State of Connecticut

Department of Energy and Environmental Protection

Recommended Reasonable Confidence Protocols

Quality Assurance and Quality Control Requirements

Volatile Petroleum Hydrocarbons

by the

Massachusetts DEP VPH Method

Version 3.0

May 2024

Written by the Connecticut DEEP QA/QC Workgroup

Revision	Comments	Date
1.0	Draft for Public Comment	November 30, 2008
2.0	Final version following public comment period	May 1, 2009
3.0	Updates to reflect CAM method updates to improve consistency between different states.	May 2024

Table of Contents

Table of Tables

Acronym List

ACRONYM	DEFINITION
APS	Additional Polluting Substances
CASN	Chemical Abstracts Service Number
CCV	Continuing calibration verification
COD	Chloro-octadecane
%D	Percent difference or percent drift
DEEP	CT Department of Energy and Environmental Protection
DF	Dilution factor
EP	Environmental Professional
FID	Flame Ionization Detection
g	grams
GC	Gas chromatograph
GC/MS	Gas chromatography/mass spectrometry
HCl	Hydrochloric acid
ICV	Initial calibration verification
LCS	Laboratory control sample
LCSD	Laboratory control sample duplicate
LLOQ	Lower Limit of Quantitation
mL	Milliliters
MD	Matrix Duplicate
MGP	Manufactured gas plant
MS	Matrix spike
MSD	Matrix spike duplicate
MSE	Microscale solvent extraction
MTBE	Methyl tertiary butyl ether
NA	Not applicable
OHM	Oil and Hazardous Materials
PAC	Peak area calculation
PFTE	Polytetrafluoroethylene
PID	Photoionization detector
QA	Quality assurance
QC	Quality control
r/r^2	Correlation coefficient
%R	Percent recovery
%RSD	Percent relative standard deviation
RCP	Reasonable Confidence Protocol
RL	Reporting limit
RPD	Relative percent difference
RSR/RSRs	Remediation Standard Regulations
SIM	Selective ion monitoring
SPE	Solid phase extraction
TPH	Total petroleum hydrocarbons
TSP	Trisodium phosphate dodecahydrate
UCM	Unresolved complex mixture
µg/kg	micrograms per kilogram
µg/L	micrograms per liter
µL	microliters
VOC	Volatile organic compound
VPH	Volatile petroleum hydrocarbons

1.0 Quality Assurance and Quality Control Requirements for MassDEP VPH Method

1.1 Method Overview

The Volatile Petroleum Hydrocarbons Method (the "VPH Method") uses purge-and-trap sample concentration, gas chromatographic ("GC") separation using photoionization and flame ionization detectors ("PID/FID") in-series. This method is designed to identify and quantify both target analytes and method-defined aliphatic and aromatic hydrocarbon fractional ranges in water, soils and sediments. Volatile aliphatic hydrocarbons are collectively quantified within two specific ranges: C_5 through C_8, and C_9 through C_{12}. Volatile aromatic hydrocarbons are collectively quantified within the C_9 to C_{10} range. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 36°C and 220°C. This method may also be used to identify and quantify benzene, toluene, ethylbenzene, xylenes ("BTEX"), naphthalene, and methyl tertiary butyl ether ("MTBE") as Target Analytes.

All references to SW-846 Methods (i.e., SW-846 8000, 8260, etc.) in this document refer to the United States Environmental Protection Agency's most recently published version. All references to "the VPH Method" in this document refer to latest promulgated version of the *Massachusetts DEP VPH Method*.

The use of the VPH Method is designed to complement and support the toxicological approach developed by the Connecticut Department of Energy and Environmental Protection ("DEEP") to evaluate human health hazards that may result from exposure to petroleum hydrocarbons. It is intended to produce data in a format suitable for evaluation by that approach.

Overall usability of data produced using this RCP protocol should be evaluated for compliance with project-specific data quality objectives, regardless of "Presumptive Certainty" status.

Petroleum products suitable for evaluation by the VPH Method include gasoline, mineral spirits, and certain petroleum naphthas. In and of itself, the VPH Method is not suitable for the evaluation of kerosene, jet fuel, heating oils, lubricating oils, and/or other petroleum products, which contain higher boiling components, or distillates of aliphatic and/or aromatic hydrocarbons that are beyond the analytical range of the VPH Method.

1.2 Summary of Method

The VPH Method is suitable for the analysis of waters, soils, sediments and non-aqueous phase liquids ("NAPL"). The method includes inert gas purging, of an aqueous sample or soil methanol extract, with concentration onto an adsorbent trap, and subsequent analyses by gas chromatography. The GC oven is temperature-programmed to facilitate separation of the analytes of interest. Detection is achieved by using a PID and FID operating in series. Quantitation is based on comparing the PID and FID detector response of a sample to a standard comprised of volatile aromatic and aliphatic hydrocarbons. The PID chromatogram is used to determine the individual concentrations of target analytes (BTEX/MTBE/naphthalene) and collective concentration of aromatic hydrocarbons within the C_9 through C_{10} range. The FID chromatogram is used to determine the collective concentration of aliphatic hydrocarbons within the C_5 through C_8 and C_9 through C_{12} ranges. The VPH method marker compounds and retention time windows are summarized in Table 1.0.

Table 1.0: VPH Method Marker Compounds

Range/ Hydrocarbon Standard	Beginning Marker Compound	Ending Marker Compound
C_5 - C_8 Aliphatic Hydrocarbons (FID)	0.1 minutes before n-Pentane	0.01 minutes before n-Nonane
C_9 - C_{12} Aliphatic Hydrocarbons (FID)	0.01 minutes before n-Nonane	0.1 minutes before Naphthalene
C_9 - C_{10} Aromatic Hydrocarbons (PID)	0.1 minutes after o-Xylene	0.1 minutes before Naphthalene

1.2.1 Sample Introduction Methods

As prescribed in the VPH Method, samples for analysis are introduced into the GC system using a purge-and-trap concentrator as described in SW-846 Methods 5030 and 5035 for aqueous and solid samples, respectively. If other sample introduction methods are utilized because of analytical circumstances, the laboratory must provide a full explanation and justification in the analytical case narrative.

