U.S. Army
Chemical Materials Agency

Programmatic
Laboratory and Monitoring Quality Assurance Plan

Final

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CHANGE PAGES FOR
CMA PROGRAMMATIC LMQAP

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NOTICE

This Programmatic Laboratory and Monitoring Quality Assurance Plan (LMQAP) for the U.S. Army Chemical Materials Agency (CMA) addresses all major requirements of chemical agent and non-stockpile industrial chemical materiel monitoring involving the CMA.

This CMA Programmatic LMQAP includes discussions of commercially available monitoring devices to detect chemical warfare materiel (CWM). The descriptions and corresponding operational requirements of these devices are provided at the government's request to illustrate the concepts discussed. Inclusion in this report does not necessarily represent endorsement of a product, nor should exclusion of additional products that may exist and be applicable to the CWM monitoring operations represent a lack of endorsement.

FOREWORD

The U.S. Army Chemical Materials Agency (CMA) Programmatic Laboratory and Monitoring Quality Assurance Plan (LMQAP) shall be used at all CMA activities to support laboratory and air monitoring programs. The CMA Programmatic LMQAP will be implemented when chemical detection (chemical agent and industrial chemicals), screening, and analyses are required. This LMQAP supercedes all other quality control/quality assurance documentation for all CMA monitoring and laboratory activities. This document provides requirements for laboratories and monitoring teams to implement a quality system in accordance with programmatic requirements. The CMA-Risk Management Directorate (RMD) is the governing authority for this document and will provide final interpretation on all requirements.

The LMQAP has been developed to: (1) require CMA laboratories and monitoring teams to perform monitoring, quality assurance, and quality control activities; (2) provide specific guidance to address requirements from the Department of the Army, environmental regulatory agencies, and Department of Health and Human Services; (3) require CMA laboratories and monitoring teams to identify and perform timely corrective actions to ensure the acceptable quality of monitoring and laboratory data; and (4) require sufficient documentation to verify the quality of sample collection and monitoring data submitted to CMA.

Adherence to the requirements of this CMA Programmatic LMQAP will ensure that the CMA mission is performed with the highest regard for the safety of the workers, communities, and environment.

Michael A. Parker
Director
U.S. Army Chemical Materials Agency

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Director, Risk Management

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1. INTRODUCTION

1.1 Purpose

The purpose of the U.S. Army Chemical Materials Agency (CMA) Programmatic Laboratory and Monitoring Quality Assurance Plan (LMQAP) is to define requirements for laboratories and monitoring teams to implement a quality system to support CMA activities.

This document shall provide requirements to ensure that all data is scientifically valid, defensible, and of known precision and accuracy (P&A). In doing this, the CMA Programmatic LMQAP shall also:

- Provide consistent framework to create a Laboratory Quality Control Plan (LQCP), ensuring the generation of quality control (QC) data that supports chemical materiel activities.
- Establish standard practices, procedures, and methods that are in control.
- Provide the specifications to ensure that QC requirements are met.

The CMA Programmatic LMQAP shall be used at each site, project, or operation that directly supports sample analyses, method development, method validation, or data analyses.

Program participants shall develop and implement a LQCP that contains the requirements and procedures outlined in this CMA Programmatic LMQAP. The LQCP shall also be in accordance with Federal, State, and local regulatory requirements. Each laboratory/monitoring group supporting CMA project site activities will revise its LQCP within a time period specified by CMA-Monitoring Office or designee, whenever (1) the CMA Programmatic LMQAP is updated; (2) new regulatory guidance is promulgated; and/or (3) new site-specific QC requirements are implemented. LQCPs and modifications shall be submitted to the CMA-Monitoring Office for review and evaluation before implementation.

1.2 Background

Public Law (PL) 99-145 requires CMA to carry out the destruction of the nation's stockpile of unitary chemical agents and munitions. PL 102-484 requires CMA to identify the locations, types, and quantities of the nation's non-stockpile chemical materiel (NSCM). CMA will accomplish these congressional mandates by implementing comprehensive programmatic guidance, including quality assurance (QA) requirements for all phases of treatment, storage, transportation, and demilitarization activities.

1.3 Scope

The scope of this CMA Programmatic LMQAP covers surety materiel, non-surety materiel, quantitative and qualitative laboratory analytical methods, and near real-time (NRT) monitors that are used to analyze solid, liquid, and air samples.

This document outlines the purpose, policy, organization, and operations of all quality assurance/quality control (QA/QC) programs that have been established to support CMA laboratories. The emphasis of this CMA Programmatic LMQAP is to ensure that all activities generate credible scientific data. This includes those aspects of field sampling that may affect the integrity of samples, as well as laboratory/monitoring group activities.

1.4 Waivers and Deviations

Requests for waivers or deviations from the minimum requirements set forth in the CMA Programmatic LMQAP must be submitted in writing to CMA-Monitoring Office for review and written approval prior to implementation. The request for a waiver shall identify the actions taken to implement the requirement, justification for why the requirement can not be implemented, and impact on operations. If the CMA Programmatic LMQAP is different from or conflicts with other codes or regulations from state and/or federal authorities, the CMA-Monitoring Office is to be notified for resolution of the conflict and the most stringent requirement shall be followed, pending resolution. A flow chart for

the waiver process is provided in figure 1-1. Any waivers and deviations issued under Section I of Program Manager for Chemical Demilitarization (PMCD) Policy Statement No. 49, for Research Development, Test, and Evaluation (RDT&E) dilute operations, will meet the intent of this requirement. Poor planning on the part of the CMA activity to comply with the requirements of the CMA Programmatic LMQAP will not justify a request for a waiver. Any recommended changes to this document are to be addressed to CMA-RMD, ATTN: AMSCM-RD, Bldg. E4585, Aberdeen Proving Ground, Maryland 21010. CMA-Monitoring Office will be the approval authority for change pages and corrections to the CMA Programmatic LMQAP.

1.5 Laboratory and Monitoring Systems

Information obtained from monitoring will be used to ensure that CMA operations are being conducted properly to mitigate a release of chemical materiel or personnel exposure.

Monitoring must be performed using instruments selected to measure the proper parameters for the specific chemical encountered at its associated monitoring level. Samples must be taken at intervals designed to ensure that useful information will be available within acceptable time limits. The instruments and methods used must be sufficiently sensitive to reliably measure threshold quantities at required levels. To accomplish these goals, instruments and methods used by CMA will include those specifically developed or approved by the Army to monitor chemical materiel under specific conditions in air, liquid, soils, and solids. Other methods may be used if they are more sensitive, specific, or faster and meet the requirements of the existing methods for precision, accuracy, and reliability, as described in this CMA Programmatic LMQAP, and upon approval by Department of the Army (DA) Safety.

An overriding requirement of the design and development of monitoring systems is reliable day-to-day performance. Reliability, in this context, relates to the ability of the instrument or method to perform its intended function when called upon to do so. Selection of monitoring and sampling locations is also critical to an effective monitoring

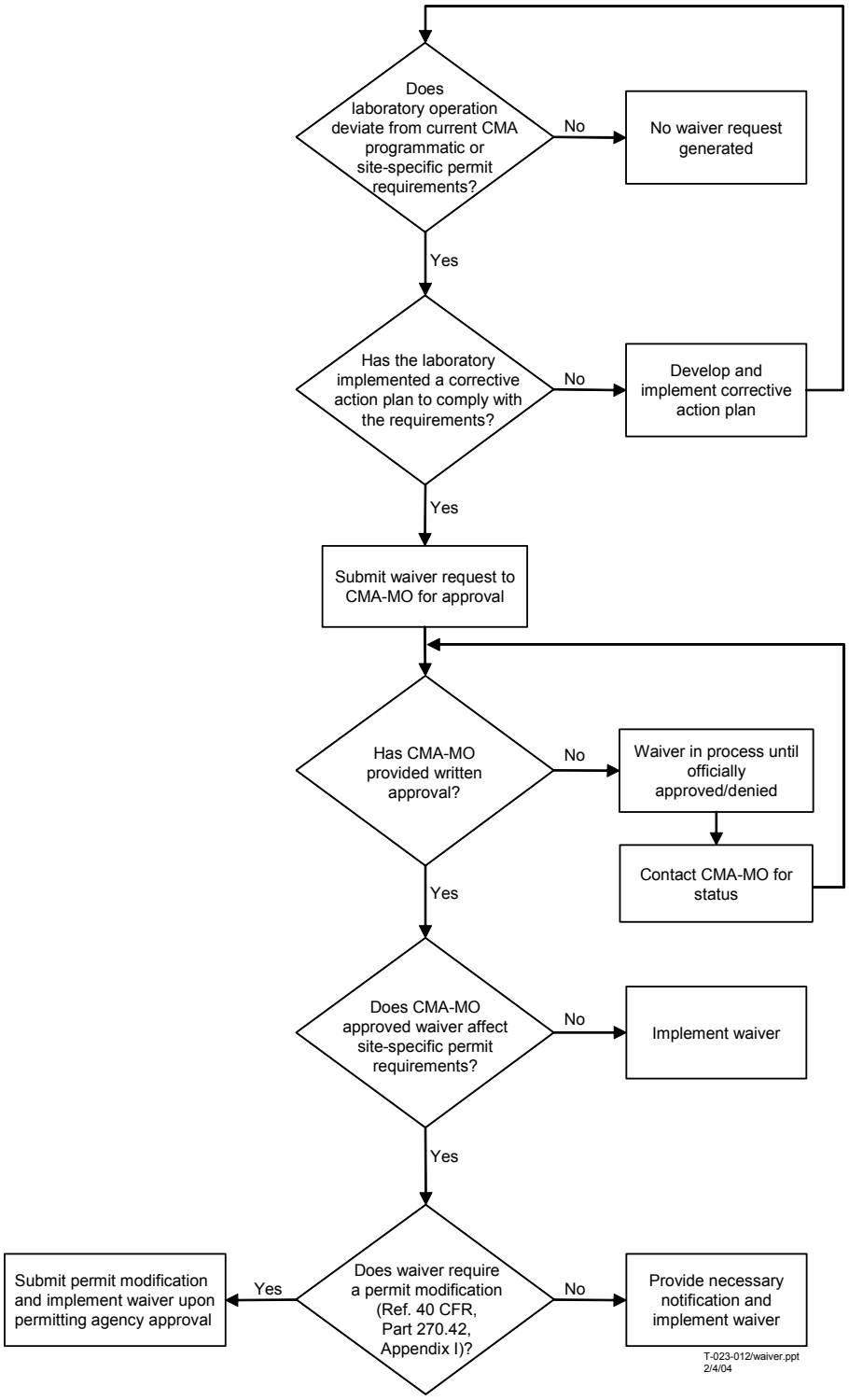

Figure 1-1. Waivers Flow Chart

program. The monitors must be positioned so that samples may be collected from representative points where any released chemical or other chemical hazard would likely be detected. Locations for ambient air monitors must be selected to provide optimum information and maximum protection for workers and the environment. Wastes must also be sampled to provide information representative of the matrix. Monitoring strategies are provided in the CMA Programmatic Monitoring Concept Plan, most current revision.

2. ORGANIZATIONS

2.1 CMA-Risk Management Directorate (RMD)

CMA-RMD is responsible for overseeing all CMA project activities and for the effective implementation of a comprehensive system of QA, monitoring, safety, and environmental activities.

2.2 CMA-RMD Monitoring Office

CMA-Monitoring Office defines QA requirements for all laboratory and monitoring activities involving chemical materiel and industrial chemicals that are used as chemical materiel. CMA-Monitoring Office will review the quality of the CMA laboratory and monitoring data such that it is representative, technically correct, and valid. CMA-Monitoring Office or representative will conduct audits and surveillances of laboratory and monitoring activities, either announced or unannounced. All documents and data produced by the CMA laboratory or monitoring groups will be eligible for inspection. CMA Monitoring Office will monitor and oversee the effective implementation for the CMA Programmatic LMQAP.

2.3 CMA Laboratories/Monitoring Groups

CMA laboratories/monitoring groups include all laboratories and monitoring groups supporting CMA laboratory and monitoring operations with support from analytical and monitoring personnel using surety and non-surety materiel.

3. QUALITY SYSTEM REQUIREMENTS

3.1 Quality Policy Statement

Laboratory/monitoring group management shall establish, implement, and maintain a quality management system (QMS) to define and document the laboratory/monitoring group's policy, objectives, procedures, instructions, and commitment to quality and to the continual improvement of their QMS for chemical materiel activities. The laboratory/monitoring group management shall ensure that these policies and objectives are documented in a LQCP. Executive management shall ensure that this policy is understood, implemented, maintained, reviewed, and communicated/understood at appropriate levels of the organization. The LQCP shall be submitted to the CMA-Monitoring Office for review and evaluation before implementation.

3.2 Organization Description

The LQCP shall identify and/or provide a reference to the organization of the laboratory/monitoring group to include sample analysis/collection and monitoring activities to convey the organization such as location, size of the groups, number of personnel in each group, types and quantity of equipment, types of services offered, and major fields of activity.

As a minimum, the organization description shall:

- Define the organization and management structure of the laboratory/monitoring group and the organizational arrangements with the overall organization, and include relevant organizational charts.
- Identify relationships between management, technical operations, support services, and the QC system.
- Provide a matrix showing job title and the required training, skills, and experience for all CMA laboratory and monitoring personnel.

- Provide job descriptions of key staff, including references to job descriptions of other CMA laboratory/monitoring group staff, and identify agent custodians.
- Identify the CMA laboratory/monitoring group's approval signatories (by position) and approval process for Standing Operating Procedures (SOPs), Internal Operating Procedures (IOPs), Laboratory Operating Procedures (LOPs) and required documents. Maintain a master list of controlled documents and implement a configuration control process overseeing controlled documents.
- Identify technical and quality managers and their acting deputies.

3.3 Management Review

Executive management shall define and document the organization's policy, objectives for and commitment to quality, and to the continual improvement of the QMS for all chemical materiel monitoring and analytical activities in the LQCP. The executive management of each organization shall establish procedures for a regular and systematic review and evaluation of the quality system to ensure its suitability, adequacy, and effectiveness. Such reviews shall be conducted a minimum of once every 12 months. The management review shall include but not be limited to the following items:

- Review and evaluate the records of internal and external audits and of follow-up actions from previous management reviews.
- Review status of preventive and corrective actions, customer suggestions, comments, concerns, complaints, and customer satisfaction.
- Consider external influences such as new technology, changing or new regulations, organizational changes, etc., that could affect product, service, and/or the quality system.
- Review and assess the entire quality system, including a determination of its suitability, adequacy, effectiveness, and process performance.
- Review subcontractor audits to ensure corrective actions have been taken and are adequate (if applicable).

- Generate recommendations and courses of action, including resource needs and actions for improvements to the quality system, processes, and products.
- Initiate actions, including periodic follow-up reviews to verify implementation and evaluate status of each action until all action items have been completed and documented.
- Evaluate the need for changes to the organization's quality system policy and objectives, review of quality objectives, and whether objectives are being met.

3.4 Quality System

3.4.1 LQCP. Each laboratory and monitoring group shall develop, implement, and maintain a LQCP that defines and/or references all plans, SOPs, IOPs, LOPs, local procedures, procedures required by this CMA Programmatic LMQAP, work instructions, and internally-developed computer programs that comprise their analytical and monitoring QC systems, as well as the interaction among them to ensure the quality of the data generated.

The LQCP shall include the scope of the quality system and a description of the interaction between processes of the QMS.

3.4.2 Quality Planning. The purpose of quality planning is to define and document how the requirements for quality shall be met within the organization.

As a minimum, quality planning shall include:

- Laboratory/monitoring group management statement to the standard of service it will provide
- Purpose of the quality system
- Personnel certification process to include understanding and training on the quality system, implementing policies, and performing procedures within their work

- Laboratory/monitoring group management commitment to good professional practice and quality of data
- Laboratory/monitoring group management commitment to compliance with CMA Programmatic LMQAP, Federal, State, and local regulations and permit conditions
- Defining the roles and responsibilities of technical and quality managers
- Specific quality objectives that shall be measurable, quantifiable, and consistent with the quality policy.

Quality planning must ensure that when changes to the quality system are required, the system maintains its integrity.

4. IMPROVEMENT

4.1 General

Management shall continually seek to improve the effectiveness and efficiency of the processes of the laboratories rather than wait for a problem to reveal opportunities for improvement. The process for improvement encompasses customer satisfaction, corrective action, and preventive action. Improvements may result in change to the product or processes and eventually to the QMS or to the laboratories.

4.1.1 Continual Improvement. The laboratories shall continually improve the effectiveness of the QMS through the use of the quality policy, quality objectives, audit results, analyses of data, corrective and preventive actions, and management review.

4.1.2 Customer Satisfaction. The laboratories shall monitor information relating to customer perception as to whether the laboratories have met customer requirements. The methods for obtaining and using this information shall be determined and specified in the LQCP. The customer is defined as any entity to which the laboratory/monitoring group is providing a service or a product.

4.2 Corrective Action

The laboratory/monitoring group shall establish a policy and procedure designating appropriate authorities to implement a corrective action when nonconforming work or departures from the policies and procedures have been identified. An aggressive and timely corrective action program shall be developed and implemented to minimize systematic error and random error. The laboratory/monitoring group shall monitor the results to ensure corrective actions taken have been effective. The implementation and effectiveness of the corrective action will be verified by personnel other than those responsible for carrying out the corrective action.

4.2.1 Immediate Corrective Actions. Immediate corrective action is required of the operators when response to a given QC sample is unacceptable (QC samples are described in detail in section 12 of this CMA Programmatic LMQAP). The effectiveness of the immediate corrective action will be verified by generating a passing monitoring level challenge and/or a continuing calibration verification (CCV). All corrective actions must be documented. If operators are unable to solve a problem using immediate corrective actions, long-term corrective actions must be employed.

4.2.2 Long-Term Corrective Actions. The steps to implementing long-term corrective actions will be as follows: (1) define the problem, (2) assign responsibility for investigation of the problem, (3) investigate and determine the cause of the problem, (4) determine the corrective action to eliminate the problem, (5) assign and accept responsibility for implementation of the corrective action, (6) implement the corrective action and determine its effectiveness, and (7) verify that the corrective action has eliminated the problem and did not create a new problem. All long-term corrective actions must be documented.

4.2.3 Root Cause. In some circumstances CMA may request a root cause analysis for critical failures. Root cause is the identification as to the cause of the noncompliance; defined as: the most basic cause that can be reasonably identified and that management has control to fix.

4.3 Preventive Action

Preventive action is directed toward eliminating the causes of potential nonconformities. Organizations shall identify and implement actions to eliminate the causes of potential nonconformities to prevent their occurrence. Preventive actions shall be documented and appropriate to the effects of the potential problems.

A documented procedure shall be established to define requirements for:

- Determining potential nonconformities and their causes
- Evaluating the need for action to prevent occurrence of nonconformities
- Determining and implementing action needed
- Recording results of action taken
- Reviewing preventive action taken.

4.4 Preventive Maintenance

To ensure calibration and test method integrity during site operations, routine preventive maintenance shall be performed on all monitoring and analytical equipment. As a minimum, initial preventive maintenance should be performed in accordance with manufacturer recommendations. If operational test data indicate that a less stringent preventive maintenance protocol is adequate or a more stringent preventive maintenance protocol is required for specific monitoring and/or analytical equipment, a new preventive maintenance schedule shall be developed and implemented for each specific piece of equipment. The CMA laboratory/monitoring group shall develop a procedure to document preventive maintenance frequency, circumstances, and recordkeeping. Tracking or trending of maintenance activities shall be performed. If a trend is observed that adversely affects the performance, a corrective action report shall be generated to maximize instrument performance if necessary. Additionally, the CMA laboratory and monitoring group shall maintain a spare parts list for all equipment relating to laboratory and monitoring operations.

4.5 Complaints and Nonconformances

The CMA laboratory/monitoring group shall have a documented nonconformance policy that includes procedures for the identification and rapid resolution of nonconformances with the CMA Programmatic LMQAP and written site-specific procedures. This policy shall be described in the LQCP. Documentation of all alleged nonconformance issues and actions taken by the CMA laboratory/monitoring group to correct or resolve the

nonconformance shall be maintained. These records will be archived for at least 40 years as part of the site's permanent record files.

