Prepared for:
Tronox LLC
Henderson, Nevada

Data Validation Summary Report

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1.0 INTRODUCTION

The purpose of limited data validation performed on laboratory results for the third quarter of 2007 was to determine the suitability of the data for future on-site environmental assessments, including the Quarterly Performance Perchlorate Report on the Perchlorate Recovery System for July-September of 2007.

MWH Laboratories in Monrovia, CA was the laboratory contracted by Tronox for the chemical analyses discussed below as a part of the routine monitoring program at the Tronox facility in Henderson, Nevada.

The specific analyses performed by the laboratory and reviewed in this report include all hexavalent chromium, total chromium, total dissolved solids (TDS), nitrate, and chloride analyses provided by MWH in the selected analytical reports and not just the perchlorate results required for the quarterly report.

2.0 DATA VALIDATION PROCESS

All the specified results contained in the laboratory reports listed in the data validation memorandum were subjected to thorough data review known as limited validation. Ten percent of the data packages were provided by the laboratory as CLP-like deliverables and these were subjected to formal full data validation as recommended in the guidance on data validation provided by NDEP for the BMI Plant Sites (NDEP, 2006). The laboratory submitted sample and batch QC results with narratives in pdf format and EQuIS format EDDs for all reports. The required extra raw data needed for full data validation was submitted for three reports The EDDs were imported into an EQuIS database, specifically created for the ongoing monitoring at the Henderson site, at Tronox. ENSR performed a limited validation on the data using the hard copy data package and subsequently entered the validation qualifiers into the database.

Limited validation consisted of reviewing the following data elements based on review of summary data forms.

- Agreement of analyses conducted with chain-of-custody (COC) requests
- Holding times and sample preservation
- Laboratory blanks/equipment blanks/ field blanks
- Laboratory control sample/ laboratory control sample duplicate (LCS/LCSD) results
- Matrix spike/matrix spike duplicate (MS/MSD) results
- Laboratory duplicate results
- Field duplicate results
- Quantitation limits and sample results

Full validation consisted in reviewing the above data elements plus the following extra elements, based on raw data review.

- Initial and continuing calibrations
- Interference check sample results

- ICP serial dilution results

- Calculations and transcription verifications

Analytical data were evaluated with reference to the National Functional Guidelines (EPA, 1999 and 2004) and other method appropriate validation guidance documents, as well as the Region 9 Superfund Data Evaluation/Validation Guidance (EPA, 2001), the above mentioned NDEP Guidance on Data Validation (NDEP, 2006), the quality control (QC) criteria provided by the laboratory. The Regional and National Functional Guidelines were modified to accommodate the non-CLP methodologies. The specific guidelines used for the various methods were as follows:

- Inorganic analytical data were evaluated with reference to "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review" (EPA, 2004)

In general, the validation qualifiers and definitions employed were based on those used by EPA in the document mentioned above. Validation qualifiers and definitions are listed in **Table D-1**. A reason code was assigned to all the applications of validation qualifiers for this project. The reason codes and their explanations are listed in **Table D-2**. These codes were entered in the project database for each application of a validation qualifier that changed a lab qualifier or result value to indicate the primary reason(s) for data qualification. Conversions of the laboratory reported "ND" (not detected) to the U qualifier (see Table D-1) in the database are not further discussed in this report. In addition, the laboratory-applied "J" qualifier to indicate results less than the reporting limit but greater than the method detection limit were not changed and are not further discussed in this report.

Data validation was organized by MWH Laboratory Report which is also identified as the sample delivery group (SDG) in the tables. Two data validation memoranda, one for all the limited validation, and one for all the full validation, were written and reviewed at ENSR's Westford office. These memoranda are included on CD-ROM as a pdf document. Each memorandum includes a list (in Appendix A) of the laboratory SDGs reviewed.

