

US EPA ARCHIVE DOCUMENT



Tier III Data Validation Report Summary

Client: Chevron Environmental Management Company	Laboratory: Air Toxics Limited (LTD)
Project Name: Risk Assessment/Hooven Vapor Investigation	Sample Matrix: Vapor
Project Number: 500-016-012	Sample Start Date: September 29, 2009
Date Validated: November 5, 2009	Sample End Date: October 2, 2009
Parameters: Volatile Organic Compounds (VOCs) by Modified Method TO-15	
Laboratory Project IDs: 0910192	
Data Validator: Justin Hildenbrand, Environmental Chemist	

DATA EVALUATION CRITERIA SUMMARY

A Tier III Data Validation was performed by Trihydro Corporation's Chemical Data Evaluation Services group on the analytical data report package generated by Air Toxics LTD evaluating samples from the Chevron Site located in Cincinnati, Ohio.

Precision, accuracy, method compliance, and completeness of this data package were assessed during this data review. Precision was determined by evaluating the calculated relative percent difference (RPD) values of samples from field duplicate pairs. Laboratory accuracy was established by reviewing the demonstrated percent recovery of laboratory control samples (LCS) to verify that none of the data were biased. Additionally, field accuracy was established by collecting trip blanks to monitor for possible ambient or cross contamination during sampling. Method compliance was established by reviewing holding times, detection limits, surrogate recoveries, method blanks, and LCS percent recoveries against method specific requirements. Completeness was evaluated by determining the overall ratio of the number of samples planned versus the number of samples with valid analyses. Determination of completeness included a review of the chain-of-custody, laboratory analytical methods, and all other necessary documents associated with this analytical data set.

Data were evaluated in general accordance with validation criteria set forth in the United States Environmental Protection Agency (USEPA) Contract Laboratory Program (CLP) National Functional Guidelines for Superfund Organic Methods Data Review, document number USEPA-540-R-08-01, June 2008, with additional reference to USEPA CLP National Functional Guidelines for Organic Data Review, document number EPA 540/R-99-008 of October 1999 and the USEPA CLP National Functional Guidelines (NFG) for Inorganic Data Review, document number EPA 540R-04-004, October 2004. Review of duplicates is conducted in accordance with USEPA Region 1 Laboratory Data Validation Functional Guidelines for Evaluation of Organic Analysis, December 1996 or as specified by the method. In addition to the above mentioned guidance documents, the USEPA Hazardous Waste Support Branch Validating Air Samples Volatile Organic Analysis of Ambient Air in Canister by Method TO-15, SOP # HW-31, October 2006, document and the applicable methods were used for verification of the data.

SAMPLE NUMBERS TABLE

Client Sample ID	Sample Number
Float 1 - 092909	0910192-01A
Float 1 - 093009	0910192-02A
Float 1 - 100109	0910192-03A
Float 1 - 100209	0910192-04A
BD 1 - 100209	0910192-05A
TB-2, 100209	0910192-06A





Tier III Data Validation Report Summary

The samples were analyzed for client-specified analytes. The samples were shipped to Air Toxics LTD under chain-of-custody (COC) documents included for work order 0910192. The laboratory data were reviewed to evaluate compliance with the required methods and the quality of the reported data. A leading check mark (✓) indicates that the referenced data was deemed acceptable. A preceding crossed circle (⊗) signifies problems with the referenced data that may have warranted attaching qualifiers to the data.

