STANDARD OPERATING PROCEDURE NO. 22

ANALYTICAL DATA VALIDATION

REVISION LOG		
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1.0 PURPOSE AND SCOPE

This Standard Operating Procedure describes the procedures to be used to conduct an independent review of environmental analytical laboratory data so that data used for all project reporting and environmental decision making for the Molycorp, Inc. (hereafter referred to as Molycorp) Rock Pile Stability Project will be of a quality appropriate for its intended use. This SOP includes two levels of data review, evaluation of sample-specific parameters and evaluation of laboratory performance parameters.

2.0 RESPONSIBILITIES AND QUALIFICATIONS

The Team Leader and Characterization Team will have the overall responsibility for implementing this SOP. They will be responsible for assigning appropriate staff to implement this SOP and for ensuring that the procedures are followed accurately.

Personnel performing data validation are required to have a complete understanding of the procedures described within this SOP and to receive specific training regarding these procedures, if necessary.

All environmental staff and assay laboratory staff are responsible for reporting deviations from this SOP to the Characterization Team Leader.

3.0 DATA QUALITY OBJECTIVES

4.0 RELATED STANDARD OPERATING PROCEDURES

- SOP 1 Data Management
- SOP 2 Sample Management
- SOP 25 Stable Isotope analyses
- SOP 26 Electron microprobe analyses
- SOP 27 X-ray diffraction analyses
- SOP 28 X-ray fluorescence analyses
- SOP 29 Clay Mineralogy analyses
- SOP 30 ICP-OES analyses
- SOP 31 ICP-MS analyses
- SOP 68 Water Analyses
- All other project SOPs dealing with analytical data generation

5.0 DATA REVIEW PROCEDURES

As noted in Section 1.0, analytical data used for reporting and environmental decision making for the Molycorp rock pile stability project will receive a review independent of the laboratory to ensure that data are of known and documented quality.

5.1 Sample-Specific Parameters

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). Sample-specific parameters shall be reviewed and evaluated.

5.2 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. 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 shall be reviewed for at least 10% of the project 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 to document the results of all data review activities, all data qualification assigned, and any limitations on the use of the data.

6.0 REVIEW OF SAMPLE-SPECIFIC PARAMETERS

6.1 Case Narrative Comments

Data validation begins with an examination of the case narrative. 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.

6.2 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.

6.3 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 the QAPP and/or respective analytical methods, as applicable. Results for analyses not performed within holding time limits will be qualified as estimated ("J/UJ" in Table 1). If the holding time is grossly exceeded (more than two times the holding time limit), the data reviewer should use professional judgment to evaluate the need to reject non-detectable results.

A qualifier code of "HT" (see Table 2) will be assigned to results qualified or rejected on the basis of holding times.

7.0 REVIEW OF LABORATORY PERFORMANCE PARAMETERS

7.1 Cation-Anion Balance

As another QC check, groundwater and surface water samples for which both cation and anion concentrations are reported shall be evaluated to determine the cation-anion balance. Concentrations of dissolved major cations (calcium, magnesium, sodium, potassium, and others as appropriate) will be compared to concentrations of major anions (sulfate, chloride, carbonate, bicarbonate, and others as appropriate). If the cation-anion ratio does not balance, the laboratory may be requested to reanalyze the subject samples.

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 samples being analyzed for major cations and major anions, the data reviewer shall evaluate whether there is an acceptable balance between anion concentrations and cation concentrations. In accordance with Standard Method #1030F, the equation used to calculate anion-cation balances is:

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

Laboratory accuracy control limits for these types of analytes typically have a bias of ±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 will use professional judgment to discern likely causes of the imbalance and any need for qualification of that data.

8.0 DOCUMENTATION

8.1 Data review worksheets

This section describes the documentation that will be generated as part of the data review procedure. Appendix 1 contains generic data review worksheets which are tools the reviewer may elect to use to facilitate the review. Data validation results will be documented in a narrative report. Section 8.2 describes the contents of the resultant data validation reports.

Figures 1 and 2 in Appendix 1 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. Because of space limitations and the number of analytical methods, the specific evaluation criteria are not included in the tables. The Molycorp Rock Pile Stability Study QAPP and/or 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.

8.2 Data Review Narrative Reports

Data review activities shall be detailed in a data validation narrative report. At a minimum, the report shall 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 shall 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. Data review reports will be peer reviewed by a qualified person to assure compliance with the procedures described in this SOP.

9.0 DATA VALIDATION TABLES

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

10.0 REFERENCES

Standard Method #1030F The accept/reject criteria for cation-to-anion balance is described in Standard Method 1030F in the EPA-approved 1992 Standard Methods for the Examination of Water and Wastewater (18 th edition).

USEPA Contract Laboratory Program (CLP) National Functional Guidelines for Inorganic Data Review, (EPA-540/R-94/013, February 1994).

USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, (EPA540/R-99/008) October 1999.

APPENDIX 1. Data Review Worksheets

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		

Parameter	Criteria	Criteria Satisfied?	Details	Actions (qualified data)
• Field Blank • Trip Blank • Other (e.g., splits)		Y N NA Y N NA Y N NA		
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		