5. PERSONNEL

The laboratory and monitoring management shall ensure the competence of all who operate, maintain, service, perform tests and/or calibration, evaluate results, and sign reports on laboratory/monitoring group equipment. The laboratory and monitoring management shall maintain descriptions of duties for all laboratory/monitoring group job positions and ensure that the individuals assigned to a job position have the requisite experience and training that are consistent with the descriptions of duties for the job position. Laboratory and monitoring training provided shall include, as a minimum, site-specific on-the-job training (OJT) on local equipment, plans, and procedures. Laboratory and monitoring management shall maintain onsite records of personnel qualifications. Laboratory and monitoring management shall develop a training plan to identify requirements for operator competency, certification, and recertification process to record operator proficiency associated with their job responsibilities. Certification of personnel shall be documented upon effective and successful completion of the job task. Personnel certification and training records shall be maintained onsite in accordance with state and federal environmental regulations.

Certified operators shall satisfy the following requirements for a monitoring and/or analytical method:

- Management certification to demonstrate proficiency to perform the operating procedure
- Maintain current certification by performing method challenges at definite frequencies to develop operator baseline.
- Demonstrate proficiency for all monitoring levels for the respective agent.
- Demonstrate proficiency to operate and maintain equipment.

6. ACCOMMODATION AND ENVIRONMENT

The laboratory/monitoring group management shall monitor, control, and record environmental working conditions as required by the relevant specifications, methods, and procedures or where they influence the quality of the results. The environment in which sample analysis occurs shall ensure the representativeness and validity of results and enhance the required accuracy of measurements. Incompatible activities shall be appropriately separated. Access to and use of areas affecting the quality of tests and/or calibrations shall be controlled. QC equipment and records shall be defined and controlled. The work environment shall be ergonomically conducive to achieve conformity of products. Adequate measures shall be taken to ensure good housekeeping in CMA laboratories and monitoring areas. The laboratory/monitoring group manager or his or her designated representative will have these responsibilities.

Computer and automated equipment shall be maintained to ensure proper function under environmental and operating conditions necessary to maintain integrity.

7. MONITORING STANDARDS AND CONTROL LIMITS

7.1 Department of Health and Human Services (DHHS)

The Centers for Disease Control and Prevention (CDC) Public Health Service, DHHS, has responsibility by PL 91-121 to oversee the Demilitarization Program and make recommendations for protecting human health and safety. On October 9, 2003, CDC published new nerve agent airborne exposure limits (AELs) to be implemented. On May 3, 2004, CDC published new mustard AELs.

7.2 DA Standards

The DA has promulgated monitoring standards and exposure levels for chemical agents in the following documents: DoD 6055.9-STD, *Ammunition and Explosive Safety Standards*; Army Regulation (AR) 385-61, *Army Toxic Chemical Agent Safety Program*; AR 50-6, *Chemical Surety*; DA Pamphlet (Pam) 385-61, *Toxic Chemical Agent Safety Standards*; DA Pam 40-8, *Occupational Health Guidelines for the Evaluation and Control of Occupational Exposure to Nerve Agents GA, GB, GD, and VX*; DA Pam 40-173, *Occupational Health Guidelines for the Evaluation and Control of Occupational Exposure to Mustard Agents H, HD, and HT*; and PMCD Pam 40-1, *Occupational Health Guidelines for the Evaluation and Control of Occupational Exposure to Lewisite*. Federal agencies such as the U.S. Environmental Protection Agency (USEPA) and Occupational Safety and Health Administration (OSHA) also require monitoring for the presence of hazardous chemicals in ambient air to ensure that site workers and the surrounding communities are not exposed to hazardous conditions during excavation, transportation, storage, and disposal of chemical materiel, and during emergency response activities. OSHA regulations are codified in the Code of Federal Regulations (CFR), Title 29 CFR, Part 1910.120 (29 CFR 1910.120). Primary USEPA regulations incorporate OSHA standards by reference and are codified in Title 40 CFR Part 311.

7.3 AELs and Process Control Levels

AELs for tabun (GA), sarin (GB), lewisite (L), O-ethyl S-(2-diisopropylaminoethyl) methylphosphonothioate (VX), soman (GD), distilled mustard (HD), Levinstein mustard (H), mustard-T mixture (HT), nitrogen mustard (HN-1 and HN-3), and mustard-L mixture (HL) at short-term exposure limits (STELs), worker population limits (WPLs), general population limits (GPLs), vapor screening limit (VSL), and source emission limits (SELs) are listed in tables 7-1 through 7-8. Industrial chemicals will be monitored to the regulatory permissible exposure limits (PELs), as specified in table 7-9.

To minimize the potential for false alarms that decrease confidence in the monitoring system, the goal is to set all WPL/STEL NRT monitor alarms at 1.0Z.[1] In order to set the alarm at 1.0Z, the NRT monitor shall meet the initial and continuing baseline requirements identified in tables 10-3 and 10-4. If baseline requirements are not satisfied due to either poor recovery, method variation, or human differences, a more conservative alarm (that is, below 1.0Z) will be set that demonstrates an equal or greater than 95 percent statistical response rate at the selected alarm level concentration (see paragraph 16.5.10).

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[1] Vapor validation of MINICAMS® for L demonstrated a negative bias. Therefore, MINICAMS® configured to monitor for L shall implement an alarm setpoint at or below 0.4Z.

Table 7-1. AELs for GA, GB, and VX

AEL (mg/m^3)	GPL[a]	WPL[a]	STEL[a]	IDLH
GA/GB	1×10^{-6}	3×10^{-5}	1×10^{-4}	0.1
VX	6×10^{-7}	1×10^{-6}	1×10^{-5} [b]	0.003
Averaging Time	24 hours	8 hours	15 minutes	≤ 30 minutes
Monitoring Method for Recommended Exposure Criteria	Historical[c]	NRT or Historical	NRT monitor	NRT monitor

Notes:

Airborne exposure limits (AELs) are taken from 68 FR 58348-58351 (9 October 03).

[a] An additional reduction factor for statistical assurance of action at the exposure limit is not needed because of safety factors already built into the derivation of the exposure limit.

[b] VX STEL has been adjusted from 4×10^{-6} mg/m^3 (up to four times per day) as proposed in the Federal Register (FR) announcement to 1×10^{-5} mg/m^3 (not more than one time per day) based on technical capabilities of existing air monitoring technologies.

[c] Historical monitoring typically refers to long-term sampling and analytical methods. Air monitoring results from historical methods are not known until laboratory analyses are complete.

Table 7-2. AELs for HD[a]

Sulfur Mustard (H, HD, HT)[b] Criteria	GPL	WPL	STEL[c]	IDLH[d]
Exposure Level	0.00002	0.0004	0.003	0.7
Averaging Time	12 hours	8 hours	≤15 minutes	≤30 minutes
Recommended Monitoring Method	Historical[e]	Historical[e] or near real-time	Near real-time	Near real-time

Notes:

[a] Although the Centers for Disease Control and Prevention (CDC) do not specifically recommend additional reduction factors for statistical assurance of action at the exposure limit, exposures to sulfur mustard should be minimized given the uncertainties in risk assessment, particularly as related to characterizing carcinogenic potency.

[b] The toxicity data for agent T is inadequate for setting exposure limits. The very low vapor pressure for agent T precludes it as a vapor hazard under normal ambient conditions. For sulfur mustard and T mixtures, air monitoring for sulfur mustard alone should be sufficient under most circumstances to prevent exposure to T.

[c] To be evaluated with a near real-time instrument using shortest practicable analytic cycle time. No more than one exposure per work-shift.

[d] The mustard IDLH is based on non-carcinogenic effects. No IDLH has been established for carcinogens.

[e] Historical monitoring typically is used for time-weighted average (TWA) monitoring where the sample analyzed represents an extended time period, for example, 8 or 12 hours. Results are not known until laboratory analysis is completed after the sampling event. AELs using historical monitoring are set at levels at which health effects are not expected to occur for most workers. Exposures above the WPL-8, but below the STEL, likewise are not expected to result in significant health effects unless such exposures occur continuously for long periods.

Table 7-3. VX AELs

VX	Averaging Time						
	GPL (24 hours)	WPL (12 hours)	WPL (8 hours)	WPL (4 hours)	WPL (2 hours)	STEL[a] (15 minutes)	Variable
General Population	6×10^{-7} mg/m^3						
No Respiratory Protection		6×10^{-7} mg/m^3	1×10^{-6} mg/m^3	2×10^{-6} mg/m^3	4×10^{-6} mg/m^3	1×10^{-5} mg/m^3	
Air-Purifying Respirator w/o Escape Bottle		3×10^{-5} mg/m^3	5×10^{-5} mg/m^3	1×10^{-4} mg/m^3	2×10^{-4} mg/m^3	5×10^{-4} mg/m^3	
Supplied-Air Respirator or Supplied-Air Respirator with Escape Bottle		6×10^{-4} mg/m^3	1×10^{-3} mg/m^3	2×10^{-3} mg/m^3	4×10^{-3} mg/m^3	1×10^{-2} mg/m^3	
Self-Contained Breathing Apparatus		6×10^{-3} mg/m^3	1×10^{-2} mg/m^3	2×10^{-2} mg/m^3	4×10^{-2} mg/m^3	1×10^{-1} mg/m^3	
Demilitarization Protective Ensemble					100 mg/m^3[b]		
Vapor Screening Limit							1×10^{-5} mg/m^3
Source Emission Limit							3×10^{-4} mg/m^3

Notes:

[a] Exposures at the STEL shall not occur more than one time per day.
[b] Implemented as a ceiling value.

Airborne exposure limits (AELs) are taken from Army Regulation 385-61 (12 October 01) and 68 FR 58348-58351 (9 October 03).

All AELs are concentration and time values, not concentration only values. Administrative controls may be used to limit potential exposure to workers. However, because administrative controls cannot be used to limit the duration of potential public exposure, only the worker population limit (WPL) protective action level is significantly affected by administrative controls, which limit the duration of potential exposure.

Table 7-3. VX AELs (Continued)

Notes: (Continued)

The maximum use concentration is the product of the AEL and the assigned protection factor for the respirator. The assigned protection factors used in this table are taken from 68 FR 34036-34119.

The demilitarization protective ensemble is only authorized for use up to 2 hours (depending on temperature). The maximum use concentration is based on extensive testing performed 1975 through 1979.

The source emission limit was previously known as the allowable stack concentration.

Table 7-4. GB/GA AELs

GB/GA	Averaging Time						
	GPL (24 hours)	WPL (12 hours)	WPL (8 hours)	WPL (4 hours)	WPL (2 hours)	STEL[a] (15 minutes)	Variable
General Population	1×10^{-6} mg/m^3						
No Respiratory Protection		2×10^{-5} mg/m^3	3×10^{-5} mg/m^3	6×10^{-5} mg/m^3	6×10^{-5} mg/m^3	1×10^{-4} mg/m^3	
Air-Purifying Respirator		1×10^{-3} mg/m^3	1.5×10^{-3} mg/m^3	3×10^{-3} mg/m^3	3×10^{-3} mg/m^3	5×10^{-3} mg/m^3	
Supplied-Air Respirator w/o Escape Bottle		2×10^{-2} mg/m^3	3×10^{-2} mg/m^3	6×10^{-2} mg/m^3	6×10^{-2} mg/m^3	1×10^{-1} mg/m^3	
Self-Contained Breathing Apparatus or Supplied-Air Respirator with Escape Bottle		2×10^{-1} mg/m^3	3×10^{-1} mg/m^3	6×10^{-1} mg/m^3	6×10^{-1} mg/m^3	1 mg/m^3	
Demilitarization Protective Ensemble					100 mg/m^3[b]		
Vapor Screening Limit							1×10^{-4} mg/m^3
Source Emission Limit							3×10^{-4} mg/m^3

Notes:

[a] Exposures at the STEL shall not occur more than four times per day, and at least 60 minutes must lapse between successive exposures.
[b] Implemented as a ceiling value.

Airborne exposure limits (AELs) are taken from Army Regulation 385-61 (12 October 01) and 68 FR 58348-58351 (9 October 03).

Table 7-4. GB/GA AELs (Continued)

Notes: (Continued)

All AELs are concentration and time values, not concentration only values. Administrative controls may be used to limit potential exposure to workers. However, because administrative controls cannot be used to limit the duration of potential public exposure, only the worker population limit (WPL) protective action level is significantly affected by administrative controls, which limit the duration of potential exposure.

The maximum use concentration is the product of the AEL and the assigned protection factor for the respirator. The assigned protection factors used in this table are taken from 68 FR 34036-34119, 6 June 2003.

The demilitarization protective ensemble is only authorized for use up to 2 hours (depending on temperature). The maximum use concentration is based on extensive testing performed 1975 through 1979.

The source emission limit was previously known as the allowable stack concentration.

Table 7-5. GD AELs

GD	Averaging Time						
	GPL (72 hours)	WPL (12 hours)	WPL (8 hours)	WPL (4 hours)	WPL (2 hours)	STEL (15 minutes)	Variable
General Population	3×10^{-6} mg/m^3						
No Respiratory Protection		3×10^{-5} mg/m^3	3×10^{-5} mg/m^3	3×10^{-5} mg/m^3	3×10^{-5} mg/m^3	3×10^{-5} mg/m^3	
Air-Purifying Respirator		1.5×10^{-3} mg/m^3	1.5×10^{-3} mg/m^3	1.5×10^{-3} mg/m^3	1.5×10^{-3} mg/m^3	1.5×10^{-3} mg/m^3	
Supplied-Air Respirator w/o Escape Bottle		3×10^{-2} mg/m^3	3×10^{-2} mg/m^3	3×10^{-2} mg/m^3	3×10^{-2} mg/m^3	3×10^{-2} mg/m^3	
Self-Contained Breathing Apparatus or Supplied-Air Respirator with Escape Bottle		3×10^{-1} mg/m^3	3×10^{-1} mg/m^3	3×10^{-1} mg/m^3	3×10^{-1} mg/m^3	3×10^{-1} mg/m^3	
Demilitarization Protective Ensemble				See note a			
Vapor Screening Limit							3×10^{-5} mg/m^3
Source Emission Limit							1×10^{-4} mg/m^3

Notes:

[a] Pending USACHPPM report, which is currently being written.

Table 7-5. GD AELs (Continued)

Notes: (Continued)

Airborne exposure limits (AELs) are taken from Army Regulation 385-61 (12 October 01).

All AELs are concentration only values, regardless of duration. Personal protective equipment (PPE) may be used to limit potential exposure to workers.

The maximum use concentration is the product of the AEL and the assigned protection factor for the respirator. The assigned protection factors used in this table are taken from 68 FR 34036-34119, 6 June 2003.

The demilitarization protective ensemble is not authorized for use with GD due to lack of testing.

The source emission limit was previously known as the allowable stack concentration.

Table 7-6. H/HD/HT AELs

H/HD/HT	Averaging Time						
	GPL (12 hours)	WPL (12 hours)	WPL (8 hours)	WPL (4 hours)	WPL (2 hours)	STEL[a] (15 minutes)	Variable
General Population	2×10^{-5} mg/m^3						
No Respiratory Protection		2.7×10^{-4} mg/m^3	4×10^{-4} mg/m^3	8×10^{-4} mg/m^3	1.6×10^{-3} mg/m^3	3×10^{-3} mg/m^3	
Air-Purifying Respirator	For sulfur mustards, air-purifying respirators are for escape purposes only.						
Supplied-Air Respirator w/o Escape Bottle		0.27 mg/m^3	0.4 mg/m^3	0.8 mg/m^3	1.6 mg/m^3	3 mg/m^3	
Self-Contained Breathing Apparatus or Supplied-Air Respirator with Escape Bottle		2.0×10^{-3} mg/m^3	4 mg/m^3	8 mg/m^3	16 mg/m^3	30 mg/m^3	
Demilitarization Protective Ensemble					100 mg/m^3[b]		
Vapor Screening Limit							3×10^{-3} mg/m^3
Source Emission Limit							3×10^{-2} mg/m^3

Notes:

[a] Exposures at the STEL shall occur not more than one time per day. The Centers for Disease Control and Prevention (CDC) may publish updated numbers.

[b] Implemented as a ceiling value.

Table 7-6. H/HD/HT AELs (Continued)

Notes: (Continued)

Airborne exposure limits (AELs) are taken from Army Regulation 385-61 (12 October 01) and 69 FR 24164-24168 03 May 04.

All AELs are concentration and time values, not concentration only values. Administrative controls may be used to limit potential exposure to workers. However, because administrative controls cannot be used to limit the duration of potential public exposure, only the worker population limit (WPL) protective action level is significantly affected by administrative controls, which limit the duration of potential exposure.

The maximum use concentration is the product of the AEL and the assigned protection factor for the respirator. The assigned protection factors used in this table are taken from 68 FR 34036-34119, 6 June 2003. For sulfur mustards and lewisite, air-purifying respirators are for escape purposes only.

The mixture HT shall be monitored as HD.

The source emission limit was previously known as the allowable stack concentration.

Table 7-7. L/HL AELs

L/HL	Averaging Time						
	GPL (12 hours)	WPL (12 hours)	WPL (8 hours)	WPL (4 hours)	WPL (2 hours)	STEL (15 minutes)	Variable
General Population	3×10^{-3} mg/m^3						
No Respiratory Protection		3×10^{-3} mg/m^3	3×10^{-3} mg/m^3	3×10^{-3} mg/m^3	3×10^{-3} mg/m^3	3×10^{-3} mg/m^3	
Air-Purifying Respirator	For lewisite, air-purifying respirators are used for escape purposes only.						
Supplied-Air Respirator w/o Escape Bottle		3 mg/m^3	3 mg/m^3	3 mg/m^3	3 mg/m^3	3 mg/m^3	
Self-Contained Breathing Apparatus or Supplied-Air Respirator with Escape Bottle		30 mg/m^3	30 mg/m^3	30 mg/m^3	30 mg/m^3	30 mg/m^3	
Demilitarization Protective Ensemble[a]							
Vapor Screening Limit							3×10^{-3} mg/m^3
Source Emission Limit							3×10^{-2} mg/m^3

Notes:

[a] The DPE is not authorized for use with L/HL based on testing.

Table 7-7. L/HL AELs (Continued)

Notes:

Airborne exposure limits (AELs) are taken from Army Regulation 385-61 (12 October 01).

All AELs are concentration only values, regardless of duration. Personal protective equipment (PPE) may be used to limit potential exposure to workers.

The source emission limit was previously known as the allowable stack concentration.

Table 7-8. HN-1/HN-3 AELs

HN-1/HN-3	Averaging Time						
	GPL (72 hours)	WPL (12 hours)	WPL (8 hours)	WPL (4 hours)	WPL (2 hours)	STEL (15 minutes)	Variable
General Population	--	--	--	--	--	--	
No Respiratory Protection		3×10^{-3} mg/m^3	3×10^{-3} mg/m^3	3×10^{-3} mg/m^3	3×10^{-3} mg/m^3	3×10^{-3} mg/m^3	
Air-Purifying Respirator	For nitrogen mustards, air-purifying respirators are for escape purposes only.						
Supplied-Air Respirator w/o Escape Bottle		3 mg/m^3	3 mg/m^3	3 mg/m^3	3 mg/m^3	3 mg/m^3	
Self-Contained Breathing Apparatus or Supplied-Air Respirator with Escape Bottle		30 mg/m^3	30 mg/m^3	30 mg/m^3	30 mg/m^3	30 mg/m^3	
Demilitarization Protective Ensemble					100 mg/m^3[a]		
Vapor Screening Limit							3×10^{-3} mg/m^3
Source Emission Limit							--

Notes:

[a] Implemented as a ceiling value.

Table 7-8. HN-1/HN-3 AELs (Continued)

Notes:

Airborne exposure limits (AELs) are taken from Army Regulation 385-61 (12 October 01).

All AELs are concentration only values, regardless of duration. Personal protective equipment (PPE) may be used to limit potential exposure to workers.

Table 7-9. Exposure Limits for Industrial Chemicals Used as CWM

Chemical	Permissible Exposure Limit as 8-Hour TWA or STEL (15 Minutes)		IDLH	
	mg/m^3	ppmv[a]	mg/m^3	ppmv[a]
DM, DA, DC	No air monitoring required[b]	No air monitoring required[b]	No air monitoring required[b]	No air monitoring required[b]
BZ	0.004	--	0.2	0.5
Chloroform[c]	9.7	2		500
Chlorine	2.90 (STEL)	1 (STEL)	29.0	10
Chloropicrin (PS)[c]	0.7	0.1	13.4	2
Cyanogen Chloride (CK)	0.6	0.3	NS	NS
Hydrogen Cyanide (AC)	11.0	10.0	55	50
Phosgene[c]	0.4	0.1	8.1	2
Diphosgene[d]	--	--	NS	NS
Triphosgene[d]	--	--	NS	NS
Arsine (SA)	--	--	--	--
Triphenylarsine (TA)	--	--	--	--
PD[b]	NS	NS	NS	NS
DF	0.008	--	--	--
QL	0.03	--	0.9	--
HF[c]	2.5	--	--	--

Notes:

[a] Parts per million by volume at 20°C and 1 atmosphere
[b] Appropriate engineering/process controls shall be used to minimize exposure.
[c] National Institute for Occupational Safety and Health (NIOSH) *Pocket Guide to Chemical Hazards*
[d] Monitored as phosgene

NS: No Army or Occupational Safety and Health Administration (OSHA) standard available. CMA-RMD shall be notified prior to operations with these chemicals and monitoring levels will be determined on a case-by-case basis in accordance with operation and protection levels applied.