- ✓ Data Completeness
- ✓ COC Documentation
- ✓ Holding Times and Preservation
- ✓ Laboratory Blanks
- ⊗ Initial and Continued Calibrations
- ✓ Instrument Calibrations
- ✓ System Monitoring Compounds (i.e. Surrogates)
- ⊗ Laboratory Control Samples (LCS)
- ⊗ Field Duplicates
- ⊗ Trip Blank

OVERALL DATA PACKAGE ASSESSMENT

Based on a data validation review, the data are acceptable as delivered. Air Toxics LTD qualified a total of one data point with a J data flag in data set 0910192. The laboratory assigned data qualifier was reviewed and found to be valid and correct. The purpose of validating data and assigning qualifiers is to assist in proper data interpretation. Data which are not qualified meet the site data quality objectives. If values are assigned qualifiers other than an "R", the data may be used for site evaluation, with the reasons for qualification being given consideration when interpreting sample concentrations. Data points which are assigned an "R" qualifier should not be used for any site evaluation purposes. The laboratory qualified one data point with a J flag, indicating estimated data. Laboratory J flags were preserved in the data and included in the Data Qualification Summary table at the end of this report. A total of 33 additional data points were qualified with J, JB, or UJ data flags as a result of this data validation review. Some of the qualified data points are useful only for qualitative purposes with the professional judgment of the project manager and associated technical staff. Data were qualified due to high field duplicate RPD values, TO-15 calibration data outside of acceptable limits, and low LCS recoveries in the TO-15 analyses.

Data qualifiers used during this validation included:

- J – Estimated concentration
- UJ – Estimated reporting limit
- JB – Estimated concentration due to blank contamination
- U – Evaluated to be undetected at the reporting limit

Data Completeness

All analyses were performed as requested on the chain-of-custody records. All samples were received by the laboratory and analyzed properly. Excluding the trip blank sample, the complete data package consisted of 330 data points, total. No data points were rejected. The data completeness measure for this data package is 100% and is acceptable.



TABLE 1. GENERAL VALIDATION CRITERIA CHECKLIST

1. Was the report free of non-conformances related to the analytical data identified by the laboratory?	No
<p>Comments: The laboratory listed the following non-conformances related to the analytical data.</p> <p>The reported CCV for each daily batch may be derived from more than one analytical file due to the client's request for non-standard compounds.</p> <p>Non-standard compounds may have different acceptance criteria than the standard TO-14A/TO-15 compound list as per contract or verbal agreement.</p> <p>The trip blank, sample TB-2, 100209, has reportable levels of target compounds present.</p> <p>The reported result for Methylene chloride in sample Float 1 - 100109 may be biased high due to co-elution with a non target compound with similar characteristic ions.</p> <p><u>The laboratory noted the following for the initial calibration.</u></p> <p>An initial calibration curve was analyzed on 09/29/09 on MSD-Z. The instrument was set up to do Full Scan and Selective Ion Monitoring (SIM) simultaneously.</p> <p>As noted on the accompanying analytical run log, point level 5 was reanalyzed due to anomalous unacceptable peak shape of 1,3-Butadiene.</p>	
2. Were data qualification flags used by the laboratory? If yes, define.	Yes
<p>Comments: The following data qualifier flag was used by the laboratory.</p> <p>J – Estimated value</p>	
3. Were sample COC forms complete?	Yes
<p>Comments: The COC records from field to laboratory were complete, and custody was maintained as evidenced by field and laboratory personnel signatures, dates, and times of receipt.</p>	
4. Were detection limits in accordance with the quality assurance project plan (QAPP), permit, or method, or indicated as acceptable by the Tier I validator?	Yes
<p>Comments: Detection limits were reviewed and determined to be acceptable. The laboratory reported required dilutions between 1.68 and 2.06 times.</p>	
5. Were the requested analytical methods in compliance with the QAPP, permit, or COC?	Yes
<p>Comments: The requested analytical methods were performed in accordance with the chain-of-custody forms.</p>	
6. Were samples received in good condition within method specified requirements?	Yes
<p>Comments: Samples were received intact and in good condition. The final vacuums from the field and receipt vacuums measured by the laboratory were compared and the vacuums appeared to be acceptable, with pressure/vacuum changes from the field to the laboratory less than five inches of mercury for each sample.</p> <p>The canisters used for sampling were 100% certified by the laboratory. The canister certification results were reviewed and found to be acceptable.</p>	
7. Were samples analyzed within method specified or technical holding times?	Yes
<p>Comments: The samples were analyzed within method specified holding times for analysis of Summa canisters and the respective method.</p>	
8. Were reported units appropriate for the associated sample matrix/matrices and method(s) of analyses?	Yes
<p>Comments: The results for Method TO-15 were reported in units of part per billion by volume (ppbv) and micrograms per cubic meter ($\mu\text{g}/\text{m}^3$). The results for Method ASTM D-1946 for fixed gases were reported as percentages (%). These units are appropriate for the air matrix and for the methods used.</p>	
9. Do the laboratory reports include all constituents requested to be reported as indicated by the Tier I validator?	Yes
<p>Comments: The requested constituents were reported as requested.</p>	



