WSC – CAM – VIII A

Quality Control Requirements and Performance Standards for the ***Analysis of Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

VIII. High Performance Liquid Chromatography (HPLC) and Ion Chromatography (IC) Methods

A. Quality Control Requirements and Performance Standards for WSC-CAM-VIII A (Nitroaromatics and Nitramines by HPLC)

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CAM	**Compendium of Analytical Methods**
CASN	**Chemical Abstracts Service Number**
CCAL	**Continuing calibration**
%D	**Percent difference or percent drift**
DF	**Dilution factor**
1,2-DNB	**1,2-Dinitrobenzene**
3,4-DNT	**3,4-Dinitrotoluene**
ECD	**Electron capture detector**
GC	**Gas chromatograph**
HMX	**Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine**
HPLC	**High performance liquid chromatography**
ICV	**Initial calibration verification**
IRAs	**Immediate Response Actions**
LC	**Liquid chromatograph**
LCS	**Laboratory control sample**
MassDEP	**Massachusetts Department of Environmental Protection**
MCP	**Massachusetts Contingency Plan**
MOHML	**Massachusetts Oil and Hazardous Materials List**
MS	**Matrix spike**
MSD	**Matrix spike duplicate**
NA	**Not applicable**
PDA	**Photodiode array**
PETN	**Pentaerythritol tetranitrate**
PTFE	**Polytetrafluoroethylene**
QA	**Quality assurance**
QC	**Quality control**
r	**Correlation coefficient**
r^2	**Coefficient of determination**
RAO	**Response Action Outcome**
RCs	**Reportable Concentrations**
RDX	**Hexahydro-1,3,5-trinitro-1,3,5-triazine**
RL	**Reporting limit**
RPD	**Relative percent difference**
RQs	**Reportable Quantities**
%RSD	**Percent relative standard deviation**
TNT	**Trinitrotoluene**
µg/kg	**micrograms per kilogram**
µg/L	**micrograms per liter**
UV	**Ultraviolet**
UXO	**Unexploded ordnance**

1.0 Quality Control Requirements and Performance Standards for WSC-CAM-VIII A

1.1 Overview of WSC-CAM-VIII A

WSC-CAM-VIII A, *Quality Control Requirements and Performance Standards for the Analysis of Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC) in Support of Response Actions under the Massachusetts Contingency Plan (MCP)*, is a component of MassDEP's Compendium of Analytical Methods (CAM). Effective July 1, 2010, this revised CAM protocol, WSC-CAM-VIII A, replaces the original Nitroaromatic and Nitramine HPLC CAM document, WSC-CAM-VIII A (effective date, August 27, 2004). Refer to WSC-CAM-I A for an overview of the CAM process. Please note that while this protocol must be followed on and after the effective date of July 1, 2010 for the purpose of "Presumptive Certainty," the revised protocol may be used optionally prior to its effective date upon its publication on April 15, 2010.

This document provides Quality Control (QC) requirements and performance standards to be used in conjunction with the required analytical method SW-846 8330A, analysis for nitroaromatics and nitramines in aqueous and solid samples by HPLC preceded by conventional sample preparation methods via SW-846 Methods, as described in Section 1.3 of this protocol. The QC requirements and performance standards specified in this document in Table VIII A-1 together with the analytical procedures described in EPA SW-846 Method 8330A, *Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)*, constitute the WSC-CAM-VIII A protocol. All protocols included in the CAM are considered "methods" published by the MassDEP pursuant to the provisions of 310 CMR 40.0017(2). Use of EPA SW-846 8330A is a "Presumptive Certainty" requirement of WSC-CAM-VIII A. However, it should be noted that if the laboratory utilizes the analytical procedures in SW-846 Method 8330B instead of 8330A, it is acceptable to answer "YES" to Question B on the MassDEP Analytical Protocol Certification Form; although 8330B is not yet promulgated, improvements in the preparation and extraction of samples in 8330B make this an acceptable method. Sample preservation, container and analytical holding time specifications for aqueous, soil, and sediment matrices for nitroaromatics and nitramines analyzed in support of MCP decision-making are presented in Appendix VIII A-1 of this document and Appendix VII-A of 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)*. Data reporting requirements are also provided in WSC-CAM-VII A.

Overall usability of data produced using this CAM protocol should be evaluated for compliance with project-specific data quality objectives, regardless of "Presumptive Certainty" status. For more guidance on data usability, refer to MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

1.1.1 Reporting Limits for WSC-CAM-VIII A

The reporting limit (RL) for an individual compound using WSC-CAM-VIII A is dependent on the concentration of the lowest non-zero standard in the initial calibration, analyzed under identical conditions as the sample, with adjustments made for the sample size, extraction concentration factor, percent solids, dilution factors, etc., as required. Except as provided in the table below, the CAM RLs for WSC-CAM-VIII A target analytes are:

- 0.2-0.8 µg/L for aqueous samples (surface water, groundwater and drinking water); and
- 250-1000 µg/kg (wet weight) for soil/sediment samples (assuming 100% solids).

These values are readily achievable using the techniques specified in CAM, including an HPLC-system equipped with a pump capable of achieving 4000 psi, a 100 µl loop injector and a 254 nm UV detector and based on the ultrasonic extraction of 2.0 grams of soil in 10.0 ml of acetonitrile and the low-level salting-out extraction method for aqueous samples.

For "Presumptive Certainty" purposes, if the CAM RLs are not achieved, respond "NO" to Question G of the "MassDEP MCP Analytical Protocol Certification Form" and address the CAM RL exceedance in the laboratory narrative.

