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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

March 2021

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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 C9-C10 aromatics not subtracted from C9-C12 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 |
| **Target Compound Identification**: Polycyclic aromatic hydrocarbons (PAHs) reported as co-eluting in samples and standards. | 1 |
| APH | **Calibration**: m/z 134 not utilized for measurement of C9-C10 aromatics. | 1 |
| **Calibration**: n-Hexane and cyclohexane not used in calibration of C5-C8 aliphatics. | 1 |
| **Calibration**: Hydrocarbon ranges calibrated in units of ppbV. | 1 |
| **Calibration**: Quadratic regression utilized for C9-C10 aromatics and select target analytes. | 1 |
| **Results Quantitation**: Mass/charge (m/z) 134 not utilized for measurement of C9-C10 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.

# 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.

# 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.

# 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.

# 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.

## 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.

## 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.

## 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.

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

## 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. |

## 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 |

## 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**  *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?* | No issues noted. | |
| **Question B**  *Were the analytical method(s) and all associated QC requirements specified in the selected CAM protocol(s) followed?* | 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. |
| SGS-Accutest | Low-level standard calculation not performed when linear or quadratic regression used for calibration. |
| **Question C**  *Were all required corrective actions and analytical response actions specified in the selected CAM protocol(s) implemented for all identified performance standard non-conformances?* | 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**  *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”?* | Phoenix | Post-sampling flow rates and RPDs not included in original data package. |
| **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 | Naphthalene not reported. |
| Phoenix | Naphthalene not reported. |
| Eurofins/Spectrum | Only halocarbons reported. |
| NETLab | Naphthalene not reported. |
| **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)?* | 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**  *Were the RLs at or below all CAM RLs specified in the selected CAM protocol(s)?* | NETLab | Select CAM RLs not met. |

# SUMMARY OF CAM NON-COMPLIANCE ISSUES FOR MASSDEP VPH

## 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. |
| 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. |

## 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 |

## 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**  *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?* | Chemserve | Elevated cooler temperature. |
| **Question B**  *Were the analytical method(s) and all associated QC requirements specified in the selected CAM protocol(s) followed?* | Chemserve | Integration of aliphatic hydrocarbon ranges not performed properly in initial and continuing calibrations.  N-decane and n-butylcyclohexane not used in calibration of C9-C12 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 C9-C10 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**  *Were all required corrective actions and analytical response actions specified in the selected CAM protocol(s) implemented for all identified performance standard non-conformances?* | Phoenix | LCS percent recoveries (%Rs) outside acceptance limits and no corrective action performed.  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. | |

# SUMMARY OF CAM NON-COMPLIANCE ISSUES FOR MASSDEP EPH

## 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 C11-C22 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 C11-C22 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 C11-C22 aromatics not reported. | No issues noted. |
| Con-test Analytical | No issues noted. | No issues noted. |
| Alpha Analytical | No issues noted. | No issues noted. |
| NETLab | Results for ranges not summarized on LCS summary form.  Results for unadjusted C11-C22 aromatics not reported. | No issues noted. |

## 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 |

## 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**  *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?* | Chemserve | Elevated cooler temperature and samples delivered one day after collected. |
| **Question B**  *Were the analytical method(s) and all associated QC requirements specified in the selected CAM protocol(s) followed?* | 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**  *Were all required corrective actions and analytical response actions specified in the selected CAM protocol(s) implemented for all identified performance standard non-conformances?* | No issues noted. | |
| **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?* | ESS | Question A was not answered. |
| Absolute Resources |
| **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. | |

# SUMMARY OF CAM NON-COMPLIANCE ISSUES FOR MASSDEP APH

## 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 C9-C10 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.  Chromatograms did not clearly show retention time markers.  Leak check report not provided. |

## 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 |

## 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**  *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?* | No issues noted. | |
| **Question B**  *Were the analytical method(s) and all associated QC requirements specified in the selected CAM protocol(s) followed?* | Phoenix | n-Hexane and cyclohexane not included in calibration of C5-C8 aliphatics. |
| SGS-Accutest | Mass/charge (m/z) 134 not utilized for determination of C9-C10 aromatics. |
| Low-level standard calculation not performed when linear regression used in calibration. |
| Eurofins/Spectrum |
| **Question C**  *Were all required corrective actions and analytical response actions specified in the selected CAM protocol(s) implemented for all identified performance standard non-conformances?* | No issues noted. | |
| **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?* | No issues noted. | |
| **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. | |