TABLE 1. GENERAL VALIDATION CRITERIA CHECKLIST

10. Were the field duplicates collected equal to at least 10% of the total number of samples, or as required by the project guidelines, QAPP, SAP, or permit, or as indicated by the Tier I validator? Yes

Comments: One field duplicate was collected for this sampling event, resulting in a collection frequency of 10% of the total number of samples. Sample BD 1-100209 was collected as a duplicate of sample Float 1-100209.

11. Were field duplicate RPD values within data validation QC limits (soil 0-50%, water 0-30%, or air 0-25%)? Yes

Comments: Precision based on field duplicate RPD results was determined to be acceptable with one exception, summarized at the end of this section. Field duplicate RPD values are reported in the Field Duplicate Summary table at the end of this data validation review. Analytes where both the parent and duplicate samples were non-detect are omitted from the Field Duplicate Summary table since precision could not be assessed for these data. **The RPDs for 2-butanone and ethanol exceeded the data validation QC limit of 25% for air samples. These analytes were qualified J for detections and UJ for non-detections for each sample reported for this data set based on professional judgment due to possible poor precision.**

If an analyte was detected in one sample but not in the other sample for the duplicate pair, a valid RPD could not be calculated and the RPD was reported as DL. **Data for carbon disulfide were qualified J for detections and UJ for non-detections for each sample reported for this data set based on professional judgment due to possible poor precision. Data for bromomethane were qualified for the parent and duplicate samples only based on professional judgment.**

For analytes where both the parent and duplicate results were detected at less than two times the reporting limit, a valid RPD could not be calculated and the result was reported as +/- RL.

12. Was the number of equipment, trip, or field blanks collected equal to at least 10% of the total number of samples, or as required by the project guidelines, QAPP, SAP, or permit, or as indicated by the Tier I validator? No

Comments: One trip blank, TB-2, 100209, was collected and submitted with the samples reported in these laboratory reports, resulting in a collection frequency of less than 10% of the total number of samples. Equipment and field blanks were not collected with the reported samples.

13. Were the trip blank, field blank, and/or equipment blank samples free of analyte contamination? No

Comments: Several detections of reported analytes were reported in the trip blank, as noted in the table below.

Analyte	Trip Blank Detection ($\mu\text{g}/\text{m}^3$)
Ethanol	1.2
Acetone	9.5
2-Butanone (Methyl Ethyl Ketone)	0.85
m,p-Xylene	1.2
o-Xylene	1.1
Propylbenzene	1.5
4-Ethyltoluene	15
1,3,5-Trimethylbenzene	6.4
1,2,4-Trimethylbenzene	23
Butylbenzene	3.2

Detections less than either the reporting limits or the blank detections were qualified U. Detections greater than or equal to both the reporting limits and the blank detections, but less than ten times the blank detections, were qualified JB. Detections greater than or equal to both the reporting limit and ten times the method blank detection were not qualified.

