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Multi-Lab Data Audit Report

Summary of Findings and Recommendations from an Audit of
Data Packages for Analyses Performed under the MassDEP's
Compendium of Analytical Methods

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EXECUTIVE SUMMARY

This report presents the findings of the Massachusetts Department of Environmental Protection's (MassDEP's) audit of analyses certified by each participating laboratory as having been performed according to MassDEP's Compendium of Analytical Methods (CAM) for Volatile Petroleum Hydrocarbons (VPH), Extractable Petroleum Hydrocarbons (EPH), Air-Phase Petroleum Hydrocarbons (APH) and Volatile Organic Compounds in Air (TO-15). This audit is one of a number of initiatives the Department has undertaken to ensure that the quality of analytical data for analyses conducted on samples from disposal sites regulated under Massachusetts General Law Chapter 21E and the Massachusetts Contingency Plan (MCP) is adequately documented and commensurate with its use in supporting assessment and cleanup decisions.

In general, the data audits demonstrated that the EPH and VPH methods exhibited more significant issues such as method deviations, calculation errors, and reporting issues. In general, significant issues were defined as those issues which could result in a change to the reported concentrations or reporting limits, the values ultimately used in the MCP decision-making process. The data audits for the TO-15 and APH methods also found several method deviations and reporting issues, but these were less significant and less frequent, indicating that overall, the laboratory community has a good understanding of the TO-15 and APH methods and the ability to clearly follow the procedures and CAM requirements for these methods.

The significant issues noted during the data audit for each method are summarized in Table ES-1 below. Tables 9-2 through 9-5 of this report summarize significant issues by parameter and laboratory. Of particular concern is that the majority of these issues would not be discovered by reviewing a typical CAM deliverable. In addition, if the data user relied solely upon the MassDEP Analytical Protocol Certification Form (Certification Form) to determine whether the data obtained Presumptive Certainty and the analyses complied with CAM requirements, such reliance would result in an inaccurate determination. In many cases, the laboratory completed the Certification Form as if data achieved a status of Presumptive Certainty (i.e., provided a "Yes" response to Question B) when in actuality, based upon the audit, the data did not achieve this status.

This data audit was designed to look across multiple labs to assess compliance with the CAM quality control requirements with respect to the four analytical methods. While data packages submitted in support of MCP response actions were reviewed, this data audit only looked at a subset of the analytical data collected and used at a given disposal sites. As such, no conclusions about the impact these issues may have on overall site characterizations can be drawn from this report.

MassDEP provided the preliminary findings of this audit to the laboratories and solicited their comments. The final report reflects MassDEP's consideration of the laboratories' responses. A number of laboratories have implemented corrective actions to address practices identified by the audit as not complying with the CAM.

Table ES-1
Significant Issues Noted During Data Audit

Method	Description of Issue	# Laboratories Affected
TO-15	Reporting Limits (RLs): RLs not verified when linear or quadratic regression used by calculating concentrations in lowest concentration standard.	2
VPH	Calibration: Calibration factors (CFs) for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range.	7
	Results Quantitation: Sample results changed when correct CFs utilized in calculations.	6
	Results Quantitation: Errors made in calculation (e.g., concentration of surrogate subtracted from range instead of area count, concentrations of target analytes <RL subtracted from ranges, concentration of C ₉ -C ₁₀ aromatics not subtracted from C ₉ -C ₁₂ aliphatics).	3
	Reporting Limits: RLs too low; RLs for hydrocarbon ranges were based on a concentration less than 100 times the lowest calibration standard.	9
	Reporting Limits: RLs not verified when linear or quadratic regression was used by calculating concentrations in lowest concentration standard.	3
	Range Retention Times: Not established per method requirements.	3
	Significant Modification: Wrong column used with no or inadequate column equivalency study provided.	3
	Peak Integration: Peaks over-integrated in continuing calibration standard to ensure passing standard; valley-to-valley integration not performed for target analytes.	2
	Sample Preservation: Soil samples not preserved in methanol.	1
EPH	Calibration: CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range.	8
	Calibration: Missing information and therefore unable to verify CFs reported.	2
	Results Quantitation: Sample results changed when correct CFs utilized in calculations.	8
	Reporting Limits: RLs too low; RLs for hydrocarbon ranges were based on a concentration less than 100 times the lowest calibration standard.	5
	Reporting Limits: RLs too high; RLs for hydrocarbon ranges were based on a concentration greater than 100 times the lowest calibration standard.	3
	Reporting Limits: RLs not verified when linear or quadratic regression used by calculating concentrations in lowest concentration standard.	4
	Range Retention Times: Not established per method requirements.	2
APH	Target Compound Identification: Polycyclic aromatic hydrocarbons (PAHs) reported as co-eluting in samples and standards.	1
	Calibration: m/z 134 not utilized for measurement of C ₉ -C ₁₀ aromatics.	1
	Calibration: n-Hexane and cyclohexane not used in calibration of C ₅ -C ₈ aliphatics.	1
	Calibration: Hydrocarbon ranges calibrated in units of ppbV.	1
	Calibration: Quadratic regression utilized for C ₉ -C ₁₀ aromatics and select target analytes.	1
	Results Quantitation: Mass/charge (m/z) 134 not utilized for measurement of C ₉ -C ₁₀ aromatics.	1
	Results Quantitation: Sample results changed when linear regression utilized in calculations instead of quadratic regression.	1
	Results Quantitation: Errors in sample result calculations (e.g., area counts of target analytes subtracted from ranges instead of concentrations, discovered integration errors when compiling full audit deliverable).	2
	Reporting Limits: RLs not verified when linear or quadratic regression used by calculating concentrations in lowest concentration standard.	2
	Reporting Limits: RLs for hydrocarbon ranges too low; not established as per method requirements.	2

As a result of the data audit, MassDEP has provided a clarification in the VPH method (February 2018) and the EPH method (December 2019) on the hydrocarbon range calibration procedures, one of most common cited audit nonconformances. Additional MassDEP actions under consideration as a result of this audit are detailed in Section 10 of the Report and include:

- Providing outreach and training for analytical laboratories and LSPs;
- Issuing Notices of Non-Compliance (NONs) to the laboratories for non-conformance with CAM requirements;
- Performing routine data audits;
- Expanding the CAM by including a mandatory third-party audit program; and
- Implementing a 21E laboratory certification program.

MassDEP intends to have additional discussions with program stakeholders on the results of this audit and actions that can be taken to continue to improve the reliability of data analyses and the application of the CAM for samples taken to support response action decisions at 21E disposal sites.

1.0 INTRODUCTION

The Massachusetts Department of Environmental Protection (MassDEP) Multi-Laboratory Data Audit project included evaluation of 38 data packages from 13 different laboratories. These 13 laboratories have performed most of the Chapter 21E analytical work in Massachusetts. Data were evaluated to ensure compliance with MassDEP Compendium of Analytical Methods (CAM) and Environmental Protection Agency (EPA) guidance for acceptable quality assurance/quality control (QA/QC) for scientifically defensible data. The data audit was conducted with the support of TRC Environmental Corporation, under a contract with MassDEP's Bureau of Waste Site Cleanup.

Four analytical methods were included in the data audit:

- Volatile petroleum hydrocarbons (VPH) by MassDEP Method, Revision 1.1, May 2004, WSC-CAM-IVA
- Extractable petroleum hydrocarbons (EPH) by MassDEP Method, Revision 1.1, May 2004, WSC-CAM-IVB
- Air-phase petroleum hydrocarbons (APH) by MassDEP Method, Revision 1, December 2009, WSC-CAM-IXA
- Volatile Organic Compounds (VOCs) by EPA method TO-15, WSC-CAM-IXB

All of the data packages from the 13 laboratories had been submitted to MassDEP after January 1, 2014, to support assessment and/or remedial actions at contaminated sites regulated by the Massachusetts Contingency Plan (MCP). Each data package contained a *MassDEP Analytical Protocol Certification Form* (Certification Form), consisting of a series of Yes or No attestations on the laboratory's compliance with CAM protocols and standards. All Certification Forms were signed by an authorized representative of the laboratory, attesting to the accuracy and completeness of responses.

After an initial review, Request for Information (RFI) letters were sent to each laboratory, requesting supplemental information on each data package (see Appendix 1 for a sample RFI letter and the Data Package Requirements). A copy of this letter was also sent to the Licensed Site Professional (LSP) and potentially responsible parties (PRPs) who had made the MCP response action submittal that contained the data package(s). The requested information was needed to conduct a detailed audit of the data submittals. Under CAM, such information is required to be generated and retained by the laboratory and provided to MassDEP upon request. The laboratory was given 30 days to provide the requested information.

After the data audits were conducted, each laboratory was provided a copy of the preliminary findings and given an opportunity to respond to these findings. The laboratories were asked to document whether or not they agreed with the finding, the corrective action that was put in place to correct the finding, and the date the corrective action was put in place. This final report reflects MassDEP's consideration of the laboratories' responses.

This Data Audit was performed with the following intentions: 1) to evaluate the validity of the results and determine if the data were generated and reported in accordance with the applicable MassDEP CAM documents and 2) to determine if the data are usable for MCP decisions based on CAM requirements for acceptable accuracy, precision, sensitivity and technical usability. Applicable MCP CAM and EPA regulatory documents upon which this audit was based are listed in Section 3.0.

This report has been compiled to summarize the types and severity of the QA/QC issues uncovered in the Data Audit (see scope of data audit in Table 2-1) and how these issues potentially impact the use of the data for MCP decisions. An overall summary of CAM-compliance can be found in Tables 9-2 through 9-5.

2.0 SCOPE OF WORK SUMMARY

Table 2-1 summarizes the laboratories included in the data audit and the data package identification numbers, organized by analytical method.

Table 2-1 Summary of Laboratories and Data Packages Included in the MassDEP Data Audits	
Laboratory Name *	Data Package ID
EPA Method TO-15 (indoor air, ambient air, and soil gas)	
SGS-Accutest (Marlborough, MA)	MC27326
Alpha Analytical	L1602185
Con-test Analytical	15J0798
New England Testing (NETLab)	C-0422
Phoenix	BK52966-BK52974
Eurofins/Spectrum	SC00448
Test America-Buffalo	200-30186
MassDEP APH (indoor air, ambient air, and soil gas)	
SGS-Accutest (Marlborough, MA)	MC41710
Alpha Analytical	L1519362
Con-test Analytical	14L0320
NETLab	B0914
Phoenix	GBK86635 (subcontracted by Eastern Analytical 154036)
Eurofins/Spectrum	SC19582
Katahdin Analytical	SH0764 (subcontracted by Test America-Buffalo 200-20778)
MassDEP VPH (soil and groundwater)	
Absolute Resources	33120 (2015-014)
SGS-Accutest (Marlborough, MA)	MC45941
Alpha Analytical	L1525444
AMRO Environmental	1503031
Chemserve	16050061
Con-test Analytical	14I1252
ESS	1503322
NETLab	B1217-25
Phoenix	GBH46059
Eurofins/Spectrum	SC18902
RI Analytical	1504-07728
Test America-Buffalo	480-37724
MassDEP EPH (soil and groundwater)	
Absolute Resources	33120 (2015-014)
SGS-Accutest (Marlborough, MA)	MC45941
Alpha Analytical	L1606629
AMRO Environmental	1601047
Chemserve	15110079
Con-test Analytical	15D1233
ESS	1505514
NETLab	B1217-25
Phoenix	GBH44795
Eurofins/Spectrum	SC06377
RI Analytical	1404-07494
Test America-Buffalo	480-57712
* Name of the laboratory at the start of the data audit.	

Backup documentation of the data audit process for each laboratory data package consisted of the following:

- A summary of all items reviewed and issues noted. This included CAM method-specific QC, overall compliance with CAM, and usability in terms of accuracy, precision, and sensitivity of results. Acceptance criteria were based on the detailed performance standards listed in CAM methods, EPA data validation guidance, the EPA or MassDEP Method, and professional judgment, when applicable.
- Spreadsheets documenting checks of calculations on calibration, sample results, and/or reporting limits (RLs).

This report provides the following information for each analytical parameter:

- A summary table of all non-conformance issues noted as compared to CAM, categorized by the types of issues noted (e.g., CAM reporting non-compliance, CAM method deviation, calculation errors, QC non-compliance, laboratory error), and the overall effect on the usability of the data for MCP decisions.
- Overall summary of audit results for all laboratory data packages.

3.0 REFERENCES

References for guidance documents used in the Data Audit are as follows:

- MassDEP WSC-CAM-IVA *Quality Control Requirements and Performance Standards for the Analysis of Volatile Petroleum Hydrocarbons (VPH) in Support of Response Actions Under the Massachusetts Contingency Plan (MCP)*, July 2010.
- MassDEP WSC-CAM-IVB *Quality Control Requirements and Performance Standards for the Analysis of Extractable Petroleum Hydrocarbons (EPH) in Support of Response Actions Under the Massachusetts Contingency Plan (MCP)*, July 2010.
- MassDEP WSC-CAM-VIIA *Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)*, January 2017.
- MassDEP WSC-CAM-IXA *Quality Control Requirements and Performance Standards for the Analysis of Air-Phase Petroleum Hydrocarbons (APH) by Gas Chromatography/Mass Spectrometry (GC/MS) in Support of Response Actions Under the Massachusetts Contingency Plan (MCP)*, July 2010.
- MassDEP WSC-CAM-IXB *Quality Control Requirements and Performance Standards for the Analysis of Volatile Organic Compounds in Air Samples (TO-15) by Gas Chromatography/Mass Spectrometry (GC/MS) in Support of Response Actions Under the Massachusetts Contingency Plan (MCP)*, July 2010.
- MassDEP *Method for the Determination of Volatile Petroleum Hydrocarbons (VPH)*, Revision 1.0, May 2004.
- MassDEP *Method for the Determination of Extractable Petroleum Hydrocarbons (EPH)*, Revision 1.0, May 2004.
- MassDEP *Method for the Determination of Air-Phase Petroleum Hydrocarbons (APH)*, Revision 1, December 2009.
- USEPA *Determination of Volatile Organic Compounds (VOCs) in Air Collected in Specially-prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry (GC/MS)*, January 1999.
- USEPA *National Functional Guidelines for Organic Superfund Methods Data Review*, EPA-540-R-2017-002, January 2017.

4.0 OVERALL SUMMARY OF CAM NON-COMPLIANCE ISSUES

This Data Audit Report includes the specific CAM compliance issues observed in the individual data audits, organized by type of non-compliance and summarized by frequency of occurrence for each parameter type.

Various CAM reporting non-compliances, CAM method deviations, calculation errors, and QC non-compliances were noted during the data audit.

Sections 5 through 9 summarize the types of issues discovered, the number of laboratories where the issues were observed, the potential effect on the usability of the data and whether or not the issue would be obvious to the data user with a typical CAM-required deliverable.

4.1 Evaluation of CAM Deliverable Requirements & Completeness

Under CAM, two types of data packages are specified. As a minimum/default requirement, data packages must contain a limited amount of QC information (e.g., method blank, laboratory control sample [LCS] data) together with the MassDEP Certification Form. The Certification Form was specifically developed to provide a simple overall summary and certification of data quality, as most LSPs (and MassDEP staff) are not proficient in detailed data review and validation.

While the default package constitutes the vast majority of data submittals made to MassDEP, CAM also specifies an expanded data submittal package that must be provided by a laboratory upon request. This expanded submittal contains detailed information on method calibration and quality control parameters, for review and evaluation by persons with expertise in this area, to ensure that the attestations provided in Certification Forms are accurate.

During this Data Audit, both the original (“CAM Deliverable”) and expanded (“Full Deliverable”) data packages were evaluated. Of particular interest was the accuracy and completeness of the original data package submittals (Table 2-1), as the QC data and representations contained in these submittals are generally the basis of the data usability and representativeness evaluations that are required by the MCP, and as further discussed in MassDEP policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

The most common CAM deliverable completeness issue involved the lack of required information in the laboratory narratives. In general, CAM protocols require that all QC nonconformances be discussed in the laboratory narrative. In general, data packages that were non-compliant with CAM deliverable requirements resulted in misleading information and omissions such that the data user would not be able to properly assess data quality. In addition, some of the full deliverables that are required to be provided by laboratories were missing; this does not adhere to CAM requirements. The absence of this information does not allow for a complete evaluation. The specific issues noted in the review of CAM deliverables are summarized in Sections 5 through 9.

