



Massachusetts Department of Environmental  
Protection Bureau of Waste Site Cleanup

WSC-CAM

Section: VI A

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Quality Control Requirements and Performance Standards for the ***Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

## **WSC – CAM – VI A**

Quality Control Requirements and Performance Standards for  
the **Analysis of Total Cyanide and Physiologically  
Available Cyanide (PAC)** in Support of Response Actions  
under the Massachusetts Contingency Plan (MCP)

|                                                                                                                                                                                                                                     |                  |                |
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|  Massachusetts Department of Environmental<br>Protection Bureau of Waste Site Cleanup                                                               | WSC-CAM          | Section: VI A  |
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## VI. Miscellaneous Wet Chemical Methods

### A. Quality Control Requirements and Performance Standards for WSC-CAM-VI A (Total Cyanide and Physiologically Available Cyanide)

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ACRONYM LIST

|                 |                                                                 |
|-----------------|-----------------------------------------------------------------|
| CAM             | Compendium of Analytical Methods                                |
| CASN            | Chemical Abstracts Service Number                               |
| CCB             | Continuing calibration blank                                    |
| CCV             | Continuing calibration verification                             |
| CN <sup>-</sup> | Cyanide ion (Free Cyanide)                                      |
| CNCl            | Cyanogen chloride                                               |
| COC             | Chain-of-custody                                                |
| DF              | Dilution factor                                                 |
| HCN             | Hydrocyanic acid                                                |
| ICB             | Initial calibration blank                                       |
| ICV             | Initial calibration verification                                |
| KCN             | Potassium cyanide                                               |
| LCS/LCSD        | Laboratory control sample / Laboratory control sample duplicate |
| LCS-N           | Negative LCS for PAC                                            |
| LCS-P           | Positive LCS for PAC                                            |
| LLCV            | Low-level calibration verification                              |
| LLOQ            | Lower limit of quantitation                                     |
| MassDEP         | Massachusetts Department of Environmental Protection            |
| MB              | Method blank                                                    |
| MCP             | Massachusetts Contingency Plan                                  |
| MD              | Matrix duplicate                                                |
| MOHML           | Massachusetts Oil and Hazardous Materials List                  |
| MS              | Matrix spike                                                    |
| NaOH            | Sodium hydroxide                                                |
| PAC             | Physiologically Available Cyanide                               |
| QA              | Quality assurance                                               |
| QC              | Quality control                                                 |
| %R              | Percent recovery                                                |
| r               | Correlation coefficient                                         |
| RCs             | Reportable Concentrations                                       |
| RL              | Reporting limit                                                 |
| RPD             | Relative percent difference                                     |
| RQs             | Reportable Quantities                                           |
| SRM             | Standard reference material                                     |
| USEPA           | United States Environmental Protection Agency                   |
| UV              | Ultraviolet                                                     |

UNITS:

|       |                        |
|-------|------------------------|
| L     | Liter                  |
| mg/L  | Milligram per liter    |
| mg/kg | Milligram per kilogram |
| mL    | Milliliter             |
| nm    | nanometer              |
| µm    | Micron                 |



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

### 1.1 Overview of WSC-CAM-VI A

WSC-CAM-VI A, *Quality Control Requirements and Performance Standards for the Analysis of Total Cyanide and Physiologically Available Cyanide (PAC) in Support of Response Actions under the Massachusetts Contingency Plan (MCP)*, is a component of MassDEP's Compendium of Analytical Methods (CAM). Effective October 15, 2024, this revised CAM protocol, WSC-CAM-VI A, replaces the previous version of the Cyanide CAM document, WSC-CAM-VI 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 October 15, 2024 for the purpose of "Presumptive Certainty," the revised protocol may be used optionally prior to its effective date upon its publication on July 15, 2024.

This document provides Quality Control (QC) requirements and performance standards to be used in conjunction with the required analytical methods SW-846 9014 (manual colorimetric using UV spectrometry), SW-846 9012B (automated colorimetric using UV spectrometry), or Standard Method 4500-CN<sup>-</sup> (manual colorimetric using UV spectrometry; *Standard Methods for the Examination of Water and Wastewater*, Part 4000, 20<sup>th</sup> Edition), for the analysis of aqueous and solid samples for Total Cyanide and Physiologically Available Cyanide (PAC). The QC requirements and performance standards specified in this document in Table VI A-1 together with the analytical procedures described in the referenced methods constitute the WSC-CAM-VI 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 9014, 9012B, or Standard Method 4500-CN<sup>-</sup> (or a subsequent/more current version of each method) is a "Presumptive Certainty" requirement of WSC-CAM-VI A.

Sample preservation, container and analytical holding time specifications for aqueous, soil, and sediment matrices for Cyanide/PAC analyzed in support of MCP decision-making are presented in Appendix VI 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 Cyanide/PAC by WSC-CAM-VI A

The reporting limit (RL) or lower limit of quantitation (LLOQ) for Cyanide/PAC using WSC-CAM-VI A is dependent on the concentration of the lowest non-zero standard in the initial calibration or the low-level calibration verification (LLCV), analyzed under identical conditions as the sample, with adjustments made for the sample size, preparation factors, percent solids, dilution factors, etc., as required. The CAM RLs/LLOQs for Cyanide/PAC using the WSC-CAM-VI A protocol are:

- 0.005-0.010 mg/L for aqueous samples (surface water, groundwater, and drinking water); and
- 1.0 mg/kg for soil/sediment samples (assuming 100% solids).



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For “Presumptive Certainty” purposes, if the typical 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 Cyanide/PAC may be required to satisfy project requirements. The RL/LLOQ (based on the concentration of the lowest calibration standard or the LLCV) for Cyanide/PAC 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 analytical modifications to improve sensitivity. All such modifications must be described in the laboratory narrative. RLs/LLOQs for Cyanide/PAC will be proportionately higher for samples that require dilution, when a reduced sample size is used, or when the sample has a relatively high percent moisture (low percent solids).

#### 1.1.2 Initial Demonstration of Proficiency for WSC-CAM-VI A

Each laboratory that uses the WSC-CAM-VI 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, the preparation/analysis of laboratory control samples (LCS) and LCS duplicates (LCSD) to assess analytical accuracy and precision. Matrix spikes (MS) or matrix duplicates (MD) may also be used to evaluate accuracy and/or 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 VI A-1 of this protocol. General requirements for performing the Initial Demonstration of Proficiency can be found in SW-846 Chapter One and Section 9.2 of SW-846 Method 9014. 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-VI A must include the following information:

| QC Element                                                                                      | Performance Criteria           |
|-------------------------------------------------------------------------------------------------|--------------------------------|
| Initial Calibration                                                                             | See WSC-CAM-VI A, Table VI A-1 |
| Continuing Calibration Verification                                                             |                                |
| Method Blank                                                                                    |                                |
| Percent Recovery for<br>Total Cyanide: LCS & MS<br>PAC: LCS-P, LCS-N, & MS                      |                                |
| Relative Percent Difference (RPD) for<br>Total Cyanide: LCSD or MD<br>PAC: LCSD-P, LCSD-N or MD |                                |
| High Concentration Distilled Standard                                                           | ±10% of true value             |
| Low Concentration Distilled Standard                                                            |                                |



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Laboratories are encouraged to actively monitor pertinent QC performance standards described in Table VI 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-VI A protocol, laboratory-specific control limits must meet or exceed (demonstrate less variability than) the performance standards for each QC element listed in Table VI A-1. It should be noted that the performance standards listed in Table VI 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 who are experienced in using colorimetric UV spectrometry as a quantitative tool for environmental analyses and knowledgeable in the minimization of interferences described in this method.