1.2.2 Analysis of Water Samples

Water samples may be analyzed directly without sample preparation. The analysis of water samples is described in detail in the VPH Method. In general, a sample aliquot is introduced to the purge chamber using a 5 mL gas-tight syringe. If necessary, samples may be diluted prior to injection into the purge chamber. In such cases, sample dilutions must be performed as expeditiously as possible, and the diluted sample should be transferred to a gas-tight syringe without delay.

1.2.3 Analysis of Soil and Sediment Samples

Soil and sediment samples are dispersed in methanol to extract the volatile organic constituents. A portion of the methanol extract is then extracted/concentrated by purge-and-trap and analyzed by GC/PID/FID. Methanol may be added in the field or in the laboratory if the samples are collected in specially designed airtight samplers. The desired ratio of methanol-to-soil is 1 mL methanol/1 gram soil, ± 25%. Highly organic matrices (e.g., peat) may require additional methanol (up to 2 mL per gram of soil). In either case, an aliquot of the methanol extract is added to reagent water to produce a 5 mL adjusted sample volume and introduced into the GC using a purge and trap concentrator. The volume of the aliquot will depend on the anticipated VPH concentration. ***Be advised that the volume of methanol aliquot added to the sparging flask should not exceed 200 µL to preclude adverse solvent front and trap breakthrough difficulties.***

1.3 Method Interferences

1.3.1 Chemical Contaminants

Refer to SW-846 Method 8260 for a detailed description of chemical contaminants, cross-contamination, and corrective actions which may be taken to eliminate contamination. Analyses of calibration and reagent blanks provide information about the presence of contaminants. When potential interfering peaks are noted in blanks, the analyst should determine the cause of the contamination before re-analysis occurs. Corrective actions may include changing the purge gas source and/or regenerating the molecular sieve purge gas filter. **Subtracting blank values from sample results is not permitted.**

Impurities in the purge gas, and from organic compounds out-gassing from the plumbing ahead of the trap, account for most system contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic tubing, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interferences and/or false positives.

Cross-contamination may occur when any sample is analyzed immediately after a sample containing high concentrations of volatile organic compounds. After the analysis of a sample containing high concentrations of volatile organic compounds (including VPH target analytes and ranges), one or more blanks should be analyzed to check for potential cross-contamination/ carryover. The laboratory must determine individual VOC concentrations that cause a cross-contamination/carryover condition. Manifestation of this condition is dependent upon the concentration and level of detector saturation for the particular analyte. Concentrations of VOCs, which exceed the upper limit of calibration, should prompt the analyst to check for potential cross-

contamination/carryover. In addition, samples containing large amounts of water-soluble materials, suspended solids, or high boiling point compounds may also present potential for cross-contamination/carryover. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later sample analysis.

1.3.2 Other Potential Interferences

Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and chlorofluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free reagent water (for aqueous samples) or methanol (for soil and sediment samples), and carried through sampling and handling protocols, serves as a check on such contamination.

1.3.3 General Precautions

As a general precaution, the laboratory where VPH and other volatile analyses are performed should be completely free of uncontained solvents. The analytical and sample storage areas should be isolated from all sources of potentially interfering volatile organics. All GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to potentially interfering volatile organics during common laboratory activities can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed can also lead to random elevated background concentrations of volatile organics and the same precautions must be taken.

1.4 Quality Control Requirements for the MassDEP VPH Method

1.4.1 Reporting Limits/Lower Limits of Quantitation for the VPH Method

The reporting limits (“RL”), or lower limits of quantitation (“LLOQ”), reflect the sampling procedures and the prescriptive analytical conditions imposed by the VPH method. The RL/LLOQs are dependent on the concentration of the lowest non-zero analytical standard in the initial calibration and/or percent solids of the sample. RL/LLOQs for VPH target analytes and hydrocarbon ranges will be proportionately higher for samples that require dilution or when a reduced sample size is used. Table 2.0 lists approximate RL/LLOQs for various matrices utilizing GC/PID/FID. Solid matrices in this table assume 100% solids.

Table 2.0: Typical Reporting Limits / Lower Limits of Quantitation[1]

Analyte	Matrix	Typical Reporting Limit
Aliphatic & Aromatic Ranges	Water	100 to 150 µg/L
	Soil	5,000 to 1,000 µg/kg
VPH Target Analytes	Water	1 to 5 µg/L
	Soil	50 to 250 µg/kg

[1]Note these values are intended to serve as guidance to EPs when planning analytical needs to achieve the data quality objectives to meet project-specific goals. These tables are not intended to dictate what RL/LLOQs laboratories must report.

Moisture content of soils and sediments will raise the RL/LLOQ, as all results must be reported on a dry weight basis for these two matrices. Sample dilution or lower sample weight/volume will also cause the RL/LLOQs to be raised. It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL/LLOQ for the target analytes to meet the project Data Quality Objectives (“DQOs”). To meet the RLs/LLOQs applicable to project DQOs, it may be necessary to modify the analytical method by using increased sample volume or mass or employing selective ion monitoring. In such cases the modifications must be noted in the laboratory report narrative.

1.4.2 General Quality Control Requirements

This protocol is restricted to use by, or under the supervision of, analysts experienced in the use of purge and trap systems, the use of GC instrumentation as a quantitative tool, and skilled in the interpretation of gas chromatograms for individual target analytes and petroleum hydrocarbon ranges in environmental matrices. Each analyst must demonstrate the ability to produce acceptable quantitative and qualitative results both for individual target analytes and petroleum hydrocarbon ranges with this method.

Refer to SW-846 Method 8000 for general quality control ("QC") procedures for all chromatographic methods, including the VPH Method. These requirements ensure that each laboratory maintain a formal QA program and records to document the quality of all chromatographic data and be certified by the Connecticut Department of Public Health for the analysis performed. QC procedures necessary to evaluate the GC system operation may be found in the VPH Method including evaluation of calibrations and chromatographic performance of sample analyses. Instrument quality control and method performance requirements for the analytical system may be found in the VPH Method, Sections 10.0 and 13.0, respectively.

