

Prepared for:
Tronox LLC
Henderson, Nevada

Revised
Data Validation Summary Report
Phase B Source Area Investigation Soil Gas
Survey – Tronox LLC Facility
Henderson, Nevada

ENSR Corporation
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Revised Data Validation Summary Report Phase B Source Area Investigation Soil Gas Survey – Tronox LLC Facility Henderson, Nevada

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Acronyms and Abbreviations

%D	Percent difference or percent drift
%R	Percent recovery
%RSD	Percent relative standard deviation
ASB	Analytical Services Branch
BHC	Hexchlorocyclohexane
CCV	Continuing calibration verification
CDD	Chlorinated Dibenzo-p-Dioxins
CDF	Chlorinated Dibenzofurans
CLP	Contract Laboratory Program
COC	Chain of custody
DCB	Decachlorobiphenyl
DQI	Data quality indicator
DRO	Diesel range organics
EDD	Electronic data deliverables
EMPC	Estimated Maximum Possible Concentrations
EPA	U.S. Environmental Protection Agency
EPN	O-Ethyl-O-p-nitrophenyl benzene thiophosphate
GC/MS	Gas Chromatography/Mass Spectrometry
GRO	Gasoline range organics
HT	Holding time
ICAL	Initial calibration
ICP	Inductively Coupled Plasma
ICP/MS	Inductively Coupled Plasma/Mass Spectrometry
ICS	Interference check sample
ID	Identification
IS	Internal standard
LCL	Lower control limit
LCS	Laboratory control sample
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
MARLAP	Multi-Agency Radiological Laboratory Analytical Protocols Manual
MBAS	Methylene-Blue Active Substances MCL Maximum contaminant levels
MDL	Method detection limit

Acronyms and Abbreviations (Cont'd)

Mn	Manganese
MS/MSD	Matrix spike/matrix spike duplicate
ND	Not detected
NDEP	Nevada Department of Environmental Protection
NFG	National Functional Guidelines
NS	Not spiked
ORO	Oil range organics
PAH	Polycyclic aromatic hydrocarbons
prep	Preparation
PRG	Preliminary Remediation Goals
QAPP	Quality Assurance Project Plan
QC	Quality control
r ²	Correlation coefficient
RCRA	Resource Conservation and Recovery Act
RL	Reporting limit
RPD	Relative percent difference
RRF	Relative response factor
SDG	Sample Delivery Group
SRC	Site-Related Chemical
STL	Severn Trent Laboratories
SVOC	Semivolatile organic carbon
TCMX	Tetrachlorometaxylene
TDS	Total dissolved solids
TOC	Total organic carbon
TSS	Total suspended solids
UCL	Upper control limit
VOC	Volatile organic carbon

1.0 Introduction

The purpose of formally validating the Phase B Source Area Investigation Soil Gas Survey laboratory results was to determine the suitability of the data for potential use in the conceptual site model, risk assessment, and other future on-site environmental assessments.

Columbia Analytical Services (hereafter abbreviated as CAS) in Simi Valley, CA was the laboratory contracted by Tronox for the Phase B Source Area Investigation Soil Gas Survey chemical analyses. All VOC analyses utilized EPA Method TO-15. All He tracer gas analyses utilized modified EPA Method 3C

The validation covered seven SDGs containing a total of 115 soil gas samples. The distribution of samples within SDGs is detailed in Table E-3. The TO-15 analyte list for all samples in this project included 71 VOC compounds as specified in Table 2 of the Phase B Source Area Investigation Soil Gas Survey Workplan (ENSR, March 2008). All field sample analytical results are provided in a separate table titled "Volatile Organic Compounds and Helium Concentrations in Soil Gas".

Attachment C contains the comments received from the Nevada Division of Environmental Protection (NDEP) dated September 17 and 30, 2008 to the initial DVSR (August 2008) and the Tronox response of September 30, 2008, respectively. This submittal incorporates revisions requested by NDEP to the initial DVSR. The revisions to the initial submittal are highlighted and provided in Attachment C

2.0 Data Validation Process

The laboratory results for the Phase B Source Area Investigation Soil Gas Survey were subjected to formal data validation following the guidance on data validation provided by the Nevada Division of Environmental Protection (NDEP) for the BMI Plant Sites (NDEP 2006). The data from the laboratory were submitted as Contract Laboratory Program (CLP)-like data packages in PDF format and EQUIS® format electronic data deliverables (EDDs). The EDDs were imported into an EQUIS® database specifically created for this project. ENSR validated the data using the pdf data packages plus EDDs and subsequently entered the validation qualifiers into the database. Results were compared to the goals stated in the Phase B Source Area Investigation Soil Gas Survey Workplan (ENSR, March 2008), hereafter referred to as the "Workplan," and the Draft Quality Assurance Project Plan (ENSR, April 2008) hereafter referred to as the "QAPP."