4.2 Evaluation of Accuracy, Precision, and Method Compliance

Evaluation of accuracy and precision was based on CAM performance standards including holding times and sample preservation, calibration QC, method blank results, media certification (for TO-15 and APH analyses), surrogate spike recoveries, LCS and LCS duplicate (LCSD) results, internal standard results, method-specific QC (e.g., breakthrough check results for EPH analysis), RL evaluations, and sample result verification. Additionally, the raw data provided by the laboratories were evaluated for compliance with CAM and to verify all calculations were performed correctly, from calibration through the final reporting

of sample results and RLs. As discussed in Sections 5 through 9, the most common issues were related to calibration processes, result quantitation, and RL calculations in the VPH and EPH methods.

4.3 Evaluation of the MassDEP Analytical Protocol Certification Form

The MassDEP Certification Form is required to be present and correctly completed for “Presumptive Certainty” under the MCP CAM program. However, many of the laboratories input “Yes” to select questions when the correct answers were “No” due to method deviations (e.g., lack of proper corrective actions) and QC issues (e.g., LCS/LCSD and continuing calibration verification [CCV] recoveries outside of CAM acceptance limits). CAM requires that these issues be included in the laboratory narrative; however, several laboratories that incorrectly filled out the Certification Form had incomplete laboratory narratives or misrepresented data quality as acceptable by incorrectly stating that no non-compliances were noted.

These issues cause significant concern since the affected laboratories provided an attestation that all information in the reports was accurate and complete. This results in an overall concern over the reliability of the Certification Form and the laboratory narratives as tools for evaluating data quality.

5.0 SUMMARY OF CAM NON-COMPLIANCE ISSUES FOR TO-15

5.1 Data Deliverables: TO-15

Table 5-1 summarizes the data deliverable issues for the TO-15 analyses. Each of the seven laboratories included in the TO-15 audit exhibited issues with the original CAM deliverable and/or the full audit data deliverable. CAM deliverables that are non-compliant with CAM requirements can result in misleading information and omissions such that the data user would not be able to properly or completely assess data quality. In general, the missing information in the full deliverables used in the audit did not hinder the assessment of the overall usability and accuracy of the data set.

Table 5-1 Data Deliverable Issues: TO-15		
Laboratory	CAM Deliverable	Full Deliverable Used in Audit
Con-test Analytical	The percent relative standard deviation (%RSD) for acetone in the initial calibration was outside of the acceptance criteria; not noted in laboratory narrative. Media certification check detected acetone above the RL; not noted in laboratory narrative.	Spectra of detected results in media certification analysis not provided.
Alpha Analytical	The percent difference (%D) for one compound was outside of the acceptance criteria in the continuing calibration; not noted in laboratory narrative.	Raw data for media certifications not originally provided; provided in resubmittal.
SGS-Accutest	No issues noted.	Extracted ion chromatograms for manual integrations not provided. Raw data for media certifications not provided.
Eurofins/Spectrum	No issues noted.	Raw data for media certifications not provided.
NETLab	Ethanol detected above calibration range in sample; not noted in laboratory narrative. Select CAM RLs not met; not noted in laboratory narrative. Flow controller calibration relative percent differences (RPDs) >20; not noted in laboratory narrative. Sample received with vacuum at 0 in Hg; not noted in laboratory narrative.	Dilution factors not listed on result summaries. Extracted ion chromatograms not provided for manually integrated compounds in calibration standards. Spectra missing for some detected results.
Phoenix	Media certification results not originally provided; provided in resubmittal. Flow controller calibration RPDs not originally provided; provided in resubmittal.	Raw data for media certifications not provided.
TestAmerica-Buffalo	No issues noted.	Raw data for media certifications not provided. Ion intensities for detected analytes not provided.

5.2 Accuracy, Precision & Method Compliance: TO-15

Evaluation of accuracy and precision was based on CAM performance standards including holding times and sample integrity, instrument tunes, calibration QC, method blank results, media certification, surrogate spike recoveries, LCS results, internal standard results, RL evaluations, and sample result verification. Additionally, the raw data provided by the laboratories were evaluated for compliance with CAM and to verify all calculations were performed correctly, from calibration through the final reporting of sample results and RLs. Table 5-2 summarizes the evaluation parameters where issues were noted.

Table 5-2 Accuracy, Precision & Method Compliance Summary: TO-15	
Evaluation Parameter	Laboratory where Issues Noted
Holding times	No issues noted.
Sample integrity (vacuums, flow controller RPDs)	Vacuums: NETLab Flow controller RPDs: NETLab
Gas chromatograph/mass spectrometer (GC/MS) tunes	No issues noted.
Initial calibration	Con-test Analytical, NETLab, SGS-Accutest
Continuing calibration	Alpha Analytical, Phoenix
Method blanks	Phoenix
LCS	Con-test Analytical, Eurofins/Spectrum, Phoenix
Internal standards	NETLab
Target compound identification	Eurofins/Spectrum, NETLab, Test America-Buffalo
Quantitation	NETLab
Media certification	Con-test Analytical
Laboratory narrative	Alpha Analytical, Con-test Analytical, NETLab
RLs	Con-test Analytical, NETLab, SGS-Accutest
Certification Form	Alpha Analytical, Con-test Analytical, Eurofins/Spectrum, NETLab, Phoenix, SGS-Accutest

5.3 MassDEP Analytical Protocol Certification Form: TO-15

The MassDEP Analytical Protocol Certification Form was present and completed as required for “Presumptive Certainty” under the MCP CAM program for all seven laboratories. Six of the seven laboratories provided incorrect “YES” responses on this form. Table 5-3 summarizes these errors; questions A through F affect the achievement of Presumptive Certainty. Data for six of the seven laboratories may be misrepresented to the end-user since Presumptive Certainty was not achieved.

Table 5-3 Summary of Errors on the MassDEP Analytical Protocol Certification Form: TO-15		
Question	Laboratory	Issue
Question A <i>Were all samples received in a condition consistent with those described on the Chain-of-Custody, properly preserved (including temperature) in the field or laboratory, and prepared/analyzed within method holding times?</i>	No issues noted.	
Question B <i>Were the analytical method(s) and all associated QC requirements specified in the selected CAM protocol(s) followed?</i>	NETLab	Only two internal standards used instead of the required minimum of three. Low-level standard calculation not performed when linear or quadratic regression used for calibration.

Table 5-3 Summary of Errors on the MassDEP Analytical Protocol Certification Form: TO-15		
Question	Laboratory	Issue
	SGS-Accutest	Low-level standard calculation not performed when linear or quadratic regression used for calibration.
Question C <i>Were all required corrective actions and analytical response actions specified in the selected CAM protocol(s) implemented for all identified performance standard non-conformances?</i>	Con-test Analytical	Media certification check detected acetone above the RL; required corrective action to re-clean canisters not followed. Low recoveries of VOCs in LCS; LCS and associated samples not reanalyzed per required corrective action.
	Phoenix	Low recovery of bromoform in LCS; LCS and associated samples not reanalyzed per required corrective action.
	Eurofins/Spectrum	Low recoveries of carbon tetrachloride and hexachlorobutadiene in LCS; LCS and associated samples not reanalyzed per required corrective action.
Question D <i>Does the laboratory report comply with all the reporting requirements specified in CAM VII A, "Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data"?</i>	Phoenix	Post-sampling flow rates and RPDs not included in original data package.
Question E <i>VPH, EPH, APH, and TO-15 only</i> <i>a. VPH, EPH, and APH Methods only: Was each method conducted without significant modification(s)? (Refer to the individual method(s) for a list of significant modifications).</i> <i>b. APH and TO-15 Methods only: Was the complete analyte list reported for each method?</i>	SGS-Accutest	Naphthalene not reported.
	Phoenix	Naphthalene not reported.
	Eurofins/Spectrum	Only halocarbons reported.
	NETLab	Naphthalene not reported.
Question F <i>Were all applicable CAM protocol QC and performance standard non-conformances identified and evaluated in a laboratory narrative (including all "No" responses to Questions A through E)?</i>	Con-test Analytical	The high %RSD for acetone in the initial calibration was not narrated.
	Alpha Analytical	The high %D for 1,2,4-trichlorobenzene in the continuing calibration was not narrated.
Question G <i>Were the RLs at or below all CAM RLs specified in the selected CAM protocol(s)?</i>	NETLab	Select CAM RLs not met.

6.0 SUMMARY OF CAM NON-COMPLIANCE ISSUES FOR MASSDEP VPH

6.1 Data Deliverables: VPH

Table 6-1 summarizes the data deliverable issues for the MassDEP VPH analyses. Eight of the 12 laboratories included in the MassDEP VPH audit exhibited issues with the original CAM deliverable and/or the full audit data deliverable. In general, CAM deliverables that were non-compliant with CAM requirements can result in misleading information and omissions such that the data user would not be able to properly or completely assess data quality. In general, with the exception of one laboratory, the missing information in the full deliverables used in the audit did not hinder the assessment of the overall usability and accuracy of the data set.

Table 6-1 Data Deliverable Issues: VPH		
Laboratory	CAM Deliverable	Full Deliverable Used in Audit
Chemserve	Elevated cooler temperature; not noted in laboratory narrative.	Chromatograms showing integration of target analytes and hydrocarbon ranges not provided; was limitation of software. Retention time windows used for hydrocarbon ranges not provided. No information provided on column used. Initial calibration factor (CF) and %RSD summary not provided.
Eurofins/Spectrum	Three samples were analyzed at 5-fold dilution with only non-detects reported; no explanation provided for dilution in laboratory narrative and no evidence of matrix interference noted.	Could not verify LCS was second-source.
Phoenix	No issues noted.	Initial calibration CF, %RSD, and standard concentration summary not provided. No information provided on how method blank prepared. Could not verify LCS was second-source. No information originally provided on column used, GC conditions, and purge & trap conditions; resubmittal included column information.
AMRO Environmental	Results of the unadjusted ranges were not reported.	No issues noted.
Alpha Analytical	No issues noted.	Could not verify LCS was second-source in original package; resubmittal provided the information.
SGS-Accutest	No issues noted.	Could not verify LCS was second-source. No information provided on column used, GC conditions, and purge & trap conditions.
Con-test Analytical	No issues noted.	No issues noted.
RI Analytical	No issues noted.	Retention time windows used for hydrocarbon ranges not provided.
ESS	No issues noted.	No issues noted.

Table 6-1 Data Deliverable Issues: VPH		
Laboratory	CAM Deliverable	Full Deliverable Used in Audit
Test America-Buffalo	No issues noted.	Individual area counts for aliphatic components not provided for calibration data.
NETLab	No issues noted.	No issues noted.
Absolute Resources	No issues noted.	No issues noted.

6.2 Accuracy, Precision & Method Compliance: VPH

Evaluation of accuracy and precision was based on CAM performance standards including holding times and preservation, GC performance, calibration QC, method blank results, surrogate spike recoveries, LCS results, RL evaluations, and sample result verification. Additionally, the raw data provided by the laboratories were evaluated for compliance with CAM and to verify all calculations were performed correctly, from calibration through the final reporting of sample results and RLs. Table 6-2 summarizes the evaluation parameters where issues were noted.

Table 6-2 Accuracy, Precision & Method Compliance Summary: VPH	
Evaluation Parameter	Laboratory where Issues Noted
Holding times	No issues noted.
Sample preservation	Chemserve, Test America-Buffalo
GC performance	No issues noted.
Initial calibration	Absolute Resources, AMRO Environmental, Chemserve, ESS, Eurofins/Spectrum, NETLab, Phoenix, Test America-Buffalo
Continuing calibration	Absolute Resources, Chemserve, ESS, Eurofins/Spectrum, NETLab, Phoenix, Test America-Buffalo
Method blanks	No issues noted.
LCS	Absolute Resources, Eurofins/Spectrum, Phoenix, Test America-Buffalo
Target compound identification	Test America-Buffalo
Surrogates	Phoenix
Retention time ranges	AMRO Environmental, Chemserve, Eurofins/Spectrum, Test America-Buffalo
Quantitation	Absolute Resources, Chemserve, ESS, Eurofins/Spectrum, NETLab, Phoenix, SGS-Accutest, Test America-Buffalo
Laboratory narrative	Chemserve
RLs	Alpha Analytical, AMRO Environmental, Chemserve, Con-test Analytical, ESS, Eurofins/Spectrum, NETLab, Phoenix, Test America-Buffalo
Significant modifications	Eurofins/Spectrum, Phoenix, Test America-Buffalo
Certification Form	Absolute Resources, AMRO Environmental, Chemserve, Con-test Analytical, ESS, Eurofins/Spectrum, NETLab, Phoenix, SGS-Accutest, Test America-Buffalo

6.3 MassDEP Analytical Protocol Certification Form: VPH

The MassDEP Analytical Protocol Certification Form was present and completed as required for “Presumptive Certainty” under the MCP CAM program for all 12 laboratories. Ten of the 12 laboratories provided incorrect “YES” responses on this form. Table 6-3 summarizes these errors; questions A through F affect the achievement of Presumptive Certainty. Data for 10 of the 12 laboratories may be misrepresented to the end-user since Presumptive Certainty was not achieved.

Table 6-3 Summary of Errors on the MassDEP Analytical Protocol Certification Form: VPH		
Question	Laboratory	Issue
Question A <i>Were all samples received in a condition consistent with those described on the Chain-of-Custody, properly preserved (including temperature) in the field or laboratory, and prepared/analyzed within method holding times?</i>	Chemserve	Elevated cooler temperature.
Question B <i>Were the analytical method(s) and all associated QC requirements specified in the selected CAM protocol(s) followed?</i>	Chemserve	Integration of aliphatic hydrocarbon ranges not performed properly in initial and continuing calibrations. N-decane and n-butylcyclohexane not used in calibration of C ₉ -C ₁₂ aliphatics.
	Absolute Resources	Integration of all hydrocarbon ranges not performed properly in initial and continuing calibrations.
	NETLab	Integration of aliphatic hydrocarbon ranges not performed properly in initial and continuing calibrations. Low-level standard calculation not performed for C ₉ -C ₁₀ aromatics when linear regression used in calibration. LCS not matrix-specific.
	Con-test Analytical	Low-level standard calculation not performed when linear regression used in calibration.
	ESS	Integration of all hydrocarbon ranges not performed properly in initial and continuing calibrations.
	Phoenix	Method of calibration could not be reproduced or understood.
	AMRO Environmental	Low-level standard calculation not performed when quadratic regression used in calibration. Retention times of hydrocarbon ranges not established in accordance with method requirements.
	Eurofins/Spectrum	Integration of aliphatic hydrocarbon ranges not performed properly in initial and continuing calibrations. Retention times of hydrocarbon ranges not established in accordance with method requirements.
	Test America-Buffalo	Integration of all hydrocarbon ranges not performed properly in initial and continuing calibrations. LCS not from a secondary source. Retention times of hydrocarbon ranges not established in accordance with method requirements.
Question C <i>Were all required corrective actions and analytical response actions specified in the selected CAM</i>	Phoenix	LCS percent recoveries (%Rs) outside acceptance limits and no corrective action performed.

Table 6-3		
Summary of Errors on the MassDEP Analytical Protocol Certification Form: VPH		
Question	Laboratory	Issue
protocol(s) implemented for all identified performance standard non-conformances?		Photoionization detector (PID) surrogate %R outside acceptance limits and no corrective action performed.
Question D Does the laboratory report comply with all the reporting requirements specified in CAM VII A, “Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data”?	No issues noted.	
Question E VPH, EPH, APH, and TO-15 only a. VPH, EPH, and APH Methods only: Was each method conducted without significant modification(s)? (Refer to the individual method(s) for a list of significant modifications). b. APH and TO-15 Methods only: Was the complete analyte list reported for each method?	SGS-Accutest	Question b: answered YES but APH or TO-15 not analyzed.
	Absolute Resources	Question a not answered.
	Eurofins/Spectrum	Question a: wrong column used: this is a significant modification.
	Phoenix	
	Test America-Buffalo	
Question F Were all applicable CAM protocol QC and performance standard non-conformances identified and evaluated in a laboratory narrative (including all “No” responses to Questions A through E)?	No issues noted.	
Question G Were the RLs at or below all CAM RLs specified in the selected CAM protocol(s)?	No issues noted.	

7.0 SUMMARY OF CAM NON-COMPLIANCE ISSUES FOR MASSDEP EPH

7.1 Data Deliverables: EPH

Table 7-1 summarizes the data deliverable issues for the MassDEP EPH analyses. Ten of the 12 laboratories included in the MassDEP EPH audit exhibited issues with the original CAM deliverable and/or the full audit data deliverable. In general, CAM deliverables that were non-compliant with CAM requirements can result in misleading information and omissions such that the data user would not be able to properly or completely assess data quality. In general, the missing calibration information in the full deliverables used in the audit did hinder a portion of the assessment of the overall usability and accuracy of the data set.