# 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 C9-C10 aromatics not subtracted from C9-C12 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 |
| **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 C9-C10 aromatics. | 1 |
| **Calibration**: n-Hexane and cyclohexane not used in calibration of C5-C8 aliphatics. | 1 |
| **Calibration**: Hydrocarbon ranges calibrated in units of ppbV. | 1 |
| **Calibration**: Quadratic regression utilized for C9-C10 aromatics and select target analytes. | 1 |
| **Results Quantitation**: m/z 134 not utilized for measurement of C9-C10 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 ≥ 10x 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 ±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. |
| Eurofins/Spectrum | 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. |
| Detected VOCs | NA | Target Compound Identification | Full deliverable omission | Ion intensities were not provided for detected VOCs; could not verify ratios were ±20%. |  | Usability of the data not significantly affected. |
| NETLab | 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. |
| 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. |
| C5-C8 Aliphatics, C9-C12 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. |
| C9-C12 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. |
| C5-C8 Aliphatics | Low | Results Quantitation | CAM method deviation | Results re-quantified using CF from summation of individual peaks. | \* | Sample result recalculated using correct CF.  C5-C8 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. |
| C9-C12 Aliphatics | Low | Results Quantitation | CAM method deviation | Results re-quantified using CF from individual peak. | \* | Sample result recalculated using correct CF.  C9-C12 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: C5-C8 aliphatics and C9-C10 aromatics biased high and C9-C12 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: C5-C8 aliphatics and C9-C10 aromatics reported by laboratory biased high by ~30% and C9-C12 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. |
| C9-C10 Aromatics, C9-C12 Aliphatics | Low and High | LCS | CAM method deviation and non-compliance | The recoveries of C9-C10 aromatics and C9-C12 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. |
| C9-C10 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:  C9-C10 aromatics: Laboratory reported result biased high: 246 ug/L (not 270 ug/L)  C5-C8 aliphatics unadjusted: Laboratory reported result biased low: 100 ug/L (not 75 ug/L): both non-detect when adjusted  C9-C12 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 C9-C10 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 C9-C12 aliphatics but not needed: during review recalculated using linear regression.  C5-C8 aliphatics: recalculated average CF during review; all CFs could be reproduced except 75 ppb standard: used correct CF in all subsequent calculations during audit.  C9-C10 aromatics: laboratory used 1,2,4-trimethylbenzene only but areas listed for C9-C10 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 C9-C12 aliphatics but all were below the RL. |
| All hydrocarbon ranges | I | Sensitivity/RLs | CAM method deviation | Laboratory did not recalculate concentration of C9-C12 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 | C5-C8 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  C9-C12 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  C9-C10 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 | \* | C5-C8 and C9-C12 Aliphatics: effect on usability of data will be dependent on petroleum product present in sample.  C9-C10 aromatics: effect on usability of data is a low bias. |
| C5-C8 Aliphatics, C9-C12 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 C5-C8 aliphatics (not 100 ug/L) and 500 ug/L for C9-C12 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 | C9-C12 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 | C5-C8 Aliphatics, C9-C12 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. |
| C5-C8 Aliphatics, C9-C12 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. |
| C5-C8 Aliphatics, C9-C12 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 (C5-C8 aliphatics: 79% vs 95% and C9-C12 aliphatics: 76% vs 111%). |  | Usability of the data not significantly affected. |
| C5-C8 Aliphatics, C9-C12 Aliphatics | Low | Results Quantitation | CAM method deviation | Results re-quantified using CFs from summation of individual peaks.  C5-C8 aliphatics and C9-C12 aliphatics results reported by laboratory lower than what was recalculated during review. | \* | Sample recalculated using correct CFs:  C5-C8 aliphatics unadjusted: Laboratory reported result biased low: 1200 ug/L (not 890 ug/L): (adjusted 930 ug/L, not 626 ug/L)  C9-C12 aliphatics unadjusted: Laboratory reported result biased low: 2390 ug/L (not 1540 ug/L) (adjusted 1596 ug/L, not 794 ug/L) |