A comprehensive ("full") data validation was performed on one of the seven laboratory Sample Delivery Groups (SDGs), and the remainder underwent a more limited validation as described below. The goal of a minimum of 10% full validation that was established for the project was exceeded in order to comprehensively evaluate a full representative SDG. Limited validation consisted of reviewing the following data elements contained in laboratory summary data forms (and did not generally include raw data review):

- Agreement of analyses conducted with chain-of-custody (COC) requests
- Holding times and sample preservation
- Initial and continuing calibrations
- Laboratory method blanks/canister blanks
- Surrogate recoveries
- Internal standard performance
- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results

- Laboratory duplicate results
- Field duplicate results
- Quantitation limits and sample results
- Helium tracer gas concentrations

Full validation consisted of reviewing to the level of raw data all of the elements covered in the limited validation plus the following elements where applicable as defined by the analytical method:

- Mass spectrometer tuning
- Gas Chromatography/Mass Spectrometry (GC/MS) performance checks
- Compound identification
- Peak integration and mass spectral matches
- Calculation and transcription verifications

Analytical data were evaluated with reference to the National Functional Guidelines (NFG; EPA 1999) as well as the Region 9 Superfund Data Evaluation/Validation Guidance (EPA 2001), the above-mentioned NDEP Guidance on Data Validation (NDEP 2006), the EPA reference method, the quality control (QC) criteria specified in the QAPP (ENSR, April 2008), and the Workplan (ENSR, March 2008). The Regional and National Functional Guidelines were modified to accommodate the non-CLP methodologies.

Helium tracer gas data was utilized to determine whether significant leaks of surface air contaminated or diluted the soil gas during collection. This data was used for screening purposes and was not provided by the laboratory in a format that permitted validation of the He data itself, however the He results were used to qualify the TO-15 data if the % average leak of helium into the soil gas exceeded 1% of the concentration introduced inside the shroud at the surface. This rule was based on a conservative interpretation of the Interstate Technology Regulatory Council (ITRC) document "Vapor Intrusion Pathway: A Practical Guideline" (ITRC, Jan 2007), and the New York State Department of Health document "[Guidance for Evaluating Soil Vapor Intrusion in the State of New York](#)" (NYSDOH, Oct 2006)

In general, the validation qualifiers and definitions employed were based on those used by the U.S. Environmental Protection Agency (EPA) in the documents mentioned above. Validation qualifiers and definitions are listed in **Table E-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 E-2**. These codes were entered in the project database for each application of a validation qualifier that changed a laboratory qualifier or modified a result value to indicate the primary reason(s) for data qualification. Where multiple reason codes were assigned to a single result then professional judgment was used to determine the most appropriate overall qualifier and bias sign, if any. Conversions of the laboratory reported "ND" for not detected to the "U" flag in the database and the laboratory-applied "J" qualifier to indicate results less than the reporting limit but greater than the method detection limit (MDL) are generally not discussed in this report. These laboratory qualifiers were standardized and migrated to the validation qualifier field so that the "J" qualifier, unless applied by a validator for other reasons discussed below and documented with a reason code, always indicates a result is estimated because it is less than the reporting limit but greater than the MDL.

Data validation was organized by laboratory report SDG. For each separate SDG a data validation memorandum was written by a validator and reviewed by a peer at ENSR's Westford, MA office. These memoranda are included as Word and Excel documents and sorted by ENSR Identification (ID), which is correlated with the laboratory SDGs, field sample IDs, and collection dates as listed in **Table E-3**. **Table E-3** is

provided as an Excel spreadsheet that can be resorted to assist the data user in locating validation information for any particular sample or SDG. Note validation information about the helium tracer results is provided in this revised DVSR and associated tables in response to NDEP comments but is not included in the original data validation memoranda.

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
- Mass spectrometer tuning
- Laboratory blanks/equipment blanks/field blanks
- Surrogate recoveries
- LCS/LCSD results
- Internal standard performance
- Laboratory duplicate results
- Field duplicate results
- Quantitation limits and sample results
- GC/MS performance checks
- Peak integration and mass spectral matches
- Calculation and transcription verifications
- Helium tracer gas concentrations

Tables E-4 through E-7 and E-9 list all the results qualified based on QC problems identified with regard to blank contamination, calibrations, field duplicates, quantitation problems, and helium tracer results, respectively. Reason codes for each qualifier assignment have been provided in each table. Where available, a numerical data quality indicator (DQI) result value, and acceptance criteria for that DQI 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 holding times, mass spectrometer tuning, surrogate recoveries, LCS recoveries, internal standard performance, laboratory duplicate results, GC/MS performance checks, compound identification, or peak integration. Results for a single sample were rejected based on data validation due to the helium tracer concentrations. This location (SG42B) was successfully resampled and provided data without detectable He. **Table E-8** was provided as part of the response to NDEP comments and details the calculated helium percent leak for soil gas samples in which the helium tracer was detected. The data validation summary results table contents are sorted by sample ID and SDG to assist the data user in locating the associated data validation memoranda. The data validation memoranda discuss the application of qualifiers in more detail. **Table E-4 through Table E-7, plus Table E-9** are provided on CD as Excel spreadsheets that can be resorted to assist the data user in locating validation information for any particular sample, SDG, method, or analyte. The results in each table will be summarized separately in sections below.