The minimum requirements for a formal QA program include Initial Demonstration of Capability ("IDOC"), ongoing analysis of standards and blanks to confirm acceptable continuing performance, and analysis of laboratory control samples ("LCS") and/ or matrix spikes ("MS") to assess accuracy and LCS duplicates ("LCSD") and matrix spike duplicates ("MSD") to assess precision. Matrix duplicates ("MD") may also be used to evaluate precision when such samples are analyzed either at discretion of the laboratory or at the request of the data-user. The use of site-specific MS/MSD's is highly recommended.

Evaluation of sample matrix effects on compound recovery is key to making informed decisions. Percent recovery data from site-specific samples allow the environmental professional ("EP") to make informed decisions regarding contamination levels at the site. Batch MS/MSD results do not give any indication of site-specific matrix interferences or analytical problems related to the specific site matrices. Field, rinsate, or other blanks should not be used for MS/MSD's.

Laboratories must document and have on file an IDOC for each combination of sample preparation and determinative analytical method in use. An IDOC must be completed and documented when a method is initially started up, whenever a method is substantially modified, or new laboratory staff is trained to perform the VPH Method. These data must meet or fall within the performance standards as presented in Section 1.4 and Table 1A of this RCP, in the VPH Method, and as presented in SW-846 Method 8000. Procedural requirements for performing the IDOC can be found in SW-846 Method 8000 and in the VPH Method. The IDOC must include the following elements provided in Table 3.0:

Table 3.0: IDOC Requirements

QC Element	Performance Criteria
Initial Calibration	Table 1A of this RCP
Continuing Calibration	Table 1A of this RCP
Laboratory Method Blanks	Table 1A of this RCP
Laboratory Control Samples	The VPH Method
Surrogate Recovery	Table 1A of this RCP

Because of the inherent difficulty in quantifying fractional hydrocarbon ranges and the number of QC elements associated with the IDOC, it should be expected that one or more of the ranges and/or target analytes may not meet the performance standard for one or more QC elements. Under these circumstances, the analyst should attempt to locate and correct the problem and repeat the analysis for all non-conformances. All non-conformances, along with the laboratory-specific acceptance criteria should be noted in the IDOC data. This information should be kept on-file at the laboratory.

Laboratories are required to generate laboratory specific performance criteria for LCS compound recovery limits, matrix spike/matrix spike duplicate compound recovery and relative percent difference (“RPD”) limits, and surrogate recovery limits. These limits must be equal to or fall within the limits specified in Table 1A.

1.4.3 Use of Surrogates, and Matrix Spikes (MS) and Matrix Spike Duplicates (MSD) with Methanol Preserved Soil/Sediment Samples

The recovery of surrogates and matrix spikes from a soil/sediment sample that has been preserved with methanol cannot be used to directly evaluate matrix-related bias/accuracy in the conventional definition of these terms. QC parameters expressed in terms of these percent recoveries (“%R”) may be more indicative of the variabilities associated with the analytical system (sample processing, introduction, and/or component separation and quantitation).

Because of this limitation, it is recommended that the laboratory consider adopting alternative quality control elements for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies. Recommended practices for additional quality assurance may be found in SW-846 Methods 5000 and 8000.

This inherent limitation associated with the evaluation of matrix spike and surrogate recoveries attributable to methanol preservation of soil and sediment samples is more than compensated for by the marked improvement in sample integrity and conservation/recoveries of the volatile analytes of concern from soil and sediment matrices.

1.4.4 Specific QA/QC Requirements and Performance Standards for the VPH Method

Specific QA/QC requirements and performance standards for the VPH Method are presented in Table 1A. Strict compliance with the QA/QC requirements and performance standards for this method, as well as satisfying other analytical and reporting requirements will provide the environmental professional with “Reasonable Confidence” regarding the usability of analytical data to support environmental decisions. The concept of "Reasonable Confidence" is explained on the DEEP website.

While optional, parties electing to utilize these protocols will be assured that agency reviewers will, generally accept "Reasonable Confidence" data. To achieve “Reasonable Confidence” parties must:

1. Comply with the applicable QC analytical requirements prescribed in Table 1A for this test procedure;

2. Evaluate and narrate all protocol non-compliances and implement, as necessary, required corrective actions and analytical response actions for all non-conforming analytical performance standards; and

3. Retain reported and unreported analytical data and information for a period of 5 years or as required under applicable accreditation criteria.

Table 1A-Specific QA/QC Requirements and Performance Standards for the VPH Method

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Capability ("IDOC")	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain all aliphatic and aromatic hydrocarbon standards listed in the VPH method. (4) Must follow procedure in the VPH Method.	No	Refer to the Appendix of the VPH by GC/PID/FID method and Section 1.4.2 of this protocol.	NA
GC Performance	Inter-laboratory consistency and comparability	(1) n-Pentane and MTBE must be resolved from solvent front. (2) Surrogate standards must be resolved from target compounds.	No	Perform instrumentation/injection port maintenance as necessary	Suspend all analyses until performance criteria are met. Report non-conformances in laboratory report narrative.
Retention Time Windows	Laboratory Analytical Accuracy	(1) Prior to initial calibration and when a new GC column is installed. (2) Calculated according to the VPH method. (3) Retention time windows must be updated with every CCV.	No	NA	NA