| 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 | C5-C8 Aliphatics: start time is 0.31 minutes before pentane (instead of 0.1 min): biased high.  C9-C12 Aliphatics: end time is 0.04 min after naphthalene instead of 0.1 min before naphthalene: biased high.  C9-C10 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 | C9-C12 Aliphatics | I | Sensitivity/RLs | CAM method deviation | Laboratory did not recalculate concentration of C9-C12 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 C5-C8 aliphatics. | \* | Sample recalculated using correct CFs:  C5-C8 aliphatics: Laboratory result biased low by ~10%: 280 mg/kg vs 250 mg/kg  C9-C12 aliphatics: Laboratory result biased high by ~3%: 1650 mg/kg vs 1700 mg/kg  C9-C10 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. |
| 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 C9-C10 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. |
| C9-C12 Aliphatics and C9-C10 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. |
| C9-C10 Aromatics | Low | Results Quantitation | CAM method deviation | Results re-quantified using CFs from summation of individual peak for C9-C10 aromatics.  Laboratory also did not correct methanol extract volume for % moisture. | \* | Sample recalculated using correct CFs:  C9-C10 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. |
| C5-C8 Aliphatics, C9-C10 Aromatics | Low | Retention Time Windows | CAM method deviation | C5-C8 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.  C9-C10 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. |
| 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. |
| 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. |
| C19-C36 Aliphatics | High | GC Performance | CAM method deviation and non-compliance | The response ratio of C28 to C20 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 analyzes (> C20) 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. |
| C9-C18 Aliphatics, C19-C36 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. |
| C9-C18 Aliphatics, C19-C36 Aliphatics | I | Results Quantitation | CAM method deviation | Results re-quantified using CFs from summation of individual peaks. | \* | Sample recalculated using correct CFs:  C9-C18 Aliphatics: Laboratory result biased low by ~7%: 15.7 mg/kg vs 14.6 mg/kg  C19-C36 Aliphatics: Laboratory result biased low by ~8.5%: 494 mg/kg vs 452 mg/kg  C11-C22 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 | C9-C18 Aliphatics: window was 2.9-12.9 (should be 3.15-13.25).  C19-C36 Aliphatics: window was 12.9-23.5 (should be 13.25-23.38).  C11-C22 Aromatics: window was 5.96-20 (should be 6.12-19.77). | \* | C9-C18 Aliphatics: effect on usability of data will be dependent on petroleum product present in sample.  C19-C36 Aliphatics and C11-C22 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 C9-C18 aliphatics although the %RSD <25 and average CF could therefore be used.  Laboratory used linear regression for C19-C36 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 C11-C22 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. |
| All hydrocarbon ranges | I | Results Quantitation | Calculation error | Results re-quantified using CFs from summation of individual peaks. | \* | Samples recalculated using correct CFs:  C9-C18 Aliphatics: not significantly affected  C19-C36 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).  C11-C22 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, C9-C18 aliphatic was acceptable, but the C19-C36 Aliphatic had a high recovery (131%), the lowest standard with acceptable recovery was the next lowest standard (20 ug/mL). C11-C22 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. |
| C19-C36 Aliphatics | High | Results Quantitation | Calculation error | Could not reproduce result for C19-C36 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. |
| Test America-Buffalo | | 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 | 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. |
| All hydrocarbon ranges | Low | Retention Time Windows | CAM method deviation | C9-C18 Aliphatics: window was 3.16-9.622 (should be 3.091-10.007): biased low.  C19-C36 Aliphatics: window was 10.082-17.786 (should be 10.007-17.817): biased low.  C11-C22 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 | | C9-C18 Aliphatics, C19-C36 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 C11-C22 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 C11-C22 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. |
| C9-C18 Aliphatics, C19-C36 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 C11-C22 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 C11- C22 aromatics as resulting CF is higher than if generated as per the method. |
| All hydrocarbon ranges | I | RLs/Sensitivity | CAM method deviation | C11-C22 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 C11-C22 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. |
