compare well with historic results were re-analyzed for dissolved metals using the applicable dilution scheme.

The re-analyses for metals was limited to the dissolved metals fraction only. As such, for samples in which the dissolved fraction was re-analyzed, the total metal sample results were rejected because they were likely to have a significant low bias to sample analyses results. The reason and bias codes assigned to the total metals results for the affected samples are "DL, Hist – L." The "DL" reason code was used because it was the serial dilution results that suggested that pH-dependent matrix-related analysis problems existed. The "Hist" reason code was added to indicate that results obtained did not compare well with historic data, which further supported the presence of an analysis problem as implied by the serial dilution results.

As a consequence of the dilution scheme, some non-detect results were reported with proportionately elevated RLs.

### *Bicarbonate Alkalinity/Total Alkalinity Matrix Spike Analyses*

Initially, bicarbonate and total alkalinity results were qualified on the basis of matrix spike recoveries outside the acceptance range of 75-125 percent in the sample-specific reviews. However, further evaluation indicated that the matrix spike recoveries for the bicarbonate and total alkalinity analyses were not a pertinent measure of accuracy on acidic samples. The highly

variable pH range of the water samples and the associated carbonate species equilibria were found to affect the matrix spike recoveries more than the expected biases or uncertainty in the method. The matrix-spiked samples were found to have significantly different pHs than the parent samples due to the presence of carbonate, confounded by a dilution effect. Therefore, the matrix spike recovery data for bicarbonate and total alkalinity were not used as a measure for accuracy.

### *Non-Valid Matrix Spikes*

As specified in the QAPP, there were certain scenarios in which matrix spike analyses were not considered appropriate for assessing accuracy for sample specific matrix effects. These are as follows:

- For metals, when the parent sample concentrations were significantly greater than the spiking concentrations (i.e., greater than or equal to four times the spiking concentration), the ability to determine accuracy in the analysis diminishes as the spike level becomes nominal compared with the original sample concentration.
- Instances in which the reporting limits were increased due to dilution factors, which adversely affected the reliable quantitation of the spiked metals. In other words, the spike concentration is diluted out of the quantifiable range of the method. In these situations, the reporting limit was typically greater than the spike concentration added.

Non-valid matrix spike results were omitted from the collective assessment of matrix QC results.

## 15.2.2 Matrix Effects Affecting Soil Samples

There was only one significant matrix effect for soil samples. At project initiation, it was known that the standard acid digestion specified in Method 3050B, "Acid Digestion of Sediments, Sludges and Soils," was not effective for antimony. The optional separate digestion included in Method 5030B, involving rigorous refluxing with a nitric and hydrochloric acid mixture was not deemed necessary because antimony is not considered to be a site-related chemical constitutent. Because the standard digestion was not effective for antimony, low matrix spike recoveries, often <30 percent, were obtained. Recognizing this effect was expected, and that an order of magnitude low bias to results and sensitivity would not jeopardize project objectives, the threshold for rejecting non-detect antimony, as specified in SOP 12.1, was lowered from <30 percent specified in SOP 12.1 to <10 percent. Most antimony results for soils samples were qualified as estimated (J/UJ MS-L) as a result of low matrix spike recoveries. The low antimony matrix spike recoveries were not unexpected and with few exceptions, the antimony data are considered usable in meeting project objectives in spite of the potential low bias. A few antimony results were rejected because the matrix spike results were <10 percent.

## 15.2.3 Evaluating Potential Matrix Effects on Dissolved Lanthanide Analysis

In addition to collecting the standard field QC samples of field duplicates, rinsate blanks, field blanks and trip blanks at a frequency of 1 per 20 field samples per matrix (as applicable to the

analysis parameters) as discussed in the DVRs, another type of field QC sample was collected during the February 2004 Specialty Sampling Event. For the dissolved lanthanides analysis, a secondary filtration was done on three samples to evaluate adsorption of lanthanides to the filter material.

The laboratory suggested doing adsorbance testing on the filter material to differentiate between filtration and adsorbance in order to have a true dissolved determination of lanthanides. At sites MMW-21, MMW-30A, and MMW-30B, two dissolved metals (lanthanides) aliquots were prepared; the filtered samples were labeled with "D01N" in the field ID. Then, for each location, one of the "D01N" filtrate aliquots was filtered a second time, using a new filter, and the resultant sample was labeled with "D02N" in the field ID. The difference between the results for the D01N samples and the D02N samples were used to provide an indication of the propensity of lanthanide adsorbance on the specific filter media.

To evaluate the magnitude of adsorption of lanthanides on the filter, the ratio between the primary and secondary filtration results (i.e., D01N/D02N) was calculated as shown in the table below.

For all three samples, all ratios are 1.00 or greater, as expected, indicating that the D02N results were always less than or equal to the D01N results. For samples MMW-21 and MMW-30B, the average ratio between the D01N and D02N results were 1.01 and 1.06, indicating that while the D02N results were generally lower, but that the difference was very small (e.g. there was very little adsorption). For sample MMW-30A, however, the average ratio between the D01N and D02N results was 1.42, suggesting that there potentially might be some adsorption of lanthanides on the filter material. However, as no adsorption was indicated for the other two samples, other cause for the difference between the D01N and D02N results for sample MMW-30B were explored.

Field duplicate samples were analyzed at MMW-30A, allowing an evaluation of the sample homogeneity and analysis precision. The Relative Percent Differences (RPDs) between the dissolved MMW-30A results and its field duplicate ranged up to 55% with the average RPD across all lanthanides being 31%. The ratio of the sample result to the field duplicate sample result averaged 1.38. These results indicate a fairly large amount of heterogeneity in the MMW-30A sample. This imprecision is about the same magnitude as the differences between the D01N and D02N samples, for which the RPDs ranged up to 41% with the average RPD across all lanthanides being 35% and the average ratio between the D01N and D02N results being 1.42. Thus, the observed differences between the sample which was filtered once (D01N) and that filtered twice (D02N) are nearly identical to the differences noted for the primary field sample and its field duplicate sample. The field duplicate results suggest that the observed differences between the D01N and D02N results for MMW-30A are attributable to sample heterogeneity rather than adsorption of the lanthanides on the filter.

| Analytes | Ratio of D01N to D02N Lanthanide Results | | |
|---|---|---|---|
| | MMW-21 | MMW-30A | MMW-30B |
| Cerium | 1.00 | 1.39 | 1.08 |
| Dysprosium | 1.02 | 1.47 | 1.04 |
| Erbium | 1.02 | 1.47 | 1.06 |
| Europium | 1.02 | 1.41 | 1.07 |
| Gadolinium | 1.01 | 1.45 | 1.05 |
| Holmium | 1.02 | 1.51 | 1.04 |
| Lanthanum | 1.00 | 1.40 | 1.08 |
| Lutetium | 1.01 | 1.33 | 1.05 |
| Neodymium | 1.01 | 1.35 | 1.04 |
| Praseodymium | 1.00 | 1.36 | 1.08 |
| Samarium | 1.01 | 1.37 | 1.12 |
| Terbium | 1.02 | 1.49 | 1.03 |
| Thulium | 1.00 | 1.46 | 1.10 |
| Ytterbium | 1.01 | 1.33 | 1.01 |
| Yttrium | 1.00 | 1.51 | 1.03 |
| **Average Ratio D01N/D02N** | **1.01** | **1.42** | **1.06** |

This conclusion is further supported by the turbidity observed for the MMW-30A sample. The turbidity measurement for MMW-30A was 29.2 NTU, much larger than that observed for either MMW-21 (8 NTU) or MMW-30B (7.4 NTU). This makes it much more likely that the MMW-30A aliquots are more likely to be highly heterogeneous than the other two samples. The table below summarizes these data suggesting that in-homogeneity is the likely cause of the difference in the D01N and D02N lanthanide results for sample MW-30A.