8. EQUIPMENT AND REFERENCE MATERIALS

8.1 Introduction

For Continuous Emission Monitoring System (CEMS) operational requirements, refer to the protocol in CFR, Title 40, Part 60, Appendix F, and the Handbook, August 1997, *Continuous Emission Monitoring System for Non-criteria Pollutants*, Center for Environmental Research Information, National Risk Management, National Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268, August 1997.

Table 8-1 identifies general CMA laboratory/monitoring requirements for equipment and reference materials.

Equipment records shall include as a minimum:

- Nomenclature
- Manufacturer
- Serial number or other unique identification (ID) number
- Date received
- Date placed into service
- Current location, where appropriate
- Condition when received (for example, new, used, reconditioned)
- Copy of the manufacturer's operations and maintenance (O&M) manual
- Dates and results of calibrations and dates of next scheduled calibration
- Details of maintenance carried out and scheduled maintenance
- History of damage, malfunction, modification, repair, and QC activities.

This section also describes the procedures for the preparation, handling, storage, and evaluation of standard solutions that are used by organizations for calibration and QC spiking.

Table 8-1. General CMA Laboratory/Monitoring Requirements for Equipment and Reference Materials

CMA Laboratory/Monitoring Responsibility	Requirement
Maintenance	• Shall ensure proper maintenance of all equipment, including reference standards required for correct performance, calibration, and operation of monitoring and sampling systems • Shall document all maintenance procedures • Should as a minimum, perform preventive maintenance in accordance with manufacturer recommendations.
Spare Parts and Consumables	• Requirement shall be defined by the laboratory/monitoring group.
Records	• Shall be maintained for the monitoring and sampling systems and for all reference materials that have a significant impact on the calibration or performance of monitoring and sampling systems • Shall be maintained for all CMA laboratory/monitoring group equipment having a significant role in sample analysis.
Labels/Identification[a]	• Shall indicate the calibration status of each item of equipment • Shall indicate equipment required to be calibrated before use and equipment that does not require calibration.

Note:

[a] For monitoring equipment this information can be contained in the logbook.

8.2 Use of Chemical Agent Standard Analytical Reference Material (CASARM) Standards

8.2.1 Introduction. CASARM standards shall be used when available. Chemical agent standards are available in forms of neat liquid or RDT&E dilute standards for use at CMA laboratories. The neat standards are of high purity and characterized by several analytical techniques. The RDT&E standards are available in specific solvents. The CMA laboratories shall complete all requirements to receive, handle, and store chemical materiel, then obtain RMD authorization that the CMA laboratory satisfied these requirements. The CMA laboratory shall submit a request to CMA-RMD to receive chemical agent standards from CASARM at least 60 days before the need for such standards. In the event CASARM standards are not available, known purity and

analytically determined stock concentrations shall be used to prepare working standards.

If working standards are provided by a contracted laboratory, it is the responsibility of the CMA laboratory to verify that working standards and/or stock standards are at the appropriate concentration.

8.2.2 CASARM Proficiency Testing Program (PTP). The CASARM PTP uses interlaboratory test results to determine performance of individual CASARM program participants and of the group as a whole. It gives participants an objective means of assessing and proving the reliability of the data they produce.

For laboratories voluntarily participating in the CASARM PTP, the frequency will be determined by the CASARM PTP. The site accountability officer and/or agent custodians shall be responsible for submitting, in writing, all CASARM and PTP standard requests to the CMA-RMD. If the laboratory results are not within the specified concentration range, the laboratory shall implement corrective actions necessary to resolve the discrepancy.

Participants shall be responsible for:

a. Notifying the Edgewood Chemical Biological Center (ECBC) Chemical Quality Assurance Team (CQAT) of types of proficiency test samples required at their respective sites

b. Analysis of proficiency test samples according to this plan and other specific instructions

c. Reporting results of proficiency test analyses to the CQAT

d. Reporting problems (if any) with samples received.

8.2.3 Standards Handling Requirements. Requirements for handling standards are located in DA Pam 385-61 and PMCD Policy Statement No. 49.

8.3 Reference Materials Description and QA/QC Procedures

The standard solutions, along with the reference materials from which they are made, play an important role in the QA process because no chemical analysis method can be more accurate or more reliable than the standard solutions used for method calibration. The storage and handling procedures shall be addressed in the LQCP.

8.3.1 Neat CASARMs. CASARMs are available in neat liquid form for use at facilities that have been authorized to store and use chemical surety materiel (CSM). These materials are of high purity and are characterized by several analytical techniques. In addition, neat CASARMs are stored at or below 4°C and are shipped in approved packaging according to procedures that minimize the likelihood of chemical deterioration (that is, in sealed ampules under a blanket of dry inert gas and in the absence of light). The ECBC CQAT provides users with quality documentation that specifies the certified purity of these CASARMs.

Upon receiving a CASARM shipment, the recipient, in addition to other regulatory requirements, shall complete Section II of the Shipment Status Report (SSR) that accompanies each shipment and return it to the CQAT (CDR, RDECOM, ATTN: AMSRD-ECB-CB-RQ, 5183 Blackhawk Road, Aberdeen Proving Ground, MD 21010-5424).

8.3.2 Dilute CASARMs. Some facilities that are not authorized as CSM laboratories are authorized to use RDT&E dilute standards, that is, dilute CASARMs that are available from ECBC. The nominal concentrations in specific solvents of the available RDT&E solutions supplied by ECBC are summarized in table 8-2. The CQAT provides users with quality documentation that specifies the certified concentration of these dilute CASARMs.

Table 8-2. RDT&E Dilute Agent Solution Concentration Levels

Agent Symbol	Nominal Concentration (mg/mL)	Solvent	Shelf Life[a]	Verification
GB	0.26	Isopropanol	12 months	Monthly
GD	0.11	n-hexane	12 months	Annually
HD	2.32	n-hexane	12 months	Annually
L[b]	2.32	n-hexane	12 months	Annually
VX	0.12	Isopropanol	12 months	Monthly

Notes:

[a] Shelf life reflects storage at or below 4°C.
[b] Lewisite is analyzed as 2-chlorovinylarsonous acid (CVAA).

Upon receiving a dilute CASARM shipment, the recipient, in addition to other regulatory requirements, shall complete Section II of the SSR that accompanies each shipment and return it to the CQAT (CDR, RDECOM, ATTN: AMSRD-ECB-CB-RQ, 5183 Blackhawk Road, Aberdeen Proving Ground, MD 21010-5424).

8.3.3 Storage and Use of CASARMs. All CASARMs supplied by ECBC shall be stored at or below 4°C. The CQAT shall be notified if CASARMs are stored above 4°C for periods longer than 96 consecutive hours. Each time CASARM is removed from cold storage for use, the vial must be allowed to equilibrate to room temperature before it is opened. This will help minimize contamination from condensation of atmospheric moisture, which would hasten agent degradation. Each time a vial of CASARM neat agent is opened for use, its headspace shall be purged with a dry inert gas prior to replacement into cold storage. The time of all operations with CASARM outside of cold storage shall be kept to a minimum.

8.3.4 Standards for Calibration and Sample Spiking. A standard solution of a chemical agent at a known concentration will be used to calibrate laboratory instruments and NRT monitors.

Stock standard solutions are prepared by adding a carefully weighed quantity of neat or dilute liquid CASARM, or an aliquot of dilute CASARM, to the appropriate solvent and diluting to a known volume. The stock standard solution is then serially diluted, by weight or volume, to produce working standards whose concentrations are appropriate for calibration or QC spiking purposes. Working standards shall be prepared with a calculated concentration within 5 percent of the required target concentration. Working standard preparation is further discussed in paragraph 8.5.3.

Table 8-3 provides the minimum frequencies and suitable solvents for standards prepared by the laboratory.

8.3.5 Organic Solvents and Glassware Used to Prepare Standard Solutions. As a minimum, reagent grade organic solvents shall be used to prepare all working standard solutions. In addition, there must be enough solvent within the lot to complete the task at hand. To ensure the quality of these solvents, the organization shall document the solvent manufacturer, solvent grade, and solvent lot number used to prepare standard solutions. While in use, organic solvents shall be kept as water-free as possible. The glassware to be used for solution preparation shall be thoroughly cleaned and dried before use, even if it is new. The procedure for preparation and cleaning of glassware used to prepare standards shall be documented.

8.3.6 Standard Solution Label and Recordkeeping. Stock solutions prepared from neat CASARM shall be labeled with the agent name, the solution concentration, the solvent name, and the date prepared or expiration date. Records shall be maintained for the preparation of agent stock solutions. These records shall include the lot number of agent used, weight of agent used, purity of agent used, solvent used, solvent grade, solvent supplier, solvent lot, date prepared, expiration date, and name and/or unique ID number of preparer.

Working solutions prepared from stock solutions shall be labeled with the agent name, solution concentration, solvent name, and the date prepared or expiration date. Records shall be maintained for the preparation and destruction of these solutions.

Table 8-3. Minimum Frequency and Approximate Concentration Ranges for Preparing and/or Verifying Agent Standards[a]

Agent Solution Type	Concentration Range (mg/mL)	Frequency of Preparation/Verification	Solvent[b]
GB	0.500 to 2.000 <0.500	Annually Monthly	IPA or N-Hexane
VX	0.500 to 2.000 <0.500	Annually Monthly	IPA
HD	0.500 to 2.000 <0.500	Annually Monthly	IPA or N-Hexane
L	0.500 to 10.000 0.005 to 0.500 <0.005	Annually Every 2 months Monthly	N-Hexane or Nonane
HN-1	0.500 to 10.000 <0.500	Monthly Monthly	N-Hexane or Nonane
HN-3	0.500 to 10.000 <0.500	Monthly Monthly	N-Hexane or Nonane
Industrial Gases	Monitoring level value for chemical	Daily	Nitrogen used as mixing/dilution gas

Notes:

[a] Additional concentration ranges other than those provided in this table may be required based on flow rate and sample time.

[b] Chloroform may also be used as a solvent.

These records shall include the agent name, the solution concentration, the solvent name, solvent grade, solvent supplier, solvent lot, the date prepared, the expiration date, the preparer's name or unique ID number, and (if applicable) the identity of the solution from which the serial dilutions were made.

8.3.7 Storage and Shelf Life of Standard Solutions. Standard solutions shall be stored at or below 4°C. Standards shall be left at room temperature only for minimal lengths of time to equilibrate and shall be promptly returned to cold storage when no further use is anticipated.

Table 8-2 defines the shelf life of solutions prepared from neat CASARM. The shelf life may be extended if the organization has data to support a longer shelf life. Agent solutions with concentrations significantly less than (factor of two or more) those in table 8-2 shall have a shelf life of 1 month unless the laboratory can demonstrate otherwise.

Standard solutions that are routinely stored in septum seal vials need not be purged with an inert gas after each use. The time of all operations with standard solutions outside of cold storage shall be kept to a minimum.

8.4 Analysis of Agent Standards

The CMA laboratory shall use the purity or concentration value listed on the certificate accompanying a CASARM or other certified stock standard to derive the concentration of serially diluted agent standards, if applicable. If working standards are provided, the certified concentration of the working standard shall be used to determine agent mass on column.

8.5 Quality Evaluation of Agent Standards

Stock standards derived from the neat agent standards shall be verified by the use of an internal standard for quality analysis. Working standards shall be verified by a separate verification standard prepared at the same time as serially-diluted standards.

8.5.1 Stock Standards. New stock standards prepared by the CMA laboratory shall be verified after preparation and monthly by preparing a solution of stock standard and stock internal standard. The following are stock standard verification requirements:

- Concentration results for new stock standards are acceptable if the relative response factor (RRF) is within 5 percent of the previous stock's initial RRF and the RRF for each individual injection is within 5 percent of each other injection.
- Monthly concentration results are acceptable if the RRF is within 5 percent of the initial RRF for that specific solution and the RRF for each individual injection is within 5 percent of each other injection.
- The RRF is calculated using the following equation (except for HD analyzed via gas chromatograph-flame photometric detector [GC-FPD], which requires forcing the non-linear sulfur response, which approximates a quadratic response to a linear response by taking the square root of the sulfur response).

$$RRF = (AREA_{agent})\,(CONC_{IS}) \,/\, (AREA_{IS})\,(CONC_{agent})$$

where

RRF	=	relative response factor
AREA	=	instrument determined peak area
CONC	=	concentration
IS	=	internal standard.

Equation 8-1. Stock Standard RRF

- Agent concentration of standards shall be corrected for purity using the most recent CASARM, or equivalent, determined concentration. If stock verification fails to satisfy the requirements, the stock standard that failed shall be decontaminated and a new stock standard shall be prepared.
- Verification results that are proven to be unacceptable shall be brought to the attention of the laboratory manager and CMA-RMD. The laboratory manager shall document and implement appropriate required corrective actions. If verification is performed by a contracted laboratory providing working standards, the analytical results shall accompany the working standards.

8.5.2 Quality Evaluation of Internal Standards. The internal standard purity verification requires the following:

- Internal standards shall be verified at a minimum of once every 6 months.
- If the purity assay(s) fails to confirm the purity within 5 percent of the manufacturer's listed purity, the purity assay(s) shall be repeated until the results of the last three trials are all within 5 percent of their average. The newly calculated purity of the internal standard will replace the purity listed on the manufacturer's certificate. No internal standard with a purity less than 85 percent will be used.

8.5.3 Verification of Working Standards. If working standards (no further dilutions necessary) are provided to laboratory and/or monitoring personnel via the CASARM group, quality evaluation of the standards is not required. The CMA laboratory shall verify via documentation, audits, or other means that working standards are prepared from stock standards that satisfy RRF requirements and are at accurate concentrations.

Working standards (challenge and calibration) that are prepared by dilution or serial dilution from a stock standard by the CMA laboratory will be prepared and/or verified at

a minimum of once per month. Verification of working standards shall require the following:

- The verification standard shall be made from a different stock than that used for calibration, or they should be made by a different agent chemist (or operator trained on the preparation SOP).
- RRF of the solution is 1 ±0.1. The RRF shall be determined by comparing a working standard against a verification standard as follows:

$$RRF = (AREA_{working})(CONC_{verification}) / (AREA_{verification})(CONC_{working})$$

where

RRF	=	relative response factor
AREA	=	instrument determined peak area
CONC	=	concentration.

Equation 8-2. Working Standard RRF

- For verification purposes, one or more injections shall be used for automated processes and three injections shall be used for manual processes. For manual processes, the area in the equation then becomes an average area for all injections made.
- Results of working standard verification shall be reviewed and approved by a QC representative prior to implementing the standards for field use.

Once the working standard concentration and dilution process have been verified, the verification working standard may be destroyed. The remaining working standard may be segregated into two separate aliquots (one for calibration and one for challenge).

8.6 Use of Commercially Available Chemical Standards

Certified commercial chemical standards shall be maintained and stored in accordance with vendor-provided recommendations. If a certificate of analysis accompanies commercially available standards, further evaluation of the standard is not required; however, the standard must remain traceable to the certificate of analysis. If a certificate of analysis is not provided, the laboratory should certify the standard in accordance with vendor recommendations.

9. MEASUREMENT TRACEABILITY AND ACCEPTANCE TESTING

9.1 Equipment Measurement Traceability and Calibration

The CMA laboratory shall have an established and documented calibration program. Laboratory equipment used for quantitation measurement shall be calibrated and/or the correctness of operation shall be verified before the equipment is placed into service. Any measurements that affect the quality of the system shall be traceable to a national standard.

Reference standards of measurement (for example, weights, chemical standards, etc.) held by the CMA laboratory shall be:

- Used for calibration and challenge purposes only
- Calibrated by a vendor that can provide traceability to a national standard
- Traced to a national standard at the specified frequency.

9.2 Acceptance Testing for Laboratory and Monitoring Equipment

Acceptance testing shall be required prior to field implementation. Acceptance testing shall only be performed by a certified individual on specific equipment. Table 9-1 provides the acceptance test requirements for laboratory and monitoring equipment for the CMA laboratory. Table 9-2 provides recommendations for conditioning and reconditioning of Depot Area Air Monitoring System (DAAMS) tubes and 3-millimeter (mm) transfer tubes. Pressure Drop Tests for DAAMS tubes are located in appendix E.

9.3 Systemization of NRT Monitor/Data Acquisition System Interface

Data acquisitions systems (that is, process data acquisition and recording system [PDARS], ChromNet, MINI-LINK™, ChromLink, etc.) shall undergo systemization testing. The following procedures are required for all CMA laboratories to systemize the

Table 9-1. Acceptance Testing Requirements for Laboratory and Monitoring Equipment

Equipment	Requirements	Additional Information
General, to Include Non-GC/FPD and Non-GC/MSD Equipment	Site-specific	Arrangements, methods, and requirements will be specified in the purchase order or specification package.
NRT Equipment	1. Functionality Testing. One-hundred percent functionality testing of all NRT monitor units by the vendor.	Acceptance test plan will be prepared by the vendor.
	2. Precision and Accuracy (P&A) Testing. Statistical sampling of each batch of NRT monitor units for agent or simulant Class I P&A tests at 1.0Z-mass equivalent.[a,b] If the sampling fails acceptance criteria, the vendor will perform P&A tests on 100 percent of the batch.	Acceptance test report will include all data and corrective actions.
	3. Challenging. NRT monitors not chosen for P&A testing must pass challenges of 1.0Z-mass equivalent simulant.	
GC-MSD/FPD	Perform an autotune and verify tune relative abundances to test the MSD. Inject a performance check standard designed specifically for the FPD. Verify column performance and detector response.	Site-specific
GC-FPD	Inject a performance check standard designed specifically for the detector. Verify column performance and detector response.	Site-specific
NO_x Pre-Filter	Verify absence of cracks, packing separation, and other physical defects.	Tube fabrication must be accomplished in accordance with laboratory approved procedures.

Table 9-1. Acceptance Testing Requirements for Laboratory and Monitoring Equipment (Continued)

Equipment	Requirements	Additional Information
DAAMS Tube Focusing Tube 3-mm Transfer Tube[c]	Verify shipping manifest; verify absence of obvious defects. Pressure drop testing. Tubes must satisfy method flow rate requirements. All tubes shall be preconditioned before use. Agent testing. Selected tubes shall be aspirated for 12 hours, post-spiked with agent, and analyzed to ensure adequate agent retention and desorption is performed.	Tubes may be purchased from a vendor or fabricated in accordance with CMA-approved procedures. CMA laboratory shall develop a plan to define activities, such as tube tracking, that must be performed to verify tube performance. Pressure drop testing can be performed by the manufacturer. Agent testing recoveries must satisfy acceptance criteria identified in table 12-1. Acceptance testing of DAAMS and transfer tubes can be found in appendix E.
PCT[c]	Verify absence of gaps in the sorbent material and fractures in the glass tubing.	If flow rate errors are encountered and are attributed to the new PCT, the new PCT shall be discarded and another new PCT shall be used. If QC data trending indicates that the PCT lot may be inadequate, the PCT vendor shall be notified for corrective actions.
AgF Conversion Pad[c]	Minimum of 75 percent conversion efficiency, as determined by agent recovery	Laboratories shall develop procedures to ensure that multiple agent monitoring for VX, GA, and HD is conducted in such a manner as not to bias GA and HD detection, when AgF conversion pads are used.
Colorimetric Tube	Visual Inspection: • Verification that the manufacturer certification accompanies the tubes • Verification of the absence of obvious defects. Chemical Testing: • Shall provide a response within ±50 percent of the target concentration at a one-half scale challenge.	

