Tier III Data Validation Report Summary

Client: Chevron Environmental Management Company – Chevron Cincinnati Facility	Laboratory: Air Toxics LTD
Project Name: Hooven Vapor Investigation	Sample Matrix: Air
Project Number: 500-016-012	Sample Start Date: 03/04/2009
Date Validated: 04/28/2009	Sample End Date: 03/05/2009
Parameters: Volatile Organic Compounds (VOCs) by Modified Method TO-15 and Helium and Fixed Gases by Modified Method ASTM D-1946	
Laboratory Project ID: 0903253A and 0903253C (TO-15 MOD) and 0903253B (ASTM D-1946 MOD)	
Data Validator: Leslie Hill, Senior 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 laboratory and field duplicate pairs. Laboratory accuracy was established by reviewing the demonstrated percent recoveries of laboratory control samples (LCS) to verify that none of the data were biased. Method compliance was established by reviewing holding times, detection limits, surrogate recoveries, method blanks, and the LCS 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 any other necessary documents associated with this analytical data set.

Data were evaluated in general accordance with validation criteria set forth in the 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 Contract Laboratory Program (CLP) National Functional Guidelines for Organic Data Review, document number EPA 540/R-99-008 of October 1999. Review of duplicates is conducted in accordance with USEPA Region 1 Laboratory Data Validation Function Guidelines for Evaluation of Organic Analysis, December 1996. 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
VW-96(55),030409[b]	0903253A-01A/B-01A, -01AA
SW-96(50),030409[a]	0903253A-02A, -02AA/B-02A
SW-96(45),030409	0903253A/B-03A
SW-96(40),030409	0903253A/B-04A
SW-96(35),030409	0903253A/B-05A
SW-96(30),030409	0903253A/B-06A
SW-96(25),030409	0903253A/B-07A

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Client Sample ID	Sample Number
SW-96(20),030409	0903253A/B-08A
SW-96(15),030409	0903253A/B-09A
SW-96(10),030409	0903253A/B-10A
SW-96(5),030409	0903253A/B-11A
BD-1,030509	0903253A/B-12A
FLOAT,030409	0903253C-13A

[a] – A laboratory duplicate was prepared from this sample for the Method TO-15 analyses.
[b] – A laboratory duplicate was prepared from this sample for the Method ASTM-D-1946 analyses.

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The samples were analyzed for client-specified analytes. The samples were shipped to Air Toxics LTD under chains-of-custody. 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 were 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
- ⊗ Calibrations
- ✓ System Monitoring Compounds (i.e. Surrogates)
- ⊗ Laboratory Control Samples (LCS)
- ✓ Laboratory Duplicates
- ✓ Field Duplicates

OVERALL DATA PACKAGE ASSESSMENT

Based on a data validation review, the data are acceptable as delivered. 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, 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. A total of 26 data points were qualified due to out of range calibration data, LCS recoveries, and matrix interference identified by the laboratory.

Data qualifiers used during this validation included:

J – Estimated concentration

UJ – Estimated reporting limit

DATA COMPLETENESS

The analyses appeared to be performed as requested on the chain-of-custody records. The associated samples were received by the laboratory and appeared to be analyzed properly. No data points were rejected. The data completeness measure for these data packages are 100%.