Thus, the difference in lanthanide results between the first (D01N) and second filtrations (D02N) generally mirrors the difference between the total lanthanide (T01N) and dissolved (D01N) lanthanide results. With the difference noted for sample MMW-30A likely being attributable to sample inhomogeneity, the comparison of the dissolved lanthanide results for the D01N and D02N is considered to indicate that there was little or no adsorption to the filter material for any of the three sets of samples.

| | MMW-21 | MMW-30A | MMW-30B |
|---|---|---|---|
| Average Ratio of D01N to D02N results | 1.01 | 1.42 | 1.06 |
| Average Ratio of D01N to D01D results | NA | 1.38 | NA |
| Average RPD of D01N to D02N results | 1% | 35% | 6% |
| Average RPD of D01N to D01D results | NA | 31% | NA |
| Turbidity, NTU | 8.0 | 29.2 | 7.4 |
| Flow Rate, L/min | 0.36 | 0.27 | 0.34 |
| Aquifer Type | Bedrock | Alluvium | Colluvium |

T01N = total lanthanide sample fraction.
D01N = dissolved lanthanide sample fraction, primary filtration.
D02N = dissolved lanthanide sample fraction, secondary filtration of the T01N sample.
NA = Not applicable because a field duplicate sample was not collected at this location.

## 15.3 SUMMARY OF VALIDATION RESULTS

As a result of the data validation effort, approximately 99.3 percent of the RI/FS data set was deemed to be usable for meeting project objectives. Data qualified as non-detect or as estimated are considered usable for meeting project objectives, whereas data qualified as unusable ("R") are not. The following section discusses the quantity of valid data, and the effect of rejected data on decision-making.

### 15.3.1 Valid Data Statement

As noted in Section 15.1, all data were validated in accordance with the provisions of the Administrative Order on Consent approved QAPP and SOP 12.1. The data validation procedures meet the minimum requirements specified in EPA's Risk Assessment Guidance for Superfund (September, 1989) (RAGS) and those specified in EPA's Guidance for Data Usability in Risk Assessment (April, 1992) (DURA). As specified in DURA, data qualified as "U" (non-detectable) or "J" (estimated) should be used for risk assessment purposes. DURA (page 5-15) further indicates that:

> "the guidance here is to use J-qualified concentrations the same way as positive data that do not have this qualifier. If possible, note potential uncertainties associated with the qualifier, so that if data qualified with a J contribute significantly to the risk, then appropriate caveats can be attached."

The table below presents the total number of field sample analyses results qualified as unusable ("R-flagged"), estimated ("J"- flagged), and non-detect ("U"-qualified) as well as the corresponding percentage.

**Qualified Data Summary**

| | Result Count | Percentage of Total |
|---|---|---|
| Total Number of Analytical Results1 | 302,871 | |
| Number of R-flagged Results (rejected) | 2,057 | 0.68 |
| Number of J-flagged Results (estimated) | 53,828 | 17.77 |
| Number of U-flagged Results (nondetect) | 13,990 | 4.62 |
| Number of Unqualified Results | 235,843 | 77.87 |
| SUM | 305,718 | 100.94 |

As shown by the table above, greater than 99.3 percent of the analytical RI/FS data were considered acceptable for use in meeting project objectives as qualified. Many sample results were qualified as non-detect ("U" flagged) on the basis of contamination identified in the laboratory blanks. Additionally, many results were qualified as estimated ("J" or "UJ") based on a variety of reasons. The sum of the percentages of total is greater than 100% because some results received qualification as both estimated (J) and nondetect (U). As noted earlier, the DVRs for each sampling event provide the detailed discussion regarding all data qualifiers assigned.

### 15.3.2 Affect of Rejected Data on Project Decision Making

Approximately 0.7 percent of the field sample analysis results were qualified as unusable ("R" flagged). As explained below, the data user should note that the vast majority (86 percent) of rejected data was due to two issues, neither of which affects the ability to make project decisions.

The first major cause of rejected data (approximately 57 percent of all rejected data) was discussed earlier in the section on Matrix Related Analysis Problems (Section 15.2.1). In this section, it was explained that the total metals data for several groundwater and surface water samples from the first two sampling events were rejected due to poor comparability with historic results in combination with matrix related effects, which were eliminated by dilution prior to analysis. Re-analyses were conducted for the dissolved metals analysis of the affected samples, as ecological risk-based evaluations would generally be made using the dissolved metals results, rather than the total metal results. For human health groundwater, the dissolved fraction generally mimics the total fraction results owing to use of low-flow purging of wells and the component of groundwater that moves through the aquifer is adequately described by the dissolved fraction results. Due to logistics and time restraints, re-analyses were not considered warranted for the total metals samples.

The second major cause of rejected data (approximately 29 percent) was improper sample location for some soil samples. In most cases, replacement samples were collected at the proper locations such that there was no effective loss of data. The replacement samples were given a unique field ID. The initial results were rejected so that they would be excluded from the useable data set. Because the vast majority of rejected data were either not crucial to risk-based

evaluations, or were compensated for by results for replacement samples, the amount of rejected data, 0.7 percent, is not considered to affect the overall robustness of the data set.

With the analytical completeness being 99.3 percent, it is considered likely that the ability to make project decisions will not be limited due to a lack of valid data.

## 15.4 FIELD OR LABORATORY CHEMICAL CONSTITUENTS

Throughout the Molycorp RI, several analytes were reported as detected in the field investigation samples and in field rinsate blank samples at a comparable frequency and concentration for various media sampled. For example, ammonia was reported as detected in 167 of the 222 rinsate blanks (75 percent) and was detected in all investigative media at a comparable frequency and magnitude.

Molycorp proposed in the February 2004 meeting with EPA that these analytes with a blank frequency of detection and range of concentrations that are comparable to site samples be excluded from consideration as chemicals of potential concern. In subsequent discussions and meetings, EPA concurred with this recommendation and revised the Screening Level Criteria tables to reflect the agreement.

### *Ammonia*

Sixty-five percent of all aqueous samples were qualified as non-detect on the basis of ammonia contamination in associated blanks. Evaluation of solid media requires a calculation of the concentration in soil or sediment equivalent to that in an aqueous blank. Taking a conservative approach for calculating equivalent concentration based on the assumptions that all contamination found in the blank aliquot analyzed would be present in the sample aliquot analyzed and taking into account the differing environmental and rinsate blank preparation procedures, the maximum likely contribution for ammonia in soil or sediment samples (in mg/kg) would be the reported blank concentration in mg/L multiplied by 300. Ninety-nine percent of all detected soil and sediment samples had equivalent concentrations of ammonia within the range of concentrations likely attributable to contamination.

The following table summarizes detectable ammonia concentrations in field blanks and various abiotic media. Evaluations of these data indicate a frequency and magnitude of detection of ammonia in blanks comparable to that for field samples.

Therefore, comparable concentrations of ammonia detected in the 35 percent of the aqueous samples not qualified or reported as non-detectable and in all of the soil or sediment samples are considered to be attributable to field or laboratory contamination and not to presence in site samples. The reporting of detectable concentrations of ammonia in site samples is not considered to be an indication of the presence of ammonia in those samples on site.

| Medium | Percent Detects | Range of Concentrations (mg/L or mg/kg) | Equivalent Blank Conc. (mg/L or mg/kg) |
|---|---|---|---|
| Rinsate Blanks | 75% | 0.03 – 0.84 | |
| Groundwater | 86% | 0.02 – 0.96* | 0.03 – 0.84 |
| Seep | 82% | 0.03 – 0.51 | 0.03 – 0.84 |
| Surface Water | 76% | 0.03 – 0.54 | 0.03 – 0.84 |
| GSI Surface Water | 75% | 0.04 – 0.18 | 0.03 – 0.84 |
| Soil | 95% | 0.05 – 256** | 9 - 250 |
| Sediment | 93% | 3 – 258*** | 9 – 250 |

*excludes 7 of 7 samples from MW-B, 2 of 3 samples from SC-1B, and 1 of 7 samples from MMW-8A with ammonia concentrations ranging from 1.2 to 7 mg/L

**excludes highest 1 percent of results (13 soil samples with concentrations ranging from 265 to 1,610 mg/kg)

***excludes highest 1 percent of results (4 sediment samples with concentrations ranging from 266 to 793 mg/kg)

### *Other Chemical constituents*

The table below summarizes benzaldehyde detects for soil samples and associated laboratory QC samples.