Table 9-1. Acceptance Testing Requirements for Laboratory and Monitoring Equipment (Continued)

Equipment	Requirements	Additional Information
Colorimetric Tube (continued)	Quantity and Frequency: • Ten percent of each sample lot shall be tested prior to using tubes from the lot. • One tube from each sample lot shall be tested during operations at 30-day intervals after the initial acceptance test.	
Sample Pumps	Pump flow requirements meet analytical method requirements	Pump specifications identify maximum flow rate and vacuum capacity.
Sample Lines, Probes, and DAAMS Manifolds	Test new sample lines for interferences prior to introduction of agent. Verify sample lines and sample probes provide a transmission efficiency of ≥75 percent.	Transmission efficiency can be demonstrated by the vendor and/or the Systems Contractor.
Automated Sorbent Tube Desorption Equipment	Verify unit's ability to pass applicable CCV or ICV acceptance criteria. Demonstrate the automated unattended analysis of multiple tubes.	

Notes:

[a] Statistical sampling will be performed in accordance with American National Standards Institute (ANSI)/American Society for Quality Control (ASQC) Z1.4-1993 at General Inspection Level II with a 5 percent acceptance quality level.

[b] Agent testing is preferred over simulant testing.

[c] Acceptance testing will be performed in accordance with a General Inspection Level I with a required acceptable quality level of 2.5 percent or less, as defined by the ANSI/ASQC Z1.4-1993. The entire sample lot shall be rejected if the number of rejects exceeds the value allowed, as determined by the testing procedure, unless items are accepted individually (on a one-at-a-time basis).

Table 9-2. Optimized Parameters for Conditioning and Reconditioning Methods[a]

Method[b]	Temperature (°C)	Time (min)	Flow Rate (mL/min)[c]	Least Critical Parameter
8-mm DAAMS Conditioning	195	120	100	Time
8-mm DAAMS Reconditioning	200	5	50	Flow rate
6-mm DAAMS Conditioning	205	15	550	Time
6-mm DAAMS Reconditioning	195	5	1,000	Flow rate
3-mm Transfer Tube Conditioning	220	3	50	Flow rate

Notes:

[a] Southwest Research Institute: *Optimizing the Conditioning and Reconditioning Methods for Sorbent Tubes for ACAMS and DAAMS Analysis of GB, VX, and HD*, Final Report, February 23, 2000, Table 8.

[b] Other diameter size DAAMS tubes may be used at a CMA facility. In the event a DAAMS tube is used that does not match one of the methods identified, the 8-mm DAAMS conditioning and reconditioning parameters shall be used as a starting point for developing optimized parameters.

[c] Optimized flow rates are based on helium. Other gases such as air and nitrogen may be used. However, the operator should take into consideration that air may cause additional oxidation to the sorbent and the density of air and nitrogen may cause channeling of the sorbent.

NRT monitor/data acquisition system interface in order to demonstrate field acceptance. Following installation of the equipment and verification that the equipment is operational, an NRT monitor will be selected for systemization. Verify that the NRT monitor is compliant with QC criteria. The systemization procedure will be described in detail in the LQCP and shall include the following general elements:

- For all challenges, verify that the data acquisition system screen displays a value within 5.0 percent of the NRT monitor display. Verify that the data acquisition system screen displays the station number, location description, and alarm status and that the alarm printer prints the date, time, station number, and alarm status.
- Verify that after the challenge has cleared the instrument, the NRT monitor shows a subsequent blank cycle value below 0.2Z and that the data acquisition system no longer displays the station number, location description, and alarm status.

- Verify that when power is removed from the NRT monitor utility bus, the NRT monitor continues to operate and that no alarms are activated resulting from loss of utility power.

10. CERTIFICATION AND VALIDATION REQUIREMENTS

10.1 Introduction

The CMA laboratories shall perform a certification and validation process for operators, instruments, and methods to confirm that analytical processes are suitable for use. Method certification will require completion of a successful P&A study (waste screening methods will require spike and recovery determinations) and initial baseline study. Method certification will be required before the method can be used in field support operations. Method validation will be demonstrated through the continuous baseline study.

10.2 Method Description

A method is a process that begins with the collection of a sample and is followed by analysis of the sample by an appropriate analytical technique. Method requirements specify the type of sampling media, airflow rates, collection time, the details of sample preparation, and the types and setpoints of instrumentation that will be used to analyze samples. Methods shall be placed under configuration control and critical parameters shall have identified tolerances that, when exceeded, will result in a “new” method.

The CMA laboratory shall define each method used to support chemical materiel operations as follows:

- Application
  - Matrix
  - Monitoring level
  - Target analyte(s).
- Sample collection
  - Sampling device(s)
  - Flow rate

- Aspiration time
- Collection media (absorbent type, mesh size, bed depth).

• Sample preparation
• Sample analysis
  - Instrument type
  - Detector type and optical filter
  - Configured instrument operational parameters (timing, temperatures, gas types, flow rates, pressures, peak parameters, error limits)
  - Column(s) (type, phase, phase thickness, length, and diameter)
  - Carrier gas or mobile phase type.

10.3 P&A Method Certification

All methods shall successfully satisfy P&A study method certification requirements and/or waste method certification as required in tables 10-1 and 10-2 before the method is allowed to support operations. All data from P&A studies, including method description as defined in paragraph 10.2, shall be submitted to CMA-Monitoring Office for review and concurrence before the method can support operations.

Table 10-1. Type of P&A Method Certification

Application	Type of Certification Required
NRT Chemical Agent Methods	Class I
NRT Industrial Chemical Methods	Class A
Historical Methods	Class I
Historical Perimeter Methods	Class II[a]
Confirmation Methods	Class III[a]
Waste Screening Methods	Spike and Recovery[b]

Notes:

[a] A Class I P&A certification may be performed.
[b] Method Detection Limit (MDL) Study, as defined in 40 CFR Part 136, Appendix B

Table 10-2. P&A Method Certification Requirements

Type of Certification	Number of Operators	Number of Instruments	Number of Days[a]	Target Concentrations[b]	Total Number of Points	Criteria
Class I	2 or more	2 or more	4	8 each at 0.0, 0.5, 0.75, 1.0, 1.5, and 2.0 times the monitoring level (Z)	48	• Target action level (TAL) is greater than the statistical calculated limit of quantitation (LOQ). • Uncertainty in found mass (UIFM) is less than or equal to ±25 percent. • Recovery at the monitoring level is within 75 to 125 percent. • For MSD methods, UIFM is less than or equal to ±40 percent.
Modified Class I	1 or more	1 or more	4			
Class A	1	1	1	6 each at 0.1, 0.5, 1.0, and 2.0Z	24	• Estimated analytical recovery must demonstrate accuracy within ±25 percent with 95 percent confidence at the monitoring level.
Class II	1	1	2	4 at 1.25Z, 8 each at 1.0 and 0.5Z	20	• All samples at 1.25 and 1.0Z yield a positive response, as defined during method development. • No more than 25 percent of the samples at 0.5Z yield a positive response.
Class III	1	1	2	16 at 1.0Z, 4 at 0.0	20	• All challenges at 1.0Z yield a positive response, as defined during method development. • None of the blank samples yield a positive response.
USEPA SW-846	1	1	1	Minimum of 7 replicate samples at the PQL	7	Recovery and precision defined by method development.

Notes:

[a] Preferably consecutive days
[b] The CMA laboratory shall request approval from the CMA-Monitoring Office to use different challenge levels than those values recommended for use.
[c] Statistically determined outliers, not to exceed the square root of the total number of data points, may be excluded from the set. Assignable cause data shall be repeated with documentation as to why the data point(s) was repeated.

When methods are identical, to include spiked mass, with the exception of the aspiration time and WPL monitoring level, only the longest sampling aspiration period shall require a P&A study. For example, a GB 4-hour WPL method with no respiratory protection and the GB 8-hour WPL method with no respiratory protection, assuming the methods are otherwise the same and the 1.0Z mass spikes are equivalent between the methods, only requires P&A certification of the GB 8-hour application.

When performing a P&A study, all sampling and analysis operations shall be performed exactly as set forth in the applicable analytical procedures. All P&A challenges will be in the form of quality plant (QP) samples. Method certification shall be performed with a representative sample matrix. Once a P&A study starts, all challenges will be part of the P&A study except for those during documented maintenance activities. For air sampling methods, the P&A challenges can be injected directly into the NRT instrument, distal end of the sample line, or sample collection media. If the P&A challenge is injected directly into the NRT instrument, this shall be performed at the beginning of the sample cycle and the sample line immediately reconnected (except for process areas where chemical materiel is present). For historical air methods, the P&A challenge shall be spiked onto the sampling media prior to air sample collection. For NRT confirmation methods, the P&A challenge may be spiked onto the sampling media before (pre-spike) or after (post-spike) air sample collection.

Multi-agent methods (more than one agent monitored by the same instrument) shall undergo a P&A study using all analytes (injected separately or as a cocktail) with randomized analyte concentrations. The P&A will demonstrate that the analytes in a cocktail have no adverse interactions and the challenge analytes are compatible. When GB and VX analysis is conducted simultaneously, the amount of G-analog in the GB standard must be corrected; otherwise, GB and VX shall be analyzed separately. For example, for a dual-agent method A and B, challenges may consist of the following: [0.0Z A; 2.0Z B], [0.5Z A; 1.5Z B], [0.75Z A; 1.0Z B], [1.0Z A; 0.75Z B], [1.5Z A; 0.5Z B], [2.0Z A; 0.0Z B].

Operators performing the study shall not be given access to the target concentration (TC) values until after the found concentration (FC) values have been reported, when practical. For quantitative methods, the concentration range must provide quantitative accuracy over the finite range of agent concentrations that the CMA laboratory is required to report. More stringent challenge levels may be specified by permit requirements, analytical method capabilities, and/or alarm level setpoint. The CMA laboratory shall request approval from the CMA-Monitoring Office to use different challenge levels than those values recommended for use.

In some cases, a Class I P&A study may not be feasible, due to lack of instrumentation, lack of trained analysts, and/or sensitivity issues associated with the chemical materiel. The CMA laboratory shall request written approval from the CMA-Monitoring Office to perform a modified Class I P&A study. A Class I P&A study takes into account the variability between instruments and operators, thus demonstrating that the method can be performed by any similarly configured instrument and by any qualified and trained operator. A modified Class I study does not take into account the variability between instruments and/or operators. CMA discourages the use of modified Class I studies but will approve its use under special situations and with the following restrictions:

- If a modified Class I P&A study is performed, then that instrument will be the only one approved to run the specified method.
- If a modified Class I P&A study is performed with only one operator, then that operator will be the only one approved to run the specified method.

For Class II and Class III P&A studies, a positive response shall be defined during method development and can be based on minimal signal height, minimal area counts, signal-to-noise ratios, or minimal percent recovery. Sample analyses for routine operations shall use the same definition of a positive response used in the certification process.

Data generated from a Class I P&A study shall be entered into the CMA mandated statistical program. For multi-agent methods, the program will be used to determine

results for each analyte separately. The results shall be evaluated by pooling all 4 days of generated data into a single group and performing a linear regression analysis of the TC versus FC for the data population. The program shall calculate limit of quantification, uncertainty of found mass, and percent recovery. Statistically determined outliers, not to exceed the square root of the total number of data points, may be excluded from the set. Assignable cause data shall be repeated with documentation as to why the data point(s) was repeated.

10.4 Baseline Method Certification and Validation

All methods shall successfully satisfy the initial baseline study method certification requirements as shown in table 10-3 before the method is allowed to support operations. All data from the initial baseline studies shall be submitted to the CMA-Monitoring Office for review and concurrence. Method validation will be demonstrated by the continuing baseline study. Failure to establish a continuing baseline study (minimum of 4 consecutive weeks of data) immediately after finishing the initial baseline study will require repeating the initial baseline study.

CMA recognizes that for certain projects/operations the above baseline requirements are impractical or impossible to implement. CMA has created alternate baseline requirements as shown in table 10-4. For routine depot surveillance operations, alternate baseline requirements are acceptable. CMA-Monitoring Office written approval is required before implementation of the alternate baseline requirements and will be based on the length of the project/operation, type of operation, and monitoring strategy.

When performing the baseline studies, all sampling and analysis operations shall be performed exactly as set forth in the applicable analytical procedures. All challenges will be in the form of QP samples. For air sampling methods, the baseline challenges can be injected directly into the NRT instrument, distal end of the sample line, or sample collection media. For NRT methods, if the challenge is injected directly into the instrument, the challenge shall be performed at the beginning of the sample cycle and

Table 10-3. Baseline Performance Criteria

Application	Challenges[a]	Performance Standard for Each Analyte
NRT	Daily, each station[b]	If alarm setpoint is 1.0Z, first challenge pass rate ≥ 95 percent for each station, or if this cannot be met, a statistical response rate at the alarm level ≥ 95 percent for each station and a first challenge pass rate ≥ 75 percent
NRT – Common Stack	6 times daily, each station[b]	Statistical response rate at alarm level ≥ 95 percent for each station and first challenge pass rate ≥ 75 percent for each station
NRT – Process Area	Daily, each station[b]	Either challenge pass rate ≥ 75 percent for each station
NRT – Industrial Chemicals	Daily, each station[b]	Either challenge pass rate ≥ 75 percent for each station
NRT – Mobile Station[c]	Initial: One for each station Continuing: Each station every 4 to 5 hours and at the end of the workday or operation	QP FC is ±25 percent of TC
Historical and Other Class I Methods	Each method daily, rotating stations with each station challenged at least once every 28 days	Statistical response rate at reportable limit ≥ 95 percent for each method, data for all stations pooled together by method
Historical – Perimeter	Each method daily, rotating stations	Pass rate ≥ 95 percent (challenges provide positive response), data for all stations pooled together by method
Confirmation of NRT – Common Stack and Process Effluents	Daily each method and each station[b]	Pass rate ≥ 95 percent (challenges provide positive response), data for all stations pooled together by method
Confirmation of NRT – Agent	Each method daily, rotating stations with each station challenged at least once every 28 days[d]	Pass rate ≥ 95 percent (challenges provide positive response), data for all stations pooled together by method
Confirmation of NRT– Industrial Chemicals	Once per month	In accordance with manufacturer specifications

Table 10-3. Baseline Performance Criteria (Continued)

Application	Challenges[a]	Performance Standard for Each Analyte
Confirmation of Historical	Daily, rotating stations and methods	Pass rate ≥ 95 percent (challenges provide positive response), data for all stations pooled together by method
Waste Screening	None	N/A

Notes:

[a] All challenges at 1.0Z of monitoring level for each analyte the station monitors. Daily challenges must be at least 12 hours apart.
[b] When multiple near real-time (NRT) monitors are used to sample the same location, each instrument shall be considered its own station.
[c] A mobile station is comprised of NRT instruments and sampling points that change location in accordance with operational requirements to include real-time analytical platforms (RTAPs) and first entry monitoring.
[d] Challenges are spiked before or after sampling period and are equivalent to the mass of analyte at 1.0Z collected during one sampling cycle of the NRT monitor.

Table 10-4. Alternate Baseline Performance Criteria[a]

Application	Challenges[b]	Performance Standard for Each Analyte
NRT – Chemical Agents/Industrial Chemicals	Initial: One for each station[c]	QP FC is ±25 percent of TC
	3-Day Initial: Three challenges per day for each station[c] (minimum 3 hours between challenges)	If alarm setpoint is 1.0Z, first challenge pass rate will be ≥ 95 percent for each station, or if this cannot be met, a more conservative alarm level will be used with an either challenge pass rate = 100 percent and first challenge pass rate ≥ 80 percent or statistical response rate at the alarm level ≥ 95 percent.
	Continuing: Each station[c] every 4 to 5 hours and at the end of the workday or operation (If a successful 3-day initial baseline has been completed, then one challenge per day for each station[c])	If alarm setpoint is 1.0Z, first challenge pass rate will be ≥ 95 percent for each station, or if this cannot be met, a more conservative alarm level will be used. If after 3 days, either challenge pass rate = 100 percent and first challenge pass rate ≥ 80 percent or statistical response rate at the alarm level ≥ 95 percent can be demonstrated, then a minimum of one daily challenge per station will be required. If the equipment has been taken offline (turned off or placed in standby mode), a successful challenge is required before the equipment can support operations.
NRT – Chemical Agents/Industrial Chemicals – Mobile Station[d]	Initial: One for each station[c] Continuing: Each station every 4 to 5 hours and at the end of the workday or operation	QP FC is ±25 percent of TC
Historical, Class I Methods	Each method daily, rotating stations	QP FC is ±40 percent
Confirmation of NRT	Each method daily, rotating stations[e]	All challenges provide positive response.
Confirmation of Historical	Daily, rotating stations and methods	All challenges provide positive response.
Waste Screening	None	N/A

Notes:

[a] CMA-Monitoring Office written approval is required before implementation.

Table 10-4. Alternate Baseline Performance Criteria[a] (Continued)

Notes: (continued)

[b] All challenges at 1.0Z of monitoring level for each analyte the station monitors. Daily challenges must be at least 12 hours apart, unless otherwise specified.

[c] When multiple NRT monitors are used to sample the same location, each instrument shall be considered its own station.

[d] A mobile station is comprised of near real-time (NRT) instruments and sampling points that change location in accordance with operational requirements to include real-time analytical platforms (RTAPs) and first entry monitoring.

[e] Challenges are spiked before or after sampling period and are equivalent to the mass of analyte at 1.0Z collected during one sampling cycle of the NRT monitor.

the sample line will be immediately reconnected (except for process areas where chemical materiel is present). For historical air methods, the challenge shall be spiked onto the sampling media prior to air sample collection. For NRT confirmation methods, the challenge may be spiked onto the sampling media before (pre-spike) or after (post-spike) air sample collection.

During baseline studies, sampling of incinerator exhaust (furnace ducts and common stack) shall be performed in a similar way as when chemical agent operations are being performed and shall require that the primary or secondary chamber of the furnace (or any furnace for the common stack) be at its operating temperature. Challenges shall be injected through the stack sampling system (where used).

For NRT stations failing two consecutive challenges (F1 and F2), corrective action shall be implemented and monitoring shall be considered unable to support operations until the instrument is back in control or a conforming NRT system is placed into service. Preventive maintenance may be performed at any time during baseline studies.

10.4.1 Initial Baseline Study. Initial baseline studies demonstrate the readiness of the monitoring system to support operations. No method shall be allowed to support operations until it can successfully satisfy the initial baseline study method certification requirements. During this period (or sooner), all sampling lines shall demonstrate a recovery of ±40 percent of the TC (at the applicable monitoring level) when challenged at the distal end.

The initial baseline study consists of air method challenges for a consecutive 28-day period. Performance standards and challenge frequencies shall be as specified by table 10-3. Because construction/systemization activities during this period may affect or prevent certain monitoring operations, the following shall be considered:

- Initial baseline data shall be entered into the CMA mandated statistical program (see section 16). A report from each initial baseline study shall be transmitted to the CMA-Monitoring Office for review and concurrence.

- Each NRT station is an independent baseline study. Passing or failing the initial baseline will be on a per station basis and not on a global basis. Not all stations need to be in the same 28-day window.
- Each historical and confirmation method is an independent baseline study, but each station may affect more than one method and all stations using that method shall be included. Passing or failing the initial baseline will be on a per method basis. Not all methods need to be in the same 28-day window.
- A minimum of 20 days of data in a 28-day period is required for each initial baseline study. Excluding days from the study is only allowed if construction/systemization activities or other operations prevented the daily challenge or if a challenge is excluded due to documented assignable cause (for example, use of the wrong standard).
- If an initial baseline study is failing, the study can be extended, as improvements are made and corrective actions take effect, shifting the 28-day window accordingly.

The alternate initial baseline consists of the following:

- For 1-day operations or daily operations in which the monitoring systems are taken offline (shutdown or put on standby) at the end of the workday, a successful initial baseline is passing one QP challenge for each NRT station and each historical and confirmation method as specified by table 10-4. The initial baseline shall be repeated each operational day before the monitoring system can support operations.
- For continuous operations or where the monitoring systems are not taken offline at the end of the day, a successful initial baseline will consist of a consecutive 3-day period for each NRT station and each historical and confirmation method as specified by table 10-4.
- Initial baseline data shall be submitted to the CMA-Monitoring Office as part of the continuing baseline data.

10.4.2 Continuing Baseline Study. The continuing baseline study validates the long-term performance of the monitoring system and starts immediately after successful completion of the initial baseline.