## 1.2 Summary of Cyanide/PAC Methods

The following determinative methods may be used for analysis of Total Cyanide or PAC in solution/distillates with WSC-CAM-VI A:

- SW-846 Method 9014 – manual colorimetric ultraviolet (UV) spectrometry for the determination of free (non-complexed) Cyanide ( $\text{CN}^-$ ) and hydrocyanic acid (HCN) in solution/distillates
  - This method also includes titrimetric determination; however, the sensitivity of the titrimetric method may not meet data quality objectives (see Section 1.6 of this WSC-CAM-VI A protocol for further information)
- SW-846 Method 9012B – automated colorimetric UV spectrometry for the determination of free  $\text{CN}^-$  and HCN in solution/distillates
- Standard Method 4500- $\text{CN}^-$  – includes colorimetric (equivalent to SW-846 Method 9014), titrimetric, and potentiometric procedures for the determination of HCN in distillates

The definitions of different Cyanide species are defined as follows.

- Total Cyanide includes Free Cyanide plus nitriles (organic cyanides), other simple cyanides such as cyanide salts, and stable metallo-cyanide complexes including iron-cyanides. Total Cyanide is defined as the sum of cyanides, as HCN, released during the aggressive catalytic, mineral acid reflux distillation procedure described in SW-846 Method 9010C.
- Free Cyanide (non-complexed) is defined as the sum of cyanide, as HCN and cyanide ion ( $\text{CN}^-$ ). Free Cyanide may be determined directly using SW-846 Method 9213 without reflux distillation. Alternatively, SW-846 Method 9213 can be used to measure free cyanide in distillates, with higher RLs/LLOQs than SW-846 Method 9014 (see Section 1.6 of this WSC-CAM-VI A protocol for further details).
- Physiologically Available Cyanide (PAC) as determined by the MassDEP PAC protocol, includes biologically available cyanides that are released during this modified distillation including free cyanide, simple cyanide salts, and some metal-cyanide complexes that are easily dissociated. This protocol will **not** release iron-cyanide complexes from samples.

Cyanides are released from samples using reflux-distillation (see Section 1.3 below), which results in HCN in the distillate. The distillate is treated with chloramine-T at pH <8 to convert the HCN into cyanogen chloride ( $\text{CNCl}$ ) and color is formed by adding pyridine-barbituric acid. Quantitation of Cyanide in the



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treated distillates is based on the color absorption at 578-nm wavelength using a UV spectrometer.

Absorbance (peak height) is measured as a function of Cyanide/PAC concentration, based on a multi-level calibration curve.

Prior to analysis, the aqueous and solid (soil/sediment) samples must be prepared and distilled according to the procedures described in Section 1.3 below.

### 1.3 Sample Distillation/Preparation Methods for WSC-CAM-VI A

The aggressiveness of the sample digestion associated with the preparation method selected determines the range of cyanide salts and insoluble cyanide complexes that will ultimately be decomposed and captured in the absorber solution and detected by the selected determinative method. Total Cyanide prepared using SW-846 Method 9010C is the most aggressive preparation procedure (measuring almost all cyanide forms), while the MassDEP PAC Protocol, simulating the human digestive system, is the least aggressive preparation procedure (measuring only simple cyanides and excluding iron-cyanide complexes).

Samples for analysis by WSC-CAM-VI A must be prepared/distilled using one of the following methods.

| Preparation /<br>Distillation Method      | Matrix                      | Description                                                                                                                                                                                                                                                                                                                                                                                                  |
|-------------------------------------------|-----------------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| SW-846 9010C                              | Aqueous/Leachate/Waste      | <i>Total and Amenable Cyanide: Distillation</i><br>Reflux-distillation for acidic extraction of all forms of Cyanide for Total Cyanide in distillate (as HCN).<br><br>Macro and Micro-distillation procedures are allowed by modifying reagent volumes.                                                                                                                                                      |
| MassDEP PAC<br>Protocol                   | Aqueous/Soil/Sediment/Waste | <i>MassDEP Physiologically Available Cyanide (PAC)<br/>Protocol</i><br>Reflux-distillation for mildly acidic extraction of free cyanide, simple cyanide salts and other biologically available cyanides for PAC in distillate (as HCN). The PAC protocol does not extract iron-cyanide complexes ("Prussian Blue").<br><br>Macro and Micro-distillation procedures are allowed by modifying reagent volumes. |
| Standard Method<br>4500-CN <sup>-</sup> C | Aqueous/Soil/Sediment/Waste | <i>Total Cyanide After Distillation</i><br>Reflux-distillation for acidic extraction of all forms of cyanide for Total Cyanide in distillate (as HCN). This method is equivalent to SW-846 Method 9010C.                                                                                                                                                                                                     |
| SW-846 9013A                              | Solids and Oily Waste       | <i>Cyanide Extraction Procedure for Solids and Oils</i><br>Sample is first extracted with water at pH ≥10 and then distilled using SW-846 Method 9010C.                                                                                                                                                                                                                                                      |

### 1.4 Method Interferences

Refer to Section 3.0 of SW-846 Method 9010C and Standard Method 4500-CN<sup>-</sup> for further information on method interferences. Many potential interferences are eliminated in the acidic reflux-distillation procedure used for Total Cyanide (SW-846 Method 9010C and Standard Method 4500-CN<sup>-</sup> C). Several common



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interferences and corrective measures are summarized as follows.

- Oxidizing agents such as chlorine decompose most cyanide compounds and complexes. Chlorine interferences can be removed by adding an excess of ascorbic acid or sodium arsenite to aqueous samples prior to preservation and storage to reduce the chlorine ( $\text{Cl}_2$ ) to non-interfering chloride ( $\text{Cl}^-$ ).
- Sulfide interference can be removed by adding an excess of bismuth nitrate to the samples (to precipitate the sulfide) before distillation. Samples that contain hydrogen sulfide, metal sulfides, or other compounds that may produce hydrogen sulfide during the distillation should be treated by the addition of bismuth nitrate.
- Positive interferences for cyanide (high bias results) may be observed for samples that contain nitrate and/or nitrite at concentrations exceeding 10 mg/L. During the distillation, nitrate and nitrite will form nitrous acid, which will react with some organic compounds to form oximes. These compounds will decompose under test conditions to generate HCN. The possibility of interference by nitrate and nitrite is eliminated by pretreatment with sulfamic acid just before distillation.
- Fatty acids, detergents, surfactants, and other compounds may cause foaming during the distillation when they are present in high concentrations. Refer to Section 6.7 of Method SW-846 9010C for an extraction procedure to eliminate this interference.
- Samples containing solids of an amount and/or size as to interfere with agitation and homogenization of the sample mixture in the distillation flask, or so much oil or grease as to interfere with the formation of a homogeneous emulsion may be extracted with water (and hexane if heavy grease is present) at pH 10 or greater to minimize this potential interference as described in SW-846 Method 9013A, *Cyanide Extraction Procedure for Solids and Oils*.