| | SAMPLE TYPE | | Laboratory Control Samples |
|---|---|---|---|
| | Soil Samples | Method Blanks | |
| Number of samples | 276 | 27 | 34 |
| Number of Benzaldehyde detections | 202 | 19 | 23 |
| Frequency of Benzaldehyde detection | 73 percent | 70 percent | 68 percent |
| Average Detected Benzaldehyde Concentration | 0.241 mg/kg | 0.278 mg/kg | 0.175 mg/kg |
| Range of Detected Benzaldehyde Concentrations | 0.018 to 4.7 mg/kg | 0.023 to 1.2 mg/kg | 0.018 to 0.720 mg/kg |
| **EPA R6 Medium Specific Screening Levels** | **Residential** | **Industrial Indoor Worker** | **Industrial Outdoor Worker** |
| mg/kg | 6,100 | 100,000 | 68,000 |

The SVOC benzaldehyde was detected in approximately 73 percent of the soils samples from the fall 2002 sampling event. However, despite the frequency of detection in the field samples, benzaldehyde is considered to be a laboratory artifact. The frequency of benzaldehyde detection in field samples is comparable to the frequency of detection in method blanks and laboratory control samples. Additionally, the range of detected concentrations was comparable between the

three populations of samples: soil samples, method blanks, and laboratory control samples. Discussions with laboratory personnel indicated that the benzaldehyde issue extended to other projects in house at the same time period as the samples from this event; the laboratory suspects that the benzaldehyde may be introduced during the GPC clean-up step. Although some benzaldehyde detections remained after data qualification was issued based on method blank results, the remaining detections of benzaldehyde are considered a laboratory artifact rather than a site-related issue.

A similar situation was encountered for the following analytes:

1. Common Laboratory Chemical constituents (all media): acetone, methylene chloride, carbon disulfide, and phthalates
2. Surface water: bis(2-ethylhexyl)phthalate, acetone, and carbon disulfide
3. Groundwater: bis(2-ethylhexyl)phthalate, diethylphthalate, di-n-butylphthalate, acetone, carbon disulfide, chloroform, methylene chloride, and tetrachloroethene
4. Soil and sediment: 1,2,3,4,6,7,8,9-octachlorodibenzofuran, 1,2,3,4,6,7,8,9-octachlorodibenzo-p-dioxin, 1,2,3,7,8-pentachlorodibenzofuran, 2,3,4,6,7,8-hexachlorodibenzofuran, bis(2-ethylhexyl)phthalate, diethylphthalate, di-n-butyl phthalate, acetone, carbon disulfide, methylene chloride, and benzaldehyde.

Figure 15-1 compares the frequency of detection of chemical constituents found in surface water samples to the frequency of detection of these chemical constituents in the associated field blank samples.

The bar graph indicates that for two of the four analytes, ammonia, and carbon disulfide, the frequency of detection in the field samples was very similar to the frequency of detection in the associated field blanks. For the other two analytes, bis(2-ethylehxyl)phthalate, and acetone the frequency of detection was significantly higher in the field blanks than in the field samples. These results strongly suggest that the presence of these analytes in field samples is due to ambient conditions in the field or laboratory. These analytes are not considered to be site-related.

Figure 15-2 compares the frequency of detection of chemical constituents found in soil and sediment samples to the frequency of detection of these chemical constituents in the associated field blank samples.

The bar graph indicates that for all analytes except acetone, the frequency of detection in the field samples was very similar to the frequency of detection in field blanks (i.e., frequency of detections differed by no more than 25 percent). Acetone is a known common laboratory chemical constitutent. As such, the higher frequency of detection in field samples relative to field blanks is not considered to indicate that these analytes are site-related. These results strongly suggest that the presence of these analytes in field samples is due to ambient conditions in the field or laboratory. These analytes are not considered to be site-related.

Figure 15-3 compares the frequency of detection of chemical constituents found in groundwater samples to the frequency of detection of these chemical constituents in the associated field blank samples.

The bar graph indicates that for all analytes, the frequency of detection in the field samples was either very similar to the frequency of detection in the associated field blanks or the frequency of detection in field blanks was much higher than for field samples. These results strongly suggest that the presence of these analytes in field samples is due to ambient conditions in the field or laboratory. These analytes are not considered to be site-related.

Molycorp proposed in the February 2004 meeting with EPA that these analytes with a blank frequency of detection and range of concentrations that are comparable to site samples be excluded from consideration as chemicals of potential concern. In subsequent discussions and meetings EPA concurred with this recommendation and revised the Screening Level Criteria tables to reflect the agreement.

Compounds by medium for which blank detection rate and magnitude are comparable to field sample detection rate and magnitude are:

1. All media: acetone, ammonia, methylene chloride, carbon disulfide, and phthalates
2. Surface water: bis(2-ethylhexyl)phthalate, acetone, and carbon disulfide
3. Groundwater: bis(2-ethylhexyl)phthalate, diethylphthalate, di-n-butylphthalate, acetone, carbon disulfide, chloroform, methylene chloride, and tetrachloroethene
4. Soil and sediment: 1,2,3,4,6,7,8,9-octachlorodibenzofuran, 1,2,3,4,6,7,8,9-octachlorodibenzo-p-dioxin, 1,2,3,7,8-pentachlorodibenzofuran, 2,3,4,6,7,8-hexachlorodibenzofuran, bis(2-ethylhexyl)phthalate, diethylphthalate, di-n-butyl phthalate, acetone, carbon disulfide, methylene chloride, and benzaldehyde.

These compounds are not included in the summary results tables in the previous sections covering the individual media, but results for analysis of these compounds are included in the printout of the RI sample analysis results in Appendix A. In addition, there are compounds such as DDT that may be considered as ubiquitous to the region. If such compounds were detected in the media being presented in the previous sections, then a discussion of their presence is included within the sections covering the individual media. The reader should note that the data tables and statistics given below are those used in the February 2004 meeting. As such, the last three sampling events out of a total of 52 events were not included in the evaluation. However, the blank results for the last three events mirrored the earlier events and their exclusion is not considered to affect the overall conclusion about target analytes that can be considered chemical constituents.

## 15.5 OVERALL DATA QUALITY ASSESSMENT

Greater than 99.3 percent of the RI/FS analytical data were considered acceptable for use in meeting project objectives as qualified. Many sample results were qualified as non-detect ("U" flagged) on the basis of contamination identified in the laboratory blanks. Additionally, many

results were qualified as estimated ("J" or "UJ") based on a variety of reasons. The DVRs for each sampling event provide the detailed discussion regarding all data qualifiers assigned.

The data quality assurance objectives, as found in Section D of the QAPP, were reviewed to verify that RI/FS data collected met data quality objectives. The results of the evaluation are presented in the following sections.

### 15.5.1 Precision

Precision measures the repeatability of data by examining the spread of individual values from the average reported values, and therefore describes the magnitude of errors. The closer the numerical values of the measurements are to one another, the more confidence there is in the precision of the analysis. Precision for a single analyte was expressed as a RPD or as an absolute difference between field duplicate or laboratory duplicate results (spike duplicate analyses were used for organic methods). Precision was measured by analyzing duplicate sample (or spiked duplicate samples) at a frequency of one duplicate analysis per 20 field samples. Table B.4.4-1a of the QAPP listed the acceptance criteria used to measure precision.

The percentage of precision measurements meeting evaluation criteria ranged from 90 percent to 100 percent for all events, with the percentage being closer to 100 percent for the vast majority of sampling events. While a few results were qualified as estimated based on imprecision, none were qualified as unusable. As such, the overall level of precision demonstrated for all events collectively was considered to be acceptable.

### 15.5.2 Accuracy

Accuracy describes how close a result is to a specific target. It is a measure of the bias in a measurement system. The closer the value of the measurement agrees with the true value, the more accurate the measurement. Accuracy was measured by spiking a control sample matrix and field samples with known quantities of target analytes and then calculating the percent recovery of the analyte. The samples made by spiking a control matrix are called laboratory control samples (LCSs). The samples made by spiking field samples are called matrix spike (MS) samples. LCSs and MSs were prepared and analyzed at a frequency of one per 20 field samples. Additionally, for organic analyses, surrogate compounds were spiked into every sample. The percent recoveries were compared to the acceptance criteria listed in QAPP Table B.4.4-1a.