3.0   DATA VALIDATION RESULTS

The data validation qualifiers and reason codes were used to select all the data in the database where results were qualified as a result of validation. This information was sorted by the quality control (QC) review elements listed below:

- Agreement of analyses conducted with chain-of-custody (COC) requests

- Initial and continuing calibrations (full validation only)

- Interference check sample results (full validation only)

- Holding times and sample preservation

- Laboratory blanks/equipment blanks/ field blanks

- Laboratory control sample/ laboratory control sample duplicate (LCS/LCSD) results

- Matrix spike/matrix spike duplicate (MS/MSD) results

- Laboratory duplicate results

- Field duplicate results

- ICP serial dilution results (full validation only)

- Quantitation limits and sample results

- Calculations and transcription verifications (full validation only)

Tables D-3 lists all the results which were qualified based on quality control issues identified with regard to holding times and laboratory duplicate results. No QC issues were identified that resulted in qualification of results based on blank contamination, LCS/LCSD results, MS/MSD results, field duplicate results, or quantitation limits. Reason codes, Data Quality Indicators (DQI), and the nonconforming DQI results are listed in the table as requested by NDEP. **Table D-4** lists all the SDGs, sample IDs, and the specific analyses reviewed. SDGs subjected to full validation are shown in bold. All other SDGs underwent limited validation.

3.1   Holding Times and Sample Preservation

Holding times were derived from the EPA methods utilized and were calculated beginning from the time of sample collection. The majority of analyses were performed within the method-specified holding times. Exceptions are listed in **Table D-3** and summarized in the validation memoranda. The DQI result value for holding time in Table D-3 is the time elapsed between sample collection and analysis. The holding time for hexavalent chromium in water is 24 hours from collection to analysis. The holding time for perchlorate in water is 28 days from collection to analysis. The holding time for TDS in water is 7 days from collection to analysis. No data were rejected on the basis of holding time exceedances but some results were qualified as estimated. Results for hexavalent chromium and TDS required qualification on the basis of holding time issues as discussed in the data review memoranda. Where the TDS holding time was exceeded, TDS results were qualified as estimated, biased low (J-) because the method specifically mentions potential biodegradation of solids as the reason samples should be filtered as soon as possible. The hexavalent chromium qualifiers for the holding time exceedance were not assigned a bias (low or high) because it is unclear which direction (positive or negative bias) the result would deviate. Hexavalent chromium concentrations can change unpredictably over time in response to absorption of gases, pH changes, and redox condition changes

3.2   Blank Contamination

In general, laboratory and field blanks were free of contamination. The equipment blanks collected on 7/31/07 and 8/1/07 contained low levels of perchlorate and/or TDS. The associated samples were all more than ten times greater in concentration for these analytes, therefore no data required qualification due to blank contamination.

3.3   Laboratory Control Samples

LCS and LCSD recoveries met QC acceptance criteria for all of the analyses reviewed

3.4   Matrix Spike Samples

MS and MSD recoveries met the QC acceptance criteria for all the analyses reviewed in this report with the exception of M-71. The spike amount was significantly lower than the sample concentration in this case that the data was not useable and therefore no validation action was taken based on the spike recovery failure.

3.5   Laboratory Duplicates

The evaluation of laboratory duplicate precision included an assessment of the agreement between LCS and LCSDs, MS and MSDs, and matrix duplicates, as measured through relative percent difference (RPD). These results met the QC acceptance criteria for all of the analyses reviewed with the exception of the TDS results for PC-18. The positive TDS results in all associated samples were therefore qualified as estimated as shown in Table D-3.

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3.6   Field Duplicates

The results of the four groundwater sample duplicate pairs collected during the third quarter of 2007 were evaluated during validation. RPDs were compared to the objectives of 30% maximum RPD for aqueous samples. No results were qualified during validation based on field duplicate precision nonconformances.

3.7   Quantitation Limits and Sample Results

No results were qualified based on QC related to quantitation limits or sample results reported.

3.8   Rejected Results

No results in the reviewed dataset were rejected based on validation criteria or QC nonconformances.

4.0   EVALUATION OF DATA QUALITY INDICATORS

Data validation information was used to evaluate the data quality indicators (DQI) of precision, accuracy, representativeness, comparability, completeness, and sensitivity for results in the dataset for the Henderson Quarterly Performance Perchlorate Report. Each of these DQI parameters is discussed in sections below.

4.1   Precision

Precision is the measure of agreement among repeated measurements of the same property under identical or substantially similar conditions. Field precision was assessed through the collection and measurement of field duplicates and expressed as the RPD of the sample and field duplicate pair results. In general the field duplicate precision was acceptable for all analytes reported.

Laboratory precision was assessed through the RPD results for matrix duplicates, LSC/LCSD pairs, and MS/MSD pairs. In general, the laboratory duplicate precision was acceptable, except as noted above in Section 3.5.