Reporting limits lower than the above-referenced CAM RLs for WSC-CAM-VIII A target analytes may be required to satisfy project requirements. The RL (based on the concentration of the lowest calibration standard) for each contaminant of concern must be less than or equal to the MCP standards or criteria that the contaminant concentrations are being compared to (e.g., Method 1 Standards, benchmark values, background, etc.). Meeting MCP standards or criteria may require method modifications, such as reducing the volume of the final extract, to improve sensitivity. All such modifications must be described in the laboratory narrative. Regardless of the modification that is used, RLs for the WSC-CAM-VIII A target analytes will be proportionately higher for samples that require dilution, when a reduced sample size is used or for an increased final extract volume.

1.1.2 Initial Demonstration of Proficiency for WSC-CAM-VIII A

Each laboratory that uses the WSC-CAM-VIII A protocol is required to operate a formal quality assurance program. The minimum requirements of this program consist of an initial demonstration of laboratory proficiency, ongoing analysis of standards and blanks to confirm acceptable continuing performance, and the analysis of laboratory control samples (LCSs) and LCS duplicates to assess analytical accuracy and precision. Matrix spikes (MS), matrix spike duplicates (MSD) or matrix duplicates may also be used to evaluate accuracy and precision when such samples are analyzed either at the discretion of the laboratory or at the request of the data user.

Laboratories must document and have on file an Initial Demonstration of Proficiency for each combination of sample preparation and determinative method being used. These data must meet or exceed the performance standards as presented in Table VIII A-1 of this protocol and SW-846 Method 8000B. Procedural requirements for performing the Initial Demonstration of Proficiency can be found in SW-846 Method 8000B (Section 8.4) and SW-846 method 8330A (Section 9.4). The data associated with the Initial Demonstration of Proficiency must be kept on file at the laboratory and made available to potential data users on request. The data associated with the Initial Demonstration of Proficiency for WSC-CAM-VIII A must include the following information:

QC Element	Performance Criteria
Initial Calibration	WSC-CAM-VIII A, Table VIII A-1
Continuing Calibration	WSC-CAM-VIII A, Table VIII A-1
Method Blanks	WSC-CAM-VIII A, Table VIII A-1
Average Recovery	SW-846 Method 8000B, Section 8.4
% Relative Standard Deviation	SW-846 Method 8000B, Section 8.4

QC Element	Performance Criteria
Surrogate Recovery	WSC-CAM-VIII A, Table VIII A-1

NOTE: Because of the number of QC elements associated with the Initial Demonstration of Proficiency, it should be expected that one or more 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-conforming analytes. All non-conforming analytes along with the laboratory-specific acceptance criteria should be noted in the Initial Demonstration of Proficiency documentation.

It is essential that laboratory-specific performance criteria for LCS, LCS duplicate and surrogate recoveries also be calculated and documented as described in SW-846 Method 8000B, Section 8.7. Experience indicates that the criteria recommended in specific methods are frequently not met for some analytes and/or matrices; the in-house performance criteria will be a means of documenting these repeated exceedances. Laboratories are encouraged to actively monitor pertinent QC performance standards described in Table VIII A-1 to assess analytical trends (i.e., systematic bias, etc) and improve overall method performance by preempting potential non-conformances.

For the WSC-CAM-VIII A protocol, laboratory-specific control limits must meet or exceed (demonstrate less variability than) the performance standards for each QC element listed in Table VIII A-1. It should be noted that the performance standards listed in Table VIII A-1 are based on multiple-laboratory data, which are in most cases expected to demonstrate more variability than performance standards developed by a single laboratory.

This protocol is restricted to use by, or under the supervision of, analysts experienced in the use of high performance liquid chromatography, interpretation of chromatograms, experienced in the handling samples containing explosives and the analysis of explosive residues.

1.2 Summary of SW-846 Method 8330A

The method provides a salting-out extraction procedure for low concentrations (parts per trillion, or nanograms per liter) of explosive residues in surface water or groundwater, a direct injection method for diluted and filtered water samples with elevated concentrations, and an ultrasonic extraction procedure with acetonitrile for the analysis of soils and sediments.

SW-846 Method 8330A provides chromatographic and ultraviolet (UV) detection conditions for the identification and quantification of explosive residues introduced into the HPLC system. All positive measurements observed on one column must be confirmed by injection under similar analytical conditions onto a dissimilar column that achieves sufficient resolution of all the method target compounds. Columns shown to demonstrate sufficient resolution are as follows:

(1) Reversed-phase HPLC column, 25 cm x 4.6 mm ID, 5 µm: Supelco LC-18;
(2) Reversed-phase HPLC column, 25 cm x 4.6 mm ID, 5 µm, Phenomenex Luna Phenyl-Hexyl;
(3) Reversed-phase HPLC column, 25 cm x 4.6 mm ID, 5 µm, Phenomenex Synergi Polar-RP; and
(4) CN column (reversed-phase HPLC column, 25 cm x 4.6 mm ID, 5 µm, Supelco LC-CN).

Analytical Note: Column (4) is known to yield numerous co-elutions for the full list of target compounds proposed in this document.

Changes in manufacturing processes to the above-listed columns may improve or diminish chromatographic efficiency. Alternate confirmatory columns (produced after 2005) may yield comparable or superior chromatographic separations.

Identification of target analytes is accomplished by comparing the sample retention time with the retention time of standards obtained under identical analytical conditions. Quantitation is accomplished by using the peak area or peak height and a calibration factor generated from a minimum five-point calibration curve.