## 1.5 Quality Control Requirements and Performance Standards for WSC-CAM-VI A

### 1.5.1 General QC Requirements

For general QC procedures for all inorganic methods, including SW-846 Methods 9012B and 9014, refer to SW-846 Chapter One. General QC procedures to evaluate the instrument's operation can also be found in SW-846 Chapter One, Section 2.0, and include evaluation of calibrations and performance of sample analyses.

### 1.5.2 Specific QC Requirements and Performance Standards for WSC-CAM-VI A.

Specific QC requirements and performance standards for Cyanide/PAC using the WSC-CAM-VI A protocol are presented in Table VI A-1. Refer to WSC-CAM-VII A for field QC requirements. ***Note that a project-specific matrix spike (MS) must be performed for Total Cyanide and PAC to evaluate accuracy in a solid matrix (soil/sediment) at a frequency of one per 20 samples per matrix.*** 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;





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- (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, respectively, 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-VI A

The following bullets highlight potential issues that may be encountered with the analysis of Cyanide or PAC using this protocol.

- Matrix Spike (MS) Recovery – ***A MS is required for WSC-CAM-VI A for Total Cyanide and PAC in solid matrices (soil/sediment) at a frequency of one per 20 samples per matrix.*** Consistent with the United States Environmental Protection Agency (USEPA) Region I data validation guidance, MassDEP requires rejection of non-detected Cyanide/PAC results with <30% recovery in the MS if the concentration of Cyanide/PAC in the unspiked sample is <4x the amount spiked. If the MS recovery is <30% and non-detected results were reported for Cyanide/PAC, the laboratory must follow the required corrective actions listed on Table VI A-1.
  - Laboratories are not required to monitor whether or not MSs are performed on soil/sediment samples at a frequency of one per 20 samples per matrix. This is the responsibility of the data user.
  - For “Presumptive Certainty” purposes, if the data user does not submit a soil/sediment sample for MS analysis, Question H of the “MassDEP MCP Analytical Protocol Certification Form” must be answered NO and this must be noted in the laboratory narrative.
- RLs/LLOQs, sensitivity, and/or the optimum linear concentration range can vary with the cyanide compound or complex, sample matrix and laboratory operating conditions.
- Ion-selective electrode analysis by SW-846 Method 9213 or the titrimetric analysis by SW-846 Method 9014 may be used in place of the colorimetric spectrometry method for samples with high levels of Cyanide/PAC (>0.01 mg/L for SW-846 Method 9213 and >0.1 mg/L for SW-846 Method 9014). Data users should note that these analytical methods are not sensitive enough to meet the lowest MCP Method 1 Cleanup Standards for Cyanide/PAC.
- The RCRA-defined “reactive” cyanide content of a waste (the cyanide content that could generate toxic fumes when exposed to mild acidic conditions) is not determined by this WSC-CAM-VI A protocol. Refer to Chapter Seven of SW-846 for additional information on reactive cyanide.
- For the colorimetric methods of analysis (SW-846 9014 and Standard Method 4500-CN<sup>-</sup>), it is important to have the same reagent concentrations in both the sample and the standards to obtain



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colors of comparable intensity.

- Some cyanide complexes, such as Potassium Ferricyanide,  $K_3 [Fe-(CN)_6]$ , may be susceptible to photodegradation when exposed to fluorescent lighting or sunlight during sample handling and/or analysis. This photodegradation can change the type of the cyanide compounds in the sample, which is critical for the PAC method (such a change will not affect Total Cyanide measurements). Therefore, if PAC is being measured, due caution must be exercised to limit the exposure of the sample to light to maintain the integrity of sample from collection through analysis.
- Appendix VI A-3 provides a typical analysis sequence for Total Cyanide or PAC analyzed using this CAM protocol.

#### 1.7 Analyte List for WSC-CAM-VI A

The MCP analyte list for WSC-CAM-VI A consists of Cyanide, Chemical Abstracts Service Number (CASN) 57-12-5, as measured in distillates for either Total Cyanide or PAC.

It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL/LLOQ for Total Cyanide or PAC. 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:  
<http://www.mass.gov/dep/cleanup/laws/regulati.htm#mcp>
- An online searchable Oil & Hazardous Materials List of RQs and RCs values may be found at the following URL: <http://eeaonline.eea.state.ma.us/DEP/MOMHL/hazmat.aspx>
- 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>

Cyanide, expressed as PAC, has promulgated MCP Method 1 groundwater/soil standards. In the absence of PAC data, the MCP standards are applicable to Total Cyanide.



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Protection Bureau of Waste Site Cleanup

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**Table VI A-1: Specific QC Requirements and Performance Standards for Total Cyanide and PAC using WSC-CAM-VI 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                           | (1) Must be performed prior to using method on samples.<br>(2) Must be performed for each matrix.<br>(3) Must follow requirements in Section 1.1.2 of this protocol.                                                                                                                                                                                                                                                                                                                                                                                                                                                                              | No                    | NA                                             | Refer to Section 1.1.2 of this protocol.                                                                                                        | NA                                                             |
| Preparation of Samples                 | Accuracy and Representativeness                                      | (1) For Total Cyanide and PAC, all aqueous and solid samples must be distilled/prepared prior to analysis. See SW-846 9010C, 9013A, Standard Method 4500-CN C, and MassDEP PAC Protocol (Appendix VI A-4) for appropriate reflux distillation procedures.                                                                                                                                                                                                                                                                                                                                                                                         | No                    | NA                                             | NA                                                                                                                                              | NA                                                             |
| Initial Calibration                    | Laboratory Analytical Accuracy                                       | (1) Daily prior to sample analysis, when daily calibration QC samples (LLCV, CCV, CCB) are not in control, or when major instrument maintenance is performed.<br>(2) Minimum calibration blank plus 5 un-distilled potassium cyanide (KCN) calibration standards; high level standard in calibration defines the upper end of the linear calibration range.<br>(3) Low-level standard in calibration must be at or below the RL/LLOQ.<br>(4) Linear regression with correlation coefficient (r) $\geq 0.995$ .<br>(5) If titration procedure is used, the silver nitrate solution must be standardized as described in Standard Method 4500-CN D. | No                    | NA                                             | Perform instrument maintenance as necessary; recalibrate as required by method.                                                                 | Suspend all analyses until initial calibration meets criteria. |
| Initial Calibration Verification (ICV) | Laboratory Analytical Accuracy                                       | (1) Immediately after each initial calibration.<br>(2) Prepared using standard source different than used for initial calibration: un-distilled.<br>(3) Concentration level near midpoint of curve.<br>(4) Percent recovery must be 85-115%.                                                                                                                                                                                                                                                                                                                                                                                                      | No                    | NA                                             | (1) Reanalyze ICV; if acceptable, no further action required.<br>(2) If reanalysis is still outside of criteria, recalibrate and reanalyze ICV. | Suspend all analyses until ICV meets criteria.                 |
| Initial Calibration Blank (ICB)        | Laboratory Analytical Sensitivity (instrument drift & contamination) | (1) Immediately after ICV.<br>(2) Un-distilled.<br>(3) Cyanide/PAC must be <RL/LLOQ.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              | No                    | NA                                             | (1) Reanalyze ICB; if acceptable, no further action required.<br>(2) If reanalysis is still outside of criteria,                                | Suspend all analyses until ICB meets criteria.                 |



Quality Control Requirements and Performance Standards for the ***Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