While several results were qualified as estimated based on poor matrix related accuracy, relatively few were qualified as unusable (0.027 percent of all data were based on matrix spike recoveries). The overall level of accuracy with respect to the site-specific sample matrix and a clean matrix demonstrated for all events collectively was considered to be acceptable.

### 15.5.3 Completeness

Completeness is a measure of the number of valid measurements obtained in relation to the total number of measurements planned. Completeness is expressed as the percentage of valid or usable measurements relative to number of measurements requested.

Greater than 99.3 percent of the RI/FS analytical data were considered acceptable for use in meeting project objectives as qualified. Many sample results were qualified as non-detect ("U" flagged) on the basis of contamination identified in the laboratory blanks. Additionally, many results were qualified as estimated ("J" or "UJ") based on a variety of reasons. As noted earlier, the DVRs for each sampling event provide the detailed discussion regarding all data qualifiers assigned.

Approximately 0.6 percent of the results were qualified as unusable ("R" flagged). The data user should remember that the vast majority (87 percent) of rejected data was due to two issues (see Section 15.3 for details), neither of which was considered to affect the ability to make project decisions. Excluding the data rejected for these two reasons from the rejected data set results in only 0.1 percent of the data set being qualified as unusable.

As 99.3 percent of the results were considered usable for project objectives, the QAPP completeness goal of 80 percent was satisfied.

### 15.5.4 Representativeness

Representativeness expresses the degree to which data accurately and precisely represent the environmental condition. Representativeness is achieved in part through using standard sampling and analytical procedures described in the QAPP and supporting FSP and SOPs (URS 2002c). Representativeness is also influenced by appropriate program design and such elements as proper well locations and sampling locations.

The agreement between the field duplicate results was used to assess representativeness for each of the sampling events. As relatively few data results were qualified on the basis of field duplicate disagreement, the samples collected were considered to be adequately representative of the medium sampled. Additionally, for groundwater, the good agreement between results from the same location over multiple sampling events also supports the conclusion that the samples collected are representative of the medium sampled.

Laboratory or method duplicates were used to evaluate how representative an aliquot taken from a sample was of a given sample. Again, the close agreement between the vast majority of laboratory duplicates results indicated that sample processing and sub-sampling procedures were acceptable.

### 15.5.5 Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared to another. Data sets are considered comparable only when precision and accuracy are considered acceptable during data validation. This goal was achieved through following

SOPs to collect and then analyze representative samples and through reporting analytical results in appropriate and consistent units. In essence, comparability was maintained by consistency in sampling conditions, selection of sampling procedures, sample preservation methods, analytical methods, data reporting units, and acceptable overall accuracy and precision.

### 15.5.6 Sensitivity

The analytical methods and laboratories used were selected taking into consideration the ability to meet the maximum allowable reporting limit requirements specified in the QAPP on a clean sample matrix. Barring dilutions required to eliminate interferences in groundwater samples with low pH readings, the reporting limits obtained generally meet the QAPP requirements. All reporting limits meeting the QAPP required reporting limits should be fully usable for project decision making. The data users, however, will need to assess the affect of non-detect results with elevated reporting limits on project decision-making.

## 15.6 QUALITY ASSURANCE – AQUATIC BIOTA

### 15.6.1 Field Sampling

#### *15.6.1.1 Fish*

All fish sampling (population and tissues) was conducted in accordance with the Molycorp RI Work Plan (URS 2002b) under the supervision of Chadwick. Personnel from URS, EPA, USFWS, NMF&G, and/or NMED were also present at all sites for oversight of fish sampling operations by Chadwick.

Fish population samples were processed on site in accordance with the Molycorp RI Work Plan (URS 2002b). All field forms were checked for completeness and signed by Chadwick. Fish tissue samples were collected and prepared as described in the Work Plan (URS 2002b). Each sample was labeled individually, placed on ice, entered on field COC forms which were checked by URS sample management personnel, and delivered to the sample management office managed by URS.

#### *15.6.1.2 Benthic Invertebrates*

All benthic invertebrate sampling (population and tissues) was conducted under the supervision of Chadwick. Also present were personnel from URS, EPA, USFWS, NMF&G, and/or NMED at all sites for oversight of benthic invertebrate sampling operations.

Benthic invertebrate population samples were collected and prepared as described in the Molycorp RI Work Plan (URS 2002b). Each sample was labeled individually, preserved with 95 percent ethyl alcohol, entered on chain of custody forms, and transferred to the laboratory at C&A. Benthic invertebrate tissue samples for chemical analysis were collected and prepared as described in the Molycorp RI Work Plan (URS 2002b). Each sample was labeled individually,

placed on ice, entered on the field COC forms which were checked by URS sample management personnel, and delivered to the sample management office managed by URS. All field forms were checked for completeness and signed by Chadwick personnel.

#### *15.6.1.3 Periphyton Populations and Macrophyte/Bryophyte Tissues*

All periphyton and macrophyte sampling (population and tissues) was conducted under the supervision of Chadwick. Also present for oversight of sampling operations were personnel from URS, EPA, USFWS, NMF&G, and/or NMED at all sites.

Periphyton population samples were collected and prepared by Chadwick as described in the Molycorp RI Work Plan (URS 2002b). Each sample was labeled individually, preserved with appropriate preservative, entered on COC forms, which were checked by laboratory personnel at C&A and submitted to C&A. Macrophyte/bryophyte tissue samples for chemical analysis were collected and prepared as described in the Molycorp RI Work Plan (URS 2002b). Each sample was labeled individually, placed on ice, entered on the field COC forms which were checked by URS sample management personnel, and delivered to the URS sample management office managed by URS. All field forms were checked for completeness and signed by Chadwick personnel.

#### *15.6.1.4 Habitat Evaluation*

All habitat evaluation was supervised by Chadwick. Present at all sites were personnel from URS, EPA, USFWS, NMF&G, and/or NMED for oversight of habitat evaluation. Habitat evaluation was conducted on site in accordance with the Work Plan (URS 2002f). All field forms were signed by Chadwick personnel.

### 15.6.2 Laboratory Analyses

#### *15.6.2.1 Benthic Invertebrate Laboratory*

For quality assurance for sorting and extraction of organisms from the samples, all samples (100 percent) were checked immediately upon completion by a C&A invertebrate taxonomist or an experienced C&A technician such that $100 \times X_{QA}/X_O < 5.0$, where $X_{QA}$ = the total number of organisms in the quality assurance check and $X_O$ = the total number of organisms counted by the original technician. Results were documented for 10 percent of the samples, chosen at random. A sample passed the quality assurance check if there was >95 percent thoroughness for sorting. Technicians who did not pass the quality assurance check on a sample continued sorting on the same sample until there was documented evidence that there was >95 percent thoroughness for sorting. This level of QC is more stringent than that suggested in the EPA Rapid Bioassessment Protocols, which is set at 90 percent thoroughness (Barbour et al. 1999).

In-house C&A quality assurance for identifications and enumerations were conducted after completion of the entire sample lot (an individual sample period, e.g., fall 2002). Quantitative

samples were randomly conducted on 10 percent of the samples, using the following community similarity index:

$$A = 100 \times \sum_{i=1}^{n} \left( \frac{\frac{x_{iO}}{X_O} + \frac{x_{iQA}}{X_{QA}}}{2} - \frac{\left| \frac{x_{iO}}{X_O} - \frac{x_{iQA}}{X_{QA}} \right|}{2} \right),$$

where $x_{iO}$ = the original taxonomist's count for the *i*th taxon, $X_O$ = the total number of organisms in the original taxonomist's count, $x_{iQA}$ = the count for the *i*th taxon in the quality assurance check, and $X_{QA}$ = the total number of organisms in the quality assurance check (Whittaker 1975, Stribling et al. 2003). This procedure provides the sum of the smaller of the original taxonomist's count and the count in the quality assurance check, proportionately, for each taxon. Samples passed the quality assurance check if the similarity index indicated >95 percent similarity between taxonomic identifications and enumerations. If a sample did not pass the quality assurance check, the reason was discussed between C&A invertebrate taxonomists to identify the taxon within the sample responsible for failure to pass the quality assurance check. All other samples from the same lot, which contained the suspect taxon were re-examined in regard to that suspect taxon. EPA Rapid Bioassessment Protocols suggest only spot checks for identification quality assurance (Barbour et al. 1999).