Continuing baseline requirements:

- Each NRT station is an independent baseline study. Passing or failing the continuing baseline will be on a per station basis and not on a global basis.
- Each historical and confirmation method is an independent baseline study. Passing or failing the continuing baseline will be on a per method basis.
- Failing to meet the performance standards described in table 10-3 will require corrective action and additional QPs until the problem is resolved. The problem and corrective action shall be documented in a Corrective Action Report, which shall be submitted to the CMA-Monitoring Office within 7 days after the end of the reporting period.
- For historical or confirmation stations failing a QP challenge, corrective actions will be required and daily QP challenges will continue until the problem is resolved and a passing QP challenge is obtained for that station. These diagnostic QP challenges shall not be included in the baseline studies, but shall be reported as part of the Corrective Action Report.
- All sampling lines, except those used to monitor process areas, shall be tested every 2 months to demonstrate a recovery of ±40 percent of the TC (at the applicable monitoring level) when challenged at the distal end.
- Certified operators shall participate in the continuing baseline study.
- All continuing baseline data shall be transmitted to the CMA mandated statistical program every 2 weeks.

Alternate continuing baseline requirements:

- For NRT stations, QP challenges will be required at the beginning of the work day, every 4 to 5 hours, and at the end of the workday or operation. If after 3 days all challenges are successful (P1 or P2) and first challenge pass rate is

greater than or equal to 80 percent or a statistical response rate at the alarm level greater than or equal to 95 percent can be demonstrated (or having successfully completed a 3-day initial baseline), then only one daily challenge per station will be required (except mobile stations, which shall continue with challenges every 4 to 5 hours and at the end of the work day).

- For NRT stations failing two consecutive challenges (F1 and F2), operations supported by this station will be suspended until the problem is resolved and a passing challenge is obtained.
- For historical or confirmation methods, one daily QP challenge shall be required for each method, rotating stations. Failing QP challenges will require corrective actions and additional QP challenges until the problem is resolved.
- For sample line challenges during first entry operations, a monthly QP challenge should be performed to support entry operations.
- All continuing baseline data shall be transmitted to the CMA-Monitoring Office every 2 weeks.

10.4.3 Baseline Recertification. Cessation of monitoring activities (taking equipment offline and/or stop doing QPs) greater than 60 days for any NRT station, historical method, or confirmation method shall require repeating the initial baseline study and re-establishment of the continuing baseline for that particular station and/or method. All the initial baseline requirements shall apply. Cessation of NRT monitoring activities for less than or equal to 60 days will require recertification in accordance with table 10-5. Cessation of historical and confirmation methods less than or equal to 60 days does not require recertification. Common stack recertification data shall be submitted to the CMA mandated statistical program.

Table 10-5. Continuing Baseline Recertification Requirements

Application	Number of Days Suspended	Number of Challenges[a] Required	Performance Standard
NRT	1 to 10	1	±25 percent of TC
NRT – Common Stack	1 to 10	6 daily[b]	All challenges ±25 percent of TC
NRT	11 to 30	2[c]	All challenges ±25 percent of TC
NRT – Common Stack	11 to 30	6 daily for two days[b]	All challenges ±25 percent of TC
NRT	31 to 60	4[c]	All challenges ±25 percent of TC
NRT – Common Stack	31 to 60	6 daily for five days[b]	Statistical response rate ≥ 95 percent

Notes:

[a] All challenges at 1.0Z of monitoring level
[b] Challenges shall be 3 to 5 hours apart.
[c] Successive challenges shall be a minimum of 12 hours apart.

11. CALIBRATION AND TEST METHODS

11.1 General Requirements

The CMA laboratories shall have an established and documented calibration program. As part of this program, the CMA laboratories shall have a calibration plan. This plan shall define calibration requirements for chemical materiel methods to include physical measurement equipment calibration requirements for equipment such as pH meters, analytical balances, thermometers, thermocouples, critical orfices, mass flowmeters, flow controllers, and other laboratory equipment that validates a measurement.

The CMA laboratory/monitoring group and/or Systems Contractor (SC) shall be responsible for all tests and calibrations performed to support CMA operations by outside laboratories. All outside work shall meet all the requirements described in this document. The CMA laboratory/monitoring group shall have written procedures to verify work performed by outside laboratories is performed in accordance with recognized national standards, when applicable.

11.2 Chemical Agent and Industrial Chemical Calibration Requirements

CMA laboratories shall use calibration requirements as identified in table 11-1 for methods/instrumentation supporting project operations. Multi-agent methods (more than one agent monitored by the same instrument) will employ calibrations using each analyte injected separately or as a cocktail. Calibration data shall be formatted to support storage and retrieval. Calibration records shall identify the following:

- Chemical name
- Date and time
- Instrument ID number
- Name or unique ID number of operator
- Calibration standard ID number (traceable back to stock solution).

Table 11-1. Chemical Agent and Industrial Chemical Calibration Requirements

Application	Frequency	Range	Acceptance Criteria	Notes
NRT	• At first deployment • If required by corrective action • Cessation of monitoring activities greater than 60 days • If instrument has been turned off for more than 10 days	Minimum 3 point calibration curve with masses covering the analytical range to be analyzed	Calibration regression lines shall provide a correlation coefficient value of 0.990 or greater.	
NRT (ACAMS)	• At first deployment • If required by corrective action • Cessation of monitoring activities greater than 60 days • If instrument has been turned off for more than 10 days	Minimum of 2 injections at the monitoring level	All calibration injections after the initial calibration injection yield a FC within ±25 percent of the TC. Carryover of the 1.0Z calibration injection is less than 0.2Z.	Calibrations of NRT agent stack/duct monitors may be performed through the stack/duct sampling system or instrument inlet.
NRT (MINICAMS®)	• At first deployment • If required by corrective action • Cessation of monitoring activities greater than 60 days • If instrument has been turned off for more than 10 days	Minimum of 2 injections at the monitoring level	All calibration injections after the initial calibration injection yield a FC within ±25 percent of the TC. Carryover of the 1.0Z calibration injection is less than 0.2Z.	Calibrations of NRT agent stack/duct monitors may be performed through the stack/duct sampling system or instrument inlet.

Table 11-1. Chemical Agent and Industrial Chemical Calibration Requirements (Continued)

Application	Frequency	Range	Acceptance Criteria	Notes
GC (Non-MSD)				
Class I	• At first deployment • Failure to pass the ICV • Failure to pass two consecutive CCVs • As defined in the Site-Specific LQCP	Minimum 3 point calibration curve with masses covering the analytical range for each chemical to be analyzed, with one mass equivalent at or below the lowest 1.0Z monitoring level	Calibration regression lines shall provide a correlation coefficient value of 0.990 or greater.	All sample analyses shall be traceable back to their most recent calibration curve. Spiking directly onto 3-mm transfer tubes rather than DAAMS tubes is allowed if the two spiking procedures agree within 5 percent.
Class II and Class III	N/A	N/A	N/A	Site-specific Demonstrate retention time and positive result based on method certification process

Table 11-1. Chemical Agent and Industrial Chemical Calibration Requirements (Continued)

Application	Frequency	Range	Acceptance Criteria	Notes
GC-MSD				
Class I	• At first deployment • Failure to pass the ICV • Failure to pass two consecutive CCVs • As defined in the LQCP	Minimum 3 point calibration curve with masses covering the analytical range for each chemical to be analyzed, with one mass equivalent at or below the lowest 1.0Z monitoring level	Calibration regression lines shall provide a correlation coefficient value of 0.990 or greater.	• PFTBA tunes shall be used for (EI). PFDTD (perfluoro-5,8-dimethyl-3,6,9-trioxidodecane) tunes shall be used for (CI). Tunes shall be performed monthly or as needed to maintain relative abundances. • Verify mass axis calibration every 12 hours. If the instrument does not meet the tune criteria, then a tune should be performed. • Spiking directly onto 3-mm transfer tubes rather than DAAMS tubes is allowed if the two spiking procedures agree within 5 percent.
Class II and Class III	N/A	N/A	N/A	Site-specific Demonstrate retention time and positive result based on method certification process.

Table 11-1. Chemical Agent and Industrial Chemical Calibration Requirements (Continued)

Application	Frequency	Range	Acceptance Criteria	Notes
Waste Screening	See calibration requirement for application	See calibration requirement for application	See calibration requirement for application	Calibration requirements are based on the instrument used in the waste screening method.
Colorimetric Tube	N/A	N/A	N/A	Use in accordance with manufacturer specifications.

11.2.1 Gas Chromatograph-Mass Selective Detector (GC-MSD) Calibration. Tables 11-2 and 11-3 provide examples of tune relative abundances for the Agilent Technologies 5973 mass selective detector (MSD) and 5973N MSD. If a bromofluorobenzene (BFB) target tune is utilized, BFB verification will be performed following a successful tune and every 12 hours thereafter to verify sensitivity in the mid-mass region. All mass ratios must state "PASS" on the verification report in order for the BFB verification standard to be accepted.

A successful BFB verification must meet the criteria listed in table 11-4. Tables 11-5, 11-6, 11-7, 11-8, 11-9, and 11-10 identify expected ion percent ratios for GB, VX as G-analog, VX, HD, L, and GA on the Agilent Technologies 5973 MSD and 5973N MSD using electron impact ionization. Table 11-11 identifies expected range of ion ratios for VX and GB using chemical ionization. If acceptance criteria cannot be satisfied, the gas chromatograph (GC)/detector system will be recalibrated. Failure of the GC/detector system to recalibrate shall require implementation of corrective actions. If different ions or ion ratios are used, the laboratory/monitoring group shall identify the ions and acceptable ion ratios in the LQCP or an SOP/IOP/LOPs.

11.2.2 Calibration of Other Equipment. Additional calibration requirements shall be specified by the laboratory/monitoring group for other equipment. Calibration requirements for other equipment are provided in table 11-12.

11.2.3 Calibration of M43A1 Chemical Agent Detector. Chemical agent calibration or challenging is not required. Simulant paddle challenging shall be performed daily in accordance with site-specific procedures.

11.3 Specified Physical Measurement Equipment

Cleaning and replacement of critical orifices and/or needle valves will be required on an as needed basis. Critical orifices and/or needle valves shall be function-checked daily with the use of a calibrated flowmeter.

Table 11-2. Example of EI Autotune Relative Abundances for AT 5973 and 5973N MSD[a]

Mass (Atomic Mass Units)	Relative Abundance (%)	Isotope Ratio (%)
69	100	Mass 70: .54 to 1.6
219	70 to 150	Mass 220: 3.2 to 5.4
502	≥ 2.5	Mass 503: 7.9 to 12.3

Note:

[a] These abundances are suggested, based on an autotune, and are instrument-specific for electron impact ionization. (Reference: p.42 AT Manual for AT course H4050A). The laboratory may use a mid-mass tune (that is, BFB Target Tune).

Table 11-3. Example of BFB Target Tune Relative Abundances for AT 5973 and 5973N MSD[a]

Mass (Atomic Mass Units)	Relative Abundance (%)	Isotope Ratio (%)
69	100	Mass 70: .54 to 1.6
131	≥35	None
219	≥30	Mass 220: 3.2 to 5.4
502	0.8 to 1.0	Mass 503: 7.9 to 12.3

Note:

[a] These abundances are suggested, based on an autotune, and are recommended by the manufacturer based on USEPA Method 624 requirements for sensitivity in the mid-mass region. The laboratory may use a mid-mass tune (that is, BFB Target Tune).

Table 11-4. BFB Verification Mass Ratio Requirements[a,b]

Target Mass	Comparison Mass	Mass Ratio Range (%)
50	95	15 to 40
75	95	30 to 60
95	95	100
96	95	5 to 9
173	174	0 to 2
174	95	50 to 100
175	174	5 to 9
176	174	95 to 101
177	176	5 to 9

Note:

[a] These mass ratios are suggested, based on an electron impact autotune, and are based on USEPA Method 624. The laboratory may use a mid-mass tune (that is, BFB Target Tune).
[b] A BFB verification sample is only required if a BFB Target Tune is being utilized.

Table 11-5. Expected GB Ion Percent Ratios

GB Ion	Comparison Ion	Expected Ion Ratio (%)[a]
81[b]	99	6 to 20
99	99	100
125	99	17 to 42

Notes:

[a] Ion ratios based on operational experience with electron impact ionization source
[b] Ion 81 is a common interference ion and may cause substantial background problems. In the event high background is present with respect to ion mass 81, ion 81 mass can be deleted and a secondary ion must be substituted.

Table 11-6. Expected VX (as G-analog) Ion Percent Ratios

VX Ion	Comparison Ion	Expected Ion Ratio (%)
81[a]	99	32 to 53
99	99	100
111[a]	99	21 to 61

Note:

[a] Ions 81 and 111 are common interference ions and may cause substantial background problems. In the event high background is present with respect to ion masses 81 and/or 111, ion 99 can be used alone.

Table 11-7. Expected VX (as VX) Ion Percent Ratios

VX Ion	Comparison Ion	Expected Ion Ratio (%)
114	114	100
127	114	10 to 20
139	114	1 to 6
167	114	2.5 to 5

Table 11-8. Expected HD Ion Percent Ratios

HD Ion	Comparison Ion	Expected Ion Ratio (%)
109	158	320 to 528
111	158	118 to 196
158	158	100
160	158	52 to 86

Note:

In instances when there are substantial background problems, an ion must be deleted and a secondary ion should be substituted. Ions are based on electron impact.

Table 11-9. Expected L Ion Percent Ratios

L Ion	Comparison Ion	Expected Ion Ratio (%)
240.9	242.9	60 to 88
242.9	242.9	100
244.9	242.9	30 to 60

Note:

In instances when there are substantial background problems, an ion must be deleted and a secondary ion should be substituted. Ions are based on electron impact.

Table 11-10. Expected GA Ion Percent Ratios

GA Ion	Comparison Ion	Expected Ion Ratio (%)
70	70	100
133	70	30 to 56
162	70	22 to 42

Note:

In instances when there are substantial background problems, an ion must be deleted and a secondary ion should be substituted. Ions are based on electron impact.

Table 11-11. Expected VX (as G-analog) and GB Ion Ratios for Chemical Ionization

Ion	Comparison Ion	Expected Ion Ratio (%)
VX 144[a] Primary Ion	144	100
VX 161[a] Qualifier Ion	144	±20 percent of expected percent relative abundance established by initial calibration
GB 158[a] Primary Ion	158	100
GB 175[a]	158	±20 percent of expected percent relative abundance established by initial calibration
VX[a,b]	258	100

Notes:

[a] Chemical ionization using 30 percent ammonia
[b] Direct VX

Table 11-12. Calibration Requirements for Other Equipment

Equipment	Calibration Requirements
High Performance Liquid Chromatograph/Mass Spectrometer	Initial calibration must be performed daily, and reference standards must be run weekly. An independent reference standard or an independent stock will be prepared to serve as a reference standard. Weekly, the reference standard will be analyzed with the initial calibration standards. The results of the reference standard must be within 25 percent of the true value for the calibration to be considered valid. If the reference standard fails, the source of the problem must be identified and corrected, and the calibration repeated.
Ion Chromatograph	Calibration will cover the method analytical range for each analyte to be monitored. Instrument hardware calibration and performance checks will be required when a successful analytical calibration range cannot be obtained.
Ion Trap Mass Spectrometer (ITMS)	ITMS tuning using a performance tuning agent will be performed.

Flowmeters and flow controllers used to support measurements shall be within the dynamic flow range of the method and shall be calibrated at site ambient conditions at least every 360 days.

12. QC SAMPLE REQUIREMENTS

12.1 Introduction

All instruments used for the analysis of chemical agents or industrial chemicals shall be subject to periodic QC sample analysis for each chemical the instrument will analyze to check the process from sample collection through analysis. Table 12-1 provides a summary of QC challenge requirements.

12.2 Air Method QC Samples

12.2.1 NRT Methods. All NRT instruments will be challenged at the frequencies identified in tables 10-3 and 10-4. All challenges will be in the form of QP samples. QP samples can be injected at the distal end of the sample line or directly into the instrument. If the challenge is injected directly into the instrument, the challenge shall be performed at the beginning of the sample cycle and the sample line will be immediately reconnected. When using a V to G conversion pad assembly to directly challenge the NRT sample inlet, allow conversion time to occur, then reconnect the sample line. Stations equipped with a stack sampling system shall be challenged through this system.

The QP challenge shall deliver the same mass of analyte (±5 percent) to the instrument as would be collected if the instrument were sampling air containing agent at 1.0Z during the entire sampling cycle. Instruments monitoring more than one analyte shall be challenged for each analyte, either with a cocktail sample or by individual samples for each analyte.

Some process area stations will require challenging when the instrument is already detecting agent. In this case, the QP challenge shall be acceptable if the challenge result is within ±25 percent of the sum of the spiked concentration plus the average of two cycles prior to the injection of the QP and two cycles immediately after the QP response, or the sample line can be disconnected and the system allowed to clear prior

Table 12-1. Summary of QC Challenge Requirements

Application	QC Type	Frequency During Operational Periods	Target Concentration	Acceptance Criteria	Failed Challenge Requirements
NRT Agent	QP	• After calibration • Daily (minimum 12 hours between challenges) • Twice daily for L monitors (minimum of 6 hours between challenges) • After F1 • After preventive maintenance • After corrective action • After an agent alarm (except process areas), on the alarming instrument only • At the discretion of the operator • For mobile stations, every 4 to 5 hours, and at the end of the workday or operation • More frequently if required by table 10-4	1.0Z ±5 percent	• FC within ±25 percent of the TC • Carryover must be less than 0.2Z	• See NRT QP Challenge Protocol

Table 12-1. Summary of QC Challenge Requirements (Continued)

Application	QC Type	Frequency During Operational Periods	Target Concentration	Acceptance Criteria	Failed Challenge Requirements
NRT Agent – Common Stack	QP	• After calibration • 6 times per day (every 3 to 5 hours) • After F1 • After preventive maintenance • After corrective action • After an agent alarm on the alarming instrument only • At the discretion of the operator	1.0Z ±5 percent	• FC within ±25 percent of the TC • Carryover must be less than 0.2Z	See NRT QP Challenge Protocol
NRT Industrial Chemicals	QP	• After calibration • Daily (minimum 12 hours between challenges) • After preventive maintenance • After corrective action • At the discretion of the operator • More frequently if required by table 10-4	1.0Z ±5 percent	FC within ±25 percent of the TC	See NRT QP Challenge Protocol
Historical/ Confirmation Class I	QP	• Each method daily, rotating stations with each station challenged at least once every 28 days • Daily for each process effluent NRT confirmation station	1.0Z ±5 percent	FC within ±40 percent of the TC	A station failing a QP challenge shall require corrective action and additional daily diagnostic QP challenges until the problem is resolved.

Table 12-1. Summary of QC Challenge Requirements (Continued)

Application	QC Type	Frequency During Operational Periods	Target Concentration	Acceptance Criteria	Failed Challenge Requirements
Historical/ Confirmation Class I (continued)	QL	• ICV immediately following an instrument calibration • CCV for every 20 samples analyzed or each operational day (every 12 hours for a GC-MSD), whichever comes first	• ICV within calibration range • CCV at the monitoring level (if more than one monitoring level, alternate between levels)	Found mass shall be within ±15 percent (±30 percent for MSD) of target. For < 0.3 ng the requirement is ±35 percent.	If the average percent recovery of two consecutive out-of-control CCV samples is ≤ 50 percent, implement corrective action, analyze duplicate samples, or use appropriate data qualification for all samples after the last in-control CCV.
Historical/ Confirmation Class II or III	QP	• Each method daily, rotating stations with each station challenged at least once every 28 days • Daily for each process effluent NRT confirmation station	1.0Z ±5 percent	Positive response (as defined during method development)	A station failing a QP challenge shall require corrective action and additional daily diagnostic QP challenges until the problem is resolved.
	QL	• Every 20 samples analyzed or each operational day (every 12 hours for a GC-MSD), whichever comes first	Monitoring level	Positive response	Implement corrective action, analyze duplicate samples, or use appropriate data qualification for all samples after the last in-control QL.
Colorimetric Tubes	QP	• Each station once per month	At one-half scale	Positive response within manufacturer's recommended accuracy	Perform additional QPs.

Table 12-1. Summary of QC Challenge Requirements (Continued)

Application	QC Type	Frequency During Operational Periods	Target Concentration	Acceptance Criteria	Failed Challenge Requirements
Waste Screening	QL	• ICV immediately following an instrument calibration • CCV for every 20 samples analyzed or each operational day (every 12 hours for a GC-MSD), whichever comes first	Within calibration range	Found mass shall be within ±15 percent (±30 percent for MSD) of target	If the average percent recovery of two consecutive out-of-control CCV samples is ≤ 50 percent, all samples since the last in-control CCV shall be re-analyzed.
	MSS	• One matrix spike per batch of 20 samples or less • One matrix spike duplicate for each matrix spike	PQL	• Method-specific accuracy and precision • MSS and MSS duplicate shall have a RPD to ≤ 25 percent	Following two consecutive failing matrix spike and matrix spike duplicates, the samples must be re-analyzed and a new matrix spike and matrix spike duplicate must be prepared.

to performing the challenge. If the line is disconnected, an acceptable challenge will be ±25 percent of the TC.