TABLE 1. GENERAL VALIDATION CRITERIA CHECKLIST	
1. Did the laboratory identify any non-conformances related to the analytical data?	Yes
Laboratory comments: 0903253A (TO-15 MOD): The laboratory noted that a ten point initial calibration was analyzed on 03/09/2009 on MSD-B. As noted on the accompanying analytical run log, the Level 2 standard was re-analyzed due to no standard injected on column. The Level 3 standard was re-analyzed using the flow controller due to 1,2,4-trichlorobenzene, Hexachlorobutadiene, and naphthalene possibly sticking in the glass syringe. The laboratory noted that a seven point initial calibration was analyzed on MSD-R on 03/05/2009. The following compounds used 0.3 ppbv as the lowest calibration concentration: 1,3-butadiene, chloroform, benzene, 1,2-dibromoethane, styrene, cumene, 1,3,5-trimethylbenzene, and 1,2,4-trimethylbenzene. The laboratory noted that the analytes sec-butylbenzene and butylbenzene were not present in the LCS spiking compound. 0903253B (ASTM D-1946 MOD): The laboratory noted that a seven point initial calibration was analyzed on GC-9 on 04/15/2008. AS noted on the accompanying analytical run log, calibration level 3 was reanalyzed due to an unacceptable linearity for compound C6-C7. Also, the laboratory noted that carbon monoxide was not detected in historic samples. 0903253C (TO-15 MOD): The laboratory noted that an initial calibration curve was analyzed on 03/19/2009 on MSD-Z. The instrument was set up to do full scan and selective ion monitoring (SIM) simultaneously.	
2. Were sample chain-of-custody forms complete?	Yes
Comments: The COC record from field to laboratory was complete, and custody was maintained as evidenced by field and laboratory personnel signatures, dates, and times of receipt.	
3. Were detection limits in accordance with the QAPP, permit, or method, or indicated as acceptable by the Tier I validator?	Yes
Comments: The detection limits were indicated to be acceptable by the Tier I validator. For Method TO-15, the laboratory reported required dilutions between 1.58 and 92.0 times. For Method ASTM D-1946, the laboratory reported required dilutions between 2.20 to 2.76 times.	
4. Were the requested analytical methods in compliance with the QAPP, permit, or COC?	Yes
Comments: The requested analytical methods were performed in accordance with the chain-of-custody form.	
5. Were samples received in good condition within method specified requirements?	Yes
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. Pressure/vacuum changes from the field to the laboratory were less than five inches of mercury for each sample. 100% of the canisters used for sampling were certified by the laboratory. The canister certification results were also reviewed and found to be acceptable. Compounds of interest were quantified in canisters used for sampling; however, the detections were at concentrations less than the reporting limit. Laboratory and field helium results were compared. The differences between the respective results were within acceptable limits.	
6. Were samples analyzed within method specified or technical holding times?	Yes
Comments: The samples were analyzed within method specified holding times.	
7. Were reported units appropriate for the associated sample matrix/matrices and method(s) of analyses?	Yes
Comments: The results for Method TO-15 were reported in units of part per billion by volume (ppbv) and micrograms per cubic meter ($\mu g/m^3$). The results for Method ASTM D-1946 for fixed gases were reported in percentages (%). These units are appropriate for the air matrix.	
8. Do the laboratory reports include all constituents requested to be reported as indicated by the Tier I validator?	Yes
Comments: The Tier I validator indicated that requested constituents were reported as requested.	
9. Were the reporting requirements for flagged data met?	Yes

TABLE 1. GENERAL VALIDATION CRITERIA CHECKLIST	
Comments: 0903253A: Method TO15: In sample VW-96(40)030409, the laboratory used an M flag to indicate that the analyte cyclohexane may have been biased due to apparent matrix interferences. This analyte was qualified as J by the data validator to indicate an estimated concentration in this sample.	
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?	No
Comments: The number of field duplicate samples collected was less than 10% of the total number of samples. Sample BD1,030509 was a field duplicate of sample VW-96(10),030509.	
11. Were field duplicate RPD values less than the upper RPD limit (soil [50%], water [30%], or air/vapor [25%]), as specified by the laboratory or method?	Yes
Comments: The field duplicate RPD values were less than the upper RPD limit of 25%, or RPD values were not applicable due to results that were undetected in both samples or results that were within two times the reporting limits. Field duplicate and native sample concentrations that were both undetected are not reflected in the table at the end of this report since RPDs are not applicable.	
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: There were no equipment blank, trip blank, or field blank samples associated with this sample set.	
13. Were detections found in trip blanks, equipment blanks, or field blanks?	N/A
Comments: There were no equipment blank, trip blank, or field blank samples associated with this sample set.	