Identification of target analytes on a single-column must be confirmed on a second column, or must be supported by at least one other independent analytical technique. It should be noted that SW-846 Method 8330A is particularly susceptible to severe co-elution problems when the detector is run at high sensitivity. Such co-elution problems affect the quantitation as well as identification of target analytes, and may result in poor agreement between quantitative results from two dissimilar columns. Although a dual-column option may satisfy this requirement, due caution should be exercised when highly contaminated samples are processed or during times of high sample throughput. Dual column confirmation is not required for samples with concentrations of all target analytes below their respective RL.

For some explosive residue analytes, their unique photodiode array (PDA) detector UV spectra or absorbance data using a different analytical (UV) wavelength may be used for confirmation. Depending on the concentration of the explosive residue, mass spectral confirmation or analysis using a dissimilar detector (GC/ECD) may also be a confirmation option.

1.3 Sample Extraction Methods for WSC-CAM-VIII A

Samples for analysis by SW-846 Method 8330A are normally prepared using one of the following methods.

Matrix	Method
Water - High Concentration	Direct Injection
Water - Low Concentration	Salting-Out with no evaporation
Water Pre-Concentration	Solid-Phase Extraction (SPE) using Porapak RDX, Sep-Pak, 6cc, 500mg
Soil/Sediment	Ultrasonic Extraction with Acetonitrile

1.4 Method Interferences

- Refer to SW-846 Methods 3500C (Section 4.0, in particular) and 8000B for a detailed discussion of interferences. Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into four broad categories.

- Contaminated solvents, reagents, or sample processing hardware,
- Contaminated HPLC carrier gas, parts, column surfaces, or detector surfaces,
- Non-target compounds simultaneously extracted from the sample matrix which cause a detector response, and
- Co-elution of target analytes.

An in-depth discussion of the causes and corrective actions for all of these interferences is beyond the scope of this guidance document. A brief discussion of the more prevalent interferences is presented below.

- Refer to SW-846 Method 8330A for a detailed description of chemical contaminants, cross-contamination, and corrective actions which may be taken to eliminate contamination. If a method blank contains a contaminant, data for samples associated with that blank must **not** undergo "blank correction" (i.e., if an associated sample also contains the contaminant, subtraction of the blank amount from the sample amount is not permitted).

- Cross-contamination may occur when any sample is analyzed immediately after a sample containing high concentrations of nitroaromatics or nitramines. After the analysis of a sample containing high concentrations of nitroaromatics or nitramines, one or more blanks should be analyzed to check for potential cross-contamination/carryover. Concentrations of nitroaromatics or nitramines which exceed the upper limit of calibration should prompt the analyst to check for potential cross-contamination/carryover. In addition, samples containing non-target, chromatographically recalcitrant compounds may also present potential for cross-contamination/carryover. Laboratories should be aware that carryover from highly-retained compounds may not appear until a later sample run.

- 2,4-Dintrotoluene and 2,6-dinitrotoluene elute at similar retention times (retention time difference of approximately 0.2 minutes). A large concentration of one isomer may mask the response of the other isomer. If it is not apparent that both isomers are present (or are not detected), an isomeric mixture should be reported.

1.5 Quality Control Requirements for WSC-CAM-VIII A

1.5.1 General QC Requirements

Refer to SW-846 Method 8000B for general QC procedures for all chromatographic methods, which includes SW-846 method 8330A. Instrument QC and method performance requirements for the HPLC system may be found in SW-846 method 8330A, Sections 9.0 and 13.0, respectively.

1.5.2 Specific QC Requirements and Performance Standards for WSC-CAM-VIII A

Specific QC requirements and performance standards for the WSC-CAM-VIII A protocol are presented in Table VIII A-1. Refer to WSC-CAM-VII A for field QC requirements. Strict compliance with the QC requirements and performance standards, as well as satisfying the CAM's other analytical and reporting requirements will provide a data user with "Presumptive Certainty" in support of Response Actions under the MCP. The concept of "Presumptive Certainty" is explained in detail in Section 2.0 of WSC-CAM-VII A.

While optional, parties electing to utilize these protocols will be assured of "Presumptive Certainty" of data acceptance by agency reviewers. In order to achieve "Presumptive Certainty" for analytical data, parties must:

(a) Use the analytical method specified for the selected CAM protocol;
(b) Incorporate **all** required analytical QC elements specified for the selected CAM protocol;
(c) Implement, as necessary, required corrective actions and analytical response actions for **all** non-conforming analytical performance standards;
(d) Evaluate and narrate, as necessary, **all** identified CAM protocol non-compliances; and
(e) Comply with **all** the reporting requirements specified in WSC-CAM-VII A, including retention of reported and unreported analytical data and information for a period of ten (10) years.

In achieving "Presumptive Certainty" status, parties will be assured that analytical data sets:

- ✓ Satisfy the broad QA/QC requirements of 310 CMR 40.0017 and 40.0191 regarding the scientific defensibility, precision and accuracy, and reporting of analytical data; and

- ✓ May be used in a data usability and representativeness assessment, as required in 310 CMR 40.1056(2)(k) for Response Action Outcome (RAO) submittals, consistent with the guidance described in MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

1.6 Special Analytical Considerations for WSC-CAM-VIII A

The following bullets highlight potential issues that may be encountered with the analysis of nitroaromatics and nitramines using this protocol.

Extraction/Analysis Issues:

- Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. All aqueous samples expected to contain Tetryl should be diluted with acetonitrile prior to filtration and acidified to pH <3. In addition, any sample expected to contain Tetryl should not be exposed to temperatures above room temperature.

- Methanol must be used as an extraction solvent whenever HMX is a target analyte and the high-level direct injection analytical procedure is utilized, as HMX quantitation is improved with the use of methanol instead of acetonitrile.

