Data Validation Summary Report Baseline Groundwater Sampling Nevada Environmental Response Trust Site Henderson, Nevada

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I hereby certify that all laboratory analytical data was generated by a laboratory certified by the NDEP for each constituent and media presented herein.

I hereby certify that I am responsible for the services described in this document and for the preparation of this document. The services described in this document have been provided in a manner consistent with the current standards of the profession and, to the best of my knowledge, comply with all applicable federal, state and local statutes, regulations and ordinances.

Susan M. Crowley, CEM 1428 Exp.:03/08/11

Crowley Environmental LLC

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ACRONYMS AND ABBREVIATIONS

Acronym	Meaning
%D	Percent Difference
BRC	Basic Remediation Company
CLP	Contract Laboratory Program
DOC	Dissolved Organic Carbon
DQI	Data Quality Indicator
DUP	Duplicate
EDD	Electronic Data Deliverable
EPA	U.S. Environmental Protection Agency
GC/MS	Gas Chromatograph/Mass Spectrometer
ICP	Inductively Coupled Plasma
LCS/LCSD	Laboratory Control Sample/ Laboratory Control Sample Duplicate
LDC	Laboratory Data Consultants
MDL	Method Detection Limit
MS/MSD	Matrix Spike/Matrix Spike Duplicate
NDEP	Nevada Division of Environmental Protection
OCP	Organochlorine Pesticides
PARCCS	Precision, Accuracy, Representativeness, Comparability, Completeness, and Sensitivity
PQL	Practical Quantitation Limit
QAPP	Quality Assurance Project Plan
QC	Quality Control
RPD	Relative Percent Difference
SDG	Sample Delivery Group
SOP	Standard Operating Procedure
SQL	Sample Quantitation Limit
TDS	Total Dissolved Solids
TOC	Total Organic Carbon
Tronox	Tronox LLC
VOC	Volatile Organic Compound

1.0 INTRODUCTION

On behalf of the Nevada Environmental Response Trust, Northgate Environmental Management, Inc. (Northgate) has prepared this Data Validation Summary Report to assess the validity (based on data validation) and usability (based on project objectives) of the Baseline Groundwater Sampling data. The Baseline Groundwater data were collected by Northgate on September 7 through 15, 2010.

Baseline Groundwater samples were collected and analyzed in accordance with the Northgate *Capture Zone Evaluation Work Plan, Tronox LLC, Henderson, Nevada, May 2010.* Groundwater collected from the 30 locations resulted in the analysis of 112 environmental and 81 field quality control (QC) samples (trip blanks, field blank, filter blanks, equipment blank, field duplicate, and matrix spike [MS]/MS duplicate [MSD] analysis). The sampling and analysis summary of the 30 groundwater samples and associated field QC samples is presented in Table 1-1.

Field samples and the associated field QC samples were logged into the laboratories in Sample Delivery Groups (SDGs). The Baseline Groundwater data are contained in six SDGs. A complete listing of the Baseline Groundwater samples and SDGs is presented in Table 1-2. Analytical services were provided by Test America, Denver, Colorado.

The analytical data were validated by Laboratory Data Consultants, Inc. (LDC) in accordance with procedures described in the Nevada Division of Environmental Protection (NDEP) *Data Verification and Validation Requirements – Supplement, Henderson, Nevada, April 13, 2009*, established for the BMI Plant Sites and Common Areas Projects. The association between the laboratory SDGs and LDC validation reports is presented in Table 1-3.



2.0 DATA VALIDATION PROCESS

A formal validation of the Baseline Groundwater analytical results was performed in accordance with the *Tronox Quality Assurance Project Plan* (QAPP; AECOM/Northgate 2009), and *NDEP Supplemental Guidance on Data Validation for the BMI Plant Sites and Common Areas Projects* (NDEP Supplemental Guidance; NDEP 2009d), all of the Baseline Groundwater data were validated. The Baseline Groundwater data are contained in six SDGs. Approximately 90% of the analytical data were validated as Stage 2B and approximately 10% were validated by Stage 4 data validation procedures. EPA Stage 2B (EPA 2009) validation evaluates the following QC criteria:

- Completeness of deliverable;
- Technical holding times and sample preservation;
- Sample integrity and cooler/sample temperature at the time of laboratory receipt;
- Laboratory and field blank contamination;
- Surrogate spike recoveries;
- MS/MSD recoveries and relative percent differences (RPDs);
- Laboratory duplicate RPDs;
- Laboratory control sample (LCS) recoveries; and
- Initial and continuing calibrations.