A voucher collection of all taxa collected was compiled using standard methodologies (Barbour et al. 1999) and submitted periodically to outside experts for verification of identifications.

Data were entered into C&A's in-house computer program using taxa codes (TSNs) provided in Appendix B of Barbour et al. (1999). If a taxon was not listed or a TSN was not provided in Appendix B of Barbour et al. (1999), a number was assigned to it based on the TSN of the next higher taxonomic category. Use of TSNs provided a quality assurance check on data entry, ensuring 100 percent accuracy for spelling and taxonomic organization. One of the C&A taxonomists responsible for the specimen identifications was also responsible for the quality assurance check of data entry to ensure that the transcription of enumerations was accurate.

Laboratory sample management quality assurance included COC forms with the same information as the sample container labels, and flow sheets recording the passage of samples through processing. These were submitted to URS and EPA in PDF in 2004.

#### *15.6.2.2 Bioassay Laboratory*

The C&A laboratory performed freshwater aquatic biomonitoring and toxicological tests utilizing EPA guidelines (EPA 2002a, b) or other accepted methodologies (e.g., ASTM 1988), as specified by the Molycorp RI Work Plan (URS 2002b). Quality assurance audits, as described in C&A's Freshwater Bioassay Laboratory Quality Assurance/Quality Control Manual, are performed on all personnel quarterly and on all phases of the bioassay process per test to ensure proper techniques and practices are being implemented. The bioassay laboratory also participates in the annual EPA DMR-QA laboratory performance evaluations, and the

documentation of all quality assurance procedures, including DMR-QA results, are kept on file at C&A. The laboratory manager functions as the quality assurance/quality control officer for the biomonitoring testing.

All forms generated during bioassay tests (i.e., COC forms, sample receipt, test preparation sheets, bench sheets, statistical analyses, and data reports) were examined and checked by the laboratory director and/or laboratory manager. Laboratory tests were considered to be valid if they met the data quality objectives for percent minimum significant difference (PMSD) and coefficient of variation (CV) for the tests, as determined using data from EPA's WET Interlaboratory Variability Study (EPA 2000). Precision of toxicity tests was measured through routine reference toxicant testing using NaCl such that the measured effect of a given reference test should fall within two standard deviations of the mean effect generated by the last 20 reference tests completed in the C&A laboratory.

In regard to ancillary laboratory procedures, deionized water used for reconstituted test water is analyzed yearly by an independent laboratory for toxic metals (Ag, Al, As, Cd, Cr, Co, Cu, Fe, Hg, Ni, Pb, Zn) and organic chemicals. Reconstituted laboratory water, using the deionized water, is prepared according to EPA protocols. Culturing of organisms is performed in a room isolated from all other testing to prevent contamination of organisms. All documentation sheets accompanying food received from an outside supplier (Aquatic Biosystems, Inc.) are kept on file at C&A; subsequent batches of yeast cerophyll trout chow (YCT) and *Artemia* cysts are evaluated for comparability to the previous batches.

Water and effluent samples were collected by either URS, Chadwick, or C&A personnel. For some tests conducted during the RI, personnel from EPA provided field, transit, and in-laboratory oversight of all phases of the bioassay testing process.

### 15.6.3 Data Validation

All data entered into computer forms were checked for transcription accuracy against original bench or field sheets, including fish population data (by Chadwick), benthic invertebrate population data (by a C&A invertebrate taxonomist responsible for identifications), and bioassay data (by the laboratory manager). Periphyton population samples and all tissue samples were analyzed by subcontracted laboratories with their own data validation and quality assurance procedures.

No data were removed from the database during data validation at Chadwick or C&A.

# SECTION 15

# QUALITY ASSURANCE SUMMARY

# TABLES

# Table 15-1
# 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 cannot 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 15-2

## 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 (or instrument detection limit [IDL]) and the sample quantitation limit. |
| RL | Reporting limit exceeds decision criterion (for nondetects) |
| LR | Over linear range without re-analysis |
| DC | Data Comparability |
| Hist | Results did not agree with historic data for the same sampling location |
| **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 |

# Table 15-2

## Data Validation Qualifier Codes And Bias Direction Codes

| Qualifier Code | Data Quality Condition Resulting In Assigned Qualification |
|---|---|
| **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 |

# Table 15-3
# Event Summary for Chemical Data

| Sampling Event | List of Packages | LAB | Matrix | Data Validation Report Number |
|---|---|---|---|---|
| Fall 2002 Soils and Sediments, Part A | SOL001 through SO046 (not dioxins) | STL | Soil and Sediment | 1 |
| Fall 2002 Soils, Part B (collected in Jan 2003) | SOL047 through SOL067 (not dioxins) | STL | Soil | 2 |
| June and September 2003 RI/FS Soils | SOL074 through SOL077, SOL087, and SOL 083 w/ make-up soils | STL | Soil | 3 |
| November and December 2003 Soils, Tailings, and Sediment | SOL095-SOL100 SPLP01 | STL | Soil, Tailings, Sediment | 4 |
| Fall 2002 and June 2003 Dioxins and Furans for Soil Samples | DIOX01 through DIOX04 | STL | Soil | 5 |
| Spring 2003 Sediment (March) | SOL068 through SOL073 | STL | Sediment | 6 |
| Summer 2003 Sediment (July) | SOL078 through SOL083 | STL | Sediment | 7 |
| Fall 2003 Sediment (September) | SOL088 through SOL093 | STL | Sediment | 8 |
| Fall 2002 Biota | BIO001 through BIO034 | STL | Biota | 9 |
| Fall 2002 Small Mammals | 829551, 829551A, 829551B | EnChem | Biota | 10 |
| Spring 2003 Benthic Tissue (BMI) | BIO035 and BIO036 | STL | Biota | 11 |
| June 2003 Small Mammals (make-up samples) | 835320A, 835320B, 835320C | EnChem | Biota | 12 |
| June 2003 Worms | BIO043 | STL | Biota | 13 |
| Fall 2003 Aquatic Biota | BIO048 through BIO057 | STL | Biota | 14 |
| June, August, and September 2003 RI/FS Plants (includes Edible Riparian Plants) | BIO037 through BIO042, BIO047 | STL | Biota | 15 |
| Choke Cherries | BIO045 and BIO046 | STL | Biota | 16 |
| | | | | |

STL = Severn Trent Laboratories, Burlington, Vermont.
Limited subcontracting to other STL laboratories was done.
Based on required analyses (i.e., Dionxin/Furnans) or instrument failure or overload.

FGS = Frontier Geosciences, Inc.
USGS = United States Geological Society
U o f AZ = University of Arizona
U of Miami = University of Miami
ACZ = ACZ Laboratories

## Table 15-3 (continued)
## Event Summary for Chemical Data

| Sampling Event | List of Packages | LAB | Matrix | Data Validation Report Number |
|---|---|---|---|---|
| WIS Plants (June and September 2003) | WISB01 through WISB14 | STL | Biota | 17 |
| WIS Soil (June and September 2003) | WISS01 through WISS05 | STL | Soil | 18 |
| Vegetable Gardens, Soil, Irrigation Water, and Riparian Soil | BIO044, SOL084 through SOL087, WAT157 and WAT162 | STL | Multiple | 19 |
| Fall 2002 Groundwater and Surface Water | WAT001 through WAT027, WATRAA1, WATRAA2, WARTRABC1, WATRAF1 | STL | Groundwater and Surface Water | 20 |
| Spring 2003 Surface Water (March) | WAT057 through WAT063, WAT087C (UFL), WATRAS1, WATRAS2, WATRAS3 | STL | Surface Water | 21 |
| Summer 2003 Surface Water (July) | WAT132 through WAT135, WAT138, WAT139, WAT141-143, WAT151, and WAT150S | STL | Surface Water | 22 |
| Fall 2003 Surface Water (September) | WAT178 through WAT185 | STL | Surface Water | 23 |
| Snowmelt (April 2003) | WAT094C through WAT102C | STL | Surface Water | 24 |
| Storm Event #1 | WAT155, WAT156 | STL | Surface Water | 25 |
| Storm Event #2 | WAT160, WAT164, WAT165 | | Surface Water | 26 |
| Storm Events #3, #4, and #5 | WAT166 through WAT168 and WAT175 (#5) | STL | Surface Water | 27 |
| Groundwater/ Surface Water Interaction Study (GSI) | WAT187 through WAT191 WAT192 through WAT195, WAT194SA (reanalysis LR-16, Day 3, PS) WAT197 - WAT199 SOL094 | STL and on-site | Surface Water Piezometer Water Chamber Water Sediment | 28 |
| Fall 2003 Hexavalent Chromium | Hex 01 through Hex 12 | STL onsite | Groundwater | 29 |

STL = Severn Trent Laboratories, Burlington, Vermont.
Limited subcontracting to other STL laboratories was done.
Based on required analyses (i.e., Dionxin/Furnans) or instrument failure or overload.