3.1 Instrument Calibration

Table E-5 lists the sample results that were qualified based on exceeded calibration criteria. The nature of the numerical DQI result value is defined by the DQI limit criteria; for instance, percent relative standard deviation (%RSD) criteria are from method initial calibration (ICAL) requirements. The one positive and nine nondetect results for 1,2-dichlorobenzene were qualified as estimated (J and UJ, respectively) due to the associated initial calibration's %RSD for this compound, which slightly exceeded the method defined criteria.

3.2 Blank Contamination

In general, laboratory and field blanks were free of contamination at significant levels. **Table E-4** lists the sample results that were qualified based on contamination in laboratory method blanks. Target compounds were not detected in the canister blanks. The blank result value associated with each qualified sample result is given in the column to the right of the reason codes.

A total of 115 sample results were negated (U) based on the presence of low levels of the common laboratory contaminants methylene chloride, acetone, and 2-butanone, as well as trace levels of benzene, carbon disulfide, ethanol, isopropylbenzene, naphthalene, and vinyl acetate in the method blanks. The majority of these negations were based on the presence of acetone. Table E-4 provides the dilution factors and sample quantitation limits (SQL) to assist the reader in understanding the blank actions. Action limits (AL) were established at 10x the method blank concentration for common lab contaminants and 5x for all other target analytes. If the sample result was < the SQL and < the AL, the result was reported as not detected (U) at the SQL. If the sample result was > SQL but < AL, the result was reported as not detected (U) at the reported concentration. If the sample result was > AL, the result was not qualified.

3.3 Field Duplicates

The results of all soil gas field duplicate pairs collected were evaluated during validation. RPDs were compared to the objectives established in the QAPP of 50% RPD for soil gas. **Table E-6** lists the results qualified during validation based on field duplicate precision nonconformances.

A total of 84 associated field sample result values in nine sample/field duplicate pairs were qualified as estimated (J) based on field duplicate result RPDs that exceeded the QAPP criteria. Twenty two different analytes and from two to ten records per analyte were qualified.

3.4 Quantitation

Table E-7 lists the results that were qualified during validation based on quantitation issues. All 25 of the qualified results were based on the laboratory qualifier M indicating a possible high bias due to matrix interferences in the GC/MS data. No other quantitation problems were discovered during data validation.

3.5 Helium tracer results

Table E-9 lists the results that were qualified based on the helium tracer concentrations detected in the soil gas samples. The DQI result is the He concentration in ppmV in the sample. The DQI limit is a calculated 1% of the He concentration in the surface shroud. Table E-8 and the associated Tronox response to comments provide additional details. If the He concentration was between 1% and 10% of the shroud average then the TO-15 VOC analyte results were qualified as estimated based on the possible contamination and dilution by surface air. If the He concentration exceeded 10% of the shroud average then the results were rejected. Four sample datasets were qualified as estimated and one was rejected based on these criteria. All but one of these soil gas samples were recollected later and He was not detected in these resample datasets (indicated by an R in the sample ID), therefore only TO-15 data from a single location (SG17B) was potentially impacted by

surface air contamination/dilution. A comparison of the original and resampled results (e.g. SG53B-05, its duplicate SG53B-05D, the resampled SG53BR-05, and its duplicate SG53BR-05D) indicates the TO-15 analyte results are very consistent regardless of the He tracer results. This confirms the assumptions used for data qualification based on He tracer results were conservative and the data quality is not significantly impacted when He results are less than 10% of the surface shroud levels.

4.0 Evaluation of Data Quality Indicators

Data validation information was used to evaluate the DQIs of precision, accuracy, representativeness, comparability, completeness, and sensitivity for results in the Henderson Phase B Source Area Soil Gas Investigation dataset. Each of these DQI parameters is discussed in the 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. The field duplicate RPD results that caused the application of validation qualifiers are discussed in Section 3.3 of this report and listed in **Table E-6**. In general the field duplicate precision was acceptable for all analytes. A limited analyte data set was qualified as estimated but usable and represents only 1% of the total field sample results dataset.

Laboratory precision was assessed through the RPD results for matrix duplicates. The laboratory duplicate precision was acceptable and no results were qualified during validation.