**Table VI A-1: Specific QC Requirements and Performance Standards for Total Cyanide and PAC using WSC-CAM-VI 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                                                                                                                |
|-------------------------------------------|-------------------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------|
|                                           |                                                                                                 |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                       |                                                | recalibrate and reanalyze ICV & ICB.                                                                                                                                                                                                                                                                                                                                                                                                                |                                                                                                                                                    |
| Low-Level Calibration Verification (LLCV) | Laboratory Analytical Sensitivity<br><br>(verify low-end of calibration range / verify RL/LLOQ) | (1) Daily prior to sample analysis if initial calibration is not performed on same day as sample analysis. If initial calibration is performed on same day as sample analysis and includes the RL/LLOQ as the low-level standard in the initial calibration curve (as required by calibration), then LLCV is not required.<br><br>(2) Prepared using same source as initial calibration standards: un-distilled.<br><br>(3) Concentration level must be at the level of the RL/LLOQ for Cyanide/PAC.<br><br>(4) Percent recovery must be 70-130%. | No                    | NA                                             | (1) Reanalyze LLCV; if acceptable, no further action required.<br><br>(2) If reanalysis is still outside of criteria and concentrations of Cyanide/PAC are $\leq 10 \times$ RL/LLOQ in associated field samples, recalibrate and reanalyze LLCV and associated samples.<br><br>(3) If concentrations of Cyanide/PAC are $> 10 \times$ RL/LLOQ in associated field samples, include explanation in laboratory narrative; no further action required. | Suspend all analyses until LLCV meets criteria unless the concentrations of Cyanide/PAC are $> 10 \times$ RL/LLOQ in the associated field samples. |
| Continuing Calibration Verification (CCV) | Laboratory Analytical Accuracy                                                                  | (1) Every 20 field samples and at the end of the analytical run.<br><br>(2) Prepared using same source as initial calibration standards: un-distilled.<br><br>(3) Concentration level near midpoint of curve.<br><br>(4) Percent recovery must be 85-115%.                                                                                                                                                                                                                                                                                        | No                    | NA                                             | (1) Reanalyze CCV; if acceptable, no further action required.<br><br>(2) If reanalysis is still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCV – unless (3) applies.<br><br>(3) If recovery is high ( $> 115\%$ ) and all associated sample results are not detected, no corrective action required.                                                                                                | If (3) applies, include explanation in laboratory narrative.                                                                                       |



Quality Control Requirements and Performance Standards for the ***Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

**Table VI A-1: Specific QC Requirements and Performance Standards for Total Cyanide and PAC using WSC-CAM-VI 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                          |
|----------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|-------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------|
| Continuing Calibration Blank (CCB)                                                                       | Laboratory Analytical Sensitivity (instrument drift & contamination) | (1) Every 20 field samples following CCV and at the end of the analytical run.<br>(2) Un-distilled.<br>(3) Cyanide/PAC must be <RL/LLOQ.                                                                                                                                                                                                 | No                    | NA                                                                                                    | (1) Reanalyze CCB; if acceptable, no further action required.<br>(2) If reanalysis is still outside of criteria, recalibrate and reanalyze all associated samples since last compliant CCB – unless (3) applies.<br>(3) If concentration of Cyanide/PAC in CCB is >RL/LLOQ but all associated sample results are either not detected or >10x concentration of Cyanide/PAC in CCB, no corrective action required. | If (3) applies, include explanation in laboratory narrative  |
| Method Blank (MB)                                                                                        | Laboratory Method Sensitivity (contamination evaluation)             | (1) One per distillation batch of ≤20 field samples.<br>(2) Must be distilled with the samples in the batch using the same preparation method as the samples.<br>(3) Cyanide/PAC must be <RL/LLOQ.                                                                                                                                       | Yes                   | NA                                                                                                    | (1) Reanalyze MB; if acceptable, no further action required.<br>(2) If reanalysis is still outside of criteria, redistill and reanalyze MB and all associated field samples in batch – unless (3) applies.<br>(3) If concentration of Cyanide/PAC in MB is >RL/LLOQ but all associated sample results are either not detected or >10x concentration of Cyanide/PAC in MB, no corrective action required.         | If (3) applies, include explanation in laboratory narrative. |
| Laboratory Control Sample (LCS) for Total Cyanide and Positive Laboratory Control Sample (LCS-P) for PAC | Laboratory Analytical Accuracy (Method Performance for PAC)          | (1) One per distillation batch of ≤20 field samples.<br>(2) <b>Total Cyanide:</b> LCS must be matrix-matched by distilling with the samples using the same preparation method as the samples. CAM requires a Standard Reference Material (SRM) be prepared and analyzed with solid field samples as the “solid LCS.” An SRM is a soil or | Yes                   | Aqueous LCS or LCS-P:<br>Recovery <50%:<br>Cyanide/PAC results in associated samples may be rejected. | (1) Reanalyze LCS: if acceptable, no further action required.<br>(2) If reanalysis is still outside of criteria and LCS-D is in-control for Cyanide/PAC, no corrective action required.                                                                                                                                                                                                                          | Report recovery exceedances in laboratory narrative.         |



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Quality Control Requirements and Performance Standards for the **Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

**Table VI A-1: Specific QC Requirements and Performance Standards for Total Cyanide and PAC using WSC-CAM-VI 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                          |
|----------------------------------------------------------------|--------------------------------------------|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------|
|                                                                |                                            | sediment matrix that contains Cyanide at a known concentration and with 95% confidence limits.<br><u>PAC</u> : LCS-P is always an aqueous matrix but is distilled with both solid and aqueous samples.<br>(3) <u>Total Cyanide</u> : Concentration level for aqueous LCS near midpoint of curve.<br><u>PAC</u> : Concentration of LCS-P = 0.1 mg/L KCN.<br>(4) LCS and LCS-P must be distilled with samples in batch.<br>(5) <u>Total Cyanide</u> : Percent recovery must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS.<br><u>PAC</u> : Percent recovery must be 80-120%.                                                                                                                                                                                                                                                                                                                                                                           |                       |                                                | (3) If LCS and LCSD are both outside of recovery criteria, redistill and reanalyze LCS/LCSD and all associated field samples in batch.                                                                                                                                                                                                                              |                                                              |
| LCS Duplicate for Total Cyanide and<br>LCS-P Duplicate for PAC | Laboratory Analytical Accuracy & Precision | (1) One per distillation batch of ≤20 field samples ONLY if not performing project-specific MD.<br>(2) <u>Total Cyanide</u> : LCS Duplicate must be matrix-matched by distilling with the samples using the same preparation method as the samples. CAM requires a solid SRM be prepared and analyzed with solid field samples as the "solid LCS." An SRM is a soil or sediment matrix that contains Cyanide at a known concentration and with 95% confidence limits.<br><u>PAC</u> : LCS-P Duplicate is always an aqueous matrix but is distilled with both solid and aqueous samples.<br>(3) Concentration levels must be same as LCS.<br>(4) LCS Duplicate and LCS-P Duplicate must be distilled with samples in batch.<br>(5) <u>Total Cyanide</u> : Percent recovery must be 80-120% for aqueous LCS and within vendor control limits (95% confidence limits) for solid LCS.<br><u>PAC</u> : Percent recovery must be 80-120%.<br>(6) Analyze immediately after LCS or LCS-P in analytical sequence | Yes                   | Same as above for LCS for recovery evaluation. | (1) Reanalyze LCSD; if acceptable, no further action required.<br>(2) If reanalysis is still outside of recovery criteria for Cyanide/PAC, and LCS is in control for Cyanide/PAC recovery, no corrective action required.<br>(3) If LCSD and LCS are both outside of recovery criteria, redistill and reanalyze LCS/LCSD and all associated field samples in batch. | Report recovery and RPD exceedances in laboratory narrative. |