Table 7-1 Data Deliverable Issues: EPH		
Laboratory	CAM Deliverable	Full Deliverable Used in Audit
Phoenix	Results for unadjusted C ₁₁ -C ₂₂ aromatics not reported.	No information originally provided to verify fractionation check performed on each new lot of cartridges; provided in resubmittal.
Eurofins/Spectrum	Laboratory narrative only discussed quadratic regression for 2 PAHs but should have been 3 PAHs. Laboratory narrative did not discuss %Ds for select PAHs outside of acceptance criteria in continuing calibration.	No information provided to verify fractionation check performed on each new lot of cartridges. Total ion chromatogram (TIC) integrations/area counts for individual PAHs in initial calibration not provided. Could not verify LCS was second-source.
AMRO Environmental	No issues noted.	TIC integrations/area counts for individual analytes in initial and continuing calibrations not provided.
Test America-Buffalo	Laboratory narrative indicated that RLs were not at or below CAM RLs for all samples due to dilutions. However, there were no dilutions performed and the RLs did meet CAM requirements.	No information provided to verify fractionation check performed on each new lot of cartridges.
ESS	No issues noted.	TIC integrations/area counts for individual PAHs in initial calibration not provided. Could not verify LCS was second-source.
Chemserve	Results for C ₁₁ -C ₂₂ aromatics and alkanes not summarized on LCS summary form. No laboratory narrative.	Initial calibration information for aliphatic hydrocarbon ranges not originally provided; provided in resubmittal. Could not verify LCS was second-source. Retention time windows used for aliphatic hydrocarbon ranges not provided.
Absolute Resources	Laboratory narrative did not note select PAHs calibrated using quadratic regression.	Could not verify LCS was second-source. Retention time windows used for hydrocarbon ranges not provided.
SGS-Accutest	No issues noted.	No information provided to verify fractionation check performed on each new lot of cartridges.
RI Analytical	Results for unadjusted C ₁₁ -C ₂₂ aromatics not reported.	No issues noted.
Con-test Analytical	No issues noted.	No issues noted.

Table 7-1 Data Deliverable Issues: EPH		
Laboratory	CAM Deliverable	Full Deliverable Used in Audit
Alpha Analytical	No issues noted.	No issues noted.
NETLab	Results for ranges not summarized on LCS summary form. Results for unadjusted C ₁₁ -C ₂₂ aromatics not reported.	No issues noted.

7.2 Accuracy, Precision & Method Compliance: EPH

Evaluation of accuracy and precision was based on CAM performance standards including holding times and preservation, GC performance, calibration QC, method blank results, surrogate spike recoveries, LCS results, breakthrough, RL evaluations, and sample result verification. Additionally, the raw data provided by the laboratories were evaluated for compliance with CAM and to verify all calculations were performed correctly, from calibration through the final reporting of sample results and RLs. Table 7-2 summarizes the evaluation parameters where issues were noted.

Table 7-2 Accuracy, Precision & Method Compliance Summary: EPH	
Evaluation Parameter	Laboratory where Issues Noted
Holding times	No issues noted.
Sample preservation	Chemserve
GC performance	Chemserve, Phoenix
Fractionation checks	Eurofins/Spectrum, Phoenix, SGS-Accutest, Test America-Buffalo
Initial calibration	Absolute Resources, AMRO Environmental, Chemserve, ESS, Eurofins/Spectrum, NETLab, Phoenix, Test America-Buffalo
Continuing calibration	Absolute Resources, AMRO Environmental, Chemserve, ESS, Eurofins/Spectrum, NETLab, Phoenix, Test America-Buffalo
Method blanks	Chemserve
LCS	Phoenix
Breakthrough	No issues noted.
Target compound identification	ESS
Surrogates	No issues noted.
Retention time ranges	Absolute Resources, AMRO Environmental, Chemserve, Eurofins/Spectrum, Phoenix, Test America-Buffalo
Quantitation	Absolute Resources, AMRO Environmental, Chemserve, ESS, Eurofins/Spectrum, NETLab, Phoenix, Test America-Buffalo
Laboratory narrative	Absolute Resources, Chemserve, Eurofins/Spectrum, Test America-Buffalo
RLs	Absolute Resources, Alpha Analytical, AMRO Environmental, Chemserve, ESS, Eurofins/Spectrum, NETLab, Phoenix, RI Analytical, Test America-Buffalo
Significant modifications	No issues noted.
Certification Form	Absolute Resources, AMRO Environmental, Chemserve, ESS, Eurofins/Spectrum, NETLab, Phoenix, Test America-Buffalo

7.3 MassDEP Analytical Protocol Certification Form: EPH

The MassDEP Analytical Protocol Certification Form was present and completed as required for “Presumptive Certainty” under the MCP CAM program for all 12 laboratories. Eight of the 12 laboratories provided incorrect “YES” responses on this form. Table 7-3 summarizes these errors; questions A through F affect the achievement of Presumptive Certainty. Data for eight of the 12 laboratories may be misrepresented to the end-user since Presumptive Certainty was not achieved.

Table 7-3		
Summary of Errors on the MassDEP Analytical Protocol Certification Form: EPH		
Question	Laboratory	Issue
Question A <i>Were all samples received in a condition consistent with those described on the Chain-of-Custody, properly preserved (including temperature) in the field or laboratory, and prepared/analyzed within method holding times?</i>	Chemserve	Elevated cooler temperature and samples delivered one day after collected.
Question B <i>Were the analytical method(s) and all associated QC requirements specified in the selected CAM protocol(s) followed?</i>	Phoenix	Integration of all hydrocarbon ranges not performed properly in initial and continuing calibrations.
	Eurofins/Spectrum	
	Test America-Buffalo	
	ESS	
	Chemserve	
	Absolute Resources	
	NETLab	
	AMRO Environmental	
	AMRO Environmental	Low-level standard calculation not performed when linear regression used in calibration.
Question C <i>Were all required corrective actions and analytical response actions specified in the selected CAM protocol(s) implemented for all identified performance standard non-conformances?</i>	No issues noted.	
Question D <i>Does the laboratory report comply with all the reporting requirements specified in CAM VII A, “Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data”?</i>	No issues noted.	
Question E <i>VPH, EPH, APH, and TO-15 only</i> <i>a. VPH, EPH, and APH Methods only: Was each method conducted without significant modification(s)? (Refer to the individual method(s) for a list of significant modifications).</i> <i>b. APH and TO-15 Methods only: Was the complete analyte list reported for each method?</i>	ESS	Question A was not answered.
	Absolute Resources	
Question F <i>Were all applicable CAM protocol QC and performance standard non-conformances identified and evaluated in a laboratory narrative (including all “No” responses to Questions A through E)?</i>	No issues noted.	
Question G <i>Were the RLs at or below all CAM RLs specified in the selected CAM protocol(s)?</i>	No issues noted.	

8.0 SUMMARY OF CAM NON-COMPLIANCE ISSUES FOR MASSDEP APH

8.1 Data Deliverables: APH

Table 8-1 summarizes the data deliverable issues for the MassDEP APH analyses. Each of the seven laboratories included in the MassDEP APH audit exhibited issues with the original CAM deliverable and/or the full audit data deliverable. In general, CAM deliverables that were non-compliant with CAM requirements can result in misleading information and omissions such that the data user would not be able to properly or completely assess data quality. In general, the missing information in the full deliverables used in the audit did not hinder the assessment of the overall usability and accuracy of the data set.

Table 8-1 Data Deliverable Issues: APH		
Laboratory	CAM Deliverable	Full Deliverable Used in Audit
Alpha Analytical	No issues noted.	Chromatograms did not clearly demonstrate how C ₉ -C ₁₀ aromatic range was integrated. No information on column used.
SGS-Accutest	No issues noted.	Could not verify LCS was second-source. Leak check report not provided.
Con-test Analytical	No issues noted.	Chromatograms did not clearly demonstrate how hydrocarbon ranges were integrated. Leak check report not originally provided; provided in resubmittal.
NETLab	No issues noted.	Media certification information not provided.
Katahdin Analytical	No issues noted.	Chromatograms did not clearly demonstrate how hydrocarbon ranges were integrated; provided in resubmittal. Flow controller information not provided. Leak check report not provided.
Phoenix	LCS summary form did not summarize the hydrocarbon ranges. Laboratory narrative did not note that vacuum of one sample was 0" Hg. Results for hydrocarbon ranges not reported in method blank.	Concentrations of standards not clearly presented for hydrocarbon ranges and their components. No documentation to verify flow controller calibration performed by laboratory before and after sample collection. Leak check report not provided. Column information not provided; provided in resubmittal.
Eurofins/Spectrum	Laboratory narrative did not note quadratic regression used for 1,3-butadiene and naphthalene.	Concentrations and units of standards not clearly presented for hydrocarbon ranges and their components. Chromatograms did not clearly demonstrate how hydrocarbon ranges were integrated.

Table 8-1 Data Deliverable Issues: APH		
Laboratory	CAM Deliverable	Full Deliverable Used in Audit
		Chromatograms did not clearly show retention time markers. Leak check report not provided.

8.2 Accuracy, Precision & Method Compliance: APH

Evaluation of accuracy and precision was based on CAM performance standards including holding times and sample integrity, GC performance, instrument tunes, calibration QC, method blank results, media certification, surrogate spike recoveries, LCS results, internal standard results, RL evaluations, and sample result verification. Additionally, the raw data provided by the laboratories were evaluated for compliance with CAM and to verify all calculations were performed correctly, from calibration through the final reporting of sample results and RLs. Table 8-2 summarizes the evaluation parameters where issues were noted.

Table 8-2 Accuracy, Precision & Method Compliance Summary: APH	
Evaluation Parameter	Laboratory where Issues Noted
Holding times	No issues noted.
Sample integrity (vacuums, flow controller RPDs)	No issues noted.
GC performance	No issues noted.
GC/MS tunes	No issues noted.
Initial calibration	Con-test Analytical, Eurofins/Spectrum, NETLab, Phoenix, SGS-Accutest
Continuing calibration	Katahdin Analytical, Phoenix, SGS-Accutest
Method blanks	Phoenix
LCS	Phoenix
Surrogates	No issues noted.
Internal standards	No issues noted.
Target compound identification	No issues noted.
Quantitation	Con-test Analytical, Eurofins/Spectrum, NETLab, Phoenix, SGS-Accutest
Media certification	No issues noted.
Laboratory narrative	Eurofins/Spectrum, Phoenix
Retention time ranges	Eurofins/Spectrum
RLs	Con-test Analytical, Eurofins/Spectrum, SGS-Accutest
Significant modifications	No issues noted.
Certification Form	Eurofins/Spectrum, Phoenix, SGS-Accutest

8.3 MassDEP Analytical Protocol Certification Form: APH

The MassDEP Analytical Protocol Certification Form was present and completed as required for “Presumptive Certainty” under the MCP CAM program for all seven laboratories. Three of the seven laboratories provided incorrect “YES” responses on this form. Table 8-3 summarizes these errors. Data for one of the seven laboratories may be misrepresented to the end-user since Presumptive Certainty was not achieved.

Table 8-3 Summary of Errors on the MassDEP Analytical Protocol Certification Form: APH		
Question	Laboratory	Issue
Question A <i>Were all samples received in a condition consistent with those described on the Chain-of-Custody, properly preserved (including temperature) in the field or laboratory, and prepared/analyzed within method holding times?</i>	No issues noted.	
Question B <i>Were the analytical method(s) and all associated QC requirements specified in the selected CAM protocol(s) followed?</i>	Phoenix	n-Hexane and cyclohexane not included in calibration of C ₅ -C ₈ aliphatics.
	SGS-Accutest	Mass/charge (m/z) 134 not utilized for determination of C ₉ -C ₁₀ aromatics.
	Eurofins/Spectrum	Low-level standard calculation not performed when linear regression used in calibration.
Question C <i>Were all required corrective actions and analytical response actions specified in the selected CAM protocol(s) implemented for all identified performance standard non-conformances?</i>	No issues noted.	
Question D <i>Does the laboratory report comply with all the reporting requirements specified in CAM VII A, “Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data”?</i>	No issues noted.	
Question E <i>VPH, EPH, APH, and TO-15 only</i> <i>a. VPH, EPH, and APH Methods only: Was each method conducted without significant modification(s)? (Refer to the individual method(s) for a list of significant modifications).</i> <i>b. APH and TO-15 Methods only: Was the complete analyte list reported for each method?</i>	No issues noted.	
Question F <i>Were all applicable CAM protocol QC and performance standard non-conformances identified and evaluated in a laboratory narrative (including all “No” responses to Questions A through E)?</i>	No issues noted.	
Question G <i>Were the RLs at or below all CAM RLs specified in the selected CAM protocol(s)?</i>	No issues noted.	

9.0 OVERALL SUMMARY OF CAM NON-COMPLIANCE ISSUES FOR ALL METHODS

In general, the data audits demonstrated that the EPH and VPH methods exhibited the more significant types of method deviations, calculation errors, and reporting issues. The data audits for the TO-15 and APH methods also exhibited several method deviations and reporting issues, but these were less significant and less frequent, indicating that overall, the laboratory community has a good understanding of these methods and the ability to clearly follow the procedures and CAM requirements for the methods. Tables 9-2 through 9-5 provide the details on the issues noted for the TO-15, VPH, EPH, and APH methods and each laboratory. These tables do not include laboratory narrative issues or issues associated with a missing deliverable that were found to be insignificant to the evaluation.

The significant issues noted during the data audit for each method are summarized in Table 9-1. Significant issues were defined as those issues which could result in a change to the reported concentrations or reporting limits, the values ultimately used in the MCP decision-making process. Of particular concern is that the majority of these issues would not be discovered with a typical CAM deliverable. In addition, if the data user relied upon the [MassDEP Analytical Protocol Certification Form](#) as a determination of whether or not the data obtained Presumptive Certainty, this would be an inaccurate determination; as noted in Sections 5 through 8, in many cases, the laboratory completed the form as if data achieved a status of Presumptive Certainty (e.g., “Yes” answer to Question B) when in actuality, the data did not achieve this status.

Table 9-1 Significant Issues Noted During Data Audit		
Method	Description of Issue	# Laboratories Affected
TO-15	Reporting Limits: RLs not verified when linear or quadratic regression used by calculating concentrations in lowest concentration standard.	2
VPH	Calibration: CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range.	7
	Results Quantitation: Sample results changed when correct CFs utilized in calculations.	6
	Results Quantitation: Errors made in calculation (e.g., concentration of surrogate subtracted from range instead of area count, concentrations of target analytes <RL subtracted from ranges, concentration of C ₉ -C ₁₀ aromatics not subtracted from C ₉ -C ₁₂ aliphatics).	3
	Reporting Limits: RLs too low; RLs for hydrocarbon ranges were based on a concentration less than 100 times the lowest calibration standard.	9
	Reporting Limits: RLs not verified when linear or quadratic regression used by calculating concentrations in lowest concentration standard.	3
	Range Retention Times: Not established per method requirements.	3
	Significant Modification: Wrong column used with no or inadequate column equivalency study provided.	3
	Peak Integration: Peaks over-integrated in continuing calibration standard to ensure passing standard; valley-to-valley integration not performed for target analytes.	2
	Sample Preservation: Soil samples not preserved in methanol.	1
EPH	Calibration: CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range.	8
	Calibration: Missing information and therefore unable to verify CFs reported.	2
	Results Quantitation: Sample results changed when correct CFs utilized in calculations.	8
	Reporting Limits: RLs too low; RLs for hydrocarbon ranges were based on a concentration less than 100 times the lowest calibration standard.	5

Table 9-1
Significant Issues Noted During Data Audit

Method	Description of Issue	# Laboratories Affected
	Reporting Limits: RLs too high; RLs for hydrocarbon ranges were based on a concentration greater than 100 times the lowest calibration standard.	3
	Reporting Limits: RLs not verified when linear or quadratic regression used by calculating concentrations in lowest concentration standard.	4
	Range Retention Times: Not established per method requirements.	2
	Target Compound Identification: PAHs reported as co-eluting in samples and standards.	1
APH	Calibration: m/z 134 not utilized for measurement of C ₉ -C ₁₀ aromatics.	1
	Calibration: n-Hexane and cyclohexane not used in calibration of C ₅ -C ₈ aliphatics.	1
	Calibration: Hydrocarbon ranges calibrated in units of ppbV.	1
	Calibration: Quadratic regression utilized for C ₉ -C ₁₀ aromatics and select target analytes.	1
	Results Quantitation: m/z 134 not utilized for measurement of C ₉ -C ₁₀ aromatics.	1
	Results Quantitation: Sample results changed when linear regression utilized in calculations instead of quadratic regression.	1
	Results Quantitation: Errors in sample result calculations (e.g., area counts of target analytes subtracted from ranges instead of concentrations, discovered integration errors when compiling full audit deliverable).	2
	Reporting Limits: RLs not verified when linear or quadratic regression used by calculating concentrations in lowest concentration standard.	2
	Reporting Limits: RLs for hydrocarbon ranges too low; not established as per method requirements.	2