TABLE 2. VALIDATION CRITERIA CHECKLIST FOR VOC ANALYSES (METHOD TO-15)	
1. Were the initial and continuing calibration verifications within acceptable limits?	No
Comments: Calibration criteria were met for all samples and analyses with the following exceptions. **0903253A: Method TO15: In the initial calibration analyzed on 03/05/2009 at 21:17 and on 03/07/2009 at 11:10 the percent relative standard deviation (%RSD) for naphthalene was outside data validation QC limits of 0-30% at 34.300%. As naphthalene was not detected in associated samples BD-1,030509, VW-96(10)030509, VW-96(15)030509, VW-96(20)030509, VW-96(25)030409, VW-96(30)030409, VW-96(35)030409, VW-96(40)030409, VW-96(45),030409, VW-96(5)030509, VW-96(50),030409, and VW-96(55),030409, it was qualified as UJ to indicate estimated reporting limits.**	
2. Were the instrument tunes within method control limits?	Yes
Comments: The GC/MS instrument tunes were within method control limits.	
3. Were the internal standards within method control limits?	Yes
Comments: The internal standard area counts differences were within TO-15 QC limits of ±40% of the internal standard area and within ±0.50 minute of the internal standard retention time.	
4. Was the total number of method blank samples prepared equal to at least 5% of the total number of samples, or analyzed as required by the method?	Yes
Comments: Method blank samples were analyzed for this data set on a greater than 5% basis.	
5. Were method blank detections reported for this data set?	No
Comments: There were no detections of target analytes in the method blank samples.	
6. Was the total number of laboratory control samples analyzed equal to at least 5% of the total number of samples, or analyzed as required by the method?	Yes
Comments: Laboratory control samples were analyzed for this data set on a greater than 5% basis.	
7. Were laboratory control recoveries within acceptable limits?	No
Comments: LCS recoveries were within acceptable limits with the following exceptions. Please note that sec-butylbenzene and butylbenzene were not added to the LCS spike mix. As these analytes are unusual target analytes, this omission has been determined to be acceptable. **0903253A: Method TO15: In the LCS analyzed on 03/13/2009 the recovery of 1,2,4-trichlorobenzene was outside laboratory QC limits of 70-130% at 68.76%. As 1,2,4-trichlorobenzene was not detected in associated samples, BD-1,030509, VW-96(10)030509, VW-96(15)030509, VW-96(20)030509, VW-96(25)030409, VW-96(30)030409, VW-96(35)030409, VW-96(40)030409, VW-96(45),030409, VW-96(5)030509, VW-96(50),030409, and VW-96(55),030409, it was qualified as UJ to indicate estimated reporting limits.** 0903253C: Method TO15: In the LCS analyzed on 03/20/2009 the recovery of ethanol was outside laboratory QC limits of 60-140% at 53.79%. As there were no ethanol analyses performed on this date, no qualification of data was required. **In the LCS analyzed on 03/21/2009 the recovery of ethanol was outside laboratory QC limits of 60-140% at 57.11%. As ethanol was detected in associated sample FLOAT,030409, it was qualified as J to indicate an estimated result.**	
8. Was the total number of matrix spike 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 are not required by Method TO-15.	
9. Were matrix spike recoveries within acceptable limits?	N/A
Comments: Matrix spike samples are not required by Method TO-15.	
10. Were surrogate recoveries within control limits?	Yes
Comments: Surrogate recoveries were within control limits.	
11. Were laboratory duplicate RPD values within laboratory-specified limits?	Yes
Comments: A laboratory duplicate was prepared from sample VW-96(50),030409. Laboratory duplicate RPD values were within laboratory specified limits.	

TABLE 3. VALIDATION CRITERIA CHECKLIST FOR METHANE AND FIXED GAS ANALYSES (ASTM D-1946 MOD)	
1. Were the initial and continuing calibration verifications within acceptable limits?	Yes
Comments: The initial and continuing calibration verifications were within acceptable limits.	
2. Were the instrument tunes within method control limits?	N/A
Comments: Instrument tunes are not required by Method ASTM D-1946 MOD.	
3. Were the internal standards within method control limits?	N/A
Comments: Internal standards are not required by Method ASTM D-1946 MOD.	
4. Was the total number of method blank samples prepared equal to at least 5% of the total number of samples, or analyzed as required by the method?	Yes
Comments: The total number of method blank samples analyzed for this data set was greater than 5% of the number of samples analyzed.	
5. Were method blank detections reported for this data set?	No
Comments: There were no detections of target analytes in the method blanks.	
6. Was the total number of laboratory control samples analyzed equal to at least 5% of the total number of samples, or analyzed as required by the method?	Yes
Comments: The total number of laboratory control samples analyzed for this data set was greater than 5% of the number of samples analyzed.	
7. Were laboratory control recoveries within acceptable limits?	Yes
Comments: Laboratory control sample recoveries were within laboratory QC limits.	
8. Was the total number of matrix spike 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 are not required by Method ASTM D-1946.	
9. Were matrix spike recoveries within acceptable limits?	N/A
Comments: Matrix spike samples are not required by Method ASTM D-1946.	
10. Were surrogate recoveries within control limits?	N/A
Comments: Surrogate recoveries are not required by Method ASTM D-1946.	
11. Were laboratory duplicate RPD values within laboratory-specified limits?	Yes
Comments A laboratory duplicate was prepared from sample VW-96(55),030409. Laboratory duplicate RPD values were within laboratory specified limits.	