The comprehensive validation, consistent with EPA designation of Stage 4 (EPA 2009), involves in-depth review of compound identification and quantification, spot-checks of calculations, and verification of summary data against the raw data. Table 1-3 is a cross-reference of laboratory SDG and associated validation reports. Field samples presented with shading were validated as Stage 4 (EPA 2009).

2.1 Data Deliverables

Analytical data deliverables were provided as an electronic data deliverable (EDD) version of the full data package, equivalent to a Contract Laboratory Program (CLP) deliverable (i.e., consisting of all the information required in a CLP package, including CLP-like summary forms). The electronic data packages were presented in PDF format with embedded text wherever possible and include complete bookmarking for all forms, tables, and sections. Each data package was also delivered as an EDD. The analytical reports for the Baseline Groundwater



data are presented in Appendix B of the Semi-Annual Remedial Performance Report for Chromium and Perchlorate, July 2010 through December 2010.

In addition to the laboratory deliverables, field information was provided to the validation staff in order to associate the field QC samples (trip blanks, field blanks, filter blanks, equipment blanks, and field duplicates) with the primary field samples prior to validation.

2.2 Validation of Analytical Deliverables

Validation of the Baseline Groundwater data was performed by LDC using the appropriate EPA guidelines (EPA 1999, 2004, 2008, 2009) or equivalent regional EPA validation guidelines such as Region 9 Superfund Data Evaluation/Validation Guidance, R9QA/006.1 (EPA 2001), the NDEP Supplemental Guidance (NDEP 2009b, 2009c, 2009d, 2009e) and the Basic Remediation Company (BRC) SOP 40, Data Review/Validation (BRC SOP; BRC 2009). These federal EPA guidelines, prepared for CLP data, were adapted to reflect the analytical methods and measurement quality objectives established for the Baseline Groundwater methods and the guidance provided by NDEP. LDC validation reports for the Baseline Groundwater data are presented in Appendix B.

Analytical data deficiencies were qualified using the data validation qualifiers in Table 2-1 and project-specific reason codes shown in Table 2-2. The finalized NDEP EDD (NDEP 2009f) for the Baseline Groundwater data is presented in Appendix D of the *Semi-Annual Remedial Performance Report for Chromium and Perchlorate, July 2010 through December 2010*.



3.0 DATA VALIDATION RESULTS

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

- Holding times and sample preservation;
- Initial and continuing calibrations;
- Serial dilution;
- Laboratory blanks/equipment blanks/field blanks;
- LCS/Laboratory Control Sample Duplicate (LCSD) results;
- MS/MSD results;
- Surrogate recoveries;
- Internal standard performance;
- Laboratory duplicate results;
- Field duplicate results; and
- Quantitation problems.

Table 3-1 presents the qualified results based on QC deficiencies identified during the validation process. Reason codes for each qualifier assignment are provided in Summary of Qualified Data, Table 3-1. Where available, a numerical data quality indicator (DQI) result value and acceptance criteria for that value have been added to the tables in columns to the right of the reason codes per NDEP's request. No QC problems were identified that resulted in qualification of results based on mass spectrometer tuning, gas chromatograph/mass spectrometer (GC/MS) performance checks, compound identification, or peak integration. The data validation memorandum presented in Appendix B discusses the application of qualifiers in detail. Table 3-1 is provided to NDEP on CD as an Excel spreadsheet that can be re-sorted to assist the data user in locating validation information for any particular sample, SDG, method, or analyte.

3.1 Holding Times and Sample Preservation

Sample preservation and analytical holding times are evaluated to assure that the sample integrity is intact for accurate sample preparation and analysis. Sample preservation and analytical hold time are presented for each method of analysis in Table B-1 of the QAPP. Holding time exceedances can cause loss of sample constituents due to biodegradation,



precipitation, volatization, and chemical degradation. In accordance with EPA guidance (USEPA 2004, 2008), sample results for organic and non-metal analyses that were performed after the method holding time but less than two times the method holding time are qualified as estimated (J- or UJ) and results for analyses performed after two times the method holding time are qualified as rejected (R). Inorganic hold time exceedances are qualified as estimated J- or R. No data were qualified based on hold time or preservation exceedances.

3.2 Initial and Continuing Calibration

Instrument performance was evaluated during the review of initial and continuing calibration for each method analyzed. No data were qualified based on calibration exceedances.

3.3 Serial Dilution

Serial dilutions were performed on results greater than 50X the instrument detection limit (IDL) to confirm matrix interference. All percent differences (% D) were acceptable with the exception of Baseline Groundwater sample M-182_201009-001 15% D for arsenic. In accordance with the EPA guidance (EPA 2004, 2008), NDEP Supplemental Guidance (NDEP 2009c,d,e), and the BRC SOP results greater than the MDL were qualified as "J/Estimated" and non-detected values were qualified as "UJ/Estimated not detected. Sample M-182_201009-001 arsenic (29 ug/L) was qualified as "J/Estimated." No other qualification was necessary.