Table 9-2
Summary of Data Audits: TO-15
* Significant Issues; I = Indeterminate

Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
Con-test Analytical	Select VOCs	Low	LCS	CAM method deviation	The LCS and associated samples were not reanalyzed per CAM “Required Corrective Action”.		Potential low bias for select VOCs. Laboratory procedure has since been revised to perform required corrective action.
	Acetone	High	Media Certification	CAM method deviation	Acetone was detected above the RL; canisters not re-cleaned prior to sending out to field per CAM “Required Corrective Action”.		Potential high bias for acetone. This was identified as an inadvertent laboratory error and not a systematic issue.
	Ethyl acetate	NA	Sensitivity	CAM method deviation	The RL for ethyl acetate was not $\geq 10\times$ the lowest initial calibration standard per CAM “Required performance Standard”. Reported RL was 10x lower than required.		Since ethyl acetate is not a CAM-analyte, there was likely no effect on the overall usability of the data.
Alpha Analytical	No issues noted.						
SGS-Accutest	Select VOCs	NA	Sensitivity and Initial Calibration	CAM method deviation	The recoveries of compounds calibrated using quadratic regression were not evaluated in the lowest calibration standard.	*	Usability of this data set may be affected as reported RLs may be lower than actual RLs.
Test America-Buffalo	Detected VOCs	NA	Target Compound Identification	Full deliverable omission	Ion intensities were not provided for detected VOCs; could not verify ratios were $\pm 20\%$.		Usability of the data not significantly affected.
Phoenix	All	NA	Initial Calibration	Deliverable Discrepancy	The initial calibration form which summarizes standard concentrations listed 0.25 ppbV for the lowest standard but all RRFs calculated using a concentration of 0.2 ppbV.		If the form was correct, reported concentrations may be slightly biased high but likely not affected by slight deviation when relative response factor (RRF) from this standard averaged with RRFs from other calibration standards. However, the laboratory subsequently noted the form was incorrect and thus all results calculations were not affected.
	Select VOCs	NA	Continuing Calibration	Full deliverable errors	The continuing calibration summary form had many errors as rows were shifted for several analytes. Calibration standard missing 4 VOCs: 1,2-dichloroethane, 1,4-dioxane, bromomethane, cis-1,3-dichloropropene.		Usability of the data affected as accuracy of 4 missing VOCs cannot be assessed.
	Acetone, ethanol, isopropanol, methylene chloride	High	Method Blank	CAM reporting non-compliance	Method blank did not detect the affected analytes but detections in sample flagged as potential contamination due to laboratory solvent.		Usability of the data affected if end-user disregarded these results with no backup data to support blank contamination.
	All VOCs	NA	Method Blank	CAM reporting non-compliance	RLs in method blank different than in samples but same sample volume analyzed.		Usability of the data may be affected as RLs were not accurate.
	Bromoform	Low	LCS	CAM method deviation	The LCS and associated samples were not reanalyzed per CAM “Required Corrective Action”.		Potential low bias for bromoform.
	Carbon tetrachloride, hexachlorobutadiene	Low	LCS	CAM method deviation	The LCS and associated samples were not reanalyzed per CAM “Required Corrective Action”.		Potential low bias for affected analytes.
Eurofins/Spectrum	Detected VOCs	NA	Target Compound Identification	Full deliverable omission	Ion intensities were not provided for detected VOCs; could not verify ratios were $\pm 20\%$.		Usability of the data not significantly affected.
	Select VOCs	NA	Sensitivity and Initial Calibration	CAM method deviation	The recoveries of compounds calibrated using linear or quadratic regression were not evaluated in the lowest calibration standard. Calculated %Rs during review for compound quantitated using linear regression and criteria not met for 1,2-dichloropropane, 1,1,2-trichloroethane, and 1,4-dioxane.	*	Usability of this data set affected as reported RLs lower than actual RLs. Laboratory procedure has since been revised to perform this calculation and evaluate %Rs.
NETLab	Select VOCs	NA	Internal Standards	CAM method deviation	Laboratory only used two internal standards and not three as per the “Required Performance Standard”.		Usability of the data not significantly affected. Laboratory procedure has since been revised to include three internal standards.
	Select Detected VOCs	NA	Target Compound Identification	Full deliverable omission	Spectra not provided for some results.		Usability of the data not significantly affected.
	Acetone, Tetrachloroethene	Low	Quantitation	Calculation error	Could not reproduce concentrations of acetone and tetrachloroethene reported in sample 14.		Usability of data may be affected if errors in concentrations exist.
	All	NA	Flow Controller RPDs	CAM reporting non-compliance	Laboratory did not calculate RPDs of pre- and post-flow rates but did provide these flow rates. Two RPDs were >20 when calculated during data audit and the end-user was not informed.		Usability of data may be affected.

Table 9-3 Summary of Data Audits: VPH * Significant Issues; I = Indeterminate							
Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
Chemserve	All	Low	Sample preservation	CAM method deviation	Cooler temperature 8°C; hand-delivered two days after collection.		Potential low bias for affected analytes.
	C ₅ -C ₈ Aliphatics, C ₉ -C ₁₂ Aliphatics	Low	Initial and continuing calibrations	CAM method deviation	CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range.	*	Potential low bias as resulting CF is higher than if generated as per the method. Laboratory procedure has since been revised to perform this calculation per the method.
	C ₉ -C ₁₂ Aliphatics	I	Initial and continuing calibrations	CAM method deviation	Decane and n-butylcyclohexane not utilized in calibration of this hydrocarbon range; only nonane used.	*	Resulting CF not representative of alkanes required by method and only based on one straight-chain alkane. Laboratory procedure has since been revised to perform the calibration with the method-required compounds.
	All hydrocarbon ranges	Low	Continuing calibration	CAM method deviation	Opening continuing calibration standard analyzed greater than 24 hours prior to samples.		Usability of the data not significantly affected.
	All	I	Target Compound Identification	Full deliverable omission	Chromatograms provided did not clearly show if target analytes were integrated valley-to-valley and if ranges were integrated from baseline.		Effect on usability of data cannot be assessed.
	C ₅ -C ₈ Aliphatics	Low	Results Quantitation	CAM method deviation	Results re-quantified using CF from summation of individual peaks.	*	Sample result recalculated using correct CF. C ₅ -C ₈ aliphatics: laboratory-reported result biased low: 1096 ug/L (not 894 ug/L) Laboratory procedure has since been revised to perform calibration and thus subsequent calculations per the method.
	C ₉ -C ₁₂ Aliphatics	Low	Results Quantitation	CAM method deviation	Results re-quantified using CF from individual peak.	*	Sample result recalculated using correct CF. C ₉ -C ₁₂ aliphatics: laboratory-reported result biased low: 323 ug/L (not <25 ug/L) Laboratory procedure has since been revised to perform calibration and thus subsequent calculations per the method.
	All hydrocarbon ranges	I	Retention Times	Full deliverable omission	Chromatograms provided did not clearly show retention times used for each range; could not verify if method requirements followed.		Effect on usability of data cannot be assessed. Response by the laboratory indicated retention time ranges were not determined properly and potential low and high biases may exist for the hydrocarbon ranges.
	All hydrocarbon ranges	I	Sensitivity/RLs	CAM method deviation	RLs were 4-6x higher than the lowest individual analyte concentrations in the range, not 100x the lowest concentration standard, as per the method.	*	RLs misrepresented as lower than allowed by method. Laboratory procedure has since been revised to use the 100x rule for hydrocarbon range RLs.
Phoenix	All hydrocarbon ranges	Low/High	Initial Calibration	CAM method deviation	The method used for calibration of ranges could not be reproduced and was not understood by the reviewer. The laboratory provided a “Peak Sum Table” at the end of the “Calibration table” included within the raw data with start and ending retention times for additional hydrocarbon ranges labeled as “C₉-C₁₀ AroPksU”, “C₉-C₁₂ AliPksU”, and C₅-C₈ AliPksU”. The laboratory provided a “response factor” for the three ranges but no %RSDs. The reviewer could not verify or recalculate how these response factors were calculated. The reviewer could also not verify what was integrated by the laboratory to obtain the “total areas” listed on the initial calibration raw data for these PksU hydrocarbon range areas. The reviewer verified that these “PksU” response factors were used to calculate the “PksU” amounts listed on the raw data. The laboratory multiplied the CFs by the “PksU” total areas. CFs were also calculated by laboratory using the inverse of the formula in the method (conc/area).		Effect on usability data noted during assessment of continuing calibration: C ₅ -C ₈ aliphatics and C ₉ -C ₁₀ aromatics biased high and C ₉ -C ₁₂ aliphatics biased low. Laboratory provided a response with a revised calibration procedure which exhibited new results for the affected field samples.
	All hydrocarbon ranges	Low and High	Continuing Calibration	CAM method deviation	When continuing calibration standard calculated using correct CFs (re-calculated during audit), all recoveries were acceptable. Using the laboratory’s CFs, all recoveries were also acceptable but different.	*	Effect on usability data noted during recalculation of continuing calibration using method-required procedure for CFs: C ₅ -C ₈ aliphatics and C ₉ -C ₁₀ aromatics reported by laboratory biased high by ~30% and C ₉ -C ₁₂ aliphatics reported by laboratory biased low by ~20%. Laboratory provided a response with a revised calibration procedure which exhibited new results for the affected field samples.

Table 9-3 Summary of Data Audits: VPH * Significant Issues; I = Indeterminate							
Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
	C ₉ -C ₁₀ Aromatics, C ₉ -C ₁₂ Aliphatics	Low and High	LCS	CAM method deviation and non-compliance	The recoveries of C ₉ -C ₁₀ aromatics and C ₉ -C ₁₂ aliphatics were reported by laboratory as 152% and 60%, respectively. No corrective actions were taken by the laboratory. When the LCS was recalculated using the correct CFs (re-calculated during audit), the recoveries were 102% and 81%, respectively.		Usability of the data not affected but this would not be known by end-user of data due to calibration errors.
	C ₉ -C ₁₀ Aromatics, VPH Target Analytes	High	Surrogate	CAM method deviation and non-compliance	The laboratory reported the surrogate recovery on the PID as high and outside the acceptance criteria with no corrective action taken. When the surrogate recovery was recalculated using the correct CFs (re-calculated during the audit), the recovery was acceptable.		Usability of the data not affected but this would not be known by end-user of data due to calibration errors.
	All hydrocarbon ranges	Low and High	Quantitation	CAM method deviation	The laboratory reported concentrations based on the summed individual peak areas for the three hydrocarbon ranges (the range areas were verified to be calculated by the laboratory using the individual peaks, not a range integration from baseline) in the sample. The laboratory also reported additional “PksU” total areas and concentrations for what appeared to be integrated using retention time windows for each hydrocarbon range. The laboratory added the total “PksU” concentrations and the summed peak area concentrations of the hydrocarbon ranges to get the “unadjusted” range values. The reviewer could not verify how the “PksU” areas were determined by the laboratory since clear chromatograms with this integration were not provided by the laboratory. The hydrocarbon range concentrations were re-calculated using the correct CFs (re-calculated during the audit).	*	Sample recalculated using correct CFs: C ₉ -C ₁₀ aromatics: Laboratory reported result biased high: 246 ug/L (not 270 ug/L) C ₅ -C ₈ aliphatics unadjusted: Laboratory reported result biased low: 100 ug/L (not 75 ug/L); both non-detect when adjusted C ₉ -C ₁₂ aliphatics unadjusted: Laboratory reported result biased low: 765 ug/L (not 501 ug/L) (adjusted 520 ug/L, not 230 ug/L) Laboratory provided a response with a revised calibration procedure which exhibited new results for the affected field samples.
	All hydrocarbon ranges	I	Sensitivity/RLs	CAM method deviation	RLs were 3.33-5x higher than the lowest individual analyte concentrations in the range, not 100x the lowest concentration standard, as per the method. RLs should be 500 ug/L for the aliphatic ranges (not 100 ug/L) and 100 ug/L for C ₉ -C ₁₀ aromatics (not 33 ug/L).	*	RLs misrepresented as lower than allowed by method. Laboratory procedure has since been revised to report RLs in accordance with the method requirements.
	All hydrocarbon ranges and target analytes	I	Column	Significant modification	The information on the column was not provided. However, some forms in the data package listed RTX-VMS column and some listed ZB MRI 0.53 mm inner diameter (ID), neither of which is the required column for the VPH method.	*	Effect on the data usability cannot be assessed. The laboratory subsequently provided a column comparability study but the results of this study were not properly evaluated and in fact, demonstrated that the column the laboratory used is likely not comparable to the method-required column. The laboratory has since switched over to the method-required column.
	Methyl tert butyl ether (MTBE)	Low	Purge & Trap Conditions	CAM method deviation	According to the method blank summary form, a heated purge was used which is a significant modification.		Heated purge with acid preservation can cause significant low bias to MTBE results. The laboratory provided a response indicating that the summary form was incorrect and a heated purge was not used.
AMRO Environmental	All hydrocarbon ranges	I	Initial Calibration	CAM method deviation	Quadratic regression used for C ₉ -C ₁₂ aliphatics but not needed: during review recalculated using linear regression. C ₅ -C ₈ aliphatics: recalculated average CF during review; all CFs could be reproduced except 75 ppb standard: used correct CF in all subsequent calculations during audit. C ₉ -C ₁₀ aromatics: laboratory used 1,2,4-trimethylbenzene only but areas listed for C ₉ -C ₁₀ aromatics did not match exact area for 1,2,4-trimethylbenzene: recalculated average CF during review and used that in all subsequent calculations during audit.		Recalculated concentrations showed <5% D for highest concentration sample (-3). At lower concentrations, higher %Ds were seen in recalculated concentrations compared to laboratory reported concentrations for C ₉ -C ₁₂ aliphatics but all were below the RL.

Table 9-3 Summary of Data Audits: VPH * Significant Issues; I = Indeterminate							
Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
	All hydrocarbon ranges	I	Sensitivity/RLs	CAM method deviation	Laboratory did not recalculate concentration of C ₉ -C ₁₂ aliphatics at lowest concentration per CAM as required for quadratic regression: recalculated during review using linear regression and lowest point (15 ppb) was 360%: lowest standard with acceptable recovery was 75 ppb (113%)	*	RLs misrepresented as lower than allowed by method.
	All hydrocarbon ranges	Low and High	Retention Time Windows	CAM method deviation	C ₅ -C ₈ Aliphatics: start time is 0.25 min before pentane (instead of 0.1 min) and end time is 0.26 minutes before nonane (instead of 0.1 min): biased high at front end and biased low at back end C ₉ -C ₁₂ Aliphatics: start time is 0.25 min before nonane (instead of 0.1 min) and end time is 0.18 min before naphthalene (instead of 0.1 min): biased high at front end and biased low at back end C ₉ -C ₁₀ Aromatics: start time is 0.28 min after o-xylene instead of 0.1 min and end time is 0.18 min before naphthalene instead of 0.1 min: biased low at front end and biased low at back end	*	C ₅ -C ₈ and C ₉ -C ₁₂ Aliphatics: effect on usability of data will be dependent on petroleum product present in sample. C ₉ -C ₁₀ aromatics: effect on usability of data is a low bias.
	C ₅ -C ₈ Aliphatics, C ₉ -C ₁₂ Aliphatics	I	Sensitivity/RLs	CAM method deviation	RLs were 20-50x higher than the lowest individual analyte concentrations in the range, not 100x the lowest concentration standard, as per the method. RLs should be 200 ug/L for the C ₅ -C ₈ aliphatics (not 100 ug/L) and 500 ug/L for C ₉ -C ₁₂ aliphatics (not 100 ug/L).	*	RLs misrepresented as lower than allowed by method.
Alpha Analytical	All hydrocarbon ranges	I	Sensitivity/RLs	CAM method deviation	RLs were 50x higher than the lowest individual analyte concentrations in the range, not 100x the lowest concentration standard, as per the method.	*	RLs misrepresented as lower than allowed by method. Laboratory procedure has since been revised to calculate RLs in accordance with the method requirements.
SGS-Accutest	C ₉ -C ₁₂ Aliphatics	I	Quantitation	CAM method deviation	Laboratory subtracted the concentration of surrogate from the range instead of the area count. Recalculated during audit and sample result was 5% lower when area counts subtracted.	*	Affects accuracy of sample results; may affect other samples differently depending on concentration.
Eurofins/Spectrum	C ₅ -C ₈ Aliphatics, C ₉ -C ₁₂ Aliphatics	Low	Initial Calibration	CAM method deviation	CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range. Laboratory used total concentrations of aromatics when calculating the CFs since timed integrations used.	*	Potential low bias as resulting CF is higher than if generated as per the method. Laboratory procedure has since been revised to perform this calculation per the method.
	C ₅ -C ₈ Aliphatics, C ₉ -C ₁₂ Aliphatics	Low	Continuing Calibration	CAM method deviation	Several alkane peaks in both aliphatic ranges were over-integrated to ensure the calibration standard was within the acceptance criteria; calibration standard would not have passed if integrated properly.	*	Misrepresentation of calibration QC.
	C ₅ -C ₈ Aliphatics, C ₉ -C ₁₂ Aliphatics	High	LCS	CAM method deviation	Aliphatic ranges not quantified per the method; used baseline integration instead of summation of individual peaks. %Rs were within acceptance criteria with both methods of integration but correct method yielded lower %Rs (C ₅ -C ₈ aliphatics: 79% vs 95% and C ₉ -C ₁₂ aliphatics: 76% vs 111%).		Usability of the data not significantly affected.
	C ₅ -C ₈ Aliphatics, C ₉ -C ₁₂ Aliphatics	Low	Results Quantitation	CAM method deviation	Results re-quantified using CFs from summation of individual peaks. C ₅ -C ₈ aliphatics and C ₉ -C ₁₂ aliphatics results reported by laboratory lower than what was recalculated during review.	*	Sample recalculated using correct CFs: C ₅ -C ₈ aliphatics unadjusted: Laboratory reported result biased low: 1200 ug/L (not 890 ug/L): (adjusted 930 ug/L, not 626 ug/L) C ₉ -C ₁₂ aliphatics unadjusted: Laboratory reported result biased low: 2390 ug/L (not 1540 ug/L) (adjusted 1596 ug/L, not 794 ug/L)