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Quality Control Requirements and Performance Standards for the **Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

**Table VI A-1: Specific QC Requirements and Performance Standards for Total Cyanide and PAC using WSC-CAM-VI 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                                                                                                  |
|------------------------------------------------------------|-----------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------|------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------------------------------------------------|
|                                                            |                                   | (7) <u>Total Cyanide</u> : RPDs must be $\leq 20$ for aqueous LCS/LCSD and $\leq 30$ for solid LCS/LCSD.<br><u>PAC</u> : RPDs must be $\leq 20$ for LCS-P/LCS-P Duplicate.                                                                                                                                                                                                                                                                                                                                                                           |                                          |                                                                                    |                                                                                                                                                                                                                                                                                                                                                                           |                                                                                                                                      |
| Negative Laboratory Control Sample (LCS-N) for PAC         | Method Performance for PAC        | (1) One per distillation batch of $\leq 20$ field samples.<br>(2) LCS-N must be distilled with samples in batch using the same preparation method as the samples.<br>(3) Concentration LCS-N = 1.0 mg/L Prussian Blue (Iron-cyanide).<br>(4) Percent recovery must be $\leq 10\%$ .<br>(5) LCS-N is always an aqueous matrix but is distilled with both solid and aqueous samples.                                                                                                                                                                   | Yes                                      | NA                                                                                 | Redistill and reanalyze LCS-N and all associated field samples in batch.                                                                                                                                                                                                                                                                                                  | Suspend all analyses until LCS-N meets criteria.                                                                                     |
| Matrix Spike (MS) Total Cyanide & PAC Project-Specific     | Method Accuracy in Sample Matrix  | (1) <u>Solid Samples (Soil/Sediment)</u> : One per distillation batch of $\leq 20$ field samples per matrix (designated by data user on chain-of-custody [COC] or at project set-up).<br><u>Aqueous Samples Frequency</u> : One per distillation batch of $\leq 20$ field samples per matrix strongly recommended (designated by data user on COC or at project set-up).<br>(2) <u>Total Cyanide</u> : Concentration level near midpoint of curve.<br><u>PAC</u> : Concentration level same as LCS-P (KCN).<br>(3) Percent recovery must be 75-125%. | Yes ONLY when requested by the data user | Recovery $< 30\%$ : affects non-detects for Cyanide/PAC in all associated samples. | (1) Reanalyze MS; if acceptable, no further action required.<br>(2) After reanalysis, if MS recovery is 30-74% or $> 125\%$ and LCS (or LCS-P) was in control, no corrective action is required.<br>(3) If MS recovery is $< 30\%$ and associated with non-detected results, redistill (homogenize sample well) and reanalyze sample/MS pair. Report results and narrate. | Report MS exceedances in laboratory narrative.<br><br>If redigested due to recoveries $< 30\%$ , report both sets of sample/MS data. |
| Matrix Duplicate (MD) Total Cyanide & PAC Project-Specific | Method Precision in Sample Matrix | (1) One per distillation batch of $\leq 20$ field samples per matrix is strongly recommended (designated by data user on COC or at project set-up).<br>(2) Prepare by distilling and analyzing an additional aliquot of the same field sample used for MS.<br>(3) RPD for Cyanide/PAC must be $\leq 20$ for aqueous and $\leq 35$ for solids.                                                                                                                                                                                                        | Yes ONLY when requested by the data user | NA                                                                                 | Narrate.                                                                                                                                                                                                                                                                                                                                                                  | Report exceedances in laboratory narrative.                                                                                          |



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Quality Control Requirements and Performance Standards for the ***Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

**Table VI A-1: Specific QC Requirements and Performance Standards for Total Cyanide and PAC using WSC-CAM-VI 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                     | <p>(1) Non-detected values must be reported with the sample-specific RL/LLOQ for Total Cyanide and PAC using all appropriate preparation/dilution factors.</p> <p>(2) The laboratory must only report values <math>\geq</math> the sample-specific RL/LLOQ.</p> <p>(3) Sample concentrations that exceed the highest calibration standard must be diluted and reanalyzed to fall within the linear calibration range.</p> <p>(4) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards.</p> <p>(5) For aqueous samples, the laboratory must note whether the results are “total” or “dissolved” in the laboratory narrative or on the report form. In addition, if “dissolved”, the laboratory must note whether the samples were filtered in the field or at the laboratory.</p> <p>Note: For the purposes of the WSC-CAM-III C protocol, “dissolved” samples are operationally defined as those samples which have been filtered through a 0.45 <math>\mu</math>m filter.</p> <p>(6) Results must be reported with 2 or more “significant figures” if <math>\geq</math>RL/LLOQ. If reporting values below the RL/LLOQ, report with 1 or more “significant figures”.<sup>2</sup></p> <p>(7) Refer to Appendix VI A-1 for COC requirements regarding preservation, cooler temperature, and holding times.</p> | NA                    | NA                                             | NA                         | <p>(1) The performance of dilutions must be documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of Cyanide/PAC, reasons for dilutions must be explained in the laboratory narrative.</p> <p>(2) If samples are not preserved properly or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory narrative.</p> <p>(3) If samples are distilled and/or analyzed outside of the holding time, note the non-conformances in the laboratory narrative.</p> <p>(4) Narrate any additional method non-compliance or sample-specific anomaly.</p> |

<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 detected results as estimated with a significant low bias.

<sup>2</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.





Quality Control Requirements and Performance Standards for the ***Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)*** 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:

<http://www.mass.gov/dep/cleanup/laws/policies.htm#finpol>.

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-VI A

### 3.1 General Reporting Requirements for WSC-CAM-VI 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-VI A

Specific QC requirements and performance standards for WSC-CAM-VI A are presented in Table VI A-1. Specific reporting requirements for WSC-CAM-VI A are summarized below in Table VI A-2 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.

Soil and sediment results must be reported on a dry-weight basis. Refer to ASTM Method D2216, Determination of Moisture Content of Soils and Sediments, for more detailed analytical and equipment specifications.



Quality Control Requirements and Performance Standards for the **Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

**Table VI A-2 Routine Reporting Requirements for WSC-CAM-VI A (Total Cyanide & PAC)**

| Parameter                                      | Required Analytical Deliverable           |
|------------------------------------------------|-------------------------------------------|
| Initial Calibration                            | <b>NO</b>                                 |
| Initial Calibration Verification (ICV)         | <b>NO</b>                                 |
| Initial Calibration Blank (ICB)                | <b>NO</b>                                 |
| Low-Level Calibration Verification (LLCV)      | <b>NO</b>                                 |
| Continuing Calibration Verification (CCV)      | <b>NO</b>                                 |
| Continuing Calibration Blank (CCB)             | <b>NO</b>                                 |
| Method Blank (MB)                              | <b>YES</b>                                |
| Total Cyanide Laboratory Control Sample (LCS)  | <b>YES</b>                                |
| PAC Positive Laboratory Control Sample (LCS-P) | <b>YES</b>                                |
| PAC Negative Laboratory Control Sample (LCS-N) | <b>YES</b>                                |
| Total Cyanide LCS Duplicate                    | <b>YES (if no MD)</b>                     |
| PAC LCS-P Duplicate                            | <b>YES (if no MD)</b>                     |
| Matrix Spike (MS)                              | <b>YES</b><br>(if requested by data user) |
| Matrix Duplicate (MD)                          | <b>YES</b><br>(if requested by data user) |
| Identification and Quantitation                | <b>NO</b>                                 |
| General Reporting Issues                       | <b>YES</b>                                |

### 3.2.2 Sample Dilution


Under circumstances that sample dilution is required because the concentration of Cyanide/PAC exceeds the concentration of the highest calibration standard or due to matrix interference, the RL/LLOQ for Cyanide/PAC must be adjusted (increased) in direct proportion to the Dilution Factor (DF).