TABLE 2. VALIDATION CRITERIA CHECKLIST FOR VOC ANALYSES (TO-15 MODIFIED)

1. Were instrument calibrations within method or data validation quality control (QC) limits?	No
Comments: Initial and continuing calibrations results were within acceptable limits, with the following exception. In the initial calibration performed on September 28, 2009, through September 30, 2009, the %RSD for 2-butanone was above the data validation limit of 30% at 36.445%. Data reported for 2-butanone were qualified J for detections and UJ for non-detections.	
2. Were the instrument tunes within method control limits?	Yes
Comments: Instrument tunes were within method control limits.	
3. Were the internal standards within method control limits?	Yes
Comments: Internal standard areas and retention times were within method control limits.	
4. Was the total number of laboratory blank samples prepared equal to at least 5% of the total number of samples, or analyzed as required by the method?	Yes
Comments: Laboratory blank samples were prepared at a frequency equal to at least 5% of the total number of samples.	
5. Were laboratory blank samples free of target analyte contamination?	Yes
Comments: Detections were not reported in the laboratory blanks.	
6. Was the total number of LCSs analyzed equal to at least 5% of the total number of samples, or analyzed as required by the method?	Yes
Comments: The LCS samples were analyzed at a frequency equal to at least 5% of the total number of samples.	
7. Were LCS/LCSD percent recoveries and LCS/LCSD RPDs within laboratory limits?	No
Comments: The LCS recoveries were within acceptable limits, with the following exceptions. The LCS recovery for styrene was outside of the laboratory limits of 70-130% at 130.08%. Associated sample data were qualified J due to a possible high bias.	
8. Was the total number of MS samples prepared equal to at least 5% of the total number of samples, or analyzed as required by the method?	N/A
Comments: Matrix spike samples were not prepared and are not required for analysis by Method TO-15 Modified.	
9. Were MS/MSD percent recoveries and MS/MSD RPDs within data validations or laboratory QC limits?	N/A
Comments: Matrix spike samples were not prepared and are not required for analysis by Method TO-15 Modified.	
10. Were surrogate recoveries within laboratory QC limits?	Yes
Comments: Surrogate recoveries were within laboratory QC limits.	
11. Were laboratory duplicate RPD values acceptable?	Yes
Comments: Laboratory duplicates were not prepared for this sample set.	

TABLE 3. DATA QUALIFICATION, CHEVRON SITE, CINCINNATI, OHIO (0910192)

Analyte	Client Sample ID	Laboratory Assigned ID	Laboratory Result	Reviewer Qualifier	Reason for Qualification
2-Butanone	Float 1 - 092909	0910192-01A	11 µg/m3	J	% RSD above QC limit
2-Butanone	Float 1 - 093009	0910192-02A	44 µg/m3	J	% RSD above QC limit
2-Butanone	Float 1 - 100209	0910192-04A	11 µg/m3	J	% RSD above QC limit
2-Butanone	TB-2, 100209	0910192-06A	0.85 µg/m3	J	% RSD above QC limit
Carbon Disulfide	Float 1 - 092909	0910192-01A	ND(2.6 µg/m3)	UJ	High field duplicate RPD value
Carbon Disulfide	Float 1 - 093009	0910192-02A	79 µg/m3	J	High field duplicate RPD value
Carbon Disulfide	Float 1 - 100109	0910192-03A	ND(3 µg/m3)	UJ	High field duplicate RPD value
Carbon Disulfide	Float 1 - 100209	0910192-04A	12 µg/m3	J	High field duplicate RPD value
Carbon Disulfide	BD 1 - 100209	0910192-05A	ND(3 µg/m3)	UJ	High field duplicate RPD value
Carbon Disulfide	TB-2, 100209	0910192-06A	ND(1.6 µg/m3)	UJ	High field duplicate RPD value
Bromomethane	Float 1 - 100209	0910192-04A	1.9 µg/m3	J	High field duplicate RPD value
Bromomethane	BD 1 - 100209	0910192-05A	ND(0.76 µg/m3)	J	High field duplicate RPD value
Ethanol	Float 1 - 093009	0910192-02A	18 µg/m3	J	High field duplicate RPD value
Ethanol	Float 1 - 100209	0910192-04A	42 µg/m3	J	High field duplicate RPD value
Ethanol	TB-2, 100209	0910192-06A	1.2 µg/m3	J	High field duplicate RPD value
Styrene	Float 1 - 093009	0910192-02A	0.81 µg/m3	J	The LCS and/or LCSD recovery(ies) were above the acceptable limits indicating a possible high bias.
1,2,4-Trimethylbenzene	Float 1 - 093009	0910192-02A	3.6 µg/m3	U	Trip blank detection
1,3,5-Trimethylbenzene	Float 1 - 093009	0910192-02A	0.95 µg/m3	U	Trip blank detection
2-Butanone	Float 1 - 100109	0910192-03A	2.6 µg/m3	JB	Trip blank detection
2-Butanone	BD 1 - 100209	0910192-05A	3 µg/m3	JB	Trip blank detection
4-Ethyltoluene	Float 1 - 093009	0910192-02A	2.6 µg/m3	U	Trip blank detection
Acetone	Float 1 - 092909	0910192-01A	58 µg/m3	JB	Trip blank detection
Acetone	Float 1 - 093009	0910192-02A	64 µg/m3	JB	Trip blank detection
Acetone	Float 1 - 100109	0910192-03A	24 µg/m3	JB	Trip blank detection
Acetone	Float 1 - 100209	0910192-04A	47 µg/m3	JB	Trip blank detection
Acetone	BD 1 - 100209	0910192-05A	41 µg/m3	JB	Trip blank detection
Ethanol	Float 1 - 092909	0910192-01A	5.7 µg/m3	JB	Trip blank detection
Ethanol	Float 1 - 100109	0910192-03A	4 µg/m3	JB	Trip blank detection
Ethanol	BD 1 - 100209	0910192-05A	5.6 µg/m3	JB	Trip blank detection
m,p-Xylene	Float 1 - 093009	0910192-02A	4.8 µg/m3	JB	Trip blank detection
m,p-Xylene	Float 1 - 100109	0910192-03A	1.1 µg/m3	U	Trip blank detection
o-Xylene	Float 1 - 093009	0910192-02A	2.2 µg/m3	JB	Trip blank detection
Naphthalene	Float 1 - 093009	0910192-02A	4.8 µg/m3	J	Flagged by the Laboratory.