Table 9-3 Summary of Data Audits: VPH * Significant Issues; I = Indeterminate							
Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
	All	Low	Sensitivity/RLs	CAM method deviation	Laboratory did not adjust RLs for dilution factor (5x).	*	RLs misrepresented as lower than allowed by method. Laboratory response indicated this occurred since all samples were routinely analyzed at 5-fold dilutions.
	All hydrocarbon ranges	High	Retention Time Windows	CAM method deviation	C ₅ -C ₈ Aliphatics: start time is 0.31 minutes before pentane (instead of 0.1 min): biased high. C ₉ -C ₁₂ Aliphatics: end time is 0.04 min after naphthalene instead of 0.1 min before naphthalene: biased high. C ₉ -C ₁₀ Aromatics: end time is 0.1 min after naphthalene instead of 0.1 min before naphthalene: biased high.	*	All hydrocarbon ranges: effect on usability of data is a high bias, depending on the petroleum product present.
	All hydrocarbon ranges	I	Sensitivity/RLs	CAM method deviation	RLs were 2.5-15x higher than the lowest individual analyte concentrations in the range, not 100x the lowest concentration standard, as per the method.	*	RLs misrepresented as lower than allowed by method.
	All	I	Analytical Column	Significant modification	Laboratory used column not listed in method with no demonstration of column equivalency, as per method. Column used: DB-MTBE, 30m x 0.45 mm ID, 2.55 um film thickness.	*	Effect on the usability of data cannot be assessed without equivalency study. The laboratory subsequently provided a column comparability study but the results of this study were not properly evaluated and could not be used to demonstrate comparability to the method-required column.
Con-test Analytical	C ₉ -C ₁₂ Aliphatics	I	Sensitivity/RLs	CAM method deviation	Laboratory did not recalculate concentration of C ₉ -C ₁₂ aliphatics at lowest concentration per CAM as required for linear regression. Calculated during audit and %R was acceptable.		Usability of the data was not adversely affected; RLs could be misrepresented as lower than allowed by method in other reports, if not performed.
	All hydrocarbon ranges	I	Sensitivity/RLs	CAM method deviation	RLs were 20x higher than the lowest individual analyte concentrations in the aliphatic ranges, not 100x the lowest concentration standard, as per the method.	*	RLs misrepresented as lower than allowed by method.
RI Analytical	No issues noted.						
ESS	All hydrocarbon ranges	Low	Initial Calibration	CAM method deviation	CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range.	*	Potential low bias as resulting CF is higher than if generated as per the method. Laboratory procedure has since been revised to perform this calculation per the method.
	All hydrocarbon ranges	Low	Initial Calibration	CAM method recommendation deviation	Concentrations used for ranges were from 50-400 ug/L instead of 1 to 200 ug/L for each component of range, as recommended in method.		RLs higher than recommended in method because concentration of lowest standard in initial calibration 50x higher than method recommended low calibration standard. Laboratory procedure has since been revised to include low concentrations for the hydrocarbon range calibrations.
	All hydrocarbon ranges	Low/High	Results Quantitation	CAM method deviation	Results re-quantified using CFs from summation of individual peaks. Laboratory did not subtract aaa-trifluorotoluene from C ₅ -C ₈ aliphatics.	*	Sample recalculated using correct CFs: C ₅ -C ₈ aliphatics: Laboratory result biased low by ~10%: 280 mg/kg vs 250 mg/kg C ₉ -C ₁₂ aliphatics: Laboratory result biased high by ~3%: 1650 mg/kg vs 1700 mg/kg C ₉ -C ₁₀ aromatics: Laboratory result biased low by ~50%: 1100 mg/kg vs 600 mg/kg Laboratory procedure has since been revised to perform this calculation per the method.
	All hydrocarbon ranges	I	Sensitivity/RLs	CAM method deviation	RLs were 2x higher than the lowest individual analyte concentrations in the range, not 100x the lowest concentration standard, as per the method.	*	RLs misrepresented as lower than allowed by method. Laboratory procedure has since been revised to calculate RLs in accordance with the method requirements.

Table 9-3 Summary of Data Audits: VPH * Significant Issues; I = Indeterminate							
Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
Test America-Buffalo	All	I	Holding Times & Sample Preservation	CAM reporting non-compliance	According to the laboratory narrative, the samples were frozen by the laboratory upon receipt at the laboratory on 5/7/2012 @ 05:15 3.5 hours (approximately 86- 92 hours after collection). However, it is not clear if EPH or VPH samples were frozen or both. CAM does not state that freezing is an option for VPH. There is no documentation provided to indicate if the samples were received in air-tight collection samples or in vials with methanol. In addition, there was no documentation on the methanol-to-soil ratio for the samples after sample collection upon receipt at the laboratory. The Form Is for the target analytes and C ₉ -C ₁₀ aromatic range data indicate a sample weight and methanol volume, but it is not clear from the documentation in the data package if the samples were preserved in methanol upon sample collection. There is no sample weight and methanol volume information on the Form Is for the two aliphatic ranges data.	*	Effect on the usability cannot be assessed due to missing information.
	All hydrocarbon ranges	Low	Initial Calibration	CAM method deviation	CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range. Could not recalculate CFs for aliphatic ranges during review since individual peak areas were not provided.	*	Potential low bias as resulting CF is higher than if generated as per the method. Laboratory procedure has since been revised to perform this calculation per the method.
	C ₉ -C ₁₂ Aliphatics and C ₉ -C ₁₀ Aromatics	I	Target Compound Identification	CAM method deviation	Butylcyclohexane and 1,2,4-trimethylbenzene reported as co-elution in initial calibration. This was clearly an error as chromatogram clearly showed two separate peaks.	*	Likely affects subsequent calculations of these hydrocarbon ranges. Laboratory procedure has since been revised to correct this co-elution issue.
	All	I	LCS	CAM method deviation	LCS was not prepared from a secondary source.		Usability of the data not significantly affected. Laboratory procedure has since been revised to use a secondary source for the LCS.
	All	I/High	Target Compound Identification	Full deliverable omission	Unable to verify target analytes integrated valley to valley unless manual integration provided. For one sample (B-39/14-16): naphthalene and surrogate included integration to baseline and not valley-to valley. Naphthalene concentration in this sample may be biased high.	*	Effect on the usability of the data will be sample-specific. Potential high bias if integrations not performed properly.
	All hydrocarbon ranges	Low	Results Quantitation	CAM method deviation	Results of target analytes which were < RL were subtracted from ranges.	*	Potential low bias due to subtraction of concentrations not allowed by method. The laboratory response indicated this procedure is utilized when the data user requests target analyte results < RL be reported.
	C ₉ -C ₁₀ Aromatics	Low	Results Quantitation	CAM method deviation	Results re-quantified using CFs from summation of individual peak for C ₉ -C ₁₀ aromatics. Laboratory also did not correct methanol extract volume for % moisture.	*	Sample recalculated using correct CFs: C ₉ -C ₁₀ aromatics: Laboratory result biased low by ~3.5-fold: 500 mg/kg vs 140 mg/kg. Laboratory procedure has since been revised to perform this calculation per the method.
	C ₅ -C ₈ Aliphatics, C ₉ -C ₁₀ Aromatics	Low	Retention Time Windows	CAM method deviation	C ₅ -C ₈ Aliphatics: start time is same time as pentane (instead of 0.1 min before): biased low; end time is 0.5 minutes before nonane (instead of 0.1 min): biased low. C ₉ -C ₁₀ Aromatics: start time is 0.5 min after o-xylene instead of 0.1 min after o-xylene: biased low.	*	Effect on usability of data is a low bias, depending on the petroleum product present. Laboratory procedure has since been revised to perform this calculation per the method.
	All hydrocarbon ranges	I	Sensitivity/RLs	CAM method deviation	RLs were 5x higher than the lowest individual analyte concentrations in the range, not 100x the lowest concentration standard, as per the method. RLs were 13-14 mg/kg but should have been 300 mg/kg. RLs not corrected for moisture correction in methanol volume.	*	RLs misrepresented as lower than allowed by method.

Table 9-3 Summary of Data Audits: VPH * Significant Issues; I = Indeterminate							
Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
	All	I	Analytical Column	Significant modification	Laboratory used column not listed in method with no demonstration of column equivalency, as per method. Column used: RTX-VMS, 60 m, 0.53 mm ID, 5 um film thickness	*	Effect on the usability of data cannot be assessed without equivalency study. The laboratory has since switched over to the method-required column.
NETLab	C9-C10 Aromatics	NA	Initial Calibration	CAM method deviation	CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range. The individual areas were > than the range areas which should not be the case but overall the difference had insignificant impact on field sample results.	*	Potential low bias as resulting CF is higher than if generated as per the method. Laboratory procedure has since been revised to perform this calculation per the method.
	Ethylbenzene, o-xylene	I	Sensitivity/RLs	CAM method deviation	Laboratory did not recalculate concentration of ethylbenzene and o-xylene at lowest concentration per CAM as required for linear regression: recalculated during review using linear regression. o-Xylene: RL based on 10 ug/L but could have been based on 5 ug/L based on linear regression recalculation of concentration at 5 ug/L. Ethylbenzene: RL based on 5 ug/L but should have been based on 20 ug/L since linear regression recalculation of low standard for 1 and 5 ug/L did not meet 70-130% limit.	*	RLs misrepresented as lower than allowed by method. Laboratory procedure has since been revised to perform this calculation and evaluation.
	Benzene	I	Initial Calibration Results Quantitation	CAM method deviation	Laboratory used quadratic regression for benzene although %RSD of CFs was within the acceptance criteria. Sample results quantified using CF still yielded detections below the RL.		For this data set, no effect on the usability of the data. May not be the case in other data sets, depending on the concentrations of benzene. Laboratory procedure has since been revised and quadratic regressions are no longer utilized.
	All	I	LCS	CAM method deviation	LCS was not matrix-specific as it was reported in aqueous units and associated samples were soil.		Usability of the data not significantly affected. The laboratory response indicated this was a laboratory information management system (LIMS) reporting issue.
	All hydrocarbon ranges	I	Sensitivity/RLs	CAM method deviation	RLs were 50x higher than the lowest individual analyte concentrations in the range, not 100x the lowest concentration standard, as per the method. RLs not corrected for moisture correction in methanol volume.	*	RLs misrepresented as lower than allowed by method. Laboratory procedure has since been revised to calculate RLs in accordance with the method requirements.
	All	I/Low	Sample Preservation	CAM method deviation	All samples were reported as exactly 10 grams which is suspect. This is not possible with the use of field preservation or EnCore samplers so it is unclear how these samples were collected.	*	Suspect results and potential low bias due to the potential that samples were not preserved.
Absolute Resources	All hydrocarbon ranges	Low	Initial Calibration	CAM method deviation	CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range.	*	Potential low bias as resulting CF is higher than if generated as per the method. Laboratory procedure has since been revised to perform this calculation per the method.
	All	I	Initial Calibration	CAM method deviation	Laboratory use linear regression for all hydrocarbon ranges and target analytes although all %RSDs were within the acceptance criteria.		Usability of the data not significantly affected.
	C9-C12 Aliphatics	High	Continuing Calibration	CAM method deviation	Results re-quantified using CF from summation of individual peaks for C9-C12 aliphatics. When re-quantified, C9-C12 aliphatics does not meet the criteria and is biased high.	*	Results for C9-C12 aliphatics biased high; end-user not aware of calibration QC issue.

Table 9-3 Summary of Data Audits: VPH * Significant Issues; I = Indeterminate							
Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
	C9-C10 Aromatics	High	LCS	CAM method deviation	Results re-quantified using CF from summation of individual peak for C9-C10 aromatics. When re-quantified, C9-C10 aromatics does not meet the criteria and is biased high (187%); also biased high when linear regression used.		Results for C9-C10 aromatics biased high.
	C9-C12 Aliphatics	High	Results Quantitation	CAM method deviation	The concentration of C9-C10 aromatics was not subtracted from the concentration of C9-C12 aliphatics.	*	Results for C9-C12 aliphatics biased high. Laboratory procedure has since been revised to perform this calculation per the method.
	All hydrocarbon ranges, naphthalene, m&p-xylenes	Low	Results Quantitation	CAM method deviation	Results re-quantified using CFs from summation of individual peaks.	*	Sample recalculated using correct CFs: C5-C8 Aliphatics: No effect C9-C12 Aliphatics: Laboratory result biased low by ~24%: 147 mg/kg vs 105 mg/kg C9-C10 Aromatics: Laboratory result biased low by ~3%: 2280 mg/kg vs 2230 mg/kg Naphthalene: Laboratory result biased low by ~45%: 3.2 vs 2.2 mg/kg m&p-Xylenes: Laboratory result biased low by ~100%: 0.2 vs 0.1 mg/kg

Table 9-4 Summary of Data Audits: EPH * Significant Issues; I = Indeterminate							
Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
Phoenix	All	Low	Holding Times and Sample Preservation	CAM method deviation	The samples were collected on 9/22/14, but were not received at the laboratory until 11/21/14. The laboratory acknowledged that the samples were received past holding time, but it is not clear in the data package if the samples were frozen upon collection and prior to receipt at the laboratory; thus, the holding time requirements as described in Appendix IV B-1 of WSC-CAM-IVB may not have been met. However, the exceedance of the holding times was not a laboratory issue since the laboratory analyzed the samples within 4 days of receipt.		Potential low bias.
	C ₁₉ -C ₃₆ Aliphatics	High	GC Performance	CAM method deviation and non-compliance	The response ratio of C ₂₈ to C ₂₀ information was not summarized by the laboratory in the data package, but was calculated during the audit based on the 40 mg/L calibration standard. The ratio was 0.79 which was < the EPH Method requirement of >0.85.		Concentrations of heavier aliphatic analytes (> C ₂₀) which are close to the RL may be biased high due to the absence of mass discrimination. Laboratory procedure has since been revised to perform this evaluation per the method.
	All hydrocarbon ranges	Low	Initial Calibration	CAM method deviation	CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range.	*	Potential low bias as resulting CF is higher than if generated as per the method. Laboratory procedure has since been revised to perform this calculation per the method.
	C ₉ -C ₁₈ Aliphatics, C ₁₉ -C ₃₆ Aliphatics	I	LCS	Calculation error	A different CF than that generated in the initial calibration was used to calculate results. Correct CF used during audit.		Usability of the data not significantly affected.
	C ₉ -C ₁₈ Aliphatics, C ₁₉ -C ₃₆ Aliphatics	I	Results Quantitation	CAM method deviation	Results re-quantified using CFs from summation of individual peaks.	*	Sample recalculated using correct CFs: C ₉ -C ₁₈ Aliphatics: Laboratory result biased low by ~7%: 15.7 mg/kg vs 14.6 mg/kg C ₁₉ -C ₃₆ Aliphatics: Laboratory result biased low by ~8.5%: 494 mg/kg vs 452 mg/kg C ₁₁ -C ₂₂ Aromatics: Laboratory result biased low by ~3%: 88 mg/kg vs 85 mg/kg Laboratory procedure has since been revised to perform this calculation per the method.
	All hydrocarbon ranges	Low and High	Retention Time Windows	CAM method deviation	C ₉ -C ₁₈ Aliphatics: window was 2.9-12.9 (should be 3.15-13.25). C ₁₉ -C ₃₆ Aliphatics: window was 12.9-23.5 (should be 13.25-23.38). C ₁₁ -C ₂₂ Aromatics: window was 5.96-20 (should be 6.12-19.77).	*	C ₉ -C ₁₈ Aliphatics: effect on usability of data will be dependent on petroleum product present in sample. C ₁₉ -C ₃₆ Aliphatics and C ₁₁ -C ₂₂ Aromatics: effect on usability of data is a high bias; also dependent on petroleum product present in sample.
	All ranges	I	Sensitivity/RLs	CAM method deviation	RLs were 5x the lowest standard, not 100x lowest standard as required in method. They were 13-14 mg/kg but should have been 300 mg/kg. RLs not corrected for moisture correction in methanol volume.	*	RLs misrepresented as lower than allowed by method. Laboratory procedure has since been revised to calculate RLs in accordance with the method requirements.
Eurofins/Spectrum	All hydrocarbon ranges	Low	Initial Calibration	CAM method deviation	CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range. Laboratory used linear regression for C ₉ -C ₁₈ aliphatics although the %RSD <25 and average CF could therefore be used. Laboratory used linear regression for C ₁₉ -C ₃₆ aliphatics but if CF calculated using summation of individual peaks, %RSD would be <25 and average CF could be used. Could not re-calculate CF for C ₁₁ -C ₂₂ aromatics because TICs for individual PAHs not provided.	*	Potential low bias as resulting CF is higher than if generated as per the method.
	Benzo(a)pyrene, indeno(1,2,3-cd)pyrene	I	Initial Calibration	CAM method deviation	Benzo(a)pyrene and indeno(1,2,3-cd)pyrene quantitated using quadratic regression but average CFs could have been used since %RSD <25.		For this data set, no effect on the usability of the data. May not be the case in other data sets, depending on the concentrations of these PAHs.