The revised RL/LLOQ for the diluted sample, RL/LLOQ<sub>d</sub>:

$$RL/LLOQ_d = DF \times \text{Lowest Calibration Standard for Cyanide/PAC}$$

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<sub>d</sub> is greater than the applicable MCP standard or criterion to which the concentration is being compared. 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 must be detected within the calibration range.

|                                                                                                                                                                                                                                  |                                                                                      |                  |                |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------|------------------|----------------|
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## Appendix VI A-1

### Sample Collection, Preservation, and Handling Procedures for Total Cyanide and PAC Analyses

Sample preservation, container and analytical holding time specifications for aqueous, soil and sediment matrices for Total Cyanide and PAC 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)*. Additional guidance may be found in SW-846, Chapter Three.



Quality Control Requirements and Performance Standards for the ***Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

| Matrix                          | Container <sup>1</sup>                                                                                                                                     | Preservation <sup>5</sup>                                                                                                                                                                                                                                                                                                     | Holding Time <sup>2</sup>                                                                |
|---------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------|
| Aqueous<br>Total Cyanide/PAC    | 250 mL Polyethylene or Glass<br>bottle for micro-distillation<br>procedure;<br><br>1 L Polyethylene or Glass<br>bottle for macro-distillation<br>procedure | Total Cyanide & PAC: NaOH to pH<br>≥ 12.0; Cool to ≤ 6°C but not<br>frozen;<br><br>0.1N sodium arsenite or 0.6 g<br>ascorbic acid per liter, if residual<br>chlorine is suspected<br><br>PAC: Keep out of direct light                                                                                                        | 14 days to<br>distillation;<br>analyze distillates<br>within 24 hours of<br>distillation |
| Aqueous<br>Dissolved Cyanide    | 250 mL Polyethylene or Glass<br>bottle for micro-distillation<br>procedure;<br><br>1 L Polyethylene or Glass<br>bottle for macro-distillation<br>procedure | Filter (0.45 µm) on site or at the<br>laboratory ( <b>prior to chemical<br/>preservation</b> ) within 24 hours of<br>collection; then preserve with<br>NaOH to pH ≥ 12 <sup>3</sup><br><br>Cool to ≤ 6°C but not frozen<br><br>0.1M sodium arsenite or 0.6 g<br>ascorbic acid per liter, if residual<br>chlorine is suspected | 14 days to<br>distillation;<br>analyze distillates<br>within 24 hours of<br>distillation |
| Soil and Sediments              | 4-ounce glass jar with inert<br>(Teflon) liner                                                                                                             | Total Cyanide & PAC: Cool to ≤<br>6°C but not frozen<br><br>PAC: Keep out of direct light                                                                                                                                                                                                                                     | 14 days to<br>distillation;<br>analyze distillates<br>within 24 hours of<br>distillation |
| Concentrated Waste <sup>4</sup> | 250 mL amber wide-mouth jar<br>with inert (Teflon) liner                                                                                                   | Total Cyanide & PAC: Cool to ≤<br>6°C but not frozen<br><br>PAC: Keep out of direct light                                                                                                                                                                                                                                     | As soon as<br>possible                                                                   |


<sup>1</sup>The collection of multiple sample containers per sample location may be required to collect enough sample for matrix QC. It is also acceptable to use smaller containers to reduce waste and as consistent with laboratory procedures.

<sup>2</sup>Holding time begins from time of sample collection. 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, data users should consider nondetect results as unusable and positive results as estimated with a significantly low bias.

<sup>3</sup>If samples are filtered and preserved at the laboratory, the laboratory must wait 24 hours prior to analysis to allow enough time for cyanide to become solubilized.

<sup>4</sup>Samples containing, or suspected of containing, cyanide or a combination of cyanide and sulfide wastes should be collected with a minimum of aeration. The sample container should be filled completely, excluding all headspace, and capped. Analysis should commence as soon as possible.

<sup>5</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.

|                                                                                                                                                                                                                                  |                                                                                      |                  |                |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------|------------------|----------------|
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Appendix VI A-2


Data Deliverable Requirements for Data Audits



Quality Control Requirements and Performance Standards for the ***Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)*** 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.

| <b>DELIVERABLE REQUIREMENTS FOR DATA AUDITS</b><br><b>WSC-CAM-VI A (Total Cyanide and PAC)</b>                        |                                                                                                                                                                                                     |
|-----------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <b>Laboratory Narrative</b>                                                                                           | Must comply with the required laboratory narrative contents as described in WSC-CAM-VII A                                                                                                           |
| <b>Sample Handling Information</b>                                                                                    | Chain-of-custody (external and internal), sample receipt logs (cooler temperatures and sample pH), correspondences                                                                                  |
| <b>Miscellaneous Logs</b>                                                                                             | Dry weight logs; Analytical logs; Freezer logs; Sample preparation logs (initial and final weights/volumes; preparation method reference); Filtration logs (if applicable)                          |
| <b>Initial Calibration Data</b>                                                                                       | Raw instrument data for initial calibration, including calculation of linear regression and correlation coefficient values; Concentrations of calibration standards used must be clearly presented. |
| <b>Initial Calibration Verification and Initial Calibration Blank Data</b>                                            | Percent recoveries for all ICVs; ICV source & true value; Results and raw instrument data for ICV and ICB                                                                                           |
| <b>Low-Level Calibration Verification, Continuing Calibration Verification, and Continuing Calibration Blank Data</b> | Percent recoveries for all LLCV and CCV; LLCV and CCV source & true value; Results and raw instrument data for LLCV, CCV, and CCB                                                                   |
| <b>Sample Results</b>                                                                                                 | Sample result forms with dilution factors, units, RLs/LLOQs, method reference, date of preparation, date of analysis; raw instrument data; percent solids results                                   |
| <b>Method Blank Results</b>                                                                                           | Method Blank results, units, RLs/LLOQs; raw instrument data                                                                                                                                         |
| <b>LCS/LCS Duplicate Results (negative and positive LCS for PAC) and/or SRM results</b>                               | Summary of results, including concentrations detected, concentrations spiked or known (vendor limits) if SRM, percent recoveries and RPDs; raw instrument data                                      |
| <b>MS Results – if analyzed</b><br><b>MD Results – if analyzed</b>                                                    | Summary of results, project-specific sample ID, unspiked sample concentration, concentration detected, concentration spiked, percent recoveries and RPDs; raw instrument data                       |

|                                                                                                                                                                                                                                     |                  |                |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------|----------------|
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Appendix VI A-3