- After the addition of the salt water during the salting-out extraction procedure for aqueous samples, the acetonitrile phase is removed for further processing. The removal of the acetonitrile phase must be performed very carefully so as to remove as little as possible of the salt water phase. The high concentration of sodium chloride in the water will produce a large peak at the beginning of the chromatogram which can interfere with the determination of HMX.

- Soil and sediment samples must be thoroughly homogenized prior to extraction by SW-846 Method 8330A. Soil samples must be air-dried at room temperature or colder to a constant weight, being careful not to expose the samples to direct sunlight. The dried sample must be thoroughly ground and homogenized in an acetonitrile-rinsed mortar to pass a 30-mesh sieve.

It is advisable to screen soil or waste samples using SW-846 colorimetric Methods 8510 or 8515 to determine whether high concentrations of explosives are present. SW-846 Method 8515 is most sensitive for 2,4,6-TNT, the analyte most often detected in high concentrations in soil samples; however, other nitroaromatics will also cause a color to be developed that will provide a rough estimation of their concentrations. SW-846 Method 8510 is most sensitive for RDX and HMX. **Highly contaminated soil or sediment samples should never be ground in a mortar and pestle.**

Visual examination of a soil or sediment sample is also important when the sample is taken from a site expected to contain explosives. Lumps of material with explosive-like appearance should be considered suspect and never ground manually. Explosives are generally a very finely ground grayish-white material.

- Degradation products of Tetryl appear as a shoulder on the 2,4,6-TNT peak. Peak heights rather than peak areas should be used when Tetryl is present in concentrations that are significant relative to the concentration of 2,4,6-TNT.

- A linear or non-linear calibration model must not be used to compensate for detector saturation or to avoid proper instrument maintenance. As such, linear or non-linear regression must not be employed for initial calibration calculations that typically meet percent relative standard deviation (%RSD) requirements specified in Table VIII A-1.

General Sampling Precautions and Safety Alerts

- To the extent practical, never expose samples for explosive residue analysis to direct sunlight.

- Sampling of media potentially contaminated with explosives should only occur after appropriate safety level clearance of the sampling site. High explosives such as RDX, HMX and TNT have chemical, physical and toxicological properties different from environmental contaminants routinely encountered at MCP sites. **The potential for explosion requires that due caution be exercised when planning and implementing field activities at sites where these materials are expected to be present at percent level concentrations. As a final caution, unexploded ordnance (UXO) may be encountered on the surface or buried in the soil at many military ranges.**

- Containers used to collect samples for the determination of explosive residues should be water washed followed by methanol (or isopropanol) rinsing. The sample containers should be of glass or Teflon, and have screw-caps with Teflon liners. In situations where Teflon is not available, solvent-rinsed aluminum foil may be used as a liner. However, acidic or basic samples may react with the aluminum foil, causing eventual contamination of the sample. Plastic containers or lids may ***never*** be used for the storage of samples for explosive residue analyses due to the possibility of sample contamination from the phthalate esters and other hydrocarbons within the plastic. Sample containers should be filled with care so as to prevent any portion of the collected sample coming in contact with the sampler's gloves, thus causing contamination. Samples should not be collected or stored in the presence of exhaust fumes. If the sample comes in contact with the sampler (e.g. if an automatic sampler is used), run organic-free reagent water through the sampler and use as a field blank.

Table VIII A-1: Specific QC Requirements and Performance Standards for Explosives (SW-846 8330A) Using WSC-CAM-VIII A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency	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 target analytes. (4) Must follow procedure in Section 8.4 of SW-846 8000B.	No	NA	Refer to Section 8.4 of SW-846 8000B and Section 1.1.2 of this protocol.	NA
Retention Time Windows	Laboratory Analytical Accuracy	(1) Prior to initial calibration and when a new LC column is installed. (2) Calculated according to the method (Section 7.6 of SW-846 8000B).	No	NA	NA	NA
Initial Calibration	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 $\leq$RL. (4) %RSD $\leq$20, r $\geq$0.99 (linear regression), or r^2 $\geq$0.99 (non-linear regression) for each target explosive. (5) If %RSD >20, linear or non-linear regression must be used. (6) Must contain all target explosives. (7) Calibration must be performed under the same conditions as the samples. (8) If linear or non-linear regression used, verify the RL by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%.	No	NA	(1) Recalibrate as required by method. (2) If recalculated concentrations from the lowest calibration standard are outside of 70-130% recovery range, either: * The RL limit must be reported as an estimated value[2], or * The RL 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. Report non-conforming compounds (%RSD >20, r <0.99, or r^2 <0.99) in laboratory narrative. If non-linear regression (i.e., quadratic equation) is used for calibration, this must be noted in the laboratory narrative along with the compounds affected.

Table VIII A-1: Specific QC Requirements and Performance Standards for Explosives (SW-846 8330A) Using WSC-CAM-VIII A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
Initial Calibration Verification	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 target explosives. (5) Percent recoveries must be between 70-130% for each target analyte.	No	NA	Locate source of problem; recalibrate if >10% of all analytes are outside of criteria.	If recovery is outside of 70-130% for any analyte, report non-conforming compounds in laboratory narrative.
Continuing Calibration	Laboratory Analytical Accuracy	(1) Prior to samples, every 12 hours or every 20 samples (10 is recommended), whichever is more frequent, and at the end of the analytical sequence. (2) Concentration level near mid-point of curve. (3) Must contain all target explosives. (4) %D must be $\leq$20 for each target analyte. (5) Verify that all analytes fall within retention time windows.	No	NA	(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 explosives were detected in the "associated samples." NOTE: "Associated samples" refers to all samples analyzed since the last acceptable continuing calibration.	Report non-conforming compounds (%D >20) and associated samples in laboratory narrative.