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Initial Calibration ("ICAL")	Laboratory Analytical Accuracy	(1) Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed. (2) Minimum of 5 standards (or 6 if non-linear regression used). (3) Low standard must be ≤RL/LLOQ. (4) Must contain all aliphatic and aromatic hydrocarbon standards listed in Table 1 of VPH by GC/PID/FID method. (5) %RSD ≤20 for target VPH analytes and ≤25 for hydrocarbon ranges, r ≥0.99 (linear) and r^2 ≥0.99 (non-regression) for each Target VPH Analyte and hydrocarbon range. (6) If %RSD >20 for target VPH analytes and >25 for hydrocarbons ranges, linear or non-linear regression must be used. (7) Must meet GC performance standards described in the VPH by GC/PID/FID method. (8) Calibration must be performed under the same conditions as the samples (heated purge). (9) If autosampler used to spike surrogates in calibration with 5 standards is acceptable for surrogates. (10) If linear or nonlinear regression used, verify the RL/LLOQ by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%. (11) If regression used, curve must not be forced through the origin.	No	(1) Recalibrate as required by method. (2) In case of linear or non-linear regression, if recalculated concentrations from the lowest calibration standard are outside of 70-130% recovery range, either: *The RL/LLOQ must be reported as an estimated value, or *The RL/LLOQ must be raised to the concentration of the next highest calibration standard that exhibits acceptable recoveries when recalculated using the final calibration curve.	Sample analysis cannot proceed without a valid initial calibration. If non-linear regression (quadratic equation) is used for calibration, this must be noted in the lab laboratory report narrative along with the target VPH analytes or hydrocarbon ranges affected.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Initial Calibration Verification ("ICV")	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Concentration level near midpoint of curve. (3) Prepared using standard source different than used for initial calibration. (4) Must contain all aliphatic and aromatic hydrocarbon standards listed in Table 1 of the VPH Method. (5) Percent recoveries must be between 70-130% for each target VPH Analyte and hydrocarbon range.	No	Locate source of problem; recalibrate if >10% of all analytes are outside of criteria.	If recovery is outside of 70-130% for any target VPH analyte or hydrocarbon range, report non-conforming analyte or hydrocarbon range in laboratory report narrative.
Continuing Calibration ("CCV")	Laboratory Analytical Accuracy	(1) Prior to samples, every 20 samples and at the end of the analytical run. (2) Concentration level near midpoint of curve. (3) Must contain all aliphatic and aromatic hydrocarbon standards listed in the VPH method. (4) Must meet GC performance standards described in the VPH method. (5) %D must be ≤20 for all target VPH analytes and ≤25 for hydrocarbon ranges except for n-nonane, which must be ≤30. (6) Verify that all analytes fall within retention time windows.	No	(1) Perform instrument maintenance, reanalyze continuing calibration and/or recalibrate as required by method. (2) Reanalyze "associated samples" if beginning or ending continuing calibration exhibited low response. (3) Reanalyze "associated samples" if beginning or ending continuing calibration exhibited high response and associated target VPH analytes and hydrocarbon ranges were detected in the "associated samples." NOTE: "associated samples" refers to all samples analyzed since the last acceptable continuing calibration.	Report non-conforming target VPH analytes (%D >20) or hydrocarbon ranges (%D >25) and "associated samples" in laboratory report narrative.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Method Blank ("MB")	Laboratory Method Sensitivity (Contamination Evaluation)	(1) Analyzed with every batch or every ≤20 field samples, whichever is more frequent. (2) Matrix specific (e.g., aqueous, soil) (3) VPH hydrocarbon ranges must be <RL/LLOQ of the most stringent applicable RSR (or "APS") standards for solid samples and aqueous samples. (4) Target VPH analytes must be <RL/LLOQ.	Yes	(1) If concentration of contaminant in sample is <10x concentration in blank, locate source of contamination; correct problem; re-analyze method blank and associated samples. (2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.	(1) If sample reanalysis is not possible, report non-conformance in the laboratory report narrative. (2) If contamination of method blanks is suspected or present, the lab, using "B" flag or some other convention, should qualify the sample results. Blank contamination should also be documented in the laboratory report narrative. (3) If re-analysis is performed within holding time and yields acceptable method blank results, the lab may report results of the re-analysis only. (4) If reanalysis is performed outside of holding time, the lab must report results of both the initial analysis and re-analysis.
Laboratory Control Sample ("LCS")	Laboratory Analytical Accuracy & Precision	(1) Analyzed with every batch or every ≤20 field samples, whichever is more frequent. (2) Prepared using standard source different than used for initial calibration. (3) Concentration level near midpoint of curve. (4) Must contain all aliphatic and aromatic hydrocarbon standards listed in the VPH method. (5) Matrix and preservative-specific (e.g., aqueous, soil). (6) Percent recoveries must be between 70-130% for target VPH analytes and hydrocarbon ranges.	Yes	(1) Locate source of problem; re-analyze LCS and associated samples if target VPH analytes or hydrocarbon ranges are outside of criteria. (2) If target VPH analytes or hydrocarbon ranges are above the acceptance criteria (>130%), re-analysis is not required if affected target VPH analytes or hydrocarbon ranges were not detected in associated samples. (3) If LCS is re-analyzed and still outside of criteria, recalibration is required. (4) Recalculate % recoveries.	(1) If sample re-analysis is not possible, report non-conformance in laboratory report narrative. (2) If recovery is outside of 70-130% for any analyte, report non-conforming target VPH analytes or hydrocarbon ranges in laboratory report narrative. (3) If re-analysis is performed within holding time and yields acceptable LCS results, the lab may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time, the lab must report results of both the initial analysis and re-analysis.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
LCS Duplicate ("LCSD")	Laboratory Analytical Accuracy & Precision	(1) Analyzed with every batch or every 20 samples, whichever is more frequent. (2) Prepared using standard source different than used for initial calibration. (3) Concentration level near midpoint of curve. (4) Must contain all aliphatic and aromatic hydrocarbon standards listed in the VPH method. (5) Matrix and preservative specific (e.g., aqueous, soil). (6) Percent recoveries must be between 70-130% for target VPH analytes and hydrocarbon ranges. (7) RPDs must be ≤25 for waters and solids.	Yes	(1) Locate source of problem; re-analyze LCS and associated samples if Target VPH analytes or hydrocarbon ranges are outside of recovery acceptance criteria. (2) If target VPH analytes or hydrocarbon ranges are above the recovery acceptance criteria (>130%), re-analysis is not required if affected target VPH analytes or hydrocarbon ranges were not detected in associated samples. (3) If LCS is re-analyzed and still outside of criteria, recalibration is required.	(1) If sample re-analysis is not possible, report non-conformance in laboratory report narrative. (2) If recovery is outside of 70-103% or RPD >25 for any analyte, report non-conforming target VPH analytes or hydrocarbon ranges in laboratory report narrative. (3) If re-analysis is performed within holding time and yields acceptable LCS results, the lab may report results of the re-analysis only. (4) If re-analysis is performed outside of holding time, the lab must report results of both the initial analysis and re-analysis.
Matrix Spike / Matrix Spike Duplicate ("MS/MSD") (Site specific)	Method Accuracy & Precision in Sample Matrix	(1) Every ≤20 samples (at discretion of lab or at request of data user). (2) Matrix and preservative specific, (e.g., aqueous, soil). (3) Must contain all aliphatic and aromatic hydrocarbon standards listed in the VPH method. (4) Percent recoveries must be between 70-130% for target analytes and hydrocarbon ranges (5) RPD's should be ≤50% for waters and solids. (6) Prepared using standard source different than used for initial calibration. (7) Concentration level near midpoint of curve. (8) Field blanks, trip blanks, etc. cannot be used for MS/MSDs.	Yes (when requested by data user).	Check LCS; if recoveries are acceptable in LCS; narrate non-conformance.	Note non-conformances in laboratory report narrative.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Matrix Duplicates ("MD")	Method Precision in Sample Matrix	(1) Every 20 samples (at discretion of lab or at request of data user). (2) Matrix and preservative specific (e.g., aqueous, soil). (3) RPDs ≤50% for waters and solids for results >5x the reporting limit.	Yes (when requested by data user).	Narrate non-conformance.	Note exceedances (RPDs >50%) in laboratory report narrative.
Surrogates	Method Accuracy in Sample Matrix	(1) Minimum of 1 surrogate. Recommended Surrogate: 2,5-dibromotoluene (2) Percent recoveries must be between 70-130% on PID and FID.	Yes (report recovery from PID and FID)	If surrogate is outside of limits, reanalyze sample unless one of the following exceptions applies: (1) Obvious interference present (e.g., UCM). NOTE: If obvious interference is present and surrogate recovery would cause rejection of data (i.e., <10%) reanalyze sample on dilution. (2) Methanol-preserved samples: re-analysis is not required if % moisture >25 and surrogate recovery is >10%. (3) If surrogate exhibits high recovery and associated target VPH analytes or hydrocarbon ranges are not detected in sample, reanalysis is not required. NOTE: Surrogate recoveries from PID affect target VPH analytes and C_9-C_{10} aromatics. Surrogate recoveries from FID affect C_5-C_8 aliphatics and C_9-C_{12} aliphatics.	(1) Report recoveries outside of 70-130% in laboratory report narrative. (Note non-conformances in laboratory report narrative). (2) If re-analysis yields similar surrogate non-conformances, the lab must report results of both analyses. (3) If re-analysis is performed within holding time and yields acceptable surrogate recoveries, the lab may report results of the re-analysis only. (4) If re-analysis is performed outside of the holding time and yields acceptable surrogate recoveries, the lab must report both analyses. (5) If sample is not reanalyzed due to obvious interference, the lab must provide the chromatogram in the data report.