4.2   Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference or true value. Laboratory accuracy was assessed during the validation using the recoveries of positive control samples (i.e., MS and MSD, LCS and LCSD). Accuracy is also indirectly addressed via the negative control samples for field activities (i.e. trip, equipment, and field blanks), as well as laboratory negative control samples (i.e., method blanks and calibration blanks). All negative control sample results were acceptable with the exceptions discussed above in Section 3.2.

Bias as a component of accuracy is also evaluated with the validation of holding time results discussed in Section 3.1 of this report. These evaluations resulted in the minor qualification of some results as described in the data validation memoranda and Section 3.1 of this report.

4.3   Representativeness

Representativeness is the measure of the degree to which data suitably represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Aspects of representativeness addressed during validation include the review of sample collection information in the chain-of-custody (COC) documentation, conformity of laboratory analyses to workplan intentions, adherence of the documented laboratory procedures to method requirements, and completeness

of the laboratory data packages. Most of the issues identified during this evaluation did not result in the qualification of laboratory data but did involve re-submittals of data from the laboratories to correct problems that were discovered during the validation process. All of these issues were resolved or were judged to have no impact on data validation. Other aspects of data representativeness such as adherence to recommended holding times are discussed in Section 3.1 of this report.

4.4   Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system, expressed as a percentage of the number of valid measurements that were or should have been collected. Valid data is defined as all the data points judged to be valid (i.e. not rejected), as a result of the validation process.

Field completeness is defined as the percentage of samples actually collected versus those intended to be collected in accordance with the plan for routine monitoring. All intended samples were collected in accordance with the monitoring schedule. All COC requests were faithfully executed by the laboratories with the minor exceptions discussed in the validation memorandum.

Laboratory completeness is defined as percentage of valid data points versus the total expected from the laboratory analyses. Actual laboratory completeness was 100% on the basis of sample analysis (i.e., all requested analyses were performed and reported by the laboratories), and 100% completeness based on valid data as a percentage of the total data points attempted.

4.5   Comparability

Comparability is a qualitative expression of the measure of confidence that two or more data sets may contribute to a common analysis. Comparability of data within the investigation was maximized by using standard methods for sampling and analysis, reporting data, and data validation. Standard water/wastewater program methods from EPA were employed by the MWH laboratory for all analyses.

4.6   Sensitivity

Sensitivity is the capability of a method or instrument to discriminate between measurement responses representing different levels of the variable of interest and particularly the capability of measuring a constituent at low levels. For the EPA methods employed in this project sensitivity is measured by the method detection limit (MDL) and reporting limit (RL). Reporting limits in general were sample quantitation limits based on the low point of calibration and adjusted for sample-specific factors such as exact aliquot size, dilutions, etc. Sensitivity of the methods employed was adequate for the routine monitoring needs and consistent with the historical data for the site.

5.0   CONCLUSIONS

One hundred percent of the laboratory data for the Quarterly Performance Report for the Perchlorate Recovery System covering July to September 2007 were subjected to a limited validation using standardized guidelines and procedures recommended by EPA and NDEP. Ten percent of the laboratory SDGs were subjected to full data validation as requested by NDEP. Ninety-eight percent of the reviewed results for this project were accepted as reported by the laboratory without additional qualification based on validation actions and should be considered valid for all decision making purposes. A subset of the laboratory results were qualified based on issues discovered during the validation process and those results are summarized in Tables D-3. The qualified data are grouped in this table based on the reason for qualification (see Table D-2), the Data Quality Indicator (DQI) involved, and the qualifier flags applied (see Table D-1). Two percent of

the results for this project were qualified as estimated due to QC problems with sample holding time and laboratory duplicate precision. These estimated results should be considered usable for decision making purposes provided the potential bias is considered when the data are used. No results were rejected as unusable due to serious QC problems. Based on the results of data validation, the overall goals for data quality were achieved for the dataset used in the Quarterly Performance Report for the Perchlorate Recovery System covering July to September 2007.

6.0   REFERENCES

EPA, 1999   USEPA "Contract Laboratory Program National Functional Guidelines for Organic Data Review"

EPA, 2001   USEPA "Draft Region 9 Superfund Data Evaluation/Validation Guidance"

EPA, 2004   USEPA "Contract Laboratory Program National Functional Guidelines for Inorganic Data Review"

ENSR, August 2006   DRAFT Quality Assurance Project Plan, Tronox LLC Facility Henderson, Nevada

NDEP, 2006   NDEP "Guidance on Data Validation, BMI Pant Sites and Common Areas Projects, Henderson, Nevada"