3.4 Laboratory Blanks/Equipment Blanks/Field Blanks

The Baseline Groundwater data were assessed using the following blanks: field blanks, filter blanks, equipment blanks, and laboratory method blanks. Data were evaluated and qualified in accordance with EPA guidance (EPA 2004, 2008), NDEP Supplemental Guidance (NDEP 2009c,d,e), and the BRC SOP. Dissolved Organic Carbon (DOC) results for sample M-181_201009-001 (0.77 mg/L) and M-181_201009-001_FD (0.65 mg/L) were modified to 1.0 mg/L and qualified as "U/Not detected" due to contamination detected in the associated equipment blank (0.83 mg/L) and field blank (0.71 mg/L).

3.5 LCS/LCSD Results

Laboratory control samples and laboratory control sample duplicates were used to assess laboratory accuracy. The Baseline Groundwater samples were evaluated in accordance with the BRC SOP. No data were qualified based on LCS/LCSD precision and accuracy.



3.6 MS/MSD Results

Matrix spike and matrix spike duplicate samples consist of aliquots of environmental samples spiked with a subset of target compounds. MS/MSD samples monitor potential interference from the site-specific sample matrix and its effect on target compounds. Additional field sample aliquots were collected for methods analyzed with the exception of Total Dissolved Solids (TDS). Samples were evaluated using the EPA guidance (EPA 2004, 2008), *NDEP Supplemental Guidance* (NDEP 2009c,d,e), the *BRC SOP*, and professional judgment.

Perchlorate/Method 314.0 field sample M-182_201009-001 was submitted for MS/MSD analysis. The perchlorate (130%) MSD recovery was outside of the laboratory establish (75-125) control limits. The associated sample M-182_201009-001 perchlorate result (6500 ug/L) was qualified as "J/Estimated." No other data were qualified based on MS/MSD precision and accuracy.

3.7 Surrogate Recoveries

Surrogate recoveries were reviewed for organic methods and evaluated using the EPA guidance (EPA 2004, 2008), NDEP Supplemental Guidance (NDEP 2009c,d,e), and the *BRC SOP*. All surrogate recoveries were within the laboratory established control limits. No data were qualified based on surrogate accuracy.

3.8 Internal Standard Performance

Internal standards were prepared for certain organic and inductively coupled plasma (ICP)/MS analyses by adding compounds similar to target compounds of interest to sample aliquots. Internal standards are used in the quantitation of target compounds in the sample or sample extract. Internal standards were reviewed using the EPA guidance (EPA 2008), NDEP Supplemental Guidance (NDEP 2009c,d,e), and the *BRC SOP*. No qualification was necessary based on internal standard performance.

3.9 Laboratory Duplicate Results

Laboratory duplicate analysis involves the preparation and analysis of an additional aliquot of a field sample. Results from duplicate sample analyses measure laboratory precision, and homogeneity of contaminants in the field matrix. The relative percent difference (RPD) of the duplicate results were evaluated in accordance with EPA guidance (EPA 2004, 2005), NDEP



Supplemental Guidance (NDEP 2009c,d,e), and the *BRC SOP*. No data were qualified based on laboratory duplicate results.

3.10 Field Duplicate Results

Field duplicates are used to evaluate sampling technique precision and homogeneity of the sample matrix. Field duplicates were collected at a frequency of 10% during the Baseline Groundwater sampling. In accordance with the QAPP, NDEP Supplemental Guidance (NDEP 2009c,d,e), and the BRC SOP, the precision goal for aqueous field duplicate analyses is \pm 30% RPD. If the field duplicate RPD exceeds the 30 % limit, non-detected sample results shall be qualified as estimated (UJ) at the sample quantitation limit (SQL) and detected results shall be qualified as estimated (J). The RPD will be calculated using the reporting limit for non-detected sample results. Similar to analytical duplicates, this limit does not apply when the result for either the sample or its duplicate is less than five times the practical quantitation limit (PQL). For this situation, the absolute value of the PQL is to be used as the control limit. No qualification was necessary based on field duplicate precision.

3.11 Quantitation Problems

During Stage 4 evaluation, all raw data was reviewed to confirm target analyte identification and quantitation. No qualification was necessary based on quantitation problems.