TABLE 4. FIELD DUPLICATE SUMMARY, CHEVRON SITE, CINCINNATI, OHIO (0910192)

Client Sample ID: Float 1-100209 Field Duplicate Sample ID: BD 1-100209			
Analyte	Laboratory Result ($\mu\text{g}/\text{m}^3$)	Duplicate Result ($\mu\text{g}/\text{m}^3$)	Relative Percent Difference (RPD)
Freon 12	2.2	2.2	0.0%
Chloromethane	1.6	1.4	13.3%
Bromomethane	1.9	ND(0.76)	DL
Freon 11	1.9	2	+/- RL
Ethanol	42	5.6	152.9%
Acetone	47	41	13.6%
2-Propanol	2.5	ND(2.4)	DL
Carbon Disulfide	12	ND(3)	DL
2-Butanone (Methyl Ethyl Ketone)	11	3	114.3%
Benzene	ND(0.66)	1	DL
1,4-Dioxane	0.79	ND(0.71)	DL
Toluene	1	1.2	+/- RL
<p>Field duplicate RPD control limits should not exceed 30% for water, 50% for soil, or 25% for air or vapor as established by USEPA Region 1 Laboratory Data Validation Function Guidelines for Evaluation of Organic Analysis, December 1996. The RPDs for 2-butanone and ethanol exceeded the data validation QC limit of 25% for air samples. These analytes were qualified J for detections and UJ for non-detections for each sample reported for this data set based on professional judgment due to possible poor precision.</p> <p>DL – Indicates that one result was detected and one was non-detect. An RPD could not be calculated. Data for carbon disulfide were qualified J for detections and UJ for non-detections for each sample reported for this data set based on professional judgment due to possible poor precision. Data for bromomethane were qualified for the parent and duplicate samples only based on professional judgment. No other data were qualified since the detections were within two times the reporting limit.</p> <p>+/-RL – Indicates that the detections in the samples are within two times the reporting limit. No qualification of data is required.</p>			