Table VIII A-1: Specific QC Requirements and Performance Standards for Explosives (SW-846 8330A) Using WSC-CAM-VIII A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
Method Blank	Laboratory Method Sensitivity (contamination evaluation)	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Matrix-specific (e.g., water, soil). (3) Target analytes must be <RL.	Yes	NA	(1) If concentration of contaminant in sample is <10x concentration in blank, locate source of contamination; correct problem; re-extract and 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 re-extraction is not possible, report non-conformance in laboratory narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" or some other convention, should qualify the sample results. Blank contamination should also be documented in the laboratory narrative. (3) If re-extraction is performed within holding time and yield acceptable method blank results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.

Table VIII A-1: Specific QC Requirements and Performance Standards for Explosives (SW-846 8330A) Using WSC-CAM-VIII A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all target explosives. (4) Matrix-specific (e.g., soil, water). (5) Percent recoveries must be between 40-140% for all target analytes with the exception of tetryl. Laboratories must develop in-house recovery control limits for tetryl. (6) Must be prepared in methanol.	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of criteria. (2) If ≤10% of compounds are outside of the acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of compounds are above the acceptance criteria (>140%), reextraction is not required if affected compounds were not detected in associated samples.	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of 40-140% for any analyte, report non-conforming compounds in laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
LCS Duplicate	Laboratory Analytical Accuracy & Precision	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all target explosives. (4) Matrix-specific (e.g., soil, water). (5) Percent recoveries must be between 40-140% for all target analytes with the exception of tetryl. Laboratories must develop in-house recovery control limits for tetryl.	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of recovery acceptance criteria. (2) If ≤10% of compounds are outside of the recovery acceptance criteria, re-extraction is not required as long as	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of 40-140% for any analyte or if RPD is outside of criteria, report non-conforming compounds in laboratory narrative.

Table VIII A-1: Specific QC Requirements and Performance Standards for Explosives (SW-846 8330A) Using WSC-CAM-VIII A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
		(6) RPDs must be ≤20 for waters and ≤30 for solids. (7) Must be prepared in methanol.			recoveries are >10%. (3) If >10% of compounds are above the recovery acceptance criteria (>140%), reextraction is not required if affected compounds were not detected in associated samples.	(3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
MS/MSD	Method Accuracy & Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data user). (2) Matrix-specific. (3) Concentration level near midpoint of curve. (4) Must contain all target explosives. (5) Percent recoveries between 40 – 140%. (6) RPDs ≤20 for waters and ≤30 for solids. (7) Must be prepared in methanol.	Yes ONLY when requested by the data user	Recovery <10%; affects nondetect result for affected analyte in unspiked sample only.	Check LCS; if recoveries are acceptable in LCS, narrate non-conformance.	Note exceedances in laboratory narrative.
Surrogates	Method Accuracy in Sample Matrix	(1) Minimum of 1 surrogate. Recommended surrogates: 1,2-DNB or 3,4-DNT (2) Percent recoveries must be between 30-130% on both columns.	Yes (report surrogate recoveries from both columns)	Recovery <10%; affects all nondetect results in affected sample.	If the surrogate is outside of limits on both columns: (1) Re-extract the sample if surrogate recoveries are low and there is no chromatographic interference. (2) Re-extract the sample if surrogate recoveries are high and explosives	(1) Report recoveries outside of 30-130% in laboratory narrative. (2) If re-extraction yields similar surrogate non-conformances, the laboratory must report results of both the initial extraction and re-extraction. (3) If re-extraction is

Table VIII A-1: Specific QC Requirements and Performance Standards for Explosives (SW-846 8330A) Using WSC-CAM-VIII A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
					were detected in the sample. NOTES: (a) If surrogate recoveries are high and target analytes are not detected in sample, re-extraction is not required. (b) If chromatographic interference is present and surrogate recovery would cause rejection of data (i.e., <10%), reanalyze sample on dilution. (c) If a surrogate is diluted to a concentration below that of the lowest calibration standard, reextraction and/or reanalysis is not required.	performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of the holding time and yields acceptable surrogate recoveries, the laboratory must report results of both the initial extraction and re-extraction. (5) If sample is not re-extracted due to chromatographic interference, the laboratory must provide the chromatogram in the data report.
Identification and Quantitation	NA	(1) Peak area is the expected default to be used for quantitation of explosives under most circumstances. Refer to Section 1.6 of this protocol for instances where it may be appropriate to use peak height. Regardless if peak area or peak height is used, the same method used for quantitation of samples must also be used for calibration standards. (2) The laboratory must use the average calibration factor, linear or non-linear regression curve generated from the associated initial calibration for quantitation of each target explosive.	NA	If RPD >100 for target explosive, reject positive result for affected explosive.	If the RPD between the dual column results >100, reanalyze the sample on dilution. Both analyses must be reported.	If the RPD between the dual column results exceeds 40, the laboratory must qualify the sample results and/or note the exceedance in the laboratory narrative. If the RPD exceedance is due to interference, the lower of the dual column values can be reported; this must be noted in the laboratory narrative.