12.2.1.1 NRT QP Challenge Protocol. The NRT QP challenge protocol shall be as follows:

- Perform first challenge.
  - If the QP challenge meets the acceptance criteria, record as P1 and collect the required data.
  - If the QP challenge fails the acceptance criteria, record as F1, perform second challenge.
- Perform second challenge.
  - If the QP challenge meets the acceptance criteria, record as P2 and collect the required data.
  - If the QP challenge fails the acceptance criteria, record as F2 and perform corrective action.
- Perform corrective action.
  - The monitoring system at this station shall be considered unable to support operations until the problem is resolved and the instrument is back in control.
  - Continue performing corrective action until a passing QP is observed; record as P3 and collect the required data.

12.2.1.2 Data Collection for NRT Methods. The following information will be recorded for each NRT QP sample:

- Identification: Instrument ID number, station location, and/or station ID number
- Challenge: Name or unique ID number of the individual performing the challenge, analyte, date and time of challenge, exact volume and ID number of the QC standard solution used to spike the sample, and target concentration
- FC.

12.2.2 Historical/Confirmation Methods.

12.2.2.1 QP Samples. All historical/confirmation method challenges shall be performed in accordance with the frequencies identified in tables 10-3 and 10-4. All challenges will be in the form of QP samples. Historical QP samples shall be prepared by spiking analyte onto the sampling media in the laboratory prior to aspiration, placed at the sampling station for a full aspiration cycle, then analyzed at the laboratory within 72 hours of sample completion. Confirmation QP samples shall be prepared by spiking onto the sampling media in the laboratory, then placed at the sampling station for the full aspiration period or aspirated at the sampling station for the full period, then spiked onto the sampling media in the laboratory. For on-demand confirmation configurations, QP samples may be aspirated when initiated by an NRT alarm. All confirmation QP samples shall be analyzed in the laboratory within 72 hours of sample completion.

Historical QP samples shall contain the same mass of analyte (±5 percent) as would be collected if the sample were aspirated with air containing agent at 1.0Z during the entire sampling cycle. Confirmation QP samples shall contain the same mass of analyte (±5 percent) as would be collected if the sample were aspirated with air containing agent at 1.0Z during one sampling period of the co-located NRT instrument. Methods monitoring more than one analyte simultaneously shall be challenged for each analyte, either with a cocktail sample or by individual samples for each analyte.

A historical or confirmation station failing a QP challenge shall require corrective action and additional daily diagnostic QP challenges until the problem is resolved. These diagnostic QP challenges shall not be included in the baseline studies, but shall be reported as part of the Corrective Action Report.

12.2.2.2 Quality Laboratory (QL) Samples. All laboratory instruments (GC-FPD, GC-MSD, etc.) used for analysis as part of a historical or confirmation method shall be challenged with QL samples (initial calibration verification [ICV] and CCV samples). Class I methods will have an ICV sample immediately following an instrument calibration. The ICV target concentration shall be within the calibration range. All

analyses shall include a CCV sample for every 20 samples analyzed or each operational day (every 12 hours for a GC-MSD), whichever comes first. The CCV target concentration shall be equivalent to the monitoring level for each method. If more than one monitoring level is covered in the calibration range, the CCV spike mass shall alternate between levels. Failing the CCV requirements shall require corrective actions (analysis of duplicate samples or appropriate data qualification) for all samples after the last in-control CCV.

The CMA laboratory may implement the use of double blind QL samples for Class I methods. Designated CMA laboratory/monitoring group personnel will spike a DAAMS tube or transfer tube with a known volume and concentration of dilute chemical agent. The blind QL sample will be introduced into the sample batch and should meet the acceptance requirements specified in table 12-1. The CMA laboratory will be responsible for tracking the double blind QL.

12.2.2.3 Laboratory Control Samples for Historical/Confirmation Methods. The LQCP shall include a description of the implementation of blank and duplicate samples into the sample load at the CMA laboratory.

12.2.2.4 Data Collection for Historical/Confirmation Methods. The following information will be recorded for each historical/confirmation QP sample:

- Same information collected for routine samples and recorded on the sample chain-of-custody (COC) sheet
- Identification: Sample ID number, station location and/or station ID number
- Aspiration: Date and time of aspiration, flow rates, and sampling media
- Challenge: Name or unique ID number of the individual performing the spiking, analyte, exact volume and ID number of the QC standard solution used to spike the sample, whether spiking was performed before or after sample aspiration, and TC
- Date and time analyzed, instrument number, and FC.

The following information will be recorded for each historical/confirmation QL sample:

- Aspiration: Aspiration media, sampling media or type (DAAMS or transfer tube)
- Challenge: Name or unique ID number of the individual performing the spiking, analyte, exact volume and ID number of the QC standard solution used to spike the sample, TC
- Date and time of analysis, instrument number, and FC.

12.3 Waste Method QC Samples

All waste screening methods shall include QC sample analyses. A matrix spike sample (MSS) and matrix spike sample duplicate (MSSD) at the applicable practical quantitation limit (PQL) shall be included per batch of 20 samples or less. An ICV sample shall be analyzed immediately following an instrument calibration. The ICV TC shall be within the calibration range. CCV samples shall be required for every 20 samples analyzed or each operational day (every 12 hours for a GC-MSD), whichever comes first, and at the end of each sample batch. The CCV TC shall be within the calibration range. Failing the CCV requirements shall require the re-analysis of all samples since the last in-control CCV.

12.3.1 Requirements for Hydrolysate Analysis. Each batch of hydrolysate will be sampled, and the analysis of each sample will be preceded by an MSS from a previous hydrolysate batch. Following sample analysis, a matrix spike duplicate from a previous hydrolysate batch or the current hydrolysate batch will be analyzed. The MSS will use the concentration of analyte used to determine the method detection limit (MDL). The matrix spike and duplicate must agree within ±25 percent. Since samples will be analyzed as received and no batching of sample runs will occur, the CCV samples will be run on a 12-hour schedule to verify that the instrument is operational.

12.3.2 Laboratory Control Samples for Waste Methods. For liquid samples, reagent-grade water or the solvent used to make the standards will be used as a blank

matrix. For solid samples, a solid that has not been exposed to the analyte(s) being analyzed or that has been certified by a vendor as being clean may be used.

The LQCP will include a description of the implementation of blank and duplicate samples into the sample load at the CMA laboratory.

12.3.3 Data Collection for Waste Methods. The following information will be recorded for each waste screening QL/MSS/MSSD sample:

- Same information collected for routine samples and recorded on the sample COC sheet
- Identification: Sample ID number
- Challenge: Name or unique ID number of the individual performing the spiking, analyte, exact volume and ID number of the QC standard solution used to spike the sample, and TC
- Date and time of analysis, instrument number, and FC.

13. OPERATIONAL CONFIGURATION CONTROL AND OPERATIONAL SPECIFICATIONS

13.1 Introduction

The CMA laboratory/monitoring group shall identify operational limits for parameters under operational configuration control and performance specifications for monitoring and laboratory equipment. Method flexibility may be used to adjust parameters under configuration controls provided that the quality of the data is not affected. The tolerance range for parameters under configuration control will be determined and documented in the LQCP. Revision of one of the identified elements outside site-specific tolerance limits requires the performance of a new P&A study. Table 13-1 provides configuration control parameters for selected CMA laboratory/monitoring group equipment.

13.2 NRT Monitoring Instruments

NRT parameters under configuration control for CMA operations are identified in table 13-2. NRT performance specifications for CMA operations are identified in table 13-3.

13.3 GC-Detector Systems

The GC and detector parameters under configuration control are listed in table 13-4. GC and detector system performance specifications are identified in table 13-5.

13.4 Software Configuration Control

All CMA laboratory/monitoring group software will be maintained under configuration control. The CMA laboratory/monitoring group shall describe in the LQCP the policies and procedures for the development of internal software applications. Software

Table 13-1. Configuration Control Parameters for Selected Monitoring Equipment

Equipment	Configuration Control Parameters
DAAMS Tube	Sorbent type, mesh size, sorbent bed depth, flow rate, aspiration time
Bubblers/Impinger	Bubbler/impinger solution concentration, volume, internal standard, flow rate, aspiration time
AgF Conversion Pad	Materials of construction, conversion/transfer efficiency: ≥75 percent, AgF pad storage to minimize photo-degradation and moisture loading
NO_x Prefilter	Materials of construction
Sample Line	Length, diameter, location of distal end, heat-trace requirements to include amps per foot, filters, flow rate, materials of construction
Dynatherm ACEM 900, ACEM 980, Multiple Tube Desorption Unit	Software, flow rates
L-Derivatization Module	Derivatizing concentration and type

Table 13-2. Configuration Control Parameters for NRT Instruments

Parameter Type	Specific Parameter
Timing	Column T1, Column T2, Cycle, Purge, Sample, Zero, Inject, Desorb
Flow Rates	Hydrogen, Nitrogen, Air, and Sample
Error Limits	Hydrogen, Nitrogen, Air, Sample, PMT Voltage, Valve Temperature, PCT Low Temperature, PCT High Temperature, Column Low Temperature 1, Column Low Temperature 2, Column High Temperature 1, Column High Temperature 2
Temperature	Flame, Ambient, FPD, Valve, PCT Low, PCT High, Column Low Temperature 1, Column Low Temperature 2, Column High Temperature 1, Column High Temperature 2
Gas Type	Hydrogen, Nitrogen, Sample, Air, Helium
Peak Parameters[a]	Agent Gate Width
PCT	Sorbent type, mesh size, sorbent bed depth
Column	Phase, internal diameter, length, film thickness
Detector	Type; optical filter

Note:

[a] The calibration height will vary, depending on NRT performance, agent mass, and PMT voltage and should be tracked.

Table 13-3. NRT Instrument Method Operational Specifications

ACAMS/MINICAMS® Parameter	Minimal Criteria
PMT Voltage	<1,000 volts for ACAMS and 1,250 volts for MINICAMS
Cycle Time	ACAMS/MINICAMS analysis time must satisfy DA Pam 385-61 monitoring requirement of 15 minutes or less analysis time.
Agent Gate	Bound a 2.0Z agent gate peak

Table 13-4. Parameters for GC and Detector Instruments

Parameter Type	Parameter
Pressure	Pressure program (if applicable), heartcut settings
Temperature	GC inlet, column, detector block, temperature program, Dynatherm temperatures
Mode	Phosphorus or sulfur (FPD only), selective ion monitoring or full scan (MSD only), ionization mode (MSD only), absorbance level (GC-AED)
Column	Phase, internal diameter, length, film thickness
Pre-column/Guard Column	Phase, internal diameter, length, film thickness
Flow Rate	Hydrogen, helium, air, nitrogen, Dynatherm flows[a]
Peak Parameters	Area reject, threshold, peak width, any additional integration functions used during the certification
Detector	Type; optical filter

Note:

[a] Gas quality/tolerances will be in accordance with manufacturer recommendations.

Table 13-5. GC and Detector System Performance Specifications

Instrument	Minimal Criteria
Retention Time	Agent peak retention times must be within the RTW.[a]
Challenge	Once calibrated, a QL shall demonstrate an FC within ±15 percent of the TC for non-GC/MSD methods (±30 percent of the TC for GC/MSD methods).

Note:

[a] Challenges shall be in the form of an initial calibration verification (ICV) or continuing calibration verification (CCV) sample. For Class II methods, a laboratory spike shall be used to challenge the system. U.S. Environmental Protection Agency (USEPA) method 8000B is recommended as an approach for determining the RTW and should be used in conjunction with method development.

developed by the CMA laboratory/monitoring group will include the following information in a software specification document:

- Name or identification code of the programmer
- Descriptions of the program
- Conditions of operation
- All calculations and algorithms
- Time, date, and description of appropriate program modifications.

New software packages and software developed by the CMA laboratory/monitoring group shall be validated/tested and documented by the CMA laboratory/monitoring group.

Configuration control will be maintained for the NRT monitor software. The CMA laboratory/monitoring group will establish written protocols for modifying NRT monitor programs, assigning review and approval authority, and identifying acceptance testing. However, if backward capability does not exist, the previous version of software shall be maintained for data reprocessing, review, etc.

Configuration control of the software used for integration systems and laboratory information systems must be maintained and documented by the CMA

laboratory/monitoring group. Changes in versions of software that affect the integration of GC data will require certification and approval by the applicable CMA project manager and CMA-Monitoring Office. Changes in software must have previous version capabilities, or a copy of the previous software must be maintained.

If an instrument is replaced with an instrument using a different data format, operating system, etc., then the laboratory/monitoring group shall maintain the capability to access the data for a minimum period of 3 years.

The Laboratory Information Management System (LIMS) and data control center will be designed and operated to prevent unauthorized access to computer records.

When LIMS raw data are collected, analyzed, processed, or maintained, laboratory/monitoring group management shall ensure that comprehensive testing of LIMS performance is conducted at least once every 24 months or more frequently as a result of software or hardware changes or modifications.

14. SAMPLE HANDLING AND STORAGE

14.1 Sample Traffic Procedures

The CMA laboratory/monitoring group will maintain SOPs to accurately record the possession, COC, and handling procedures for each sample from collection through disposal.

14.2 Sample Collection

Individuals collecting samples shall follow published USEPA-approved procedures (*Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods*, [SW-846] most current version) and site-specific procedures, as approved by state or local agencies. The individual collecting the sample shall initiate the transfer of possession and sign the sample collection record. The following types of information, as a minimum, shall be recorded on the sample collection record: (Some items may not be applicable to all types of sampling procedures.) (1) unique sample ID, (2) date and time, (3) start and stop flow rates or solid/liquid collection times, (4) start and stop times of the sampling period, (5) weight (gram [g]) or volume (milliliter [mL]) (Weight may be determined by personnel performing the analysis.), (6) collection location, (7) operator's name or unique ID number, (8) agent and monitoring level, (9) sample type, and (10) preservatives. Deviations from SOPs, security, and unusual environmental conditions shall be included in the sample collection record.

14.3 Sample Identification

A unique sample ID number will be affixed to the sample or sample container. Permanent ink must be used to prepare labels to ensure full protection and continued legibility of recorded data.

14.4 Receipt of Samples at the Laboratory

CMA laboratory/monitoring group sample recipients will be trained in the receipt, handling, protection, and retention of samples. The sample recipient shall ensure that all samples are accounted for, undamaged, and properly labeled; damaged samples shall be documented by the CMA laboratory/monitoring group.

The CMA laboratory/monitoring group will be responsible for determining the sample analysis schedule based on the priority of samples. The CMA laboratory shall maintain records on the identification of the analyst and the instrumentation used to analyze a given sample. DAAMS samples shall be analyzed within 72 hours of sample completion. Waste screening samples shall be analyzed in accordance with their specific holding time requirements. Samples used for the confirmation of agent shall have priority over all other samples.

14.5 Sample Storage

The laboratory/monitoring group shall be responsible for sample storage to ensure that all environment, security, and holding time requirements are met.

14.6 Offsite Shipment

Samples collected for offsite shipment and analysis will be handled in accordance with AR 50-6 and DA Pam 385-61.

15. DOCUMENTATION

The focus of documentation is to develop and implement effective processes (documented in procedures, plans, and methods) for the QMS that will generate records, providing evidence that an activity has been accomplished or that a requirement has been achieved.

Processes must be documented in the form of procedures, methods, and plans. Review and approval (including changes) are required to ensure that documents are adequate prior to issue for use. Therefore, quality documents must be controlled to ensure that the correct and latest requirements are available to personnel.

Obsolete documents retained for legal and/or historical purposes shall be suitably marked.

15.1 Site Plans

Site plans will be submitted to the CMA-Monitoring Office for review.

All activities shall, at a minimum, have the following plans/procedures that apply to site-specific situations:

- LQCP
- Site Monitoring Plan (SMP) or Laboratory Analysis and Monitoring Plan (LAMP)
- SOPs/IOPs/LOPs (laboratory and monitoring procedures) and method descriptions
- Training Plans
- Chemical Hygiene Plan (CHP)
- Waste Analysis Plan
- CEMS QC Plan.

15.2 QC Reports

The QC report shall include data, statistical reports, and corrective action reports. The CMA laboratory/monitoring group shall provide electronic QP data to the CMA-Monitoring Office from the initiation of the baseline survey through closure (see section 16). The electronic QP data will be transmitted to the CMA-Monitoring Office every two weeks.

15.3 Records

The CMA laboratory/monitoring group shall maintain a record system that is defined in the LQCP to suit its particular site-specific circumstances; satisfy Federal, State, and local environmental regulators' requirements; and comply with requirements promulgated by the LMQAP. The LQCP shall establish procedures for identification, collection, indexing, filing, storage, maintenance, and disposition of quality records. Records shall be a part of the 40-year storage requirement (29 CFR 1910.1020, OSHA, Department of Labor, 1 July 2001) and shall include the identity of personnel involved in sampling, preparation, calibration, QC challenges, and maintenance of laboratory/monitoring group instrumentation. All records shall be held secure and confidential to CMA. All records shall be legible and shall be stored and retained in such a way that they are readily retrievable in facilities that provide a suitable environment to prevent damage or deterioration and to prevent loss. Record storage locations shall be identified in the LQCP. Records are used to provide information on the condition (conformity or nonconformity) of a process or product. The individual who performs the activity is responsible for documenting it. Any occurrences or conditions that may affect the results of the measurements must also be documented.

The CMA laboratory/monitoring group shall maintain records for the purchase, receipt, and storage of consumable materials used for technical operations.

15.4 Data Storage

A high priority should be placed on storing as much of the required information as possible in computer databases. Data stored in computer databases must be backed up and archived according to requirements established in the LQCP. The CMA laboratory/monitoring group shall maintain the data storage in accordance with the 40-year requirement. CMA laboratories shall also include software used to access data and perform calculations for data storage to readily retrieve archived data.

15.5 SOPs

Prior to the start of analytical and monitoring operations at a site, the CMA laboratory or monitoring teams will develop SOPs. The SOPs, as approved by the CMA Site Project Manager or other approving manager, will provide the official method for performing certain routine or repetitive tasks. The CMA laboratory/monitoring group will verify and document that operators are familiar with the SOPs and that the operators can effectively perform the tasks described.

15.6 Quality System Plans

The LQCP shall address the following items, or as a minimum, state where the information is located in other support activity documents (SMP, SOPs, and IOPs, etc.):

a. *Development of Subordinate Plans.* The information required by the following paragraphs may be contained in any number of documents and may be referenced when applicable. Each requirement does not require its own plan.

(1) Plan for ensuring that all new sample collection and analysis work is reviewed to verify that the proper resources and facilities are available prior to commencing the work

(2) Plan for requesting and approving departures from approved SOPs

(3) Plan for feedback and corrective action whenever testing discrepancies are detected or departures from documented policies or procedures occur

(4) Reference to contingency procedures and CMA laboratory/monitoring group limiting conditions of operation (LCOs)

(5) Reference to procedures for generating and evaluating QL/QP data

(6) Plan for achieving traceability of measurements

(7) Reference to calibration, verification, and/or test procedures used

(8) CHP in accordance with CFR, 29 CFR 1910.1450

(9) Plan for protecting security of analytical results

(10) Training plan and procedures for maintaining records of the relevant qualifications, training, skills, and experience of the technical personnel

(11) Plan for calibration, verification, and maintenance of equipment

(12) Data management plan for QA/QC procedures, including software verification

(13) Plan for review, control, transfer, and release of analytical and monitoring data.

b. *Certification Criteria.*

(1) Verification practices including the CASARM PTP use of standard reference materials and internal QC schemes

(2) Plan and criteria for certifying and recertifying agent and industrial chemical monitoring/analysis systems in baseline studies

(3) Plan and criteria for certifying agent and industrial chemical monitoring systems not included in baseline studies

(4) Plan and criteria for certifying and recertifying personnel operating monitoring/analysis systems

(5) Plan for configuration control of the chemical materiel monitoring/analysis systems.

c. *Auditing and Performance Evaluation.*

(1) Procedures for addressing CMA, CMA project managers, state, and DHHS concerns on CMA laboratory/monitoring group performance

(2) Plan for auditing and reviewing CMA laboratory/monitoring group functions, including procedures for maintaining audit files (reports and corrective actions)

(3) Plan for capturing lessons learned about monitoring systems

(4) Plan and criteria for internal and external audits

(5) Identification of major equipment and reference measurement standards.

d. *Regulatory Requirements.*

(1) Additional and/or unique requirements established by state and federal law/regulation and/or the site environmental permits

(2) Scope of sample collection and analysis established by state and federal law/regulation and/or the site environmental permits.

