

WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 1 of 27

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)

### WSC - CAM - VIII A



WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 2 of 27

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)

### **Table of Contents**

	Acronym Lis	st .	3
1.0	Quality Con	trol Requirements and Performance Standards for WSC-CAM-VIII A	4
	1.1 Overvie	w of WSC-CAM-VIII A	4
	1.2 Summa	ry of SW-846 Method 8330A	6
	1.3 Sample	Extraction Methods for WSC-CAM-VIII A	7
	1.4 Method	Interferences	7
	1.5 Quality	Control Requirements and Performance Standards for WSC-CAM-VIII A	8
	1.6 Special	Analytical Considerations for WSC-CAM-VIII A	9
	1.7 Analyte	List for WSC-CAM-VIII A	18
2.0	Data Usabil	ty Assessment	21
3.0	Reporting R	equirements for WSC-CAM-VIII A	21
	3.1 General	Reporting Requirements for WSC-CAM-VIII A	21
	3.2 Specific	Reporting Requirements for WSC-CAM-VIII A	21
_ist of T	ables and Ap	<u>pendices</u>	
Table	VIII A-1	Specific QC Requirements and Performance Standards for WSC-CAM-VIII A	11-17
Table	VIII A-2	Analyte List for WSC-CAM-VIII A	20
Table	VIII A-3	Routine Reporting Requirements for WSC-CAM-VIII A	22
Apper	ndix VIII A-1	Sample Collection, Preservation and Handling Procedures for Nitroaromatic and Nitramine Analyses	23-24
Appe	ndix VIII A-2	Data Deliverable Requirements for Data Audits	25-27



WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 3 of 27

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)

#### **ACRONYM LIST**

CAM Compendium of Analytical Methods
CASN Chemical Abstracts Service Number
%D Percent difference or percent drift

DF Dilution factor
1,2-DNB 1,2-Dinitrobenzene
3,4-DNT 3,4-Dinitrotoluene
Electron capture de

ECD Electron capture detector GC Gas chromatograph

HMX Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine HPLC High performance liquid chromatography

IRAs Immediate Response Actions
LCS Laboratory control sample
LLOQ Lower limit of quantitation

Massachusetts Department of Environmental Protection

MCP Massachusetts Contingency Plan

MD Matrix duplicate

MOHML Massachusetts Oil and Hazardous Materials List

MS Matrix spike

MSD Matrix spike duplicate

NA Not applicable nm Nanometer PDA Photodiode array

PETN Pentaerythritol tetranitrate
Psi Pounds per square inch
PTFE Polytetrafluoroethylene
QA Quality assurance
QC Quality control
r Correlation coefficient

r<sup>2</sup> Coefficient of determination 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

SPE Solid phase extraction

TNT Trinitrotoluene

μg/kg micrograms per kilogram μg/L micrograms per liter

μΙ Microliter UV Ultraviolet

UXO Unexploded ordnance



WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 4 of 27

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)

### 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 January 1, 2026, this revised CAM protocol, WSC-CAM-VIII A, replaces the previous version of the Nitroaromatic and Nitramine HPLC CAM document, WSC-CAM-VIII A (effective date, July 1, 2010). 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 January 1, 2026 for the purpose of "Presumptive Certainty," the revised protocol may be used optionally prior to its effective date upon its publication on October 1, 2025.

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 EPA SW-846 Method 8330B instead of 8330A, it is acceptable to answer "YES" to Question B on the MassDEP Analytical Protocol Certification Form; although EPA SW-846 Method 8330B is not yet promulgated, improvements in the preparation and extraction of samples in EPA SW-846 Method 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 or Lower Limits of Quantitation for WSC-CAM-VIII A

The reporting limit (RL) or lower limit of quantitation (LLOQ) 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. The CAM RLs/LLOQs for WSC-CAM-VIII A target analytes are:



WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 5 of 27

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)

- > 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 this CAM protocol, including an HPLC-system equipped with a pump capable of achieving 4000 pounds per square inch (psi), a 100 µl loop injector and a 254 nm ultraviolet (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/LLOQs are not achieved, respond "NO" to Question G of the "MassDEP MCP Analytical Protocol Certification Form" and address the CAM RL/LLOQ exceedance in the laboratory narrative.