Table VIII A-1: Specific QC Requirements and Performance Standards for Explosives (SW-846 8330A) Using WSC-CAM-VIII A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
		(3) Secondary column analysis: Laboratory must utilize a second dissimilar column to confirm positive results. The laboratory must report the higher of the two results. All required QC parameters (e.g., calibrations, LCSs, etc.) must be met on the secondary column as well. (4) Results must be reported with 2 or more "significant figures" if ≥ RL. If reporting values below the RL, report with 1 or more "significant figures".[3]				
General Reporting Issues	NA	(1) The laboratory must only report values ≥ the sample-specific reporting limit. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for each analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported. (3) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards. (4) Refer to Appendix VIII A-1 for chain-of-custody requirements regarding preservation, cooler temperature, and holding times.	NA	NA	NA	(1) Complete analytical documentation for diluted and undiluted analyses must be made available for review during an audit. (2) The performance of dilutions must be documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboratory narrative. (3) If samples are not preserved properly or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory narrative. (4) If samples are extracted and/or

Table VIII A-1: Specific QC Requirements and Performance Standards for Explosives (SW-846 8330A) Using WSC-CAM-VIII A

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350[2]	Required Corrective Action	Required Analytical Response Action
						analyzed outside of the holding time, note the non-conformances in the laboratory narrative.

[1]As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, September 2007, if these results are observed, data users should consider nondetect results as unusable and positive results as estimated with a significant low bias.

[2]If the RL is estimated due to unacceptable recovery of the lowest standard, the CAM RL has not been achieved; Question G of the "MassDEP MCP Analytical Protocol Certification Form" must be answered "NO" and this must be addressed in the laboratory narrative.

[3]Reporting protocol for "significant figures" is a policy decision included for standardization and consistency for reporting of results and is not a definition of "significant" in the scientific or mathematical sense.

1.7 Analyte List for WSC-CAM-VIII A

The MCP analyte list for WSC-CAM-VIII A is presented in Table VIII A-2. The analyte list is comprised of sixteen (16) nitroaromatic and nitramine compounds that are routinely encountered at locations impacted by military ordnance (artillery target areas, demolition ranges, etc.) and are readily analyzable by SW-846 Method 8330A. The analyte list contains both high explosive residues and the environmental metabolites of trinitrotoluene (TNT). These compounds, with the exception of 2,6-Diamino-4-nitrotoluene and 2,4-Diamino-6-nitrotoluene (two additional environmental metabolites of TNT), are specifically identified as explosive residue analytes in Section 1 of SW-846 Method 8330A.

It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL for the target analytes. Sources of various MassDEP standards and criteria are as follows:

- Reportable Quantities (RQs) and Concentrations (RCs) as described in 310 CMR 40.1600, The Massachusetts Oil and Hazardous Materials List (MOHML), in Subpart P of the MCP may be found at the following URL: https://www.mass.gov/site-cleanup-regulations-policies-forms-more.
- An online searchable Oil & Hazardous Materials List of RQs and RCs values may be found at the following URL: https://www.mass.gov/service-details/oil-hazardous-material-list.
- An updated list of MCP Method 1 Standards may be found at the following URL: https://www.mass.gov/site-cleanup-regulations-policies-forms-more.

Most of the analytes listed in Table VIII A-2 do not have a promulgated MCP Method 1 groundwater/soil standard as of the publication date of this revision. Of the listed WSC-CAM-VIII A target analytes, only HMX, RDX, and 2,4-Dinitrotrotoluene have MCP Method 1 groundwater/soil standards as described in 310 CMR 40.0974 and 40.0980, respectively. The remaining analytes, with the exception of the four (4) TNT environmental metabolites (4-Am-DNT, 2-Am-DNT, 2,6-DAm-4NT and 2,4-DAm-6NT), have compound-specific Reportable Concentrations (RC) as described in 310 CMR 40.0360 and 40.1600.

1.7.1 Additional Analytically Amenable Compounds by WSC-CAM-VIII A

SW-846 Method 8330A may also be utilized for the determination of additional nitroaromatic and nitramine explosive compounds not specifically identified as explosive residue analytes in Section 1 of SW-846

Method 8330A. These additional analytes, shown as “Additional Analytically Amenable Compounds” in Table VIII A-2 include, but are not limited to, PETN, Nitroglycerin, and Picric Acid. These compounds are mentioned in this guidance for informational purposes only and need only be evaluated at MCP disposal sites that have known or suspected impacts by these additional compounds. Of these three (3) analytically amenable compounds, only PETN does not have an MCP Reportable Concentration.

1.7.2 Analyte List Reporting Requirements for WSC-CAM-VIII A

While it is not necessary to request and report all the WSC-CAM-VIII A analytes listed in Table VIII A-2 to obtain “Presumptive Certainty” status, it is necessary to document use and reporting of a reduced analyte list, for site characterization and data representativeness considerations. MassDEP strongly recommends use of the full SW-846 Method 8330A analyte list, including the four (4) TNT metabolites, when previously uncharacterized ordnance-impacted sites with unknown or complex history and/or disposal practices are

characterized for explosive residue analytes. These assessment activities may include but are not limited to:

- ✓ Immediate Response Actions (IRAs) performed in accordance with 310 CMR 40.0410;
- ✓ Initial Site Investigation Activities performed in accordance with 310 CMR 40.0405(1);
- ✓ Phase I Initial Site Investigation Activities performed in accordance with 310 CMR 40.0480 through 40.0483; and
- ✓ Phase II Comprehensive Site Investigation Activities performed in accordance with 310 CMR 40.0830

In a limited number of cases, the use of the full analyte list for a chosen analytical method may not be necessary, with respect to data representativeness concerns, including:

- ✓ Sites where substantial site/use history information is available to rule-out all but a limited number of contaminants of concern, and where use of the full analyte list would significantly increase investigative costs; or

- ✓ Well-characterized sites where initial full-analyte list testing efforts have sufficiently narrowed the list of contaminants of concern.

Note: a data user who avoids the detection and quantitation of a contaminant that is present or likely present at a site above background levels by limiting an analyte list could be found in criminal violation of MGL c. 21E or any regulations or orders adopted or issued thereunder.