15.7 Confirmed Responses Report

The CMA laboratory/monitoring group is required to generate a report describing all confirmed sample analysis results greater than the monitoring level. The following information is provided as a minimum:

- Identification of the chemical materiel
- Sample ID Number\Sample Station
- Found concentration, in mg/m^3
- Associated NRT reading (if applicable)
- Date, time, and location of reading or measurement
- QC sample results supporting the analytical results
- Statement on the quality of monitoring data and printouts of the actual data
- Any chemical materiel readings at any relevant stations at the site, destruction facility, or storage facility
- Description of site operations during the sample aspiration period
- A statement of the potential chemical compound's source
- Explanation of response or operator comments
- Name/unique ID number of operator collecting and analyzing the sample
- Analytical method used for analysis
- Analytical instrument ID.

15.8 Sample Analysis Report

Results of CMA laboratory sample analysis shall be reported accurately, clearly, and objectively. Each report shall be provided to CMA-Monitoring Office upon request and shall include at least the following information:

- Title
- Project name and site address
- Unique ID of the report
- Report recipient name and address
- Description of each sample analyzed
- Characterization and condition of sample
- Date of sample collection and analysis
- Identification of or reference to the analytical method used
- Identification of or reference to the sampling method used
- Identifications of deviations from approved methods and other information relevant to the sample
- Analytical results supported by tables, charts, sketches or photos
- QL and QP recoveries and other appropriate QC samples for the sample collection period
- Signature and title of persons accepting responsibility for report contents
- Results identified as performed by outside labs or vendors
- COC.

Amendments to a report after issuance shall be made only in the form of a further document, including the statement identifying the amendment as a supplement to the report and identifying the specific title and serial number of the report being amended.

15.9 Corrective Action Report

Failing to meet the performance standards for continuing baseline studies will require corrective action until the problem is resolved. The problem and corrective action shall

be documented in a corrective action report, which shall be submitted to the CMA-Monitoring Office within 7 days after the end of the reporting period. A corrective action report shall include as a minimum:

- Problem to be resolved
- Initial action performed
- Planned actions performed if problem remains unresolved
- Actions that may be taken to prevent reoccurrence of the problem.

16. STATISTICAL VALIDATION REPORTS

Statistical validation reports shall include QC data, statistical analysis, and corrective actions. The CMA laboratory/monitoring group shall submit the QC data to the CMA mandated statistical program from initial baseline through closure. The CMA mandated statistical program is a Web-based program and can be accessed with an appropriate account at https://homeqa.pmcd.army.mil/qcdrs/.

16.1 QA/QC Data Statistical Reports

Monthly out-of-control QL data shall be tracked by GC column type as an internal QC report and transmitted to CMA-Monitoring Office upon request.

Some statistical parameters (for example, inaccuracy, standard deviation [SD], etc.), other than those identified in section 10, do not have acceptance criteria and are for troubleshooting purposes only.

The Web-based CMA mandated statistical program will be used to generate biweekly reports for submission to the CMA-Monitoring Office for initial and continuing baseline studies in accordance with requirements in paragraphs 10.4.1 and 10.4.2. Baseline recertification data is not required to be submitted to the statistical program with the exception of common stack data. The CMA laboratory/monitoring group is responsible for using the mandated software to produce statistically valid and defensible reports.

Each project site will capture all data and track all assignable causes for internal QA purposes. Assignable cause and diagnostic QP data will be entered into the program as flagged entries and will not be used for statistical calculations. All remaining QP challenges are part of the statistical calculations.

16.2 Statistical Analysis of Calibration Curve Data

Calibration curve data for the analytical equipment used in the sample measurement process shall be analyzed statistically as required by table 11-1. If the CMA laboratory/monitoring group has defined additional calibration requirements for an instrument, acceptance criteria for the additional requirements shall be defined.
The chemical materiel raw calibration data shall be generated from CMA laboratory/monitoring group-specified calibration curves. Algorithms used for calibration curves shall satisfy the calibration requirements specified for each analytical method.

16.3 Data Validation and Qualification

The rigorous QC requirements identified throughout this plan are intended to ensure that data validation efforts are built into the overall quality process and include validation vehicles such as participation in a PTP, multiple QC samples, and internal and external audits. To ensure laboratory and monitoring data are used effectively, data qualification of laboratory and monitoring data shall be performed when data integrity has been or is suspected to have been compromised. Compromised data, meaning data captured did not satisfy applicable data quality requirements for various reasons (equipment failure, poor recoveries, disconnected sample lines, etc.) must be qualified appropriately and corrective actions shall be implemented to ensure that future data have probability of satisfying applicable data quality requirements.

The CMA laboratory/monitoring group shall, as a minimum, define and document their qualification procedure. The qualification procedure shall identify:

- Personnel/organization responsible to perform data qualification
- Procedure for qualifying laboratory and monitoring sample data that are not bracketed by acceptable CCVs
- Procedure for qualifying laboratory/monitoring sample data that do not have acceptable QP recoveries
- Procedure for qualifying NRT data back to the last successful challenge

- Procedure for qualifying NRT data that were generated during NRT equipment failure or inappropriate configuration/setup
- Reporting procedures for compromised data.

16.4 Sample Statistical Report

Statistical software programs are used to aid in evaluating the performance of agent monitoring systems. The following are examples of statistical programs:

a. *Inaccmo.pgm Program.* The *inaccmo.pgm* program calculates performance parameters for each NRT monitor using first challenge data. These performance parameters are calculated using the CMA mandated statistical program, defined in this section.

b. *Acomp.pgm Program.* The *acomp.pgm* program generates a composite plot of all QP challenges for all NRT monitors.

c. *Staplot.pgm Program.* The *staplot.pgm* program generates simple performance plots of all challenge events and indicates the corrective action taken for a given NRT monitor station.

d. *Perform.pgm Program.* The *perform.pgm* program tabulates the pass rates by station and by the QP challenger's ID code.

16.5 Statistical Calculations

Evaluation of performance is performed using statistical software programs. Calculations performed, such as those performed by the *inaccmo.pgm*, should incorporate classical statistical calculations for the mean ($\overline{X}$) and standard deviation (SD).

16.5.1 SD and Mean. Classical statistics will be used to calculate the SD and mean using the following equations:

$$SD = \sqrt{\frac{\Sigma (X_i - \overline{X})^2}{(n - 1)}}$$

where

Σ	=	summation of the numbers
X_i	=	first-challenge result
$\overline{X}$	=	average first-challenge response
n	=	total number of first challenges
S	=	standard deviation for 1.0Z challenges.

Equation 16-1. Standard Deviation

$$\overline{X} = \frac{\Sigma X_i}{n}$$

where

Σ	=	summation of the numbers
X_i	=	first-challenge result
n	=	total number of first challenges
$\overline{X}$	=	average monitor response to 1.0Z challenges.

Equation 16-2. Mean

16.5.2 Percent Coefficient of Variation (CV). The CV is a relative value or expression of the precision of a data set. It can be used to compare various data sets.

$$CV\ \% = \frac{SD}{\overline{X}}(100\%)$$

where

SD = standard deviation of the 1.0Z first challenges

$\overline{X}$ = average percent recovery to 1.0Z first challenges

CV % = percent coefficient of variation.

Equation 16-3. Percent CV

16.5.3 Relative Standard Deviation (RSD). The RSD is calculated by the following equation:

$$RSD = \frac{SD}{\overline{X}}$$

where

SD = standard deviation

$\overline{X}$ = mean of measurements.

Equation 16-4. RSD

16.5.4 Inaccuracy. Inaccuracy is a measure of the absolute value of the difference between the average response and a perfect response (1.0Z) plus the SD.

$$\text{Inaccuracy} = |1.0 - \overline{X}| + SD$$

where

$\overline{X}$ = average response to 1.0Z first challenges

SD = standard deviation of the 1.0Z first challenges.

Equation 16-5. Inaccuracy

16.5.5 Pass Rate for Class I Methods. The pass rate is used to determine the historical/confirmation performance standard for Class I Methods.

$$\text{Pass Rate} = [P / (n)] (100)$$

where

P = number of challenges that have a recovery within ±40 percent of the TC

n = total number of challenges.

Equation 16-6. Pass Rate for Class I Methods

16.5.6 Pass Rate for Class II/Class III Methods. The pass rate is used to determine the historical/confirmation performance standard for Class II/Class III Methods.

$$\text{Pass Rate} = [P / (n)] (100)$$

where

P = number of challenges that provide a positive response

n = total number of challenges.

Equation 16-7. Pass Rate for Class II/Class III Methods

16.5.7 First-Challenge Pass Rate. The first-challenge pass rate (PR_1) is the percent of challenges that pass on the first attempt for each day or challenge cycle or event.

$$PR_1 = \frac{P_1}{(P_1 + F_1)}(100)$$

where

P_1 = number of challenges that passed on the first challenge

F_1 = number of challenges that failed on the first challenge

PR_1 = first-challenge pass rate.

Equation 16-8. First-Challenge Pass Rate

16.5.8 Second-Challenge Pass Rate. The second-challenge pass rate (PR_2) is the percentage of challenges that pass on the second-challenge attempt for each day.

$$PR_2 = \frac{P_2}{(P_2 + F_2)}(100)$$

where

P_2 = number of challenges that passed on the second challenge

F_2 = number of challenges that failed on the second challenge

PR_2 = second-challenge pass rate.

Equation 16-9. Second-Challenge Pass Rate

16.5.9 Either-Challenge Pass Rate. The either-challenge pass rate (PR_T) is the total percent of challenges that pass. It combines the first- and second-challenge passes and divides it by the total number of challenge events.

$$PR_T = \frac{(P_1 + P_2)}{(P_1 + P_2 + F_2)}(100)$$

where

P_1 = number of challenges that passed on the first challenge

F_2 = number of challenges that failed on the second challenge

P_2 = number of challenges that passed on the second challenge

PR_T = either-challenge pass rate.

Equation 16-10. Either-Challenge Pass Rate

16.5.10 Statistical Response Rate at the Alarm Level. The statistical response rate at the alarm level (SRR_{AL}) is the probability that a 1.0Z first challenge to the NRT monitor will generate a response greater than or equal to the alarm level.

$$SRR_{AL} = P\,(Y \geq AL) = P\,(Y \geq \frac{AL - \overline{X}}{SD})$$

where

$P(Y \geq AL)$	=	the probability that the normally distributed random variable Y (as percent recovery) is greater than or equal to the AL
AL	=	alarm level
$\overline{X}$	=	average response to 1.0Z first challenges
SD	=	standard deviation of the 1.0Z first challenges.

Equation 16-11. Response Rate at Alarm Level

16.5.11 Observed Response Rate at the Alarm Level. The observed response rate at the alarm level (ORR_{AL}) is the percent of samples at 1.0Z first challenge with a recovery reading equal to or greater than the alarm level.

$$ORR_{AL} = \frac{(n - n_{AL})}{n}(100\%)$$

where

n_{AL}	=	total number of occurrences of first challenges, at 1.0Z, with recovery less than the alarm level
n	=	total number of 1.0Z first challenges
ORR_{AL}	=	observed response rate at the alarm level.

Equation 16-12. Observed Response Rate at the Alarm Level

16.5.12 Percent Recovery. The percent recovery is calculated for every QP and QL sample that is analyzed by the CMA laboratory/monitoring group. The purpose of calculating percent recovery is to identify any bias in the sample measurement process that would cause a sample to become contaminated or lose analyte prior to analysis.

$$\%C = \left(\frac{\text{Found Concentration}}{\text{Target Concentration}}\right)(100)$$

Equation 16-13. Percent Recovery

16.5.13 Statistical Response Rate at the Reportable Limit. The statistical response rate at the reportable limit (SRR_{RL}) is the probability that a 1.0Z QP challenge will generate a response greater than or equal to the reportable limit.

$$SRR_{RL} = P\,(Y \geq RL) = P\,(Y \geq \frac{RL - \overline{X}}{SD})$$

where

$P(Y \geq RL)$	=	the probability that the normally distributed random variable Y (as percent recovery) is greater than or equal to the RL
RL	=	reportable limit
$\overline{X}$	=	average response to 1.0Z first challenges
SD	=	standard deviation of the 1.0Z first challenges.

Equation 16-14. Response Rate at the Reportable Limit

17. OUTSIDE SUPPORT SERVICES AND PURCHASING

Where outside services are used to assist a CMA laboratory/monitoring group, the SC or designee shall ensure that the service organization complies with the appropriate requirements of this document, the LQCP, and all applicable environmental regulatory agencies. In order to ensure requirements are enforced, all contracts/purchase orders shall identify the QC requirements and should satisfy the procurement and validation requirements. The CMA laboratory/monitoring group shall have policies and procedures for the selection, purchase, reception, and storage of consumable materials relevant to the quality of the laboratory and/or monitoring results. Supplies and consumables used shall comply with specified requirements. The CMA laboratory/monitoring group shall review and approve purchasing documents for technical content prior to release. The laboratory/monitoring group shall ensure that purchased supplies and consumable materials that affect the quality of laboratory and/or monitoring results are not used until they have been inspected or otherwise verified as complying with standard specifications or requirements as defined in the LQCP. The CMA laboratory/monitoring group shall maintain records of all support services utilized during laboratory/monitoring group operations and shall maintain accountability of its vendors and procurement system.

APPENDIX A

ACRONYMS/ABBREVIATIONS

APPENDIX A
ACRONYMS/ABBREVIATIONS

ABB	Asea Brown Boveri, Inc.
AC	hydrogen cyanide
ACAMS	Automatic Continuous Air Monitoring System
ACEM	Automatic Continuous Emissions Monitor
ACGIH	American Conference of Governmental Industrial Hygienists
AEC	U.S. Army Environmental Center
AEL	airborne exposure limit
AgF	silver fluoride
AMC	U.S. Army Materiel Command
ANSI	American National Standards Institute
AR	Army Regulation
ASC	allowable stack concentration; also known as the source emission limit (SEL)
ASQC	American Society for Quality Control
BFB	bromofluorobenzene
BZ	3-quinuclidinyl benzilate
CAIS	chemical agent identification set
CASARM	Chemical Agent Standard Analytical Reference Material
CBDCOM	U.S. Army Chemical and Biological Defense Command
CCV	continuing calibration verification
CDC	Centers for Disease Control and Prevention
CEMS	Continuous Emission Monitoring System
CFR	Code of Federal Regulations
CHP	Chemical Hygiene Plan
CI	chemical ionization
CK	cyanogen chloride

CMA	U.S. Army Chemical Materials Agency
COC	chain of custody
CQAT	Chemical Quality Assurance Team
CRDEC	Chemical Research, Development and Engineering Center (now ECBC)
CSDP	Chemical Stockpile Disposal Project
CSM	chemical surety materiel
CV	coefficient of variation
CVAA	2-chlorovinylarsonous acid
CWM	chemical warfare materiel
DA	Department of the Army
DAAMS	Depot Area Air Monitoring System
DC	diphenylarsine
DF	methylphosphonic difluoride
DHHS	Department of Health and Human Services
DM	adamsite
DoD	Department of Defense
DOL	Department of Labor
ECBC	Edgewood Chemical Biological Center
EI	electron ionization
ERDEC	Edgewood Research, Development and Engineering Center
FC	found concentration
FM	Field Manual
FPD	flame photometric detector
FR	Federal Register
g	gram
g/mole	gram per mole
GA	tabun; ethyl N,N-dimethylphosphoroamidocyanidate

GB	sarin; isopropyl methylphosphonofluoridate
GC	gas chromatograph
GC-AED	gas chromatograph-atomic emission detector
GC-FPD	gas chromatograph-flame photometric detector
GC-MSD	gas chromatograph-mass selective detector
GC-MSD/FPD	gas chromatograph-mass selective detector/flame photometric detector
GD	soman; pinacolyl methyl phosphonofluoridate
GPL	general population limit
H	Levinstein mustard
HBESL	Health Based Environmental Screening Levels
HD	distilled mustard; bis(2-chloroethyl)sulfide
HF	hydrogen fluoride
Hg	mercury
HL	mustard-lewisite mixture
HN-1	nitrogen mustard; bis-(2-chloroethyl)ethylamine
HN-3	nitrogen mustard; tris(2-chloroethyl)amine
HT	a mixture of 60 percent HD and 40 percent T
ICV	initial calibration verification
ID	identification
IDLH	immediately dangerous to life and health
IOP	internal operating procedure
IPA	isopropyl alcohol
ISO	International Organization for Standardization
ITMS	ion trap mass spectrometry
JACADS	Johnston Atoll Chemical Agent Disposal System
L	lewisite; chlorovinyl dichloroarsine
L/mole	liters per mole

LAMP	Laboratory Analysis and Monitoring Plan
LCO	limiting condition of operation
LDRUG	Land Disposal Restrictions – Utah Group
LIMS	Laboratory Information Management System
LMQAP	Laboratory and Monitoring Quality Assurance Plan
LOP	Laboratory Operating Procedure
LOQ	limit of quantitation
LQCP	Laboratory Quality Control Plan
LQCPP	Laboratory Quality Control Plan and Procedures
m^3	cubic meter
MDL	method detection limit
µg/mL	microgram per milliliter
mg	milligram
mg/m^3	milligram per cubic meter
mg/mL	milligram per milliliter
mL	milliliter
mL/min	milliliter per minute
mm	millimeter
MSD	mass selective detector
MSS	matrix spike sample
MSSD	matrix spike sample duplicate
N/A	not applicable
ng	nanogram
ng/L	nanogram per liter
NIOSH	National Institute for Occupational Safety and Health
NO_x	nitrogen oxide
NRT	near real-time
NS	not specified
NSCM	non-stockpile chemical materiel

O&M	operations and maintenance
OJT	on-the-job training
ORNL	Oak Ridge National Laboratory
OSHA	Occupational Safety and Health Administration
P&A	precision and accuracy
Pam	pamphlet
PCT	preconcentrator tube
PD	phenyldichloroarsine
PDARS	process data acquisition and recording system
PEL	permissible exposure limit
PFDTD	perfluoro-5,8-dimethyl-3,6,9-trioxidodecane
PFPD	pulsed flame photometric detector
PFTBA	perfluorotributylamine
PL	public law
PMCD	Program Manager for Chemical Demilitarization
PMT	photomultiplier tube
PPE	personal protective equipment
ppm	parts per million
ppmv	parts per million volume
PQL	practical quantitation limit
PS	chloropicrin
psia	pounds per square inch absolute
PTP	Proficiency Testing Program
QA	quality assurance
QA/QC	quality assurance/quality control
QC	quality control
QL	quality laboratory
QL	O-(2-diisopropylaminoethyl) O'-ethyl methylphosphonite
QMS	quality management system
QP	quality plant

RCRA	Resource Conservation and Recovery Act
RDT&E	Research Development, Test, and Evaluation
RL	reportable limit
RMD	Risk Management Directorate
RPD	relative percent difference
RRF	relative response factor
RSD	relative standard deviation
RTAP	real-time analytical platform
RTW	retention time window
SA	arsine
SARM	standard analytical reference material
SBCCOM	U.S. Army Soldier and Biological Chemical Command
SC	Systems Contractor
SCF	standard cubic feet
SD	standard deviation
SEL	source emission limit; also known as the allowable stack concentration (ASC)
SMP	Site Monitoring Plan
SOP	Standing Operating Procedure
SPM	Site Project Manager
SRR_{AL}	statistical response rate at alarm level
SRR_{RL}	statistical response rate at the reportable limit
SSR	Shipment Status Report
STEL	short-term exposure limit
T	bis[2-(2-chloroethylthio)ethyl] ether
TAL	target action level
TC	target concentration
TM	Technical Manual
TOCDF	Tooele Chemical Agent Disposal Facility

TPA	triphenylarsine
TWA	time-weighted average
UCAR	Utah Chemical Agent Rule
UIFM	uncertainty in found mass
USACE	U.S. Army Corps of Engineers
USACHPPM	U.S. Army Center for Health Promotion and Preventive Medicine
USACMDA	U.S. Army Chemical Materiel Destruction Agency
USATHAMA	U.S. Army Toxic and Hazardous Materials Agency
USC	United States Code
USEPA	U.S. Environmental Protection Agency
VSL	vapor screening limit
VX	O-ethyl S-(2-diisopropylaminoethyl)methylphosphonothioate
WPL	worker population limit
Z	monitoring level

APPENDIX B
GLOSSARY OF TERMS

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GLOSSARY OF TERMS

ACCURACY: The agreement of a measurement with an accepted reference of true value. Accuracy is usually expressed in terms of percent recovery.