RLs/LLOQs lower than the above-referenced CAM RLs/LLOQs for WSC-CAM-VIII A target analytes may be required to satisfy project requirements. The RL/LLOQ (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/LLOQs 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 (QA) 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 (MD) 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 8000D. Procedural requirements for performing the Initial Demonstration of Proficiency can be found in SW-846 Method 8000D (Section 9.3) 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 8000D, Section 9.3
% Relative Standard Deviation	SW-846 Method 8000D, Section 9.3
Surrogate Recovery	WSC-CAM-VIII A, Table VIII A-1



WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 6 of 27

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)

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 8000D, Section 9.6. 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 HPLC, interpretation of chromatograms, handling of 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 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 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).



January 1, 2026	Revision No. 2 Page 7 of 27
WSC-CAM	Section: 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)

<u>Analytical Note</u>: 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 may yield comparable or superior chromatographic separations.

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

As noted above, 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/LLOQ.

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 (gas chromatograph/electron capture 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 8000D 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.



WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 8 of 27

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)

- 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 crosscontamination/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 and Performance Standards for WSC-CAM-VIII A

#### 1.5.1 General QC Requirements

Refer to SW-846 Method 8000D 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.



WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 9 of 27

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)

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) and 40.1057(2)(k) for Permanent and Temporary Solution 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.</li>
- 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.



Final	Page 10 of 28
January 1, 2026	Revision No. 2
WSC-CAM	Section: 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)

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 <u>never</u> 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 collect as an equipment blank.



WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 11 of 27

Table VII	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 <sup>1</sup>	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency	Laboratory Analytical Accuracy & Precision	<ul> <li>(1) Must be performed prior to using method on samples.</li> <li>(2) Must be performed for each matrix.</li> <li>(3) Must contain all target analytes.</li> <li>(4) Must follow procedure in Section 9.3 of SW-846 8000D and Section 9.4 of SW-846 8330A.</li> </ul>	No	NA	Refer to Section 9.3 of SW- 846 8000D and Section 1.1.2 of this protocol.	NA
Retention Time Windows	Laboratory Analytical Accuracy	<ul> <li>(1) Prior to initial calibration and when a new HPLC column is installed.</li> <li>(2) Calculated according to the method (Section 11.6 of SW-846 8000D).</li> </ul>	No	NA	NA	NA
Initial Calibration	Laboratory Analytical Accuracy	<ul> <li>(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.</li> <li>(2) Minimum of 5 standards (or 6 if non-linear regression used).</li> <li>(3) Low standard must be ≤RL/LLOQ.</li> <li>(4) %RSD ≤20, r ≥0.99 (linear regression), or r² ≥0.99 (non-linear regression) for each target explosive.</li> <li>(5) If %RSD &gt;20, linear or non-linear regression must be used.</li> <li>(6) Must contain all target explosives.</li> <li>(7) Calibration must be performed under the same conditions as the samples.</li> <li>(8) If linear or non-linear regression used, verify the RL/LLOQ by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%.</li> </ul>	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/LLOQ must be reported as an estimated value², 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.  Report non-conforming compounds (%RSD >20, r <0.99, or r² <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.



-	VSC-CAM January 1, 2026	Section: VIII A  Revision No. 2

Table VII	Table VIII A-1: Specific QC Requirements and Performance Standards for Explosives (SW-846 8330A) Using WSC-CAM-VIII A					AM-VIII A
Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350 <sup>1</sup>	Required Corrective Action	Required Analytical Response Action
Initial Calibration Verification	Laboratory Analytical Accuracy	<ol> <li>(1) Immediately after each initial calibration.</li> <li>(2) Concentration level near midpoint of curve.</li> <li>(3) Prepared using standard source different than used for initial calibration.</li> <li>(4) Must contain all target explosives.</li> <li>(5) Percent recoveries must be between 70-130% for each target analyte.</li> </ol>	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	<ul> <li>(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.</li> <li>(2) Concentration level near mid-point of curve.</li> <li>(3) Must contain all target explosives.</li> <li>(4) Percent difference or percent drift (%D) must be ≤20 for each target analyte.</li> <li>(5) Verify that all analytes fall within retention time windows.</li> </ul>	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.



WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 13 of 27

Table VII	Table VIII A-1: Specific QC Requirements and Performance Standards for Explosives (SW-846 8330A) Using WSC-CAM-VIII A					AM-VIII A
Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350 <sup>1</sup>	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 lloq.<="" td=""><td>Yes</td><td>NA</td><td>(1) If concentration of contaminant in sample is &lt;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 &gt;10x concentration in blank or if contaminant not detected in sample.</td><td>(1) If sample re-extraction is not possible, report nonconformance 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 yields 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.</td></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 nonconformance 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 yields 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.



January 1, 2026	Revision No. 2
Final	Page 14 of 27

Table VIII A-1: Specific QC Requirements and Performance Standards for Explosives (SW-846 8330A) Using WSC-CAM-VIII A					AM-VIII A	
Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350 <sup>1</sup>	Required Corrective Action	Required Analytical Response Action
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	<ul> <li>(1) Extracted with every batch or every 20 samples, whichever is more frequent.</li> <li>(2) Concentration level near midpoint of curve.</li> <li>(3) Must contain all target explosives.</li> <li>(4) Matrix-specific (e.g., soil, water).</li> <li>(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.</li> <li>(6) Must be prepared in methanol.</li> </ul>	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%), re-extraction is not required if affected compounds were not detected in associated samples.	(1) If sample re-extraction is not possible, report nonconformance 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 reextraction 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	<ul> <li>(1) Extracted with every batch or every 20 samples, whichever is more frequent.</li> <li>(2) Concentration level near midpoint of curve.</li> <li>(3) Must contain all target explosives.</li> <li>(4) Matrix-specific (e.g., soil, water).</li> <li>(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.</li> <li>(6) Relative percent differences (RPDs) must be ≤20 for waters and ≤30 for solids.</li> <li>(7) Must be prepared in methanol.</li> </ul>	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LCS.	<ul> <li>(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if &gt;10% of all analytes are outside of recovery acceptance criteria.</li> <li>(2) If &lt;10% of compounds are outside of the recovery acceptance criteria, re-extraction is not required as long as recoveries are &gt;10%.</li> <li>(3) If &gt;10% of compounds are above the recovery acceptance criteria (&gt;140%), re-extraction is not required if affected</li> </ul>	(1) If sample re-extraction is not possible, report nonconformance 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.  (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the reextraction only.



WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 15 of 27

Table VII	Table VIII A-1: Specific QC Requirements and Performance Standards for Explosives (SW-846 8330A) Using WSC-CAM-VIII A				AM-VIII A	
Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350 <sup>1</sup>	Required Corrective Action	Required Analytical Response Action
					compounds were not detected in associated samples.	(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	<ul> <li>(1) Every 20 samples (at discretion of laboratory or at request of data user).</li> <li>(2) Matrix-specific.</li> <li>(3) Concentration level near midpoint of curve.</li> <li>(4) Must contain all target explosives.</li> <li>(5) Percent recoveries between 40 – 140%.</li> <li>(6) RPDs ≤20 for waters and ≤30 for solids.</li> <li>(7) Must be prepared in methanol.</li> </ul>	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-Dinitrobenzene (1,2-DNB) or 3,4-Dinitrotoluene (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 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%), re-analyze sample	(1) Report recoveries outside of 30-130% in laboratory narrative. (2) If re-extraction yields similar surrogate nonconformances, the laboratory must report results of both the initial extraction and re-extraction. (3) If re-extraction is 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 may report results of the re-extraction only.



WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 16 of 27

Table VII	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 <sup>1</sup>	Required Corrective Action	Required Analytical Response Action
					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.	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 NA	<ol> <li>(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.</li> <li>(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.</li> <li>(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.</li> <li>(4) Results must be reported with 2 or more "significant figures" if &gt; RL/LLQQ, report with 1 or more "significant figures".<sup>3</sup></li> </ol>	NA	If RPD >100 for target explosive, reject positive result for affected explosive.	If the RPD between the dual column results >100, re-analyze the sample on dilution. Both analyses must be reported.	(1) 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.  (2) 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.



WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 17 of 27

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 <sup>1</sup>	Required Corrective Action	Required Analytical Response Action
General Reporting Issues	NA NA	<ul> <li>(1) The laboratory must only report values ≥ the sample-specific RL/LLOQ.</li> <li>(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.</li> <li>(3) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards.</li> <li>(4) Refer to Appendix VIII A-1 for chain-of-custody requirements regarding preservation, cooler temperature, and holding times.</li> </ul>	NA	NA NA	NA	(1) Complete analytical documentation for dilute 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 or the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboraton 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 analyzed outside the holding time, note the non-conformances in the laboratory narrative.

<sup>&</sup>lt;sup>1</sup>As per Appendix IV of MassDEP Policy #WSC-07-350, MCP Representativeness Evaluations and Data Usability Assessments, if these results are observed, data users should consider nondetect results as unusable and positive results as estimated with a significant low bias.

<sup>&</sup>lt;sup>2</sup>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.

<sup>3</sup>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.



WSC-CAM	Section: VIII A	
January 1, 2026	Revision No. 2	
Final	Page 18 of 27	

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)

### 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/LLOQ for the target analytes. Sources of various MassDEP standards and criteria are as follows:

- Reportable Quantities (RQs) and Reportable 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: <a href="http://www.mass.gov/dep/cleanup/laws/regulati.htm#mcp">http://www.mass.gov/dep/cleanup/laws/regulati.htm#mcp</a>
- An online searchable Oil & Hazardous Materials List of RQs and RCs values may be found at the following URL: <a href="http://eeaonline.eea.state.ma.us/DEP/MOMHL/hazmat.aspx">http://eeaonline.eea.state.ma.us/DEP/MOMHL/hazmat.aspx</a>
- An updated list of MCP Method 1 Standards may be found at the following URL: https://www.mass.gov/regulations/310-CMR-4000-massachusetts-contingency-plan

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-Dinitrotoluene 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 RCs 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 RC.

### 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 (16) analyte list, including the four (4) TNT metabolites, when previously uncharacterized <u>ordnance-impacted sites</u> 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:



WSC-CAM	Section: VIII A	
January 1, 2026	Revision No. 2	
Final	Page 19 of 27	

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)

- ✓ 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.



WSC-CAM	Section: VIII A	
January 1, 2026	Revision No. 2	
Final	Page 20 of 27	

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)

Analyte	CASN
HMX	2691410
RDX (Cyclonite) <sup>1</sup>	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	78115
Picric Acid (Trinitrophenol)	88891

 $^{1}\underline{RDX}$  is identified as Cyclonite in the MOHML in Subpart P of the MCP

**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.

<u>HMX</u> - Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine <u>RDX</u> - Hexahydro-1,3,5-trinitro-1,3,5-triazine

 $\underline{\underline{\mathsf{Tetryl}}} \text{ -} \underline{\mathsf{Methyl-2,4,6-trinitrophenylnitramine}} \qquad \underline{\underline{\mathsf{PETN}}} \text{ -} \underline{\mathsf{Pentaerythritol}} \text{ tetranitrate}$ 



WSC-CAM	Section: VIII A	
January 1, 2026	Revision No. 2	
Final	Page 21 of 27	

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)

### 2.0 Data Usability Assessment

Specific guidance applicable to all Permanent and Temporary Solutions, including Permanent and Temporary Solutions on a portion of a disposal site, for preparation of Representativeness Evaluations and Data Usability Assessments pursuant to 310 CMR 40.1056(2)(k) and 40.1057(2)(k), respectively, 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 a Permanent or Temporary Solution submittal. The most current version of this document may be found at the following URL: <a href="http://www.mass.gov/dep/cleanup/laws/policies.htm#finpol.">http://www.mass.gov/dep/cleanup/laws/policies.htm#finpol.</a>

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)". Requirements listed as "YES" must always be included as part of the laboratory deliverable for this method. It should be noted that data for those items listed as "NO" under "Required Analytical Deliverables" must be available for review during an audit and may also be requested for inclusion in the analytical deliverable on a client-specific basis.