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., LCS and surrogate spikes). All positive control sample recoveries were acceptable and no results were qualified based on LCS or surrogate recoveries.

Accuracy is also indirectly addressed via the negative control samples for field activities, as well as laboratory negative control samples such as method blanks and calibration blanks. Based on blank results validation, 115 results were qualified as described in Section 3.2, which represents only 1.4% of the total data points collected. No data were rejected based on blank results.

Bias as a component of accuracy is also evaluated with the validation of HT, calibration, internal standard performance, and quantitation results. Collectively these evaluations resulted in the qualification of only 0.4% of the total data points. No data were rejected based on these aspects of bias.

Evaluation of the remaining 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 points during validation.

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 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 resubmittals of data from the laboratories to correct problems that were discovered during the validation process. All of these issues were resolved. Other aspects of data representativeness, such as adherence to recommended HTs, instrument calibration requirements, as well as field and laboratory precision assessments, are discussed above in this report. The possible entrainment of contaminants and dilution by surface air also could impact the representativeness of the soil gas and this is discussed above in this revised DVSR report as well as the Tronox response to NDEP comments on the original DVSR for the soil gas dataset. Very low levels of the helium tracer and consistency between the original and recollected sample results for VOC analytes both indicate the sampling was representative of the subterranean soil gas.

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 are defined as all the data points judged to be usable (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 per the Workplan. The goal stated in the QAPP for this project was greater than 90% field completeness. A comparison of the Workplan sample tables with the database sample IDs indicates that actual field completeness was 100%, exceeding the goal established for the project. This field completeness calculation is based on the total sample locations scheduled in the Workplan compared to the COC requests sent to the laboratories. All COC requests were faithfully executed by the laboratories with the minor exceptions detailed in the data validation memoranda.

Laboratory completeness is defined as percentage of valid data points versus the total expected from the laboratory analyses. The objective stated in the QAPP for this project was greater than 95% laboratory completeness. Actual laboratory completeness was 100% on the basis of sample analysis (i.e., all requested analyses were performed and reported by the laboratories), and 99% completeness based on valid data.

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. Because this project was an initial site investigation for all of the TO-15 parameters, involving new soil gas sampling locations, there was no well characterized historical data set for comparisons. Comparability of data within the investigation was maximized by using standard methods for sampling and analysis, reporting data, and data validation. A single laboratory performed all the analyses to eliminate interlaboratory variability.

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 MDL and reporting limit (RL). Both nominal MDLs and RLs were provided by the laboratories in the laboratory data packages and were verified during validation. Reporting limits in general were adjusted for sample quantitation limits based on the low point of calibration and corrected for sample-specific factors such as exact aliquot size, canister pressure, dilutions, etc. The laboratories were instructed to report estimated (J flagged) results if concentrations above the MDL but below the RL were detected.

To determine if the adjusted reporting limits for all project analytes were low enough to meet the project sensitivity requirements, a comparison of the project regulatory comparison levels, based on 1/10 of the EPA Region 9 Preliminary Remediation Goals (PRGs) for ambient air, adjusted for vapor intrusion dilution, was made with the nominal laboratory RLs. Risk assessment will be based on the EPA Region VI MSSL values per

NDEP request. In general the methods selected were sufficiently sensitive to meet the risk-based comparison level goals in soil gas samples and support potential vapor intrusion evaluation. The RLs and MDLs provided by the laboratories for this analyte set are typical of the TO-15 method employed and significantly lower detection limits are not routinely achievable using certified methods.

5.0 Conclusions

One hundred percent of the laboratory data for the Phase B Source Area Soil Gas Investigation were validated using standardized guidelines and procedures recommended by EPA and NDEP. Ninety four percent of the 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 was qualified during validation, and those results are summarized in **Tables E-4 to E-7**. The qualified data are grouped in these tables based on the reason for qualification (see **Table E-2**) and the qualifier symbols or flags applied (see **Table E-1**). Six percent of the results of the total analytical dataset for this project were qualified as estimated due to minor QC problems with precision, accuracy, and representativeness. Based on guidance in the EPA data usability document (EPA 1992), estimated data are considered usable with the appropriate interpretation (e.g., consideration of the potential bias). All results for a single sample were rejected during data validation based on helium tracer concentrations exceeding 10% of the surface shroud value. This sample was successfully recollected and reanalyzed.

All the qualified results were evaluated with respect to the data quality indicators and compared to the QAPP and Workplan goals. Details of this evaluation are discussed in Section 4 of this report. Based on the results of data validation, the overall goals for data quality were achieved for this project.

6.0 References

- EPA. 1992. Guidance for Data Usability in Risk Assessment. Part A.
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- NYSDOH, October 2006. New York State Department of Health "Final: Guidance for Evaluating Soil Vapor Intrusion in the State of New York"

Tables

Attachments

(See CD for Attachments A, B and C)