DATA QUALIFICATION SUMMARY

Analyte	Method	Field Sample ID	Lab Sample ID	Result (µg/m^3)	Reviewer Qualifier	Reviewer Qualifier Reason
1,2,4-Trichlorobenzene	TO-15	BD-1,030509	0903253A-12A	ND (3100)	UJ	The LCS and/or LCSD recovery(ies) were below the acceptable limits indicating a possible low bias.
1,2,4-Trichlorobenzene	TO-15	VW-96(10)030509	0903253A-10A	ND (3700)	UJ	The LCS and/or LCSD recovery(ies) were below the acceptable limits indicating a possible low bias.
1,2,4-Trichlorobenzene	TO-15	VW-96(15)030509	0903253A-09A	ND (1100)	UJ	The LCS and/or LCSD recovery(ies) were below the acceptable limits indicating a possible low bias.
1,2,4-Trichlorobenzene	TO-15	VW-96(20)030509	0903253A-08A	ND (390)	UJ	The LCS and/or LCSD recovery(ies) were below the acceptable limits indicating a possible low bias.
1,2,4-Trichlorobenzene	TO-15	VW-96(25)030409	0903253A-07A	ND (3600)	UJ	The LCS and/or LCSD recovery(ies) were below the acceptable limits indicating a possible low bias.
1,2,4-Trichlorobenzene	TO-15	VW-96(30)030409	0903253A-06A	ND (7200)	UJ	The LCS and/or LCSD recovery(ies) were below the acceptable limits indicating a possible low bias.
1,2,4-Trichlorobenzene	TO-15	VW-96(35)030409	0903253A-05A	ND (13000)	UJ	The LCS and/or LCSD recovery(ies) were below the acceptable limits indicating a possible low bias.
1,2,4-Trichlorobenzene	TO-15	VW-96(40)030409	0903253A-04A	ND (6500)	UJ	The LCS and/or LCSD recovery(ies) were below the acceptable limits indicating a possible low bias.

Analyte	Method	Field Sample ID	Lab Sample ID	Result (µg/m^3)	Reviewer Qualifier	Reviewer Qualifier Reason
1,2,4-Trichlorobenzene	TO-15	VW-96(45),030409	0903253A-03A	ND (11000)	UJ	The LCS and/or LCSD recovery(ies) were below the acceptable limits indicating a possible low bias.
1,2,4-Trichlorobenzene	TO-15	VW-96(5)030509	0903253A-11A	ND (36)	UJ	The LCS and/or LCSD recovery(ies) were below the acceptable limits indicating a possible low bias.
1,2,4-Trichlorobenzene	TO-15	VW-96(50),030409	0903253A-02A	ND (12000)	UJ	The LCS and/or LCSD recovery(ies) were below the acceptable limits indicating a possible low bias.
1,2,4-Trichlorobenzene	TO-15	VW-96(55),030409	0903253A-01A	ND (14000)	UJ	The LCS and/or LCSD recovery(ies) were below the acceptable limits indicating a possible low bias.
Cyclohexane	TO-15	VW-96(40)030409	0903253A-04A	3400	J	Laboratory indicated that result was biased due to matrix interference.
Naphthalene	TO-15	BD-1,030509	0903253A-12A	ND (2200)	UJ	% RSD above QC limit
Naphthalene	TO-15	VW-96(10)030509	0903253A-10A	ND (2600)	UJ	% RSD above QC limit
Naphthalene	TO-15	VW-96(15)030509	0903253A-09A	ND (800)	UJ	% RSD above QC limit
Naphthalene	TO-15	VW-96(20)030509	0903253A-08A	ND (280)	UJ	% RSD above QC limit
Naphthalene	TO-15	VW-96(25)030409	0903253A-07A	ND (2500)	UJ	% RSD above QC limit
Naphthalene	TO-15	VW-96(30)030409	0903253A-06A	ND (5100)	UJ	% RSD above QC limit
Naphthalene	TO-15	VW-96(35)030409	0903253A-05A	ND (9000)	UJ	% RSD above QC limit
Naphthalene	TO-15	VW-96(40)030409	0903253A-04A	ND (4600)	UJ	% RSD above QC limit
Naphthalene	TO-15	VW-96(45),030409	0903253A-03A	ND (7800)	UJ	% RSD above QC limit
Naphthalene	TO-15	VW-96(5)030509	0903253A-11A	ND (25)	UJ	% RSD above QC limit
Naphthalene	TO-15	VW-96(50),030409	0903253A-02A	ND (8100)	UJ	% RSD above QC limit
Naphthalene	TO-15	VW-96(55),030409	0903253A-01A	ND (9600)	UJ	% RSD above QC limit
Ethanol	TO-15	FLOAT,030409	0903253C-13A	2.7	J	The LCS and/or LCSD recovery(ies) were below the acceptable limits indicating a possible low bias.

FIELD DUPLICATE SUMMARY

Client Sample ID: VW-96(10)030509			
Field Duplicate Sample ID: BD-1,030509			
Analyte	**Lab Result**	**Duplicate Result**	**Relative Percent Difference (RPD)**
2,2,4-trimethylpentane	280000 µg/m^3	280000 µg/m^3	0%
Butane	ND (1200 µg/m^3)	1100 µg/m^3	DL
Isopentane	7900 µg/m^3	8100 µg/m^3	2.5%
CO2 by Headspace	5.20%	5.20%	0%
Methane	0.49%	0.48%	2%
Nitrogen	84%	84%	0%
Oxygen	10%	10%	0%
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.			
DL – indicates one was detected and one was non-detect and an RPD could not be calculated, no data were qualified since the detection was within two times the reporting limit.			