In cases where a reduced list of analytes is requested, laboratories must still employ the specified QC requirements and performance standards in WSC-CAM-VIII A to obtain “Presumptive Certainty” status.

Table VIII A-2: Analyte List for WSC-CAM-VIII A (SW-846 8330A)

Analyte	CASN
HMX	2691410
RDX (Cyclonite)[1]	121824
1,3,5-Trinitrobenzene (1,3,5-TNB)	99354
1,3-Dinitrobenzene (1,3-DNB)	99650
Nitrobenzene (NB)	98953
Tetryl	479458
2,4,6-Trinitrotoluene (TNT)	118967
4-Amino-2, 6-dinitrotoluene (4-Am-DNT)	1946510
2-Amino-4, 6-dinitrotoluene (2-Am-DNT)	35572782
2,6-Diamino-4-nitrotoluene (2,6-DAm-4NT)	59299753
2,4-Diamino-6-nitrotoluene (2,4-DAm-6NT)	6629294
2,6-Dinitrotoluene (2,6-DNT)	606202
2,4-Dinitrotoluene (2,4-DNT)	121142
2-Nitrotoluene (2-NT)	88722
4-Nitrotoluene (4-NT)	99990
3-Nitrotoluene (3-NT)	99081
Additional Analytically Amenable Compounds:	
Nitroglycerin	55630
PETN [d]	78115
Picric Acid (Trinitrophenol)	88891

[1]RDX is identified as Cyclonite in Subpart P, the Massachusetts Oil and hazardous Materials List (MOHML)

CASN – Chemical Abstracts Service Numbers (CASN)

NOTE: Other nitroaromatics and nitramines may also be analyzed using the WSC-CAM-VIII A Protocol but are not considered part of the CAM target analyte list.

HMX - Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine **RDX - Hexahydro-1,3,5-trinitro-1,3,5-triazine**

Tetryl - Methyl-2,4,6-trinitrophenylnitramine **PETN - Pentaerythritol tetranitrate**

2.0 Data Usability Assessment

Specific guidance applicable to all Class A, B or C RAO Statements, including partial RAOs, for preparation of Representativeness Evaluations and Data Usability Assessments pursuant to 310 CMR 40.1056(2)(k) of the MCP is provided in *MCP Representativeness Evaluations and Data Usability Assessments* (Policy #WSC-07-350). This document provides general information regarding the purpose and content of these required evaluations as a component of and in support of an RAO submittal. The most current version of this document may be found at the following URL: https://www.mass.gov/site-cleanup-regulations-policies-forms-more.

Overall usability of data produced using this CAM protocol should be evaluated for compliance with project-specific data objectives using MassDEP Policy #WSC-07-350, regardless of "Presumptive Certainty" status.

3.0 Reporting Requirements for WSC-CAM-VIII A

3.1 General Reporting Requirements for WSC-CAM-VIII A

General environmental laboratory reporting requirements for analytical data used in support of assessment and evaluation decisions at MCP disposal sites are presented in WSC-CAM-VII A, Section 2.4. This guidance document provides limited recommendations for field QC, as well as the required content of the laboratory report, which includes:

- Laboratory identification information,
- Analytical results and supporting information,
- Sample- and batch-specific QC information,
- Laboratory Report Certification Statement,
- Copy of the Analytical Protocol Certification Form,
- Laboratory narrative contents, and
- Chain-of-custody form requirements.

3.2 Specific Reporting Requirements for WSC-CAM-VIII A

Specific QC requirements and performance standards for WSC-CAM-VIII A are presented in Table VIII A-1. Specific reporting requirements for WSC-CAM-VIII A are summarized below in Table VIII A-3 as "Required Analytical Deliverables (**YES**)". These routine reporting requirements must always be included as part of the laboratory deliverable for this method. It should be noted that although certain items are not specified as "Required Analytical Deliverables (**NO**)", these data must be available for review during an audit and may also be requested on a client-specific basis.

Table VIII A-3 Routine Reporting Requirements for WSC-CAM-VIII A (SW-846 8330A)

Parameter	Required Analytical Deliverable
Retention Time Windows	***NO***
Initial Calibration	***NO***
Initial Calibration Verification	***NO***
Continuing Calibration (CCAL)	***NO***
Method Blank	***YES***
Laboratory Control Samples (LCSs)	***YES***
LCS Duplicates	***YES***
Matrix Spike (MS)	***YES (if requested by data user)***
Matrix Spike Duplicate (MSD)	***YES (if requested by data user)***
Matrix Duplicate (MD)	***YES (if requested by data user)***
Surrogates	***YES***
Identification and Quantitation	***NO***
General Reporting Issues	***YES***

3.2.2 Sample Dilution

Under circumstances that sample dilution is required because either the concentration of one or more of the target analytes 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 for each nitroaromatic or nitramine must be adjusted (increased) in direct proportion to the Dilution Factor (DF).

The revised RL for the diluted sample, RL_d:

$$RL_d = DF \times \text{Lowest Calibration Standard for Target Analyte}$$

It should be understood that samples with elevated RLs as a result of a dilution may not be able to satisfy MCP standards/criteria in some cases if the RLd is greater than the applicable MCP standard or criterion to which the concentration is being compared. Such increases in RLs are the unavoidable but acceptable consequence of sample dilution that enable quantification of target analytes which exceed the calibration range. All dilutions must be fully documented in the laboratory narrative.

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

Appendix VIII A-1

Sample Collection, Preservation, and Handling Procedures for Nitroaromatic and Nitramine Analyses

Sample preservation, container and analytical holding time specifications for aqueous, soil, and sediment matrices for nitroaromatics and nitramines analyzed in support of MCP decision-making are summarized below and presented in Appendix VII A-1 of WSC-CAM-VII A, *Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data Conducted in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)*.