WSC-CAM	Section: VIII A	
January 1, 2026	Revision No. 2	
Final	Page 22 of 27	

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)

Parameter	Required Analytical Deliverable
Retention Time Windows	NO
Initial Calibration	NO
Initial Calibration Verification	NO
Continuing Calibration	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/LLOQ for each nitroaromatic or nitramine must be adjusted (increased) in direct proportion to the Dilution Factor (DF).

The revised RL/LLOQ for the diluted sample, RL/LLOQd:

RL/LLOQ<sub>d</sub> = DF X Lowest Calibration Standard for Target Analyte

It should be understood that samples with elevated RLs/LLOQs as a result of a dilution may not be able to satisfy MCP standards/criteria in some cases if the RL/LLOQ $_d$  is greater than the applicable MCP standard or criterion to which the concentration is being compared. Such increases in RLs/LLOQs 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 50% of its associated highest calibration standard. This will avoid unnecessarily high RLs/LLOQs for other target analytes which did not require dilution.



WSC-CAM	Section: VIII A	
January 1, 2026	Revision No. 2	
Final	Page 23 of 27	

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)

### **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).



WSC-CAM	Section: VIII A	
January 1, 2026	Revision No. 2	
Final	Page 24 of 27	

Matrix	Container <sup>1</sup>	Preservation <sup>7</sup>	Holding Time <sup>3,6</sup>
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 <sub>4</sub> (1.2 g/L)	7 days to extraction; 40 days from extraction to analysis <sup>5</sup>
Aqueous Samples, with Residual Chlorine <sup>4</sup>	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Add 1-mL 10% sodium thiosulfate solution per container (or 0.008%) <sup>4</sup> . Addition of thiosulfate solution to sample container may be performed in the laboratory prior to field use.	7 days to extraction; 40 days from extraction to analysis <sup>5</sup>
		Store in dark; Cool to ≤ 6°C but not frozen.	
Soil/Sediment Samples	(1) 8-oz. amber glass jar w/ a Teflon-lined screw cap <sup>2</sup>	Store in dark; Cool to ≤ 6°C <sup>2</sup>	14 days to extraction; 40 days from extraction to analysis <sup>2,5</sup>
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 <sup>5</sup>

<sup>&</sup>lt;sup>1</sup>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.

<sup>&</sup>lt;sup>2</sup>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. <u>Sampling container should only be filled to 2/3 of capacity to avoid breakage caused by expansion during freezing</u>. 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. Once the thawing process begins, samples must be kept at 0-6°C until extraction.

<sup>&</sup>lt;sup>3</sup>Holding time begins from time of sample collection or date thawed (see note #2 above).

<sup>&</sup>lt;sup>4</sup>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.

<sup>&</sup>lt;sup>5</sup>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.

<sup>&</sup>lt;sup>6</sup>As per Appendix IV of MassDEP Policy #WSC-07-350, MCP Representativeness Evaluations and Data Usability Assessments, 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.

<sup>&</sup>lt;sup>7</sup>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.



WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 25 of 27

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)

### **Appendix VIII A-2**

**Data Deliverable Requirements for Data Audits** 



WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 26 of 27

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)

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: SW-846 8330A)		
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 <sup>1</sup>	
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 initial calibration	
	verifications	
	Quantitation reports for all initial calibration	
	verifications	
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	



WSC-CAM	Section: VIII A
January 1, 2026	Revision No. 2
Final	Page 27 of 27

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)

	<b>,</b>	
DELIVERABLE REQUIREMENTS FOR DATA AUDITS		
WSC-CAM-VIII A (Nitroaromatics and Nitramines by HPLC: SW-846 8330A)		
	Quantitation reports for all sample analyses, reanalyses, and dilutions	
	Percent solids results	
	Summary of results, including RLs/LLOQs 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 RLs/LLOQs	
	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	
l 🕳		

Quantitation reports must exhibit peak area counts or peak heights, as appropriate, of target compounds and surrogates.

<sup>1</sup>Must clearly indicate sample weights or volumes, final extract volumes, extraction method used, extraction times where appropriate for the method, etc.