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
Quantitation	NA	(1) The lab must use the average calibration factor, linear or non-linear regression curve generated from the associated initial calibration for quantitation of each target VPH analyte and hydrocarbon range. (2) Do not report concentrations below the RL/LLOQs.	NA	NA	NA
Identification	NA	(1) Refer to the VPH method.	NA	NA	NA

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable	Required Corrective Action	Required Analytical Response Action
General Reporting Issues	NA	(1) The lab must only report values ≥ the sample-specific R/LLOQ. (2) Dilutions: If diluted and undiluted analyses are performed, the lab should report results for the lowest dilution within the valid calibration range for each target VPH analyte and hydrocarbon range.The associated QC (method blanks, surrogates, etc.) for each analysis must be reported. (3) All soil/sediment sample results must be corrected for the methanol dilution as per the VPH method. (4) Results for soil/sediments must be reported on a dry-weight basis. (5) The lab must report the GC column used (manufacturer, column name, length, ID and film thickness). (6) The lab must report the trap used in the purge-and-trap system (manufacturer, trap contents).	NA	NA	(1) The performance of dilutions must be documented in the laboratory report narrative. Unless due to elevated concentrations of target VPH analytes or hydrocarbon ranges, reasons for dilutions must be explained in the laboratory report narrative. (2) Complete analytical documentation for diluted and undiluted analyses must documented in laboratory report narrative and be maintained in laboratory records. (3) If samples are not properly preserved (pH>2 for aqueous samples, solid samples not completely covered with methanol preservative, and/or solid sample/methanol ratio outside 1:1 ±25%) or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory report narrative. (4) If samples are preserved and/or analyzed outside of holding time, note the non-conformance in the laboratory report narrative.

1.5 Special Analytical Considerations

Appropriate surrogates and full matrix spikes must be added to the methanol extract through the septum seal prior to equilibration of the sample to room temperature. All samples should be shaken for 2 minutes to assure adequate mixing prior to analysis. A 100 microliter (µL) aliquot (or other appropriate volume) of the methanol extract must then be removed and added to reagent water to provide a 5 mL "adjusted" sample volume.

1.6 Analyte List for the VPH Method

The analyte list for the VPH Method is presented in Table 1B. The list is comprised of three (3) collectively quantified volatile hydrocarbon ranges and eight (8) Target Analytes, as identified in the VPH Method, that are readily analyzable by the method using conventional purge-and-trap sample introduction via SW-846 Methods 5030 (ambient temperature) and/or 5035 for aqueous and solid samples, respectively. Use of the VPH Method to identify and quantify the listed Target Analytes is optional at the discretion of the environmental professional.