FGS = Frontier Geosciences, Inc.
USGS = United States Geological Society
U o f AZ = University of Arizona
U of Miami = University of Miami
ACZ = ACZ Laboratories

## Table 15-3 (continued)
## Event Summary for Chemical Data

| Sampling Event | List of Packages | LAB | Matrix | Data Validation Report Number |
|---|---|---|---|---|
| December 2002 Monthly Groundwater and Surface Water | WAT027 - WAT030, WATRAA1, WATRABC1, WATRAF1 | STL | Groundwater and Surface Water | 30 |
| January 2003 Quarterly Groundwater and Surface Water | WAT031-WAT045 WATRAS1 | STL | Groundwater and Surface Water | 31 |
| February 2003 Monthly Groundwater and Surface Water | WAT046-WAT051, WATRAF2, WATRAS1 | STL | Groundwater and Surface Water | 32 |
| March 2003 Monthly Groundwater and Surface Water | WAT052-WAT056, WATRAF2, WATRAS1, WATRAS2, WATRAS3 | STL | Groundwater and Surface Water | 33 |
| April 2003 Quarterly Groundwater and Surface Water | WAT064-WAT093 (only RBs in WAT087C), WAT095, WAT099, WAT103, WATRAF2, WATRAS3, WATRAS4 (Outfall 002 Al, As, Cd) | STL | Groundwater and Surface Water | 34 |
| May 2003 Monthly Groundwater and Surface Water | WAT104-WAT112 | STL | Groundwater and Surface Water | 35 |
| June 2003 Monthly Groundwater and Surface Water | WAT113-WAT121 | STL | Groundwater and Surface Water | 36 |
| July 2003 Quarterly Groundwater and Surface Water (US and DS of Springs 13 and 39) | WAT122-WAT154 WATRAS4 (Outfall 002 Al, As, Cd) | STL | Groundwater and Surface Water | 37 |
| August 2003 Monthly Groundwater and Surface Water | part of WAT157, WAT158-WAT161, WAT163-WAT165 (not WAT162) | STL | Groundwater and Surface Water | 38 |
| September 2003 Monthly Groundwater and Surface Water | WAT166-WAT177 | STL | Groundwater and Surface Water | 39 |

STL = Severn Trent Laboratories, Burlington, Vermont.
Limited subcontracting to other STL laboratories was done.
Based on required analyses (i.e., Dionxin/Furnans) or instrument failure or overload.

FGS = Frontier Geosciences, Inc.
USGS = United States Geological Society
U o f AZ = University of Arizona
U of Miami = University of Miami
ACZ = ACZ Laboratories

## Table 15-3 (continued)
## Event Summary for Chemical Data

| Sampling Event | List of Packages | LAB | Matrix | Data Validation Report Number |
|---|---|---|---|---|
| October 2003 Quarterly Groundwater and Surface Water | WAT186, WAT196, WAT200-WAT224 | STL | Groundwater and Surface Water | 40 |
| November 2003 Monthly Groundwater and Surface Water | WAT225-WAT231 | STL | Groundwater and Surface Water | 41 |
| December 2003 Monthly Groundwater and Surface Water | WAT232-WAT238 | STL | Groundwater and Surface Water | 42 |
| January 2004 Quarterly Groundwater and Surface Water | WAT239-WAT260 | STL | Groundwater and Surface Water | 43 |
| April 2004 Quarterly Groundwater and Surface Water | WAT272-WAT293 | STL | Groundwater and Surface Water | 46 |
| February 2004 Specialty Sampling | WAT261-WAT265 | STL<br>U of AZ<br>U of Miami<br>FGS | Groundwater | 44 |
| March 2003 GSI (Round 2) | WAT267-WAT271 | STL | Groundwater and Surface Water | 45 |
| May Groundwater (Spring 13, MMW-50A, Douglas) | WAT298 | STL | Groundwater | No R47; data were included with R48 |
| Historic Tailings Spill Investigation & Hunts Pond (and May GRWs, Spring 13, MMW-50A, Douglas) | WAT297, WAT299 SOL103-SOL109 SPLP02 and WAT298 | STL | Soil, Sediment, Groundwater, and Surface Water | 48 |
| Supplemental Sampling South of Tailings | WAT294-WAT297 BIO058-BIO060 SOL102 | STL | Groundwater, Soil, Plants | 49 |
| September 2004 Serial Dilution Study | WAT301-WAT303 WAT307, WAT310, WAT314 | STL | Surface Water Mixing Water | 50 |

STL = Severn Trent Laboratories, Burlington, Vermont.
Limited subcontracting to other STL laboratories was done.
Based on required analyses (i.e., Dionxin/Furnans) or instrument failure or overload.

FGS = Frontier Geosciences, Inc.
USGS = United States Geological Society
U o f AZ = University of Arizona
U of Miami = University of Miami
ACZ = ACZ Laboratories

## Table 15-3 (continued)
## Event Summary for Chemical Data

| Sampling Event | List of Packages | LAB | Matrix | Data Validation Report Number |
|---|---|---|---|---|
| September 2004 Benthic Survey Study | WAT302, WAT304-WAT306 SOL110-SOL111 | STL | Surface Water Sediment | 51 |
| September 2004 GSI Round 3 Study | WAT306, WAT308, WAT309, WAT311, WAT312, WAT313, WAT315-WAT318 SOL114 | STL | Surface Water Piezometer Water Chamber Water Sediment | 52 |
| September 2004 Radon Tracer Study | WAT319, WAT320 | STL & USGS | Surface Water | 53 |

STL = Severn Trent Laboratories, Burlington, Vermont.
Limited subcontracting to other STL laboratories was done.
Based on required analyses (i.e., Dionxin/Furnans) or instrument failure or overload.

FGS = Frontier Geosciences, Inc.
USGS = United States Geological Society
U o f AZ = University of Arizona
U of Miami = University of Miami
ACZ = ACZ Laboratories

# Table 15-4
## Analyses Strategy For Lower pH Samples

| Analyte | pH Class A (pH < 5.6) | Dilution Adjusted IDL, ug/l | Lowest Evaluation Criterion, ug/l | QAPP RL (ug/L) | ICP | ICPMS |
|---|---|---|---|---|---|---|
| Al | ICP analysis at 100x dilution | 2,260 | 87 | 40 | 22.6 | 5.3 |
| Sb | ICP analysis at 10x dilution | 50 | 6.0 | 0.7 | 5 | 0.3 |
| As | ICP analysis at 10x dilution | 67 | 50 (0.010) | 2 | 6.7 | 0.2 |
| B | ICP analysis at 10x dilution | 27 | 1.6 | 160 | 2.7 | |
| Ba | ICP analysis at 10x dilution | 84 | 4.0 | 130 | 8.4 | 0.5 |
| Be | ICP analysis at 10x dilution | 3 | 4.0 | 2 | 0.3 | 0.1 |
| Cd | ICP analysis at 100x dilution | 80 | 0.25 | 0.9 | 0.8 | 0.1 |
| Ca | ICP analysis at 100x dilution | 23,400 | -- | 200 | 234 | 17.4 |
| Co | ICP analysis at 100x dilution | 230 | 50 | 10 | 2.3 | 0.1 |
| Cr | ICP analysis at 100x dilution | 370 | 50 | 5 | 3.7 | 0.1 |
| Cu | ICP analysis at 100x dilution | 170 | 8.9 | 4 | 1.7 | 0.3 |
| Pb | ICPMS analysis at 10x dilution | 1 | 2.5 | 1 | 1.7 | 0.1 |
| Fe | ICP analysis at 100x dilution | 4,890 | 1,000 | 500 | 48.9 | 5.5 |
| Mg | ICP analysis at 100x dilution | 26,940 | -- | 200 | 269.4 | 5.2 |
| Mn | ICP analysis at 100x dilution | 280 | 200 | 60 | 2.8 | 0.1 |
| Ni | ICP analysis at 100x dilution | 340 | 52 | 20 | 3.4 | 0.3 |
| Mo | ICP analysis at 10x dilution | 17 | 180 | 3 | 1.7 | 0.2 |
| Ag | ICPMS analysis at 10x dilution | 16 | 0.32 | 1 | 1.6 | 0.1 |
| Tl | ICPMS analysis at 10x dilution | 29 | 1.7 | 0.1 | 2.9 | 0.1 |
| K | ICP analysis at 100x dilution | 31,410 | -- | 200 | 314.1 | |
| Se | ICPMS analysis at 10x dilution | 8 | 5 | 2 | 2.8 | 0.8 |
| V | ICPMS analysis at 10x dilution | 2 | 19.0 | 10 | 1.8 | 0.2 |
| Na | ICP analysis at 100x dilution | 36,560 | -- | 200 | 365.6 | |
| Zn | ICP analysis at 100x dilution | 390 | 117 | 50 | 3.9 | 0.5 |
| | QAPP Table acknowledged that criteria below 50 ug/l would not be met by conventional methods. | | | | | |