Analysis Sequence for Total Cyanide and PAC by WSC-CAM-VI A

|                                                                                                                                                                                                                                  |                                                                                      |                  |                |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------|------------------|----------------|
|                                                                                                                                                 | Massachusetts Department of Environmental Protection<br>Bureau of Waste Site Cleanup | WSC-CAM          | Section: VI A  |
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Typical analytical sequence for Total Cyanide and PAC using WSC-CAM-VI A:

- Initial Calibration
- ICV
- ICB
- LLCV – only required if initial calibration curve not performed on same day as analysis or performed on the same day but does not have a low-level standard at the level of the RL/LLOQ
- MB
- Total Cyanide: LCS / PAC: LCS-P and LCS-N
- Total Cyanide: LCSD / PAC: LCSD-P (LCSD only required if not performing a project- specific MD)
- Total Cyanide: 17 samples – include the project-specific MS and/or MD if applicable
- PAC: 15 samples – include the project-specific MS and/or MD if applicable
- CCV
- CCB
- 20 samples
- CCV
- CCB
- Etc. (continue 20 samples and CCV/CCB pairs)
- CCV – ending
- CCB – ending



|                                                                                                                                                                                                                                     |                                                                                      |                  |                |
|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------|------------------|----------------|
|                                                                                                                                                    | Massachusetts Department of Environmental<br>Protection Bureau of Waste Site Cleanup | WSC-CAM          | Section: VI A  |
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**Appendix VI A-4**

**Method for the Determination  
of Physiologically Available Cyanide (PAC)**



Massachusetts Department of Environmental  
Protection Bureau of Waste Site Cleanup

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Quality Control Requirements and Performance Standards for the ***Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

# **METHOD FOR THE DETERMINATION OF PHYSIOLOGICALLY AVAILABLE CYANIDE (PAC)**

**Massachusetts Department of Environmental Protection**

**Division of Environmental Analysis  
(Senator William X. Wall Experiment Station)**

**Office of Research and Standards**

**Bureau of Waste Site Cleanup**

**December 2009**

**Revision 1**

**(Replaces the PAC Method published on August 13, 2004)**



Quality Control Requirements and Performance Standards for the ***Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

**MassDEP**  
**Physiologically Available Cyanide (PAC) Method**

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**DISCLAIMER**

Mention of trade names or commercial products does not constitute endorsement by the Massachusetts Department of Environmental Protection (MassDEP). Trade names and commercial products specified within this method are based upon their use in validation studies conducted by MassDEP. Equipment and materials cited in this method may be replaced by similar products, as long as adequate data exist or have been produced documenting equivalent or superior performance.



Quality Control Requirements and Performance Standards for the ***Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

## 1.0 PAC Overview

The MassDEP Physiologically Active Cyanide (PAC) Method is a performance based reflux-distillation procedure that is used for the determination of biologically available cyanides and is intended to simulate the interaction of the human digestive system and ingested cyanide complexes. PAC is defined as the quantity of cyanide released during distillation under the temperature and pH conditions specified by this protocol. The cyanide forms released in this distillation include free cyanide, simple cyanide complexes (such as cyanide salts) and certain metal cyanides; with the exception of iron-cyanide complexes (including “Prussian Blue”) that are not released under the analytical conditions of this protocol.

Cyanide in the form of hydrocyanic acid (HCN) is released from environmental samples during the PAC distillation under acidic conditions of pH 2 and controlled temperature (to mimic stomach conditions) and captured into an alkaline scrubber solution. The concentration of cyanide in the scrubber solution is then determined spectrophotometrically or titrimetrically by SW-846 Method 9014, SW-846 9012B, or Standard Method 4500-CN-. Analysis by the WSC-CAM-VI A protocol is recommended in support of MCP decisions.

Method performance is verified by the evaluation of the percent recovery of a Positive LCS (LCS-P) and a Negative LCS (LCS-N). Potassium Cyanide (KCN) is used as the LCS-P and Prussian Blue is used as the LCS-N for the PAC method. Acceptable percent recovery criteria for LCS-P and LCS-N are listed in Section 7 below, and in Table VI A-1 of the WSC-CAM-VI A protocol.

This document details instructions for the manual macro (large-volume) PAC cyanide distillation (macro-distillation) procedure. For alternative micro semi-automatic PAC cyanide distillation (micro-distillation) procedures, follow manufacturer’s instructions regarding the adjustment of analytical reagent volumes.

## 2.0 Sample Preparation

### 2.1 Soil and Sediment Analysis

Soil and sediment samples are prepared as-received. Pass the soil/sediment sample through a #10 sieve. Discard any material retained on the sieve. Homogenize the sieved sample by stirring with a stainless steel spoon or placing in a mechanical stirring device. Place 10 g of sieved soil/sediment in the reaction flask for the macro-distillation (1 g for the micro-distillation). The PAC concentration in the reaction flask must not exceed 20 mg/L. Perform a percent solids analysis on a separate soil/sediment aliquot. Use the percent solids to report soil/sediment PAC results on a dry-weight basis.

### 2.2 Aqueous Analysis

Aqueous samples for PAC require no additional preparation prior to distillation. Place 500 mL of the aqueous sample for macro-distillation (50 mL for micro-distillation) directly into the reaction flask. The PAC concentration in the reaction flask must not exceed 20 mg/L. For the macro-distillation, a sample aliquot may be diluted appropriately with distilled water to a final volume of 500 mL to accommodate this requirement.



Quality Control Requirements and Performance Standards for the ***Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

## 2.3 Waste Analysis

Solid and oily waste samples should be extracted prior to PAC distillation using SW-846 Method 9013 *Cyanide Extraction Procedure for Solids and Oils*. The waste sample is first extracted with water at pH  $\geq 10$ . The resultant extract is then distilled using this PAC method, following the aqueous preparation/distillation procedure.

## 3.0 Reagent Preparation

The following reagent preparation guidelines are for the ***macro-distillation*** procedure. For the *micro-distillation* procedure, adjust reagents according to manufacturer's instructions.

Phosphate Buffer Solution - Dissolve 500 g of  $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O}$  to 500 mL of distilled water in a one-liter beaker. With the electrode of a pH meter immersed in the solution, adjust the pH of the solution to 1.25 with concentrated phosphoric acid while stirring.

Magnesium Chloride Solution - Dissolve 510 g of  $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$  in 1-liter of distilled water to prepare a 2.5M  $\text{MgCl}_2$  solution.

Sodium Hydroxide Solution - Dissolve 50 g of NaOH in 1-liter of distilled water to prepare a 1.25N NaOH solution.

Sulfamic Acid ( $\text{H}_2\text{NSO}_3\text{H}$ )

Cadmium Carbonate ( $\text{CdCO}_3$ )

## 4.0 Apparatus and Materials

The required apparatus and materials for the PAC distillation (macro-distillation) are described in Section 4.0, SW-846 Method 9010C, *Total and Amenable Cyanide: Distillation*. Cyanide, as HCN, is released from samples by acid hydrolysis/distillation and captured in an alkaline mediated gas scrubber under vacuum. Additional requirements are as follows:

- ✓ Samples should be analyzed in a fume hood,
- ✓ A flow meter should be placed at the air inlet, and
- ✓ Equipment should be set-up in a thermostatically-controlled constant temperature bath, or equivalent (such as heating blocks or heating mantles), to maintain constant temperature of distillation. Manual temperature control is less preferable, but allowable if fully documented.