Matrix	Container[1]	Preservation[7]	Holding Time[3,6]
Aqueous Samples, with no Residual Chlorine	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Store in dark; Cool to ≤ 6°C but not frozen; pH 2.0 w/ $NaHSO_4$ (1.2 g/L)	7 days to extraction; 40 days from extraction to analysis[5]
Aqueous Samples, with Residual Chlorine[4]	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Add 1-mL 10% sodium thiosulfate solution per container (or 0.008%)[4]. Addition of thiosulfate solution to sample container may be performed in the laboratory prior to field use. Store in dark; Cool to ≤ 6°C but not frozen.	7 days to extraction; 40 days from extraction to analysis[5]
Soil/Sediment Samples	(1) 8-oz. amber glass jar w/ a Teflon-lined screw cap[2]	Store in dark; Cool to ≤ 6°C [2]	14 days to extraction; 40 days from extraction to analysis[2,5]
Waste Samples	Collect sample in one (1) x 500 mL amber wide mouth jar with a teflon-lined screw cap.	No special preservation required	14 days to extraction; 40 days from extraction to analysis[5]

[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]Alternatively, soil/sediment samples for nitroaromatic or nitramine analyses may be held for up to one (1) year if frozen within 24 hours of collection at <-10°C. Sampling container should only be filled to 2/3 of capacity to avoid breakage caused by expansion during freezing. Preparation or extraction must be commenced within 24 hours of thawing. Temperature must never be allowed to go below – 20°C to avoid damage to seals, etc.

[3]Holding time begins from time of sample collection or date thawed (see note #2 above).

[4]Presence of chlorine residual is usually associated with drinking water samples. Confirm dechlorination. If residual chlorine >5 mg/L, additional dechlorination agent may be required.

[5]Nitroaromatic and nitramine sample extracts must be stored at 4°C, protected from light, and stored in sealed vials (e .g., screw-cap or crimp-capped vials) with un-pierced PTFE-lined septa.

[6]As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, September 2007, if the holding time is exceeded by >2x the allowable holding time, data users should consider nondetect results as unusable and positive results as estimated with a significantly low bias.

[7]If samples were received by the laboratory on the same day of collection and were stored and transported to the laboratory on ice, cooler temperatures above 6°C are acceptable.

Appendix VIII A-2

Data Deliverable Requirements for Data Audits

If requested by MassDEP, submission of the information listed below may be required to perform a data audit to verify compliance with the analytical methods and to evaluate accuracy and reliability of the reported results. These deliverables represent a "full data package" including all sample documentation from receipt through preparation, analysis, and data reporting. The laboratory must ensure that these deliverables are available, in the event a data audit is performed. The laboratory is required to retain these deliverables for a period of 10 years from the date generated.

DELIVERABLE REQUIREMENTS FOR DATA AUDITS **WSC-CAM-VIII A (Nitroaromatics and Nitramines by HPLC)**	
Laboratory Narrative	Must comply with the required laboratory narrative contents as described in WSC-CAM-VII A
Sample Handling Information	Chains-of-custody (external and internal), sample receipt logs (cooler temperatures and sample pH), correspondences
Miscellaneous Logs	Dry weight logs Injection logs Soil/sediment sample weight logs Freezer logs Sample preparation/cleanup logs[1]
Initial Calibration Data (both columns)	Summary of calibration factors for all standards in initial calibration; average calibration factors, %RSDs, correlation coefficients, and coefficients of determination for all target compounds Chromatograms for all standards used in initial calibration Quantitation reports for all standards used in initial calibration Concentrations of standards used must be clearly presented
Initial Calibration Verification Data (both columns)	Summary of percent recoveries for all target compounds Chromatograms for all ICVs Quantitation reports for all ICVs
Continuing Calibration Data (both columns)	Summary of %Ds and calibration factors Chromatograms for all continuing calibration standards Quantitation reports for all continuing calibration standards Concentrations of standards used must be clearly presented
Sample Results (both columns)	Chromatograms for all sample analyses, reanalyses, and dilutions

DELIVERABLE REQUIREMENTS FOR DATA AUDITS **WSC-CAM-VIII A (Nitroaromatics and Nitramines by HPLC)**	
	Quantitation reports for all sample analyses, reanalyses, and dilutions Percent solids results Summary of results, including reporting limits for each sample Date of analysis
Method Blank Results (both columns)	Chromatograms for all method blanks Quantitation reports for all method blanks Summary of results, including reporting limits Summary of how method blank was prepared in solid and aqueous matrices, as appropriate
LCS/LCS Duplicate Results (both columns)	Chromatograms for all LCS and LCS Duplicates Quantitation reports for all LCS and LCS Duplicates Summary of results, including concentrations detected, concentrations spiked, percent recoveries and RPDs Summary of how LCS/LCS Duplicates were prepared in solid and aqueous matrices, as appropriate
MS/MSD Results (if performed) (both columns)	Chromatograms for all MS/MSDs Quantitation reports for all MS/MSDs Summary of results, including unspiked sample concentrations, concentrations detected, concentrations spiked, percent recoveries and RPDs Summary of how MS/MSDs were prepared in solid and aqueous matrices, as appropriate
QC Summaries (both columns)	Surrogate recoveries Retention time windows Dual column RPDs
Other Information	Demonstration that ICV prepared from second source standard
Quantitation reports must exhibit peak area counts or peak heights, as appropriate, of target compounds, internal standards, and surrogates. [1]Must clearly indicate sample weights or volumes, final extract volumes, extraction method used, extraction times where appropriate for the method, etc.	