# Table 15-5
## Analyses Strategy For Moderate pH Samples

| Analyte | pH Class B (5.6<pH<6.7) pH Class C (pH > 6.7) | Dilution Adjusted IDL, ug/l | Lowest Evaluation Criterion, ug/l | QAPP RL (ug/L) | ICP | ICPMS |
|---|---|---|---|---|---|---|
| Al | ICP analysis at 10x dilution | 226 | 87 | 40 | 22.6 | 5.3 |
| Sb | ICPMS analysis at 2x dilution | 0.6 | 6.0 | 0.7 | 5 | 0.3 |
| As | ICPMS analysis at 2x dilution | 0.4 | 50 (0.010) | 2 | 6.7 | 0.2 |
| B | ICP analysis straight | 2.7 | 1.6 | 160 | 2.7 | |
| Ba | ICP analysis straight | 8.4 | 4.0 | 130 | 8.4 | 0.5 |
| Be | ICP analysis straight | 0.3 | 4.0 | 2 | 0.3 | 0.1 |
| Cd | ICP analysis straight | 0.8 | 0.25 | 0.9 | 0.8 | 0.1 |
| Ca | ICP analysis at 10x dilution | 2,340 | -- | 200 | 234 | 17.4 |
| Co | ICP analysis straight | 2.3 | 50 | 10 | 2.3 | 0.1 |
| Cr | ICP analysis straight | 3.7 | 50 | 5 | 3.7 | 0.1 |
| Cu | ICP analysis straight | 1.7 | 8.9 | 4 | 1.7 | 0.3 |
| Pb | ICPMS analysis at 2x dilution | 0.2 | 2.5 | 1 | 1.7 | 0.1 |
| Fe | ICP analysis at 10x dilution | 489 | 1,000 | 500 | 48.9 | 5.5 |
| Mg | ICP analysis at 10x dilution | 2,694 | -- | 200 | 269.4 | 5.2 |
| Mn | ICP analysis at 10x dilution | 28 | 200 | 60 | 2.8 | 0.1 |
| Ni | ICP analysis straight | 3.4 | 52 | 20 | 3.4 | 0.3 |
| Mo | ICP analysis straight | 1.7 | 180 | 3 | 1.7 | 0.2 |
| Ag | ICPMS analysis at 2x dilution | 0.2 | 0.32 | 1 | 1.6 | 0.1 |
| Tl | ICPMS analysis at 2x dilution | 0.2 | 1.7 | 0.1 | 2.9 | 0.1 |
| K | ICP analysis straight | 314.1 | -- | 200 | 314.1 | |
| Se | ICPMS analysis at 2x dilution | 1.6 | 5 | 2 | 2.8 | 0.8 |
| V | ICPMS analysis at 2x dilution | 0.4 | 19.0 | 10 | 1.8 | 0.2 |
| Na | ICP analysis at 10x dilution | 3,656 | -- | 200 | 365.6 | |
| Zn | ICP analysis at 10x dilution | 39 | 117 | 50 | 3.9 | 0.5 |
| | QAPP Table acknowledged that criteria below 50 ug/l would not be met by conventional methods. | | | | | |

# SECTION 15

# QUALITY ASSURANCE SUMMARY

# FIGURES

**Figure 15-1**
**Summary of Surface Water Contaminant**
**Frequency of Detection**

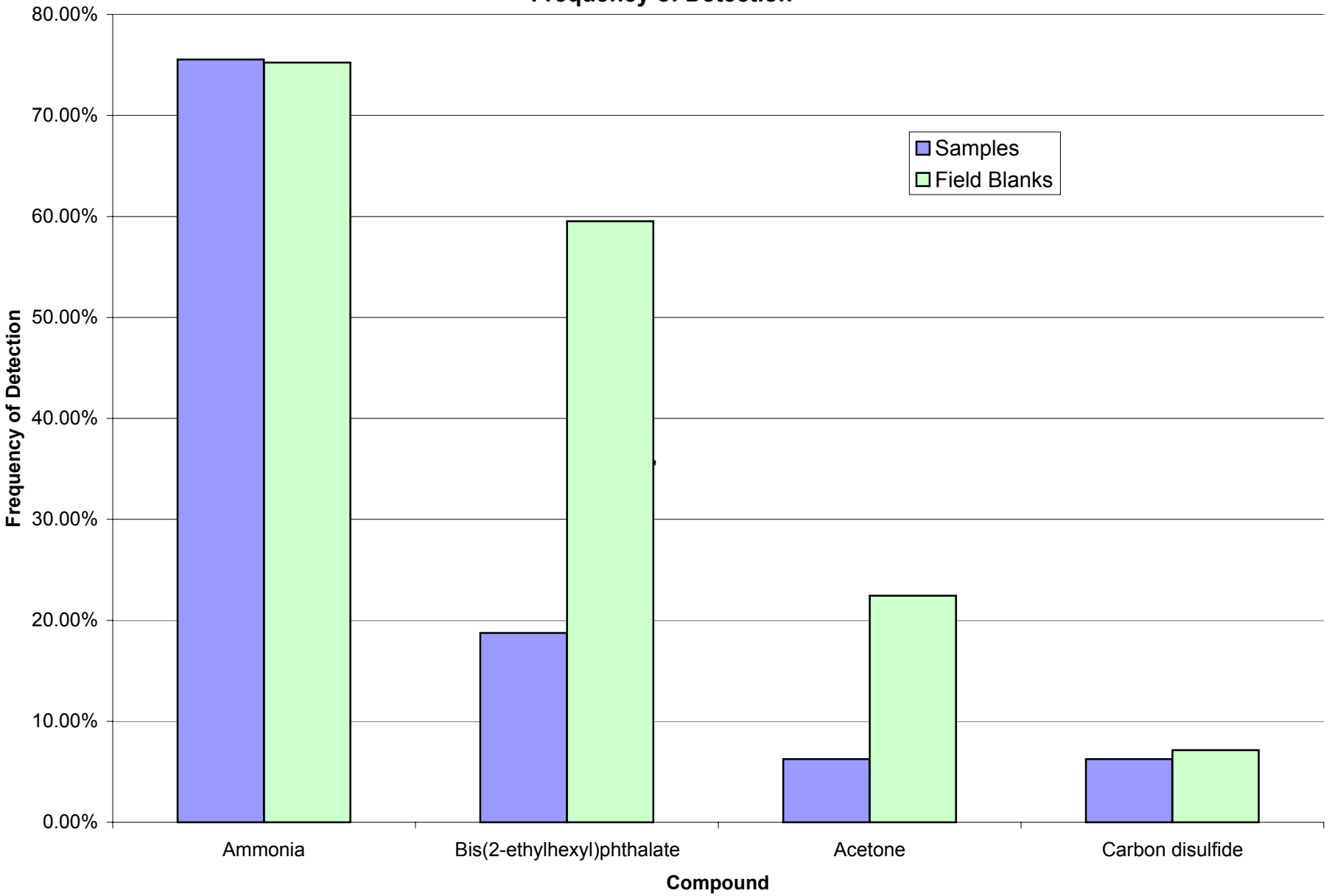


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**Figure 15-2**
**Summary of Soil/Sediment Contaminant**
**Frequency of Detection**