Table 9-4
Summary of Data Audits: EPH
* Significant Issues; I = Indeterminate

Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
	All hydrocarbon ranges	I	Results Quantitation	Calculation error	Results re-quantified using CFs from summation of individual peaks.	*	Samples recalculated using correct CFs: C ₉ -C ₁₈ Aliphatics: not significantly affected C ₁₉ -C ₃₆ Aliphatics: not significantly affected except at lower concentrations: Sample 3 reported result biased low by ~73% (70.6 mg/kg vs 19.2 mg/kg). C ₁₁ -C ₂₂ Aromatics: not significantly affected.
	All	I	Sensitivity/RLs	CAM method deviation	RLs for ranges were 33x the lowest standard, not 100x lowest standard as required in method. They were 10 mg/kg but should have been 33 mg/kg. RLs for target analytes 5x higher than lowest standard (therefore 5x higher than necessary).	*	RLs misrepresented as lower than allowed by method for ranges and higher than necessary for target analytes. Laboratory procedure has since been revised to calculate RLs in accordance with the method requirements.
AMRO Environmental	All hydrocarbon ranges	Low	Initial Calibration	CAM method deviation	CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range. CFs could not be re-calculated since individual areas provided were not based on TIC integration.	*	Potential low bias as resulting CF is higher than if generated as per the method.
	All hydrocarbon ranges	I	Sensitivity/RLs	CAM method deviation	The laboratory used linear regression for all ranges since the %RSDs were >25; the laboratory did not provide evidence that the RL was recalculated per CAM; the RLs were recalculated during review using the lowest standard, C ₉ -C ₁₈ aliphatic was acceptable, but the C ₁₉ -C ₃₆ Aliphatic had a high recovery (131%), the lowest standard with acceptable recovery was the next lowest standard (20 ug/mL). C ₁₁ -C ₂₂ Aromatics: the %Rs for the 2 lowest standards were 17 and 29%. The %R at 170 ug/mL was 105%. Linear regression used for benzo(a)anthracene and benzo(b)fluoranthene because %RSDs >25; RL recalculation not performed by laboratory: checked during review and acceptable %Rs.	*	RLs misrepresented as lower than allowed by method.
	Benzo(a)pyrene, dibenz(a,h)anthracene, indeno(1,2,3-cd)pyrene	I	Initial Calibration	CAM method deviation	Linear regression used for benzo(a)pyrene, dibenz(a,h)anthracene, and indeno(1,2,3-cd)pyrene even though %RSDs <25.		Usability of the data not significantly affected.
	C ₁₉ -C ₃₆ Aliphatics	High	Results Quantitation	Calculation error	Could not reproduce result for C ₁₉ -C ₃₆ aliphatics.	*	Laboratory result biased high by ~20% for sample -1 (2700 vs 3300 mg/kg).
	All hydrocarbon ranges	I	Retention Time Windows	CAM method deviation	Based on timed integration documentation in data package, it appears EPH range retention times not determined according to method.		Effect on the usability of the data cannot be assessed.
	All hydrocarbon ranges	Low	Initial Calibration	CAM method deviation	CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range.	*	Potential low bias as resulting CF is higher than if generated as per the method. Laboratory procedure has since been revised to perform this calculation per the method.
Test America-Buffalo	All hydrocarbon ranges	Low	Results Quantitation	CAM method deviation	Results re-quantified using CFs from summation of individual peaks.	*	Sample results approximately 3% higher with correct CFs. Laboratory procedure has since been revised to perform this calculation per the method.
	All hydrocarbon ranges	I	Results Quantitation	CAM method deviation	Laboratory reported detected results below the RL for the ranges.		Potential false positive results reported; likely no significant effect on the overall usability of the data.

Table 9-4 Summary of Data Audits: EPH * Significant Issues; I = Indeterminate							
Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
	All hydrocarbon ranges	Low	Retention Time Windows	CAM method deviation	C ₉ -C ₁₈ Aliphatics: window was 3.16-9.622 (should be 3.091-10.007); biased low. C ₁₉ -C ₃₆ Aliphatics: window was 10.082-17.786 (should be 10.007-17.817); biased low. C ₁₁ -C ₂₂ Aromatics: window was 5.758-15.690 (should be 5.671-15.754); biased low.	*	All hydrocarbon ranges: effect on usability of data is a low bias; also dependent on petroleum product present in sample. Laboratory procedure has since been revised to perform this calculation per the method.
	All	NA	Laboratory Narrative	Reporting error	The laboratory narrative indicated that the RLs were not at or below CAM RLs for all samples due to dilutions. However, there were no dilutions performed and the RLs did meet CAM requirements.		Usability of the data not affected but misleading to end user of the data.
	All	I	Sensitivity/RLs	CAM method deviation	RLs for ranges were 10x the lowest standard, not 100x lowest standard as required in method. Some detected values for ranges would be “J” values if correct RLs reported. RLs for target analytes below the lowest standard (0.2 mg/kg but should be 1 mg/kg based on 5 ug/mL standard).	*	RLs misrepresented as lower than allowed by method. Detected results below the true RL should have been flagged as estimated to the end user of the data.
ESS	C ₉ -C ₁₈ Aliphatics, C ₁₉ -C ₃₆ Aliphatics	Low	Initial Calibration	CAM method deviation	CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range. Could not re-calculate CF for C ₁₁ -C ₂₂ aromatics because TICs for individual PAHs not provided. Note that aliphatic range CFs recalculated using summation of individual standards; %RSDs acceptable. Laboratory method resulted in linear regression due to high %RSDs.	*	Potential low bias as resulting CF is higher than if generated as per the method. Laboratory procedure has since been revised to perform this calculation per the method.
	Phenanthrene, anthracene, benzo(b)fluoranthene, benzo(k)fluoranthene	I	Target Compound Identification	Identification error	Samples showed phenanthrene and anthracene identified at the same retention time; these two compounds do not co-elute and were therefore mis-identified. Benzo(b)fluoranthene and benzo(k)fluoranthene identified at the same retention time in two of the initial calibration standards.	*	CFs for benzo(b)fluoranthene, benzo(k)fluoranthene, and C ₁₁ -C ₂₂ aromatics may not be accurate but overall effect on usability of the data is likely minimal. Reporting of phenanthrene and/or anthracene in the samples may be false positives. Laboratory has implemented further review procedures to prevent this from occurring.
	C ₉ -C ₁₈ Aliphatics, C ₁₉ -C ₃₆ Aliphatics	Low	Results Quantitation	CAM method deviation	Results re-quantified using CFs from summation of individual peaks.	*	Sample results approximately 1-2% higher with correct CFs. Laboratory procedure has since been revised to perform this calculation per the method.
	All	I	Sensitivity/RLs	CAM method deviation	RLs for ranges were 500x the lowest standard, not 100x lowest standard as required in method. RLs for target analytes 5x higher than lowest standard (therefore both target analytes & ranges 5x higher than necessary).	*	RLs misrepresented as higher than recommended by method; potential false negative results. Laboratory procedure has since been revised to calculate RLs in accordance with the method requirements.
Chemserve	All hydrocarbon ranges	Low	Initial Calibration	CAM method deviation	There was no initial calibration information provided in the data package for the aliphatic hydrocarbon range and individual alkanes; thus, the auditor could not determine if the laboratory used a RT range or sum of individual components. The auditor was unable to reproduce the IC calculations for the aromatic range. However, it appears that the aromatic range was not calculated as sum of individual analyte responses, but rather using timed integration. The auditor recalculated the CF for the C ₁₁ -C ₂₂ aromatic hydrocarbons using the sum of the individual standards and the %RSD was acceptable; thus, linear regression would not have been necessary.	*	Potential low bias for C ₁₁ - C ₂₂ aromatics as resulting CF is higher than if generated as per the method.

Table 9-4 Summary of Data Audits: EPH * Significant Issues; I = Indeterminate							
Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
	All hydrocarbon ranges	I	RLs/Sensitivity	CAM method deviation	C ₁₁ -C ₂₂ Aromatics: linear regression was used since the %RSD >25%; the laboratory did not provide evidence that the RL was recalculated per CAM. The auditor could not verify if the aliphatic RLs for the lowest standard would have had acceptable recovery since aliphatic initial calibration information was not provided. The C ₁₁ -C ₂₂ aromatic RL was recalculated during review using the lowest standard (330 ug/kg) which had a low recovery; the lowest standard with acceptable recovery was 10x higher than the lowest initial calibration standard (3,333 ug/kg).	*	RLs misrepresented as lower than allowed by method.
	C ₉ -C ₁₈ Aliphatics	I	Method Blank/GC Performance	Faulty chromatography	C ₉ -C ₁₈ aliphatics reported as nondetect but baseline for this range had a dip from octadecane through eicosane.		Potential effect on the usability of the data cannot be assessed.
	All hydrocarbon ranges	Low	Results Calculations	CAM method deviation	C ₁₁ -C ₂₂ Aromatics results re-quantified using CFs from summation of individual peaks. Could not verify aliphatic ranges since initial calibration information not provided.	*	Sample results approximately 3% higher with correct CFs (970 vs 940 ug/L).
	All hydrocarbon ranges	I	Sensitivity/RLs	CAM method deviation	The RLs for C ₉ -C ₁₈ aliphatic range, C ₁₉ -C ₃₆ aliphatic range, and C ₁₁ -C ₂₂ aromatic range were 33,000 µg/kg, 17,000 µg/kg, and 28,000 µg/kg, respectively, which are greater than 100x the lowest standard.	*	RLs misrepresented as higher than recommended by method; potential false negative results.
Absolute Resources	All hydrocarbon ranges	Low	Initial Calibration	CAM method deviation	CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range. The auditor recalculated the CFs using the sum of the individual standards for the aliphatic and aromatic ranges and the %RSD were acceptable for these 2 ranges; thus, linear regression would not have been necessary.	*	Potential low bias as resulting CF is higher than if generated as per the method. Laboratory procedure has since been revised to perform this calculation per the method.
	All hydrocarbon ranges	I	RLs/Sensitivity	CAM method deviation	The laboratory used linear regression for all ranges since the %RSDs were >25%; the laboratory did not provide evidence that the RL was recalculated per CAM; the RLs were recalculated during review using the lowest standard, C ₉ -C ₁₈ aliphatic and C ₁₁ -C ₂₂ aromatic were acceptable, but the C ₁₉ -C ₃₆ aliphatic had a low recovery using the next lowest standard with acceptable recovery.	*	RLs misrepresented as lower than allowed by method for C ₁₉ -C ₃₆ aliphatics. Although not provided in the full deliverable, the laboratory stated that re-quantitation of the lowest calibration standard is standard operating procedure.
	Target PAHs	I	Initial Calibration and Results Quantitation	CAM method deviation	The target PAHs were quantitated using linear regression or quadratic regression. However, the average CFs for all target PAHs had acceptable %RSDs except for benzo(a) anthracene; thus, average CF could have been used for all target PAHs except for benzo(a)anthracene rather than linear and quadratic regression. The laboratory did not provide evidence that the RL was recalculated per CAM; the RL was recalculated for benzo(a)anthracene during review and the RL was acceptable.	*	RLs misrepresented as lower than allowed by method. Although not provided in the full deliverable, the laboratory stated that re-quantitation of the lowest calibration standard is standard operating procedure. Potential effect on the usability of the data due to the unnecessary use of linear or quadratic regression was minimal. The auditor recalculated the results using the average CF and the results were similar to the laboratory results but may have been slightly lower or higher depending on the analyte.
	C ₉ -C ₁₈ Aliphatics, C ₁₉ -C ₃₆ Aliphatics	High	Results Quantitation	CAM method deviation	Results re-quantified using CFs from summation of individual peaks.	*	Sample recalculated using correct CFs: C ₉ -C ₁₈ Aliphatics: Laboratory result biased high by ~7%: 350 mg/kg vs 375 mg/kg. C ₁₉ -C ₃₆ Aliphatics: Laboratory result biased high by ~10%: 180 mg/kg vs 190 mg/kg.
SGS-Accutest	No issues noted						

Table 9-4 Summary of Data Audits: EPH * Significant Issues; I = Indeterminate							
Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
RI Analytical	All	I	Sensitivity/RLs	CAM method deviation	RLs for ranges were ~150x the lowest standard, not 100x lowest standard as required in method. RLs for target analytes 3x higher than lowest standard (therefore both target analytes & ranges higher than necessary).	*	RLs misrepresented as higher than recommended by method; potential false negative results.
Con-test Analytical	No issues noted.						
Alpha Analytical	All hydrocarbon ranges	I	Sensitivity/RLs	CAM method deviation	RLs for ranges were 50x the lowest standard, not 100x lowest standard as required in method.	*	RLs misrepresented as lower than allowed by method.
NETLab	All hydrocarbon ranges	Low	Initial Calibration	CAM method deviation	CFs for hydrocarbon ranges were not established by the summation of peak areas for the analytes of interest in the associated range. Instead, the CFs were established by integrating all peaks within the retention time window of the associated range. Laboratory used linear regression for both aliphatic ranges although %RSDs were <25. The auditor recalculated the CFs using the sum of the individual standards for the ranges and the %RSD were acceptable for the ranges; thus, linear regression would not have been necessary.	*	Potential low bias as resulting CF is higher than if generated as per the method. Laboratory procedure has since been revised to perform this calculation per the method.
	C ₉ -C ₁₈ Aliphatics, C ₁₉ -C ₃₆ Aliphatics	I	RLs/Sensitivity	CAM method deviation	The laboratory used linear regression for aliphatic ranges; the laboratory did not provide evidence that the RL was recalculated per CAM; the RLs were recalculated during review using the lowest standard and were acceptable		No effect on the usability of the data. Laboratory procedure has since been revised to perform this calculation and evaluation.
	C ₁₉ -C ₃₆ Aliphatics	I	Continuing Calibration	Calculation error	The C ₁₉ -C ₃₆ Aliphatic continuing calibration %D was high, but the surrogate was included in the range area; the laboratory manually verified that the %D was acceptable, but did not provide the revised %D results.		Potential misleading results to end user; no effect on the usability of the data.
	All hydrocarbon ranges	Low	Results Quantitation	CAM method deviation	Results re-quantified using CFs from summation of individual peaks.	*	Sample recalculated using correct CFs and results reported by laboratory biased low: C ₉ -C ₁₈ Aliphatics: 21% higher. C ₁₉ -C ₃₆ Aliphatics: 6% higher. C ₁₁ -C ₂₂ Aromatics: 11% higher. Laboratory procedure has since been revised to perform this calculation per the method.
	All	Low	Results Quantitation	Calculation error	The laboratory used the wrong %solids for a client sample. (93.8% was used but should be 83.8%). The result reported was biased low based on using the wrong %solids value.	*	Low bias in reported results.
	All	I	Sensitivity/RLs	CAM method deviation	RLs for ranges were 40x the lowest standard, not 100x lowest standard as required in method. RLs for target analytes 5x higher than lowest standard.	*	RLs misrepresented as lower than allowed by method.