4.0 EVALUATION OF QUALITY INDICATORS

Data quality indicators of precision, accuracy, representativeness, comparability, completeness, and sensitivity (PARCCS) were used to verify that sampling and analytical systems used in support of project activities are effective and that the quality of the data generated for the project is appropriate for making decisions affecting future activities. This section discusses the DQIs for the Baseline Groundwater sampling dataset. DQIs address the field and analytical data quality aspects as they affect uncertainties in the data collected for site characterization and risk assessment. The PARCCS parameters definition and assessment are presented in the *Tronox Revised Phase B QAPP* (Revised QAPP; AECOM/Northgate 2009). All data not meeting the established PARCCS criteria were qualified during the validation process using the guidelines presented in the QAPP, *National Functional Guidelines* (EPA 2004, 2005, 2008), BRC SOP, each analytical method employed, and professional judgment.

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. The assessment of field duplicate precision is discussed in Section 3.10. Field duplicate precision (30% RPD) was met for the three field duplicate associated with the Baseline Groundwater sampling data.

Laboratory precision evaluates DQIs such as calibration, surrogates, MS/MSD, laboratory duplicate (DUP), LCS/LCSD and interference check samples previously discussed in Section 3 of this report. All laboratory precision was acceptable.

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 following QC parameters:

- Holding times and sample temperatures;
- Calibration;
- LCS percent recovery;
- MS/MSD percent recovery (organics);
- Serial dilution recovery (inorganics);



- Surrogate spike recovery; and
- Blank sample results.

Accuracy was evaluated for each of the DQIs in Sections 3.1 through 3.7. Evaluation of the Stage 4 QC elements that contribute to accuracy – such as mass spectrometer tuning, compound or element identification, peak integration and mass spectral matches, and calculation/transcription verifications did not result in the qualification or rejection of any data during validation.

4.3 Representativeness

Representativeness is a qualitative parameter defined by the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, or a process or environmental condition. There is no formula for evaluating representativeness. Aspects of representativeness addressed during validation include the review of sample collection information in the chain-of-custody documentation, conformity of laboratory analyses to Work Plan intentions, adherence of the documented laboratory procedures to method requirements, and completeness of the laboratory data packages. All representativeness deficiencies were resolved during the actual field sampling event and/or data validation process. No qualification was necessary based on representativeness.

4.4 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system, compared to the amount expected under normal conditions. "Normal conditions" are defined as the conditions expected if the program specific work plan was implemented as proposed.

Field completeness is defined as the percentage of samples actually collected versus those intended to be collected per the Work Plan. The field completeness goal established in the QAPP is 90%. A comparison of the Capture Zone Evaluation Work Plan with the database sample IDs indicates that actual field completeness was 100%, exceeding the goal established for the project. Field completeness was assessed using the total sample locations scheduled in the Work Plan compared to actual number submitted for analysis.

Laboratory completeness is defined as percentage of valid data points versus the total expected from the laboratory analyses. Valid data are defined as all the data points judged to be usable (i.e., not rejected as a result of the validation process). The laboratory completeness goal established in the QAPP is 95%. Actual laboratory completeness was 100% on the basis of



sample analysis (i.e., all requested analyses were performed and reported by the laboratories), and no data were qualified as rejected (R).

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 Baseline Groundwater was maximized by using standard methods for sampling and analysis, reporting data, and data validation.

4.6 Sensitivity

Sensitivity is the capability of a method to discriminate an actual deflection or response above instrument noise. For the EPA methods employed in this project, sensitivity is measured by the Method Detection Limit (MDL) and PQL. Both nominal MDLs and PQLs were provided by the laboratories in the laboratory data packages and were verified during validation. MDLs in general were adjusted for each Baseline Groundwater sample to include the necessary dilution factors, preparation factors, and sample volume of an individual sample as the SQL. The sensitivity requirements were based on the laboratory's ability to detect and report consistent and reliable limits.



5.0 CONCLUSIONS

One hundred percent of the laboratory data for the Baseline Groundwater sampling were validated using standardized guidelines and procedures recommended by EPA and NDEP. Based on the validated data, 100% of the results for Baseline Groundwater data were determined usable and considered valid for all decision-making purposes.

Laboratory results qualified during validation are summarized in Table 3-1. Data qualifiers and qualifier reason codes are presented as Table 2-1 and 2-2, respectively.

All qualified results were evaluated with respect to the data quality indicators and compared to the QAPP and Capture Zone Evaluation Work Plan. Details of this evaluation are discussed in Section 4 of this report. Based on the results of data validation, actual laboratory completeness was 100% on the basis of sample analysis, and 100% completeness based on valid data. The overall goals for data quality were achieved for the Baseline Groundwater dataset.



6.0 REFERENCES

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TABLES

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APPENDIX A ELECTRONIC DATA DELIVERABLE

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APPENDIX B VALIDATION REPORTS

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