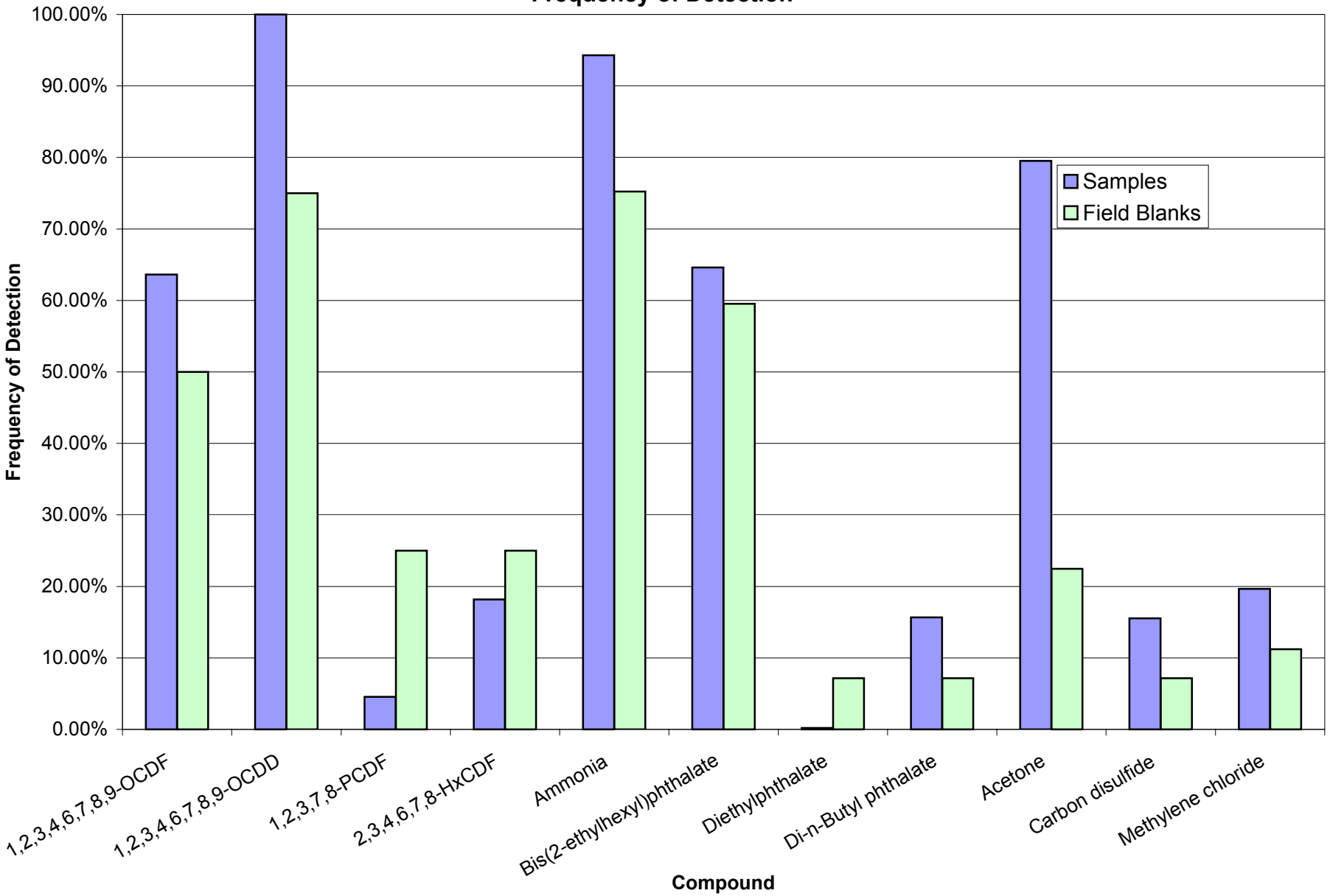
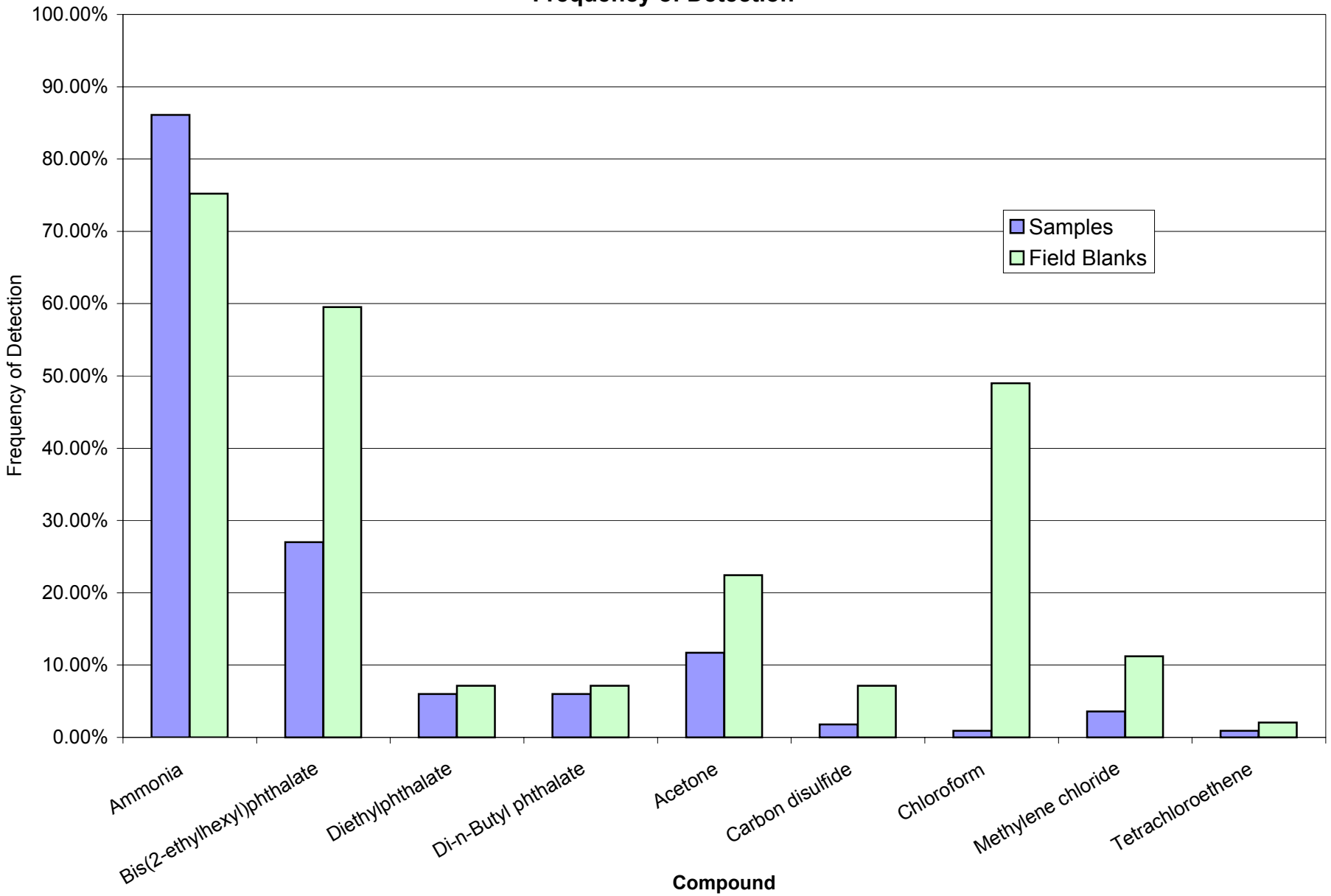


**Figure 15-3**
**Summary of Groundwater Contaminant**
**Frequency of Detection**

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# APPENDIX A-15

# SECTION 15
# QUALITY ASSURANCE

# PLACEHOLDER