| C9-C18 Aliphatics | I | Method Blank/GC Performance | Faulty chromatography | C9-C18 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 | C11-C22 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 C9-C18 aliphatic range, C19-C36 aliphatic range, and C11-C22 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, C9-C18 aliphatic and C11-C22 aromatic were acceptable, but the C19-C36 aliphatic had a low recovery using the next lowest standard with acceptable recovery. | \* | RLs misrepresented as lower than allowed by method for C19-C36 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. |
| C9-C18 Aliphatics, C19-C36 Aliphatics | High | Results Quantitation | CAM method deviation | Results re-quantified using CFs from summation of individual peaks. | \* | Sample recalculated using correct CFs:  C9-C18 Aliphatics: Laboratory result biased high by ~7%: 350 mg/kg vs 375 mg/kg.  C19-C36 Aliphatics: Laboratory result biased high by ~10%: 180 mg/kg vs 190 mg/kg. |
| SGS-Accutest | | No issues noted | | | | | | |
| 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. |
| C9-C18 Aliphatics, C19-C36 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. |
| C19-C36 Aliphatics | I | Continuing Calibration | Calculation error | The C19-C36 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:  C9-C18 Aliphatics: 21% higher.  C19-C36 Aliphatics: 6% higher.  C11-C22 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. |
| C9-C10 Aromatics | I | Initial Calibration | CAM method deviation | Quadratic regression used for C9-C10 aromatics although linear regression could have been used.  C9-C10 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. |
| C9-C10 Aromatics | Low | Results Quantitation | CAM method deviation | C9-C10 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 C9-C10 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.  C9-C12 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. |
| 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.  C5-C8 aliphatics: calculated 2.423; laboratory reported 2.95.  C9-C12 aliphatics: calculated 3.008; laboratory reported 4.04.  C9-C10 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 | C9-C12 Aliphatics | NA | Continuing Calibration | Calculation error | %D outside of acceptance criteria for C9-C12 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 | C5-C8 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.  C5-C8 Aliphatics: (original/final) ug/m3  Sample 1: 110/120  Sample 2: 76/91  Sample 3: 63/74  Sample 4: 84/90  C9-C12 Aliphatics: (original/final) ug/m3  Sample 1: 220/390  Sample 2: 120/230  Sample 3: 110/190  Sample 4: 140/260  C9-C10 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. |
| 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. |
| C5-C8 Aliphatics, C9-C12 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. |
| C5-C8 Aliphatics, C9-C12 Aliphatics | Low/High | Results Quantitation | CAM method deviation | For the adjusted C5-C8 aliphatic and C9-C12 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 C5-C8 aliphatics. If detected concentrations were subtracted, the adjusted C5-C8 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 C5-C8 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 C5-C8 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 C5-C8 aliphatics would be 130 ug/m3 and not 1720 ug/m3.  Sample 2: The adjusted C5-C8 aliphatics would be 11,009 ug/m3 and not 10,200 ug/m3.  Sample 3: The adjusted C5-C8 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 C5-C8 aliphatics, C9-C12 aliphatics, and C9-C10 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. |

# 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.

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* 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**

 

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

LAB

Attn: Lab Director Dear :

RE: Request For Information – MassDEP Data Audit

LAB

Data Package:

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-](http://www.mass.gov/eea/agencies/massdep/cleanup/regulations/wsc10-320-compendium--quality-control-reqs.html) [reqs.html](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](mailto: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

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

«LAB\_NAME»

«ADDRESS1» «ADDRESS2»

«CITYSTATE»

**Attn:** «LAB\_MANAGER» or Lab Director Dear«LAB\_MANAGER»:

RE: Request for Information – MassDEP Multi-Lab Data Audit Draft Audit Findings

«LAB\_NAME»

**(Identified as “Lab C”)**

Data Package: [Redacted]

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- reqs.html](http://www.mass.gov/eea/agencies/massdep/cleanup/regulations/wsc10-320-compendium--quality-control-reqs.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](http://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](mailto: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)

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