## 5.0 Sample Distillation

The following distillation procedure is for the ***macro-distillation***. For the micro-distillation, adjust reagents, amounts, and times according to manufacturer's instructions.



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5.1 An aliquot of  $10.0 \pm 1.0$  g (w/w) of soil/sediment or 500 mL of aqueous sample (or sample diluted to 500 mL) is placed directly in the 1-liter reaction flask. For soil/sediment samples, rinse the weighing container with distilled water to ensure complete sample transfer and bring to a final analytical volume of 500 mL. Add the distilled water with manual swirling to ensure complete surface contact.

5.2 Add 50 mL of the sodium hydroxide solution (1.25N). Connect the reaction flask, condenser, gas scrubber and vacuum trap. If high sulfides are expected in the sample, add 10 to 15 mg of  $\text{CdCO}_3$  to the trap. Set temperature of thermostatically controlled constant temperature bath to  $78 \pm 3^\circ\text{C}$ . When bath has equilibrated at  $78^\circ\text{C}$ , place the reaction flask in the bath and apply vacuum to initiate gas flow through gas scrubber.

5.3 Add 75 mL of the phosphate buffer solution, 2 g of sulfamic acid ( $\text{H}_2\text{NSO}_3\text{H}$ ), and 20 mL of  $\text{MgCl}_2$  solution through the inlet tube. Test the pH of the solution in the reaction flask to verify that the  $\text{pH} \leq 2$ . Place the thermometer in the inlet tube to record the temperature of the solution in the reaction flask.

Note: If using the micro-distillation procedure, pH cannot be checked until the reaction is complete as the micro-distillation apparatus is a closed-system.

5.4 Record the reaction flask temperature every 15 minutes. Maintain the temperature at  $78 \pm 3^\circ\text{C}$  for a period of **one hour**. Record total reaction time (the total time that the flask is in the water bath). Airflow should be measured at least every 15 minutes. Adjust the vacuum rate so that approximately two bubbles of air per second enter the flask through the air inlet tube (this is the same flow requirement as for Total Cyanide by SW-846 Method 9010C). This will translate to a flow rate of 290 to 350 mL/min, depending upon laboratory equipment used, and should be maintained for the total one-hour reaction time. Care should be taken that the sodium hydroxide solution in the gas scrubber does not bubble over.

5.5 After the one hour of reaction time, turn off heating source and allow sample to come to room temperature. Measure the pH of the solution in the reaction flask electrochemically (probe) to verify that the pH was maintained at  $\leq 2$  during the reaction. Test the absorber solution with lead acetate paper for the presence of excess sulfide before analysis. Treat with bismuth nitrate, as necessary (see SW-846 Method 9010C Section 7.2 for details on treatment of distillates for sulfide).

Analytical Note: The analyst must be continually aware of the potential for cross-contamination. Measured PAC concentrations are generally at or near the method RL/LLOQ. As such, the method is particularly susceptible to positive interferences from the presence of low level cyanide residues on glassware from previous analyses of samples and/or various QC samples and standards with elevated PAC concentrations. Thus, frits, absorber tubes, and condensers should be cleaned with dilute HCl and then rinsed with distilled water between samples.

## 6.0 Analysis

For the macro-distillation procedure, the entire content of the absorber tube is transferred to a 250 mL volumetric flask. The tube is rinsed with distilled water, with the rinsate added to the flask. The absorbing solution is then brought to volume with distilled water.



Quality Control Requirements and Performance Standards for the ***Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

The following determinative methods may be used for analysis of PAC in distillates:

- SW-846 Method 9014 – manual colorimetric UV spectrometry for the determination of free (non-complexed) Cyanide ( $\text{CN}^-$ ) and HCN in solution/distillates.
  - This method also includes titrimetric determination; however, the sensitivity of the titrimetric method may not meet data quality objectives (see Section 1.6 of this WSC-CAM-VI A protocol for further information)
- SW-846 Method 9012B – automated colorimetric UV spectrometry for the determination of free  $\text{CN}^-$  and HCN in solution/distillates
- Standard Method 4500- $\text{CN}^-$  – includes colorimetric (equivalent to SW-846 Method 9014), titrimetric, and potentiometric procedures for the determination of HCN in distillates

For colorimetric methods, the distillate is treated with chloramine-T at pH <8 to convert the HCN into cyanogen chloride ( $\text{CNCl}$ ) and color is formed by adding pyridine-barbituric acid. Quantitation for PAC in the treated distillates is based on the color absorption at 578-nm wavelength using a UV spectrometer. Absorbance (peak height) is measured as a function of PAC concentration, based on a multi-level calibration curve.

Analysis by WSC-CAM-VI A protocol is recommended in support of MCP decisions.

## 7.0 Quality Control

Required QC, frequency, and acceptance criteria for PAC can be found in WSC-CAM-VI A, Table VI A-1 Specific QC Requirements and Performance Standards for Total Cyanide and PAC using WSC-CAM-VI A. Some of the required QC parameters are highlighted below:

One method blank (MB) sample must be distilled with each PAC batch of up to 20 samples. The PAC result must be less than the RL/LLOQ.

Accuracy of the PAC method is measured by two required LCSs, which must be distilled with each PAC batch of up to 20 samples:

- Positive Laboratory Control Sample (LCS-P) = 0.1 mg/L Potassium Cyanide (KCN) standard. LCS-P percent recovery (%R) must be 80-120% of the true value (0.1 mg/L).
- Negative Laboratory Control Sample (LCS-N) = 1.0 mg/L Prussian Blue (iron-cyanide) standard. LCS-N percent recovery must be  $\leq 10\%$  of the true value (1.0 mg/L).

In addition to the LCS-P and LCS-N, a project-specific Matrix Spike (MS) is required for PAC in solid matrices (soil/sediment) for WSC-CAM-VI A compliance. For PAC, the spiking solution used for the MS is the LCS-P (KCN standard) and the recovery must be 75-125%.

See WSC-CAM-VI A protocol, Table VI A-1 for additional details on PAC performance standards and required corrective actions.



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## 8.0 Method Performance Criteria

Compliance with the LCS-P, LCS-N, and MS QC requirements described in Section 7.0 above and Table VI A-1 of this WSC-CAM-VI A protocol is the basis for evaluation of satisfactory PAC performance. Conformity with the PAC method temperature and flow rate specifications in Section 5, together with other specific analytical specifications, is expected under most circumstances to produce acceptable PAC QC results. However, it is expected that some laboratories will be able to satisfy PAC QC requirements even with modifications of this method's temperature and flow rate specifications. For example, one laboratory that assisted MassDEP in method development was able to achieve acceptable recoveries for both LCS-P and LCS-N QC standards for samples distilled at 72°C with a flow rate of 325- 350 mL/min.

## 9.0 Sample Collection, Preservation, and Holding Time Specifications

Sample preservation, container and analytical holding time specifications for surface water, groundwater, soil/sediment and waste samples for PAC analyses conducted in support of MCP decision-making are described in Appendix VI A-1 of this WSC-CAM-VI A protocol.





Quality Control Requirements and Performance Standards for the **Analysis of Total Cyanide and Physiologically Available Cyanide (PAC)** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

**Figure 1 PAC Preparation/Distillation Flow Chart**