Table 1B: Analyte List for the VPH Method

Range/ Target Analyte	CAS No.
Volatile Petroleum Hydrocarbon Ranges	
C_5 - C_8 Aliphatic Hydrocarbons	NA
C_9 - C_{12} Aliphatic Hydrocarbons	NA
C_9 - C_{10} Aromatic Hydrocarbons	NA
Target Analytes	
Benzene	71-43-2
Ethylbenzene	100-41-4
Methyl-tert-butylether (MTBE)	1634-04-4
Naphthalene	91-20-3
Toluene	108-88-3
o-Xylene[2]	95-47-6
m-Xylene[1,2]	108-38-3
p-Xylene[1,2]	106-42-3
[1]May not be resolvable under chromatographic conditions required under this Method. [2]May be reported and evaluated as mixed isomers	

1.6.1 Additional Reporting Requirements for the VPH Method

While it is not necessary to request and report all the VPH Analytes listed in Table 1B, it is required to quantify the VPH aliphatic and aromatic hydrocarbon ranges, in the same table, to obtain Reasonable Confidence status. Such limitations must be documented for site characterization and data representativeness considerations. DEEP strongly recommends use of the full analyte list during the initial stages of site investigations, and/or at sites with an unknown or complicated history of uses of oil or hazardous materials.

In cases where a shortened list of analytes is selected, the laboratory must still meet the method specific quality control requirements and performance standards associated with the requested analytes list to obtain Reasonable Confidence.

1.7 Routine Reporting Deliverables for the VPH Method

The following table (Table 4.0) lists the routine report deliverables. Note that while laboratories are not required to report certain items, they must keep the data on file and may be required to report these items in special circumstances.

Table 4.0: Report Deliverables

Parameter	Deliverable	Comments
Retention Time Windows	NO	Note non-conformances in laboratory report narrative
Initial Calibration	NO	Note non-conformances in laboratory report narrative
Continuing Calibration Verification	NO	Note non-conformances in laboratory report narrative
Method Blanks	YES	Note non-conformances in laboratory report narrative. Flag all positive results above RL/LLOQ with “B” flag.
Lab Control Sample	YES	Note non-conformances in laboratory report narrative
Site Specific Matrix Spike/ Matrix Spike Duplicate	YES (If requested)	Note non-conformances in laboratory report narrative
Surrogate Recoveries	YES	Note non-conformances in laboratory report narrative
Internal Standard Areas	NO (If used)	Note non-conformances in laboratory report narrative
General Reporting Issues	YES	Note non-conformances in laboratory report narrative. Required data reporting content is presented in the VPH Method
QA/QC Certification Form	YES	Signed by laboratory director or their designee.
Chain-of-Custody Form	YES	Signed by sample collector, courier, and laboratory.

1.7.1 Reporting and Flagging of Results

The following rules apply to reporting results:

- Non-Detects: Report all non-detects and results below the reporting limit as “ND” (Not Detected at the Specified RL/LLOQ). The RL/LLOQ for each compound in each sample must be listed on the report, based upon the lowest calibration standard, the exact sample mass, any dilution factors, percent moisture, etc.

- Compounds detected above the RL/LLOQ in blanks and in samples shall be flagged with a “B” suffix (e.g., 25B).

- All soil/sediment results shall be reported on a dry weight basis.

1.8 Sample Collection, Preservation and Holding Times

Sample preservation, container and analytical holding time specifications for surface water, groundwater, soil, and sediment matrices for VPH samples are listed in Table 5.0. Samples should be collected in accordance with the CTDEP *Guidance for Collecting and Preserving Soil and Sediment Samples for Laboratory Determination of Volatile Organic Compounds*, Version 2.0, February 28, 2006.

Methanol preservation of soil/sediment samples is mandatory.

Table 5.0: Sample Containers, Preservation, and Holding Times

Matrix	Analyte	Container[1]	Preservation[2]	Holding Time
Aqueous (ambient temperature)	All VOC's with purge & trap ≤40ºC.	(2) x 40-mL VOC vials with Teflon lined screw caps protected from light	Adjust to pH < 2 with either HCl or sodium bisulfate at time of collection. Store at 4 ± 2º C.	14 days
Aqueous (using heat purge >40°C)	VOC's + MTBE with purge & trap >40ºC.[3]	(2) x 40-mL VOC vials with Teflon lined screw caps protected from light	Adjust to pH > 11 with 0.7 g trisodium phosphate at time of collection. Store at 4 ± 2º C.	14 days
Soil and Sediment samples[6]	All VOC's with purge & trap ≤ 40ºC.	Extrude soil/sediment sample directly into a pre-weighed vial* w/ Teflon-lined septa screw caps: Vials must contain 1 mL purge-and-trap grade methanol for every gram soil/sediment. *(1) x 60-mL vial or (1) x 40-mL vial	1 mL methanol for every gram soil/sediment; add methanol before or at time of sampling; methanol must cover soil/sediment sample. Store at 4 ± 2º C. 60-mL vial: 25g soil/sediment and 25 mL methanol 40-mL vial: 15g soil/sediment and 15 mL methanol	28 days if preserved. 48 hours if unpreserved[5] (EnCore™ sampler[4]).
High Conc. Waste Samples	All VOC's	Collect in screw top jar protected from light.	Cool 4 ± 2º C.	14 days

[1]The number of sampling containers specified is not a requirement. For specific analyses, the collection of multiple sample containers is encouraged to avoid resampling if sample is consumed or compromised during shipping and/or analysis.

[2]If samples effervesce upon addition of hydrochloric acid or sodium bisulfate, samples must be collected unpreserved and stored at 4 ± 2º C. Holding time is 7-days from collection.

[3]Heated purge (>40ºC) is considered a significant modification to the method, as per the VPH method.

[4]EnCore™ Type samplers may not be suitable for all soil types. See Method 5035 in SW-846 and the DEP *Guidance For Collecting And Preserving Soil and Sediment Samples for Laboratory Determination of Volatile Organic Compounds, ver. 2.0 Feb. 28, 2006* for guidance.

[5]If the freezing option is selected, the sample must be frozen within 48 hours of collection. The holding time recommences when thawing begins. The total holding time is calculated from the time of collection to freezing plus the time allowed for thawing. The total elapsed time must be less than 48 hours. Samples must be transferred to methanol prior to analysis.

[6]An extra aliquot of sample must be collected in a 4 oz. glass jar with no preservative so that the laboratory can perform a percent solids analysis. If the same sample is being submitted to the laboratory for additional analyses, which require no preservative, the percent solids analysis can be measured using an aliquot from these bottles. Otherwise, a separate bottle will be needed.