Table 9-5 Summary of Data Audits: APH * Significant Issues; I = Indeterminate							
Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
Alpha Analytical	No issues noted						
SGS-Accutest	Target APH Analytes	I	Initial Calibration	CAM method deviation	Quadratic regression used for o-xylene and m&p-xylenes although linear regression could have been used. Quadratic regression used for naphthalene although this is not a “difficult” analyte.		See Results Quantitation below for effect on usability of data for o-xylene and m&p-xylenes. Overall effect on usability of data due to use of quadratic regression for naphthalene cannot be assessed.
	C ₉ -C ₁₀ Aromatics	I	Initial Calibration	CAM method deviation	Quadratic regression used for C ₉ -C ₁₀ aromatics although linear regression could have been used. C ₉ -C ₁₀ Aromatics based on m/z 120 only (m/z 134 not listed on quantitation report).	*	See Results Quantitation below for effect on usability of data due to use of quadratic regression. Lack of use of m/z 134 leads to an incorrect representation of this range in the calibration and an inaccurate RRF.
	All hydrocarbon ranges	I	Initial Calibration	CAM method deviation	Concentrations of ranges in calibration curve in units of ppbV instead of ug/m3.	*	Accuracy of reported results for all hydrocarbon ranges affected as it is unclear how the hydrocarbon range results were converted to ug/m3.
	MTBE	I	Continuing Calibration	Calculation error	According to the initial calibration summary form, the average RRF was used for quantitation. The continuing calibration summary form reported a percent drift instead of percent difference which implies that linear or quadratic regression was used; could not reproduce results for MTBE in continuing calibrations.		Overall effect on the usability of the data cannot be assessed.
	C ₉ -C ₁₀ Aromatics	Low	Results Quantitation	CAM method deviation	C ₉ -C ₁₀ Aromatics based on m/z 120 only (m/z 134 not listed on quantitation report). Results requantified using linear regression instead of quadratic regression used by laboratory.	*	Potential low bias as C ₉ -C ₁₀ aromatics not wholly represented by only m/z 120. Sample recalculated using linear regression and results reported by laboratory using quadratic regression biased low: 3 of 4 samples were higher by 146-166% (one sample was higher by 109%).
	M&p-Xylenes	Low	Results Quantitation	CAM method deviation	Results requantified using linear regression instead of quadratic regression used by laboratory.		Sample recalculated using linear regression and results reported by laboratory biased low: 3 of 4 samples were higher by 90-109% (one sample was nondetect and would not have changed).
	o-Xylene	High	Results Quantitation	CAM method deviation	Results requantified using linear regression instead of quadratic regression used by laboratory.		Sample recalculated using linear regression and results reported by laboratory biased high: 3 of 4 samples were lower by 3-21% (one sample would not have changed).
	All	I	RLs/Sensitivity	CAM method deviation	The laboratory used quadratic and linear regression for target analytes and ranges; the laboratory did not provide evidence that the RL was recalculated per CAM; the RLs were recalculated during review using the lowest standard for linear regression analytes (this could not be performed for analytes calibrated using quadratic regression). Toluene, ethylbenzene, o-xylene, m&p-xylenes: did not meet criteria for lowest standard (0.4 ppbV) but did meet for 2 ppbV standard. C ₉ -C ₁₂ Aliphatics: did not meet criteria for lowest standard (2.4 ppbV) but did meet for 30 ppbV standard.	*	RLs misrepresented as lower than allowed by method.

Table 9-5
Summary of Data Audits: APH
* Significant Issues; I = Indeterminate

Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
Con-test Analytical	All hydrocarbon ranges	Low	Initial Calibration and Results Calculations	Calculation/reporting error	The relative response factors (RRFs) could not be exactly reproduced during the audit. C ₅ -C ₈ aliphatics: calculated 2.423; laboratory reported 2.95. C ₉ -C ₁₂ aliphatics: calculated 3.008; laboratory reported 4.04. C ₉ -C ₁₀ aromatics: calculated 0.373; laboratory reported 0.39. Using the laboratory’s reported RRFs, the reported results for the ranges could not be reproduced. Using the auditor-generated RRFs, reported results for the ranges were closer to the calculated results.		Overall effect on the usability of the data is minimal. It was determined that the RRFs reported by the laboratory were in error and not what they used in calculations.
	All hydrocarbon ranges	I	RLs/Sensitivity	CAM method deviation	RLs 2x higher than necessary based on low standards.		RLs misrepresented as higher than required by method; potential false negative results.
NETLab	All hydrocarbon ranges	I	Initial Calibration and Results Quantitation	Calculation error	The auditor could not verify the RRFs for the 3 ranges; the laboratory’s RRFs in the data package appeared to be off by a factor of 2. However, when all results were calculated with the auditor’s RRFs, the results matched what the laboratory reported (potentially, the RRFs reported by the laboratory were in error and not what they used).		Overall effect on the usability of the data is minimal. It was determined that the correct RRFs were used in the calculations.
Katahdin Analytical	C ₉ -C ₁₂ Aliphatics	NA	Continuing Calibration	Calculation error	%D outside of acceptance criteria for C ₉ -C ₁₂ aliphatics but if correct true concentration used on continuing calibration form, the %D would be acceptable. The true concentration used was not based on sum of individual alkanes.		Overall effect on the usability of the data is minimal; misleading to end user of data. It was determined this was a one-time error.
Phoenix	C ₅ -C ₈ Aliphatics	I	Initial Calibration	CAM method deviation	n-Hexane and cyclohexane not included in calibration of range.	*	CF not wholly representative of range; overall effect on the usability of the data cannot be assessed. Laboratory procedure has since been revised to perform the calibration with the method-required compounds.
	All hydrocarbon ranges	I	Initial Calibration	Reporting error	Could not verify RRFs since concentrations of standards not provided.		Overall effect on the usability of the data cannot be assessed.
	All hydrocarbon ranges	NA	Method Blank	Calculation error	The Form 1 in package showed all 3 ranges detected in method blank. Based on the quantitation report in the raw data, it appears that the results for the ranges on the Form I were not adjusted and included surrogate/internal standard areas. Therefore, the ranges were not actually detected in the method blank.		Potential misleading results to end user; no effect on the usability of the data.
	All hydrocarbon ranges	Low	Integration	CAM method deviation	As laboratory was compiling information for MassDEP audit, it was discovered that the hydrocarbon ranges were not properly integrated in samples and did not always include the entire unresolved complex mixture. Results changed from the original CAM deliverable.	*	Revised results are higher than originally reported. C ₅ -C ₈ Aliphatics: (original/final) ug/m3 Sample 1: 110/120 Sample 2: 76/91 Sample 3: 63/74 Sample 4: 84/90 C ₉ -C ₁₂ Aliphatics: (original/final) ug/m3 Sample 1: 220/390 Sample 2: 120/230 Sample 3: 110/190 Sample 4: 140/260 C ₉ -C ₁₀ Aromatics: (original/final) ug/m3 Sample 1: 13/13 Sample 2: ND/11 Sample 3: ND/12 Sample 4: 14/14 At the time that the error was discovered, a letter and revised report was sent by the laboratory to every affected client.

Table 9-5
Summary of Data Audits: APH
* Significant Issues; I = Indeterminate

Laboratory	Analyte	Bias	Evaluation Parameter	Type of Non-Compliance	Description of Non-Compliance/Review Comments		Effect or Potential Effect on Usability of Data
Eurofins/Spectrum	1,3-Butadiene, naphthalene	I	Initial Calibration	CAM method deviation	Quadratic regression used for 1,3-butadiene and naphthalene but %RSDs <30; average RRF should have been used.		Overall effect on the usability of the data cannot be assessed.
	1,3-Butadiene, naphthalene	I	RLs/Sensitivity	CAM method deviation	The laboratory used quadratic regression; the laboratory did not provide evidence that the RL was recalculated per CAM; this calculation could not be performed during the review for analytes calibrated using quadratic regression.	*	RLs may be misrepresented as lower than allowed by method.
	C ₅ -C ₈ Aliphatics, C ₉ -C ₁₂ Aliphatics	I	Initial Calibration	Calculation	Units of concentrations for standards not clearly presented. No units were shown on the quantitation reports for individual alkane concentrations but the labels on the header of the quantitation reports listed as 2,4,10,20,40,50,250, 500 ppbV. Units on quantitation reports for hydrocarbon range concentrations were shown as ug/m3. Initial calibration standard concentrations listed as ppbV on analytical run log but continuing calibration standard concentrations listed as ug/m3 on analytical run log. To reproduce the RRFs used by laboratory, used concentrations from header labels in ppbV and internal standard concentrations in ug/m3. When used in sample calculations, this results in units of ppbV.		Overall effect on the usability of the data is minimal. Laboratory response clarified units.
	C ₅ -C ₈ Aliphatics, C ₉ -C ₁₂ Aliphatics	Low/High	Results Quantitation	CAM method deviation	For the adjusted C ₅ -C ₈ aliphatic and C ₉ -C ₁₂ aliphatic ranges, laboratory subtracted the area counts of target analytes rather than reported concentrations. If concentrations had instead been subtracted, significant changes in the final reported concentrations would have been seen. See below for specific calculation errors. Sample 1: Laboratory subtracted area counts of target analytes even if reported as nondetect on final results. Also, laboratory did not subtract area count of toluene from unadjusted C ₅ -C ₈ aliphatics. If detected concentrations were subtracted, the adjusted C ₅ -C ₈ aliphatics would be 130 ug/m3 and not 1720 ug/m3 (reason for major difference is because laboratory did not subtract area count of toluene and toluene was the highest concentration target analyte in this sample). Sample 2: Sample analyzed at 20-fold dilution. Most target analytes nondetect except toluene and o-xylene. Laboratory subtracted area counts of target analytes even if reported as nondetect on final results. If detected concentrations were subtracted, the adjusted C ₅ -C ₈ aliphatics would be 11,009 ug/m3 and not 10,200 ug/m3. Sample 3: Sample analyzed at 344-fold dilution. All target analytes nondetect. Laboratory subtracted area counts of target analytes even if reported as nondetect on final results. If detected concentrations were subtracted, the adjusted C ₅ -C ₈ aliphatics would be 413,000 ug/m3 and not 399,000 ug/m3.	*	Significant effect on final results reported by the laboratory. Sample 1: The adjusted C ₅ -C ₈ aliphatics would be 130 ug/m3 and not 1720 ug/m3. Sample 2: The adjusted C ₅ -C ₈ aliphatics would be 11,009 ug/m3 and not 10,200 ug/m3. Sample 3: The adjusted C ₅ -C ₈ aliphatics would be 413,000 ug/m3 and not 399,000 ug/m3.
	All hydrocarbon ranges	I	RLs/Sensitivity	CAM method deviation	RLs for all hydrocarbon ranges not based on low standards. For C ₅ -C ₈ aliphatics, C ₉ -C ₁₂ aliphatics, and C ₉ -C ₁₀ aromatics, RLs were reported as 12, 12, and 10 ug/m3, respectively. Based on low standards in the initial calibration, the RLs should be 46, 71, and 50 ug/m3, respectively.	*	RLs misrepresented as lower than allowed by method. Laboratory procedure has since been revised to calculate RLs in accordance with the method requirements.

10.0 RECOMMENDATIONS FOR IMPROVEMENT TO GENERATE CAM-COMPLIANT DATA

In general, the data audits demonstrated that the EPH, and VPH methods exhibited the more significant types of method deviations, calculation errors, and reporting issues. The data audits for the TO-15 and APH methods also exhibited several method deviations and reporting issues, but these were less significant to the overall usability and accuracy of the reported data and also less frequent, indicating that the laboratory community has a good understanding of the TO-15 and APH methods and the ability to clearly follow the procedures and CAM requirements for these methods.

As a result of the data audit, MassDEP has provided a clarification in the VPH method (February 2018) and the EPH method (December 2019) on the hydrocarbon range calibration procedures, one of most common cited audit nonconformances. Based on the observations of the type and frequency of other QA/QC issues noted during the data audits and using technical judgment on how to minimize the incidences of these issues in the future, the following actions are being considered by MassDEP to improve the overall compliance of CAM data and consistently produce scientifically defensible data for MCP decisions.

- Providing outreach and training for analytical laboratories and LSPs;
 - General: provide training on how to correctly fill out and review the MassDEP Analytical Protocol Certification Form and interpret laboratory narrative issues. Lack of information in the laboratory narratives on QC issues can be confusing and misleading for data users in evaluating potential impact of QC issues on usability of data.
 - Specific: provide training concerning the most common EPH and VPH method non-compliances and QA/QC performance standard issues observed during the Data Audit process.
- Conducting Enforcement for CAM non-compliance;
 - Issue Notices of Non-Compliance (NONs) to the laboratories for incorrectly certifying CAM compliance.
- Performing routine data audits;
 - MassDEP and/or its contractors could perform routine reviews of laboratory data packages such that a certain percentage of data packages submitted for MCP decisions are evaluated each year. This will allow MassDEP to keep track of potential data quality issues and non-compliance trends so that outreach, training, and/or communication can be implemented to correct systematic QA/QC issues uncovered.
- Expanding the CAM by including a mandatory third-party audit program;
 - While use of CAM would remain voluntary, MassDEP could specify a mandatory auditing program for laboratories that certify that they are implementing the CAM. The frequency of audits could be a function of how well a laboratory performs in a preceding audit, e.g., every year in cases where significant non-compliance was noted, or every 2 to 3 years where no significant noncompliance issues were noted.
 - A list of qualified third-party auditors would be established by MassDEP, based upon an evaluation of the expertise and experience of candidates.
- Implementing a 21E laboratory certification program.

MassDEP intends to have additional discussions with program stakeholders on the results of this audit and actions that can be taken, including those outlined above, to continue to improve the reliability of data analyses and the application of the CAM for samples taken to support response action decisions at 21E disposal sites.

APPENDIX 1
Example Request for Information (RFI) Letters and Data Package
Requirements Sent to Laboratories



Department of Environmental Protection

Charles D. Baker
Governor

Karyn E. Polito
Lieutenant Governor

Kathleen A. Theoharides
Secretary

Martin Suuberg
Commissioner

CERTIFIED MAIL – RETURN RECEIPT REQUESTED

DATE: November 4, 2016

RE: Request For Information –
MassDEP Data Audit
LAB
Data Package:

LAB

Attn: Lab Director

Dear :

As you know, the Massachusetts Department of Environmental Protection (MassDEP) Bureau of Waste Site Cleanup (BWSC) has undertaken a number of initiatives to ensure that the quality of analytical data for analyses conducted on samples from disposal sites regulated under MGL chapter 21E and the Massachusetts Contingency Plan (MCP) is adequately documented and commensurate with its use in supporting assessment and cleanup decisions. Personnel from commercial laboratories were key contributors to these efforts, which included the publication of MassDEP's Compendium of Analytical Methods (CAM) available on the Department's web site at:

<http://www.mass.gov/eea/agencies/massdep/cleanup/regulations/wsc10-320-compendium--quality-control- reqs.html>.

This letter is to inform you that as part of MassDEP's ongoing obligation to audit MCP response action submittals pursuant to 310 CMR 40.1100, MassDEP is conducting a multi-laboratory audit of analyses purportedly performed according to the CAM. This audit is not directed at any particular laboratory, data package or project. MassDEP established the scope of this audit to look at data for VPH, EPH, APH and TO- 15 analyses performed by 14 laboratories after April 2013 using the CAM. The Licensed Site Professionals (LSPs) and Potentially Responsible Parties (PRPs) for the above-referenced data packages are copied on this request.

As part of this effort, MassDEP is requesting information for analyses performed by your laboratory.

As stated in the CAM (WSC-CAM-VII A; Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP); July 1, 2010; Section 2.4):

“It is required that both reported and non-reported analytical data and information be retained by the laboratory for ten (10) years to facilitate further in-depth review or for audit support.”

In this regard and pursuant to M.G.L. c. 21E, §§ 2, 4, and 8 and 310 CMR 40.0165, MassDEP requests that LAB provide the information listed in the attached “Data Package Requirements” within 45 days of the date of this letter for the applicable data package(s) and analyses identified below:

Laboratory	Data Package ID (RTN#)	Date Samples Collected (Matrix)	VPH	EPH	APH	TO-15
LAB		(soil)	X	X		

Along with the information requested above, please include any further information and documentation for each data package, as the laboratory deems necessary in order to substantiate and describe data quality.