Appendix 1: VPH Data Usability

A-1 Data Usability Assessment for the VPH Method

Overall data usability is influenced by uncertainties associated with both sampling and analytical activities. This document provides detailed quality control requirements and performance standards for the VPH Method, which may be used to directly assess the analytical component of data usability. The sampling component of data usability, an independent assessment of the effectiveness of sampling activities to meet data quality objectives, is not substantively addressed in this document.

A-1.1 Specific Guidance Regarding the Interpretation and Use of VPH Data

The VPH Method produces both analyte-specific (target analytes) and method defined (hydrocarbon fractions) data. An analyte-specific approach produces data by comparing the response of a known analyte with an unknown concentration to the response of a standard for the same analyte with a known concentration under the same analytical conditions. A method defined approach produces data by prescriptively defining both analytical conditions and assumptions used to calibrate and interpret the data produced. Such an approach is particularly useful in determining average characteristics for a limited set of analytes with similar physical, chemical and toxicological properties (i.e., the collective concentration of a limited range of hydrocarbons). However, a clear understanding of the analytical limitations of the method and assumptions used to interpret data are required to maximize the potential of using this approach. Both VPH Target Analytes and hydrocarbon ranges are subject to potential "false positive" bias associated with non-specific gas chromatographic analysis. That is (1) other compounds coeluting at the specified retention time may be incorrectly identified and/or quantified (false positive) as a Target Analyte; (2) compounds not meeting the regulatory definition of the aromatic and/or aliphatic fractions defined in the VPH Method, that may elute within the method-defined retention time window would be included in the Peak Area Calculation (“PAC”) and result in an overestimation of a fraction’s concentration; or, (3) as described in the VPH Method, non-aromatic compounds that may elute between o-xylene and naphthalene and elicit a positive response on the PID would be included in the PAC, resulting in an overestimation of the C_9 through C_{10} aromatic fraction’s concentration.

Confirmatory analysis by a Gas Chromatography/Mass Spectroscopy (“GC/MS”) procedure or other suitable method, is recommended in cases where a VPH Target Analyte reported by this method exceeds an applicable reporting or cleanup standard, and/or where co-elution of a hydrocarbon compound not meeting the regulatory definition of a specific hydrocarbon fraction is suspected. Dual-column confirmation is suitable for Target Analytes only.

The following definitions are provided to assist in the interpretation and evaluation of Volatile Petroleum Hydrocarbon data:

Aliphatic Hydrocarbon: Any organic compound comprised solely of carbon and hydrogen characterized by a straight, branched or cyclic chain of carbon atoms. By definition, this class of organic compounds includes alkanes, alkenes, alkynes, cycloalkanes or cycloalkenes for the VPH methodology.

Aromatic Hydrocarbon: Any cyclic and conjugated organic compound comprised solely of carbon and hydrogen. Aromatic compounds of environmental significance are benzoids that contain benzene or fused benzene rings.

Volatile Petroleum Hydrocarbon: Any hydrocarbon that elutes within the C_5 through C_8, and C_9 through C_{12} aliphatic ranges or the C_9 through C_{10} aromatic ranges defined by the method. The definition of Volatile Petroleum Hydrocarbon specifically **excludes** all substituted aliphatic or aromatic hydrocarbon derivatives (non-hydrocarbons as defined by the VPH Method), the individual VPH Method Target Analytes and/or surrogates that co-elute within these method specific ranges. The VPH Method is suitable for the separation and quantification of the aliphatic and non-target aromatic components of gasoline, mineral spirits, certain petroleum naphthas and components of kerosene, jet fuel, heating oils, lubricating oils, and/or other petroleum products contained within the aforementioned method-defined ranges.

A-1.1.1 Interfering Peaks in Specified Aliphatic Hydrocarbon Ranges

Hydrocarbons (and non-hydrocarbons), even with elution times within the defined chromatographic windows for the aliphatic hydrocarbon ranges specified by the VPH Method, need not be included in the PAC for these ranges unless they meet the definitions of aliphatic hydrocarbon and volatile petroleum hydrocarbon, as defined above. If the concentration of a hydrocarbon range is based on one (or just a few) peaks within the range and an indicative petroleum hydrocarbon peak pattern is not apparent, the laboratory should provide this information and alert the data user of the potential for a false positive result in the laboratory report narrative. Sites with chlorinated hydrocarbons, ketones, and/or commingled non-petroleum hydrocarbons are subject to this interference.

A-1.1.2 Interfering Peaks in Specified Aromatic Hydrocarbon Range

The VPH Method should be used with caution at sites with an uncertain history, particularly closed or abandoned Manufactured Gas Plants ("MGPs"). Styrene, a common contaminant of concern ("COC") at many MGP sites, cannot be satisfactorily resolved from o-xylene under the chromatographic conditions specified for the VPH Method. If encountered, co-eluting styrene could cause an overestimation of o-xylene and a subsequent underestimation of the C_9-C_{10} aromatic range when the overestimated o-xylene peak is subtracted from the PAC for the range. Other contaminant pairs routinely encountered at sites that are difficult to resolve under the chromatographic conditions specified for the VPH Method include 1,2-dichloroethane/benzene and 1,1,1,2-tetrachloroethane/ethylbenzene.

A-1.1.3 Evaluation of Interfering Compounds Not Associated with a Petroleum Product

In general, it may be prudent to confirm all PID/FID data by SW-846 Method 8260 (GC/MS) if critical decision making (notification, compliance with cleanup standards, risk assessment, etc.) is based solely on the VPH Method (or any other non-specific GC analysis). If a positive interference is suspected from hydrocarbons and/or non-hydrocarbons not associated with VPH in either an aliphatic or aromatic fraction or with a Target Analyte, and such interference would adversely affect decision making, if confirmed, then SW-846 Method 8260, Volatile Organics by GC/MS, should be employed to accurately identify and quantify the components that comprise the fraction or to resolve the analyte pairs. It is recommended that the chromatographic conditions specified under SW-846 Method 8260 be modified for consistency with the conditions specified by the VPH Method to better allow for a direct comparison of the suspect PID/FID peaks with the GC/MS system. This is particularly useful when comparing suspect aliphatic hydrocarbons. The electron impact mass spectra for aliphatic hydrocarbon homologues are not particularly unique and chromatographic relative retention time data may also be required to confirm VPH data.