Submittal of information and data on a PDF format via electronic means (e.g., CD-ROM and/or email) is encouraged. In addition, MassDEP may request an appointment to inspect the laboratory at a future date.

In responding to this Request for Information (RFI), please be aware that 310 CMR 40.0009(6) requires any person responding to an RFI issued by MassDEP to execute a written Declaration as set forth in 310 CMR 40.0009(1). For your convenience, a copy of the requisite Declaration is attached to this RFI as a separate page. This Declaration should be executed by the party to whom this RFI is directed and, therefore, should be signed by LAB and/or anyone authorized to act on its behalf.

Please do not hesitate to contact Ken Marra at (617) 292-5966 or Kendall.Marra@state.ma.us if you have any questions regarding this request.

We appreciate your cooperation in this matter.

Sincerely,

R. Kendall Marra, PE
Environmental Engineer
Bureau of Waste Site Cleanup
Policy and Program Development

cc: Paul W. Locke – Assistant Commissioner, Bureau of Waste Site Cleanup
Elizabeth J. Callahan – Acting Director, Division of Policy and Program Development
Jennifer L. Davis – Senior Counsel, Office of General Counsel

Attach.

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CERTIFICATION OF SUBMITTAL

301 CMR 40.0009(1) and (6)

LAB

REQUEST FOR INFORMATION

I, _____, attest under the penalties of perjury (i) that I have personally examined and am familiar with the information contained in this submittal, including any and all documents accompanying this submittal, (ii) that, based on my inquiry of those individuals immediately responsible for obtaining the information, the material information contained in this submittal is, to the best of my knowledge and belief, true, accurate and complete, and (iii) that I am fully authorized to make this attestation on behalf of the person or entity legally responsible for this submittal. I/the person or entity on whose behalf this submittal is made am/is aware that there are significant penalties, including, but not limited to, possible fines and imprisonment, for willfully submitting false, inaccurate or incomplete information.

Date: _____

By: _____
Signature

Title



Commonwealth of Massachusetts
Executive Office of Energy & Environmental Affairs

Department of Environmental Protection

One Winter Street Boston, MA 02108 • 617-292-5500

Charles D. Baker
Governor

Karyn E. Polito
Lieutenant Governor

Matthew A. Beaton
Secretary

Martin Suuberg
Commissioner

CERTIFIED MAIL – RETURN RECEIPT REQUESTED

DATE: November 30, 2018

RE: Request for Information –
MassDEP Multi-Lab Data Audit
Draft Audit Findings
«LAB_NAME»
(Identified as “Lab C”)
Data Package: [Redacted]

«LAB_NAME»
«ADDRESS1» «ADDRESS2»
«CITYSTATE»

Attn: «LAB_MANAGER» or Lab Director

Dear«LAB_MANAGER»:

As part of our ongoing obligation to audit MCP response action submittals pursuant to 310 CMR 40.1100, the Massachusetts Department of Environmental Protection (MassDEP) Bureau of Waste Site Cleanup (BWSC) has been conducting a multi-laboratory audit of analyses certified in each case by the laboratory to have been performed according to MassDEP’s Compendium of Analytical Methods (CAM)¹. This audit is one of a number of initiatives the Department has undertaken to ensure that the quality of analytical data for analyses conducted on samples from disposal sites regulated under MGL chapter 21E and the Massachusetts Contingency Plan (MCP) is adequately documented and commensurate with its use in supporting assessment and cleanup decisions.

In this regard, MassDEP issued a Request for Information (RFI) in November 2016 to the participating laboratories, and in response «LAB_NAME» provided information for the applicable CAM-certified data package(s) and analyses identified below:

¹ The CAM is available on the Department’s web site at:

<http://www.mass.gov/eea/agencies/massdep/cleanup/regulations/wsc10-320-compendium--quality-control-regs.html>.

This information is available in alternate format. Contact Michelle Waters-Ekanem, Director of Diversity/Civil Rights at 617-292-5751.

TTY# MassRelay Service 1-800-439-2370

MassDEP Website: www.mass.gov/dep

Printed on Recycled Paper

Laboratory	Data Package ID (RTN#)	Date Samples Collected (Matrix)	VPH	EPH	APH	TO-15
<<LAB_NAME>>	[redacted]	[redacted] (soil)	X	X		

This letter is to provide you with a draft summary of audit findings for the laboratories subject to this review, and, as a formal RFI pursuant to M.G.L. c. 21E, §§ 2, 4, and 8 and 310 CMR 40.0165, request your responses to the CAM non-compliance issues identified for your laboratory. **«LAB_NAME» is identified as “Lab C”** in the attached summary tables. For each of the CAM non-compliance issues identified for Lab C in the attached summary tables, your response must indicate: (a) whether you agree or disagree with the non-compliance listed and why; (b) whether the non-compliance is the result of your standard practices at the time of the analyses; and (c) if, when and how you have changed your standard practices to correct such non-compliance.

The deadline for your response is January 30, 2019. In responding to this Request for Information (RFI), please be aware that 310 CMR 40.0009(6) requires any person responding to an RFI issued by MassDEP to execute a written Declaration as set forth in 310 CMR 40.0009(1). For your convenience, a copy of the requisite Declaration is attached to this RFI as a separate page. This Declaration should be executed by the party to whom this RFI is directed and, therefore, should be signed by «LAB_NAME» and/or anyone authorized to act on its behalf.

Please do not hesitate to contact Ken Marra at (617) 292-5966 or Kendall.Marra@state.ma.us if you have any questions regarding this request.

We appreciate your cooperation in this matter.

Sincerely,

R. Kendall Marra, PE
Environmental Engineer
Bureau of Waste Site Cleanup
Policy and Program Development

cc: Paul W. Locke – Assistant Commissioner, Bureau of Waste Site Cleanup
Elizabeth J. Callahan – Acting Director, Division of Policy and Program Development
Jennifer L. Davis – Senior Counsel, Office of General Counsel
Attach.

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«LAB_NAME»

Request for Information - MassDEP Data Audit

Page 4 of 4

CERTIFICATION OF SUBMITTAL

301 CMR 40.0009(1) and (6)

Re: «LAB_NAME»

REQUEST FOR INFORMATION

I, _____, attest under the penalties of perjury (i) that I have personally examined and am familiar with the information contained in this submittal, including any and all documents accompanying this submittal, (ii) that, based on my inquiry of those individuals immediately responsible for obtaining the information, the material information contained in this submittal is, to the best of my knowledge and belief, true, accurate and complete, and (iii) that I am fully authorized to make this attestation on behalf of the person or entity legally responsible for this submittal. I/the person or entity on whose behalf this submittal is made am/is aware that there are significant penalties, including, but not limited to, possible fines and imprisonment, for willfully submitting false, inaccurate or incomplete information.

Date: _____

«LAB_NAME»

By: _____
Signature

Title

Volatile Petroleum Hydrocarbons (VPH) Data Package Requirements for MassDEP Data Audit

Inclusion of the information listed below is requested to perform a data audit to verify compliance with the analytical methods and to evaluate accuracy and reliability of the reported results. These deliverables represent a “full data package” including all sample documentation from receipt through preparation, analysis, and data reporting.

1. Case Narrative – Must comply with the required laboratory case narrative contents as described in WSC-CAM-VIIA (*Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted under the Massachusetts Contingency Plan (MCP)*, Final, Revision 1, July 1, 2010), Section 2.4.2. Be sure to include documentation for any performance standards that did not meet acceptance criteria stated in Table IV A-2, Specific QC Requirements and Performance Standards for VPH using WSC-CAM-IVA (included in WSC-CAM-IVA, *Quality Control Requirements and Performance Standards for the Analysis of Volatile Petroleum Hydrocarbons (VPH)*, MADEP-VPH-04-1.1 in support of Response Actions under the Massachusetts Contingency Plan (MCP), Final, Revision 1, July 1, 2010). Corrective actions, if required in the performance standard in Table IV A-2, must also be explained. A complete list of samples included in the data package, including the field sample ID associated with the laboratory ID, would also be helpful for data review.
2. Sample Handling Information - Chain of custodies (including internal), sample receipt logs (cooler temperatures and sample pH upon receipt should also be included), correspondences and any telephone logs, if applicable
3. Miscellaneous logs
 - a. Dry weight log forms (soil/sediment samples)
 - b. Injection/analytical run logs (must include volume of water samples analyzed and volume of methanol extracts analyzed)
 - c. Soil sample weight logs
4. Calibration Information
 - a. Summary of calibration factors from the initial calibration for each standard and analyte and hydrocarbon range in the initial calibration, average calibration factors, percent relative standard deviations (%RSDs), and correlation coefficients
 - b. Calibration curves for calibrations performed using linear or quadratic regression
 - c. Summary of percent differences and calibration factors for continuing calibration standards
 - d. Chromatograms for all initial and continuing calibration standards clearly demonstrating how hydrocarbon ranges, target analytes, and surrogates were integrated
 - e. Quantitation reports for all initial and continuing calibration standards exhibiting the area counts of hydrocarbon ranges, target analytes, and surrogates
 - f. Concentrations of standards used in initial and continuing calibration should be clearly presented

Extractable Petroleum Hydrocarbons (EPH) Data Package Requirements for MassDEP Data Audit

Inclusion of the information listed below is requested to perform a data audit to verify compliance with the analytical methods and to evaluate accuracy and reliability of the reported results. These deliverables represent a “full data package” including all sample documentation from receipt through preparation, analysis, and data reporting.

1. Case Narrative – Must comply with the required laboratory case narrative contents as described in WSC-CAM-VIIA (*Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted under the Massachusetts Contingency Plan (MCP)*, Final, Revision 1, July 1, 2010), Section 2.4.2. Be sure to include documentation for any performance standards that did not meet acceptance criteria stated in Table IV B-2, Specific QC Requirements and Performance Standards for EPH using WSC-CAM-IVB (included in WSC-CAM-IVB, *Quality Control Requirements and Performance Standards for the Analysis of Extractable Petroleum Hydrocarbons (EPH)*, MADEP-EPH-04-1.1 in support of Response Actions under the Massachusetts Contingency Plan (MCP), Final, Revision 1, July 1, 2010). Corrective actions, if required in the performance standard in Table IV B-2, must also be explained. A complete list of samples included in the data package, including the field sample ID associated with the laboratory ID, would also be helpful for data review.
2. Sample Handling Information - Chain of custodies (including internal), sample receipt logs (cooler temperatures and sample pH upon receipt should also be included), correspondences and any telephone logs, if applicable.
3. Miscellaneous logs
 - a. Dry weight log forms (soil/sediment samples)
 - b. Injection/analytical run logs
 - c. Extraction/preparation/fractionation logs (must clearly indicate sample weights or volumes, final extract volumes, extraction method used, date of extraction, extraction times where appropriate for the method, etc.)
4. Calibration Information
 - a. Summary of calibration factors from the initial calibration for each standard and analyte and hydrocarbon range in the initial calibration, average calibration factors, percent relative standard deviations (%RSDs), and correlation coefficients
 - b. Calibration curves for calibrations performed using linear or quadratic regression
 - c. Summary of percent differences and calibration factors for continuing calibration standards
 - d. Chromatograms for all initial and continuing calibration standards clearly demonstrating how hydrocarbon ranges, aliphatic components, target analytes, and surrogates were integrated
 - e. Quantitation reports for all initial and continuing calibration standards exhibiting the area counts of hydrocarbon ranges, aliphatic components, target analytes, and surrogates
 - f. Concentrations of standards used in initial and continuing calibrations should be clearly presented
 - g. Demonstration of absence of mass discrimination (i.e., acceptable C_{28}/C_{20} ratio) in all aliphatic continuing calibration standards
 - h. Demonstration of adequate resolution of naphthalene and dodecane in the aliphatic calibration standards
5. Analytical Results
 - a. Quantitation reports for all sample analyses exhibiting the area counts of hydrocarbon ranges, target analytes, and surrogates

Air-Phase Petroleum Hydrocarbon (APH) Data Package Requirements for MassDEP Data Audit

Inclusion of the information listed below is requested to perform a data audit to verify compliance with the analytical methods and to evaluate accuracy and reliability of the reported results. These deliverables represent a “full data package” including all sample documentation from receipt through preparation, analysis, and data reporting.

1. Case Narrative – Must comply with the required laboratory case narrative contents as described in WSC-CAM-VIIA (*Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted under the Massachusetts Contingency Plan (MCP)*, Final, Revision 1, July 1, 2010), Section 2.4.2. Be sure to include documentation for any performance standards that did not meet acceptance criteria stated in Table IX A-2, Specific QC Requirements and Performance Standards for Air-Phase Petroleum Hydrocarbons (APH) using WSC-CAM-IXA (included in WSC-CAM-IXA, *Quality Control Requirements and Performance Standards for the Analysis of Air-Phase Petroleum Hydrocarbons (APH) by Gas Chromatography/Mass Spectrometry (GC/MS) in Support of Response Actions under the Massachusetts Contingency Plan (MCP)*, Final, July 1, 2010). Corrective actions, if required in the performance standard Table IX A-2, must also be explained. A complete list of samples included in the data package, including the field sample ID associated with the laboratory ID, would also be helpful for data review.
2. Sample Handling Information – Chains-of-custody (including internal), sample receipt logs, correspondences, and telephone logs, if applicable
3. Miscellaneous logs
 - a. Injection/analytical run logs, clearly showing the volume of sample analyzed
 - b. Canister vacuums (before and after sampling, as measured by the laboratory):
 - c. Flow controller calibration (before and after sampling)
 - d. Results of leak checks performed on autosampler for each canister
4. Calibration Information
 - a. Summary of response factors from the initial calibration for each standard and analyte and hydrocarbon range in the initial calibration, average response factors, percent relative standard deviations (%RSDs), and correlation coefficients
 - b. Calibration curves for calibrations performed using linear or quadratic regression
 - c. Summary of percent differences and response factors for continuing calibration standards
 - d. Total ion chromatograms for all initial and continuing calibration standards clearly demonstrating how aliphatic hydrocarbon ranges, target analytes, and internal standards were integrated
 - e. Extracted ion chromatograms for C₉-C₁₀ aromatics clearly demonstrating how hydrocarbon range was integrated
 - f. Quantitation reports for all initial and continuing calibration standards exhibiting the area counts of hydrocarbon ranges, target analytes, and internal standards
 - g. Extracted ion chromatograms for any manual integrations
 - h. Concentrations of standards used in initial and continuing calibrations should be clearly presented

TO-15 Data Package Requirements for MassDEP Data Audit

Inclusion of the information listed below is requested to perform a data audit to verify compliance with the analytical methods and to evaluate accuracy and reliability of the reported results. These deliverables represent a “full data package” including all sample documentation from receipt through preparation, analysis, and data reporting.

1. Case Narrative – Must comply with the required laboratory case narrative contents as described in WSC-CAM-VIIA (*Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted under the Massachusetts Contingency Plan (MCP)*, Final, Revision 1, July 1, 2010), Section 2.4.2. Be sure to include documentation for any performance standards that did not meet acceptance criteria stated in Table IX B-1, Specific QC Requirements and Performance Standards for VOCs in Air (EPA Method TO-15) using WSC-CAM-IXB (included in WSC-CAM-IXB, *Quality Control Requirements and Performance Standards for the Analysis of Volatile Organic Compounds in Air Samples (TO-15) by Gas Chromatography/Mass Spectrometry (GC/MS) in Support of Response Actions under the Massachusetts Contingency Plan (MCP)*, Final, July 1, 2010). Corrective actions, if required in the performance standard Table IX B-1, must also be explained. A complete list of samples included in the data package, including the field sample ID associated with the laboratory ID, would also be helpful for data review.
2. Sample Handling Information – Chains-of-custody (including internal), sample receipt logs, correspondences, and telephone logs, if applicable
3. Miscellaneous logs
 - a. Injection/analytical run logs, clearly showing the volume of sample analyzed
 - b. Canister vacuums (before and after sampling, as measured by the laboratory):
 - c. Flow controller calibration (before and after sampling)
 - d. Results of leak checks performed on autosampler for each canister
4. Calibration Information
 - a. Summary of response factors from the initial calibration for each standard and analyte in the initial calibration, average response factors, percent relative standard deviations (%RSDs), and correlation coefficients
 - b. Calibration curves for calibrations performed using linear or quadratic regression
 - c. Summary of percent differences and response factors for continuing calibration standards
 - d. Total ion chromatograms for all initial and continuing calibration standards
 - e. Quantitation reports for all initial and continuing calibration standards exhibiting the area counts of target analytes and internal standards
 - f. Extracted ion chromatograms for any manual integrations
 - g. Concentrations of standards used in initial and continuing calibrations should be clearly presented