A-1.1.4 PID Response to Non-Aromatic Compounds

Although not a predominant component in petroleum hydrocarbon mixtures, alkenes and other non-aromatic hydrocarbons can elicit a positive PID response. In general, the PID response to these non-aromatic compounds is weaker than the response for the same mass of an aromatic hydrocarbon. However, at elevated concentrations, these non–aromatic compounds may interfere or yield false positives (high positive bias) to aromatic target analytes or range concentrations. This condition can be somewhat mitigated by using a lower energy lamp in the PID assembly of the gas chromatograph. Such a change would result in a loss of sensitivity and is considered a major instrument modification that would require re-calibration, a redemonstration of performance and documentation in the laboratory report narrative.

A-1.2 Substitution of GC/MS for the Identification and Quantification of VPH Ranges and Target Analytes

Consistent with the VPH Method, substitution of GC/MS for conventional GC detection for the quantification of VPH ranges is considered a “significant modification”. Modifications to the VPH Method are permissible, provided that adequate documentation exists or has been developed, to demonstrate an equivalent or superior level of performance. Be advised, however, that any adaptation to the VPH Method that constitutes a “significant modification” will preclude obtaining “Reasonable Confidence” status for any analytical data produced using such modification and must be disclosed and described on the data report form, as detailed in the VPH Method.

Any major modification to the VPH Method is deemed to satisfy the requirement “to demonstrate an equivalent or superior level of performance” for the determination of the collective concentrations of specified VPH aliphatic and aromatic ranges in water and soil/sediment matrices when:

1. The analytical data produced by the candidate method modification is in a format that is suitable for the evaluation using the toxicological approach developed by the Connecticut Department of Environmental Protection to evaluate human health hazards that may result from exposure to petroleum hydrocarbons;

2. The analytical data produced by the candidate method modification for both the VPH aliphatic and aromatic ranges and target analytes must have the requisite accuracy and precision to be compared to reporting and cleanup standards (which will be site specific alternative criteria until such time as specific reporting and cleanup standards are promulgated in the Remediation Standard Regulations) and consistent with the analytical data quality requirements of the Reasonable Confidence Protocols;

3. The reported concentration for the C_5 -C_8 Aliphatic Hydrocarbon range includes the preponderance of the individual C_5 through C_8 aliphatic hydrocarbon compounds contained in the subject petroleum product in the matrix of interest associated with a release to the environment;

4. The reported concentration for the C_9 - C_{12} Aliphatic Hydrocarbon range includes the preponderance of the individual C_9 through C_{12} aliphatic hydrocarbon compounds contained in the subject petroleum product in the matrix of interest associated with a release to the environment; and,

5. The reported concentration for the C_9 - C_{10} Aromatic Hydrocarbon range includes the preponderance of individual C_9 through C_{10} aromatic hydrocarbon compounds.

Appendix 2: VPH Dilution Effects and Data Correction

A-2 Specific Reporting Requirements for the VPH Method

A-2.1 Data Correction for VPH Concentration Calculations for Methanol Preservation Dilution Effect for Soils and Sediments

Based on the requirements of the VPH Method and of SW- 846 Method 8000, VPH analytical results for soil and sediment samples must be corrected for the Methanol Preservation Dilution Effect. The potential for under reporting VPH concentrations is more pronounced as the "as-received" % moisture content of the soil/sediment sample increases, if this correction is neglected. VPH concentrations and the recovery of matrix spikes and/or surrogates in solid samples preserved with methanol are subject to a systematic negative bias if the potential increase of the total solvent volume during the methanol extraction process is not considered. This increase in extraction solvent volume is a direct result of the solubility of the entrained sample moisture (water) in the methanol. The total solvent volume is the additive sum of the volume of methanol and the entrained sample moisture that partitions into the methanol during extraction. The volume of water partitioned is estimated from the % moisture determination (and the assumption that 1 g of water occupies a volume of 1 mL). This is a conservative correction regarding calculated VPH concentrations because some fraction of the sample's % moisture may not partition into the methanol, due to various physiochemical-binding forces.

The total solvent/water volume (Vt) is calculated using the following equation:

mL solvent/water (Vt) = mL of methanol + ((% moisture/100) × g of sample)

This "corrected" Vt value should be substituted directly for the Vt value shown in the VPH Method. It should be noted that whether corrected or uncorrected, the Vt value used to calculate VPH concentrations must also take into consideration the volume of any surrogate/spiking solution added to soil/sediment samples.

A-2.1 Sample Dilution

Under circumstances that sample dilution is required because either the concentration of one or more of the VPH target analytes or ranges exceed the concentration of their respective highest calibration standard, or any non-target peak exceeds the dynamic range of the detector (i.e., "off scale"), the RL/LLOQ for each VPH target analyte or range must be adjusted (increased) in direct proportion to the Dilution Factor ("DF"). Where:

$$DF = \frac{\text{Sample Aliquot Volume (mL) + Diluent Volume (mL)}}{\text{Sample Aliquot Volume (mL)}}$$

And the revised RL for the diluted sample, RL_d:

RL_d = DF x Lowest Calibration Standard for Target Analyte

Samples with elevated RL/LLOQs as a result of a dilution may not be able to satisfy RSR reporting limits in some cases if the RL_d is greater than the applicable RSR standard or criterion to which the concentration is being compared. Such increases in RLs are the unavoidable but acceptable consequence of sample dilution that enables quantification of target analytes or ranges, which exceed the calibration range. All dilutions must be fully documented in the laboratory report narrative.

Analytical Note: Over dilution is an unacceptable laboratory practice. The post-dilution concentration of the highest concentration target analyte must be at least 60 to 80% of its highest calibration standard. This will avoid unnecessarily high reporting limits for other target analytes, which did not require dilution.

If a sample analysis results in a saturated detector response for any target or non-target compound, the analysis must be followed by a blank reagent water analysis. If the blank analysis is not free of interferences, the system

must be decontaminated. Sample analysis may not resume until a blank demonstrates the lack of system interferences.