AIRBORNE EXPOSURE LIMITS: Allowable concentrations in the air for occupational and general population exposures.

ALLOWABLE STACK CONCENTRATION (ASC): A non-regulatory ceiling value that serves as a source emission limit (SEL) and not as a health standard. It is used for monitoring the furnace ducts and common stack.

ANALYTE: The substance to be detected and/or measured when performing the chemical analysis of a sample.

ASSIGNABLE CAUSE: Assignable cause is defined as a known reason for the occurrence, usually determined to be operator or systematic error that does not correctly indicate the performance of the instrument or monitoring system.

AUDIT: A planned and documented investigative evaluation of an item or process to determine its adequacy, effectiveness, and compliance with established procedures, instructions, drawings, quality control (QC) plans, and/or other applicable documents.

CALIBRATION: The process of determining response factors used to calculate absolute concentrations by injecting specially prepared calibration samples. Establishing a relationship between known concentrations of analyte and the detector response.

CASARM: Chemical Agent Standard Analytical Reference Material (CASARM) is a high purity certified chemical agent standard used by the U.S. Army Chemical Materials

Agency (CMA) laboratory/monitoring group to prepare stock agent solutions and subsequent working standards.

CEILING VALUE: The maximum exposure concentration at any time, for any duration. Practically, it may be an average value over the minimum time required to detect the specified concentration.

CHAIN OF CUSTODY (COC): An unbroken trail of accountability that ensures the physical security of samples, data, and records.

CHALLENGE: An injection of a known standard at a required monitoring level to validate that the instrument is still in control and that the calibration is valid.

CHEMICAL WARFARE MATERIEL (CWM): Equipment, munitions, devices, and containers designed for use directly in connection with the employment of chemical weapons or containerization of chemical agents or industrial chemicals. This term includes the chemical weapons stockpile; chemical weapons production facilities; binary weapons and components; buried, range recovered, or found chemical munitions, containers, or chemical agent identification sets (CAIS).

COLORIMETRIC TUBES: Small glass tubes filled with solid adsorbents, such as silica gel, activated alumina, or inert granules, and impregnated with detecting chemicals through which air is aspirated at a controlled rate. The detector chemical undergoes a color change in the presence of the contaminant; the contaminant concentration is proportional to the intensity of color change or the length of the stain within the colorimetric tube.

CONFIDENCE (OR SIGNIFICANCE) LEVEL: A value corresponding to the probability that a single future measurement will yield a particular result or fall within a particular interval of values.

CONTINUING CALIBRATION VERIFICATION (CCV): A quality laboratory (QL) sample analyzed at prescribed intervals throughout the entire analytical run and used to verify the continued accuracy of the instrument calibration and to monitor instrument drift and overall instrument performance.

CORRECTIVE ACTION: Any action taken to rectify adverse conditions, and where possible, to preclude their recurrence.

DATA VALIDATION: An analyte- and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance (that is, data verification) to determine the analytical quality of a specific data set as specified in Guidance on *Environmental Data Verification and Data Validation*, USEPA QA/G-8, November 2002.

DEMILITARIZATION: The mutilation, destruction, or neutralization of chemical materiel, rendering it harmless and ineffectual for military purposes.

DIAGNOSTIC QUALITY PLANT (QP) CHALLENGE: QP challenges performed as part of corrective actions to determine if a problem has been resolved and are not included in the baseline calculations.

DILUTE RESEARCH DEVELOPMENT, TEST, AND EVALUATION (RDT&E) STANDARDS: Solutions in concentrations and quantities not exceeding the levels defined in Army Regulation 50-6, Chapter 6, *Research Chemical Agents*.

DOUBLE BLIND QL: Spiked sample prepared by QC personnel that is placed with the analytical sequence at a location unknown to the analyst. This process ensures that the analyst does not know the location or the spike level during analysis.

DUPLICATE SAMPLES: Also known as replicate samples or split samples, duplicate samples are two aliquots taken from the same sample container and analyzed separately to test repeatability of an analysis.

FOUND CONCENTRATION (FC): Concentration of a standard analyte solution measured by a sampling and analysis method after a challenge with a known standard concentration (target concentration [TC]).

GENERAL POPULATION LIMIT (GPL): The maximum concentration to which the general population may be exposed 24 hours per day, 7 days a week, for a 70-year lifetime. Applies to the entire general population, including all ages and medical conditions.

HOLDING TIME: The maximum time allowable between sample collection and/or extraction and analysis. Some samples may have a time limit to be analyzed/extracted from the start time when the sample was collected. Some samples may have a time limit to be analyzed once they have been extracted.

IMMEDIATELY DANGEROUS TO LIFE AND HEALTH (IDLH):

a. A condition posing an immediate threat to life or health, or an immediate threat of severe exposure to contaminants likely to have adverse delayed effects on health. This condition includes atmospheres where oxygen content by volume is less than 19.5 percent.

b. The maximum concentration from which, in the event of a respirator failure, one could escape within 30 minutes without a respirator and without experiencing any escape-impairing (for example, severe eye irritation) or irreversible health effects.

IDLH levels have not been established for vesicants because workers are required to wear supplied air or self-contained breathing apparatus at vesicant concentrations much lower than IDLH levels. IDLH levels for industrial chemicals that may be encountered during non-stockpile operations are adopted from the National Institute for Occupational Safety and Health (NIOSH).

INDUSTRIAL COMPOUNDS: Chemicals developed or manufactured for use in industrial operations or research; these chemicals are not primarily manufactured for the specific purpose of producing human casualties or rendering equipment, facilities, or areas dangerous for use by man.

INITIAL CALIBRATION VERIFICATION (ICV): A QL sample that is analyzed immediately following instrument calibration and is used to verify the accuracy of the instrument calibration and to monitor instrument drift and overall instrument performance.

LABORATORY/MONITORING GROUP: Person or person(s) responsible for performing all environmental, analytical, and safety laboratory/monitoring activities at a given site. This group has the responsibility to collect, analyze, and document samples, preserve samples, prepare samples for offsite transportation, calibrate and challenge monitoring instruments, review sample analysis results, and report sample analysis results from laboratory/monitoring instruments to the Site Project Officer.

LABORATORY QUALITY CONTROL PLAN: A quality assurance/quality control (QA/QC) plan developed to implement the requirements of the Laboratory Monitoring and Quality Assurance Plan (LMQAP) in support of CMA laboratory and monitoring activities for each site, project, or operation.

MATRIX: The component or substrate that contains the analyte of interest.

MATRIX SPIKE SAMPLE (MSS): A sample that is spiked with the appropriate analyte prior to sample preparation and analysis.

MATRIX SPIKE SAMPLE DUPLICATE (MSSD): A second spiked sample that is spiked with the appropriate analyte prior to sample preparation and analysis.

METHOD: A set of procedures and techniques for systematically performing an activity (for example, sampling, chemical analysis, quantification). A method will encompass

certain parameters that, when changed significantly, may result in a new method. Methods shall be placed under configuration control and critical parameters shall identify tolerances that, when exceeded, will result in a “new” method.

METHOD BIAS: A systematic error inherent in a method or caused by some artifact or idiosyncrasy of the measurement system. Examples are temperature effects, extraction inefficiencies, contamination, mechanical losses, and calibration errors. Bias may be both positive and negative, and several types can exist concurrently so that net bias is all that can be evaluated, except under special conditions.

METHOD BLANK: Analyte-free water or soil, processed in the exact manner as the samples within a batch, using identical reagents and solvents.

METHOD DETECTION LIMIT (MDL): The MDL refers to waste methods only. The minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyzed concentration is greater than zero and is determined from analysis of a sample in a given waste matrix containing the analyte. The MDL is the lowest level at which an analyte may be reported using that method (source is 40 CFR, Part 136, Appendix B)

METHOD OR MEASUREMENT ACCURACY: The degree of agreement of a measured value with the true or expected value of the quantity of concern. Method accuracy depends on the lack of bias and imprecision of the method.

MOBILE STATION: A mobile station is comprised of near real-time (NRT) instruments and sampling points that change location in accordance with operational requirements to include real-time analytical platforms (RTAPs) and first entry monitoring.

MONITORING: The continued or periodic act of seeking to determine whether a chemical agent is present (Department of the Army [DA] Pamphlet [Pam] 385-61).

MONITORING LEVEL: The level to which monitoring is performed. Responses at or above the monitoring level indicate the monitoring level has been met or exceeded and corrective actions are required. For waste screening purposes, the monitoring level is the negotiated treatment value for a specific analyte within a specific matrix.

MONITORING PLAN: A detailed, site-specific plan that covers all laboratory and monitoring objectives and strategies for a given site. The plan describes methods and equipment used, locations, number and type of samples, safety requirements, transportation and shipping instructions, scheduling, and any other site-related monitoring requirements.

NEAR REAL-TIME (NRT) CONFIRMATION: Confirmation of the detection of agent at the required monitoring levels in the event of an NRT monitor chemical agent alarm.

NEAT CHEMICAL AGENT: An undiluted, full-strength (as manufactured) chemical agent or agent at concentrations above RDT&E dilute level. Chemical agent manufactured by the binary synthesis route will also be considered a neat agent, regardless of purity.

NRT MONITOR: A monitor that has the capability to automatically collect a sample, analyze the sample, and report/display the sample analysis results within 15 minutes or less.

PERMISSIBLE or PUBLISHED EXPOSURE LIMIT (PEL): The exposure inhalation or dermal PEL specified in 29 CFR Part 1910, Subparts G and Z.

pH: A measure for the activity of the hydrogen ions in a substance. The hydrogen ion activity determines the acidic, neutral, or alkaline character of a substance.

PRACTICAL QUANTITATION LIMIT (PQL): The lowest concentration that can be reliably determined with specified limits of precision and accuracy for a given analytical

method. The PQL is 10 times the method detection limit (MDL) as defined by USEPA SW-846. The PQL is applied to waste screening methods.

PRECISION: A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions, generally expressed in terms of the standard deviation.

PROFICIENCY TESTING PROGRAM (PTP): This program plan specifies the program requirements adopted by the CASARM Quality Assurance Team (CQAT) for conducting a PTP for chemical agent monitoring. The PTP describes actions and activities required to operate an effective proficiency test plan to support the Army's CASARM Quality Assurance Program. The PTP may be used to check the consistency and comparability of data for individual testing personnel, establish the effectiveness and comparability of test methods, achieve systemic improvement, and assist with the determination of reasons for interlaboratory differences.

PUBLISHED EXPOSURE LEVEL: The exposure limits published in *NIOSH Pocket Guide to Chemical Hazards*, dated 1994, incorporated by reference, or if none are specified, the exposure limits published in the standards specified by the American Conference of Governmental Industrial Hygienists in their publication, *Threshold Limit Values and Biological Exposure Indices*, dated 1994, incorporated by reference.

QUALITY: The totality of features and characteristics of a product or service that bear on its ability to meet the stated or implied needs and expectations of the user.

QUALITY ASSURANCE (QA): An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the customer.

QUALITY CONTROL (QC): The overall system of technical activities that measure the attributes and performance of a process, item, or service against defined standards to verify that it meets the stated requirements established by the customer.

QUALITY LABORATORY (QL) SAMPLE: A sample media that has been spiked with a solution of dilute chemical standard analytical reference material (SARM). The exact amount of SARM is recorded and documented with the sample identification. The purpose of the sample is to verify the in-control status of the laboratory instrument.

QUALITY PLANT (QP) SAMPLE: A sample media that has been spiked with a solution of dilute chemical SARM prior to being placed in the field or following aspiration of the blank tube in the field. The sample is spiked and then carried out to the sample collection point and exposed to the sample collection point atmosphere. The exact amount of SARM is recorded and documented with the sample identification (target concentration). Upon analysis in the laboratory, the QP's found mass must be within an acceptable tolerance. The purpose of the sample is to identify sources of sample contamination or sample degradation in the field at the sample collection location.

RANDOM ERROR: A component of total error that is not assignable to appropriate specific source and the magnitude of which can be predicted only in terms of probability.

REPORTABLE LIMIT (RL): A predetermined value for historical method, that when equaled or exceeded will be reported as chemical materiel that may have exceeded the monitoring level.

SAMPLING PLAN: A detailed, site-specific plan that covers all sampling objectives and strategies for a given site. The plan describes methods and equipment used, locations, number and type of samples, safety requirements, transportation and shipping instructions, scheduling, and any other site-related sampling requirements.

SHORT-TERM EXPOSURE LIMIT (STEL): The maximum concentration to which unprotected chemical workers may be exposed for up to 15 minutes continuously.

SOLID WASTE: Discarded material, including solid, liquid, semisolid, or contained gaseous material resulting from industrial, commercial, mining, and agricultural operations, and from community activities, but does not include solid or dissolved materials in irrigation return flows or industrial discharges that are point sources subject to permits under Section 402 of the Federal Water Pollution Control Act, as amended.

SOURCE EMISSION LIMIT (SEL): The SEL replaces the previously used allowable stack concentration (ASC). SELs are identified in section 7.

SPIKED QC SAMPLE: A separate aliquot of a sample that is spiked with a known and documented amount of reference material to check for matrix or sampling effects on percent recovery.

STANDARD: A known concentration of a known chemical that is used to perform quantitative analysis.

STANDING OPERATING PROCEDURE (SOP): A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps, and that is officially approved as the method for performing certain routine or repetitive tasks.

TARGET ACTION LEVEL (TAL): The highest target or true analyte concentration that can be distinguished as lower than the monitoring level 97.5 percent of the time using a test whose probability of a false positive response is 2.5 percent. The TAL is determined by an approved statistical software package and applies to air methods only.

TARGET CONCENTRATION (TC): The expected concentration based on 100 percent recovery.

TREATMENT LEVEL: A negotiated concentration for a specified contaminant in a specified extract or total waste that must be met by any method designed to physically or chemically change the nature of a hazardous waste.

UNCERTAINTY IN FOUND MASS (UIFM): In the regression of FC versus TC, this is one-half the difference between the upper and lower 95 percent prediction bounds of the TC when the TC is equal to the monitoring level.

WORKER POPULATION LIMIT (WPL): Maximum allowable 8-hour TWA concentration that an unmasked worker could be exposed to for an 8-hour workday and 40 hours per week for 30 years without adverse effect. WPL values are identified in section 2. WPL values are identified in section 7.

Z: Generic designation for the applicable monitoring level.

APPENDIX C
REFERENCES

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REFERENCES

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APPENDIX D
EQUATIONS FOR CALCULATING POSITIVE CHEMICAL MATERIEL RESPONSES

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D.1 Introduction

The U.S. Army Chemical Materials Agency (CMA) follows a strict protocol for reporting any chemical materiel response at or above the alarm setpoint. The following paragraphs include the calculations for converting alarm responses from detection units to reportable quantity units.

Responses greater than the site-specific negotiated levels will be reported in monitoring level units (fraction of Z). This value will be read from the near real-time (NRT) display or from the analytical instrument's recorded value. For monitors or methods that report the response as concentrations (for example, parts per million [ppm] or milligrams per cubic meter [mg/m^3]), the observed values will be converted to monitoring level units using the calculations described in the following paragraphs.

For stack monitoring, total milligrams of agent may be requested to be reported. If this is the case, the stack flow (as determined by a calibrated monitoring device) must be corrected to the same temperature and pressure conditions used in the calibration of the NRT flow-control device.

For example, the NRT mass flowmeters have been re-calibrated (from factory calibration) at Tooele Chemical Agent Disposal Facility (TOCDF) to read volume flow at "standard TOCDF conditions," namely, 24°C and 24.7 inches mercury (Hg), to allow direct determination of exposure levels. Therefore, allowable stack concentrations (ASCs) are expressed in mg/m^3 at these conditions. (1 ASC = 0.0003 mg/m^3 at standard TOCDF conditions)

To calculate the stack flow rate, the Panametrics unit, Model CEM 68 (used to measure the common stack flow rate at TOCDF), measures kSCF/min (wet gas) at "standard conditions"; that is, 68°F (20°C) and 14.707 pounds per square inch absolute (psia). The stack flow must be corrected to 24°C and 24.7 inches Hg, that is, standard TOCDF conditions, so that the identical volume basis for the NRT readings and the stack gas exist. This allows the operator to multiply the NRT concentration (mg/m^3) times the stack flow rate (m^3/min) times the sampling time (min) to calculate the agent emission (mg). (Note: 1m^3 = 35.315 cubic feet)

To convert from SCF/min at 68°F and 14.707 psia to standard TOCDF conditions:

$$(V_1 \text{ SCF/min}) \times \frac{(14.707 \text{ psia}) \times (273 + 24)^\circ\text{C} \times (2.03602'' \text{ Hg})}{(2.47 \text{ '' HG}) \times (273 + 20)^\circ\text{C} \times (1 \text{ psia})} = 1.2288 \times (V_1 \text{ SCF/min})$$

and

where $1.2288 \times (V_1 \text{ SCF/min}) = V_2 \text{ SCF/min}$

V_1 = the stack flow rate measured in SCF/min at "standard conditions" (20°C and 14.707 psia)

V_2 = the stack flow rate expressed in SCF/min at "standard TOCDF conditions (24°C and 24.7 inches Hg)."

The flow values reported on the process data acquisition and recording system (PDARS) are considered instantaneous rates. Because the Panametrics unit captures an instantaneous flow rate as it sweeps through its cycle (every 30 seconds), the five reported flow rates for an NRT measuring cycle (0, 30, 60, 90, 120 seconds) were averaged to obtain the flow rate for the sampling period. This results in a minor change to the previously reported stack flow rates for a given sampling period.

D.2 Conversion from Milligrams per Cubic Meter

The airborne hazard levels are given in mg/m^3. To convert from the observed concentration in mg/m^3 to monitoring level units, divide the observed concentration in mg/m^3 by the Z equivalency in mg/m^3:

$$\frac{\text{concentration observed } (mg/m^3)}{\text{concentration of 1.0Z } (mg/m^3)} = Z$$

For example, the sarin (GB) time-weighted average (TWA) = 1×10^{-4} mg/m^3 = 1.0Z, and the monitor indicated the concentration of chemical materiel present as 1×10^{-3} mg/m^3; the recorded value is 10 TWA. Actual reported values take into account the overall method performance for that particular method.

Because the Automatic Continuous Air Monitoring System (ACAMS) reports in units of Z, another example is:

$$Z \times \text{concentration of a 1.0Z } (mg/m^3) = \text{observed concentration } (mg/m^3)$$

To convert from mg/m^3 to ppm:

$$0.0001\ mg/m^3\ GB \times [24.1\ L/mole/140\ g/mole] \times 1\ m^3/1{,}000\ L \times$$
$$1\ g/1{,}000\ mg \times 10^6\ parts/1\ part = 1.7 \times 10^{-5}\ ppmv$$

The 24.1 liters per mole (L/mole) factor used in the equation is the mole volume of GB at 20°C and 1 atmosphere pressure, and 140 grams per mole (g/mole) is the molecular weight of GB.

D.3 Conversion from Parts Per Million

Certain chemical-specific monitors indicate airborne chemical concentrations in units of ppm based on volume. To convert from the observed concentration in ppm to monitoring level units, first convert the ppm to mg/m^3. Since the gas density is a function of temperature and pressure, this conversion must be performed at a specified temperature and pressure. The following conversion may be used, assuming reference parameters of 20°C (the common workplace temperature) and standard pressure:

concentration observed (ppm) = concentration (mg/m^3) × [mole volume (L/mole)/mole mass (g/mole) × 1 m^3/1,000 L × 1 g/1,000 mg × 10^6 parts/1 part]

where

g/mole = grams per mole

L/mole = liters per mole.

Once the concentration has been converted from ppm to mg/m^3, convert from Z equivalency, in mg/m^3, to the fraction of Z, as shown previously:

$$\frac{\text{concentration observed (mg/m}^3\text{)}}{\text{concentration of 1.0Z (mg/m}^3\text{)}} = Z$$

For example, for hydrogen cyanide (AC), the molar mass (also known as the gram molecular weight) is 27.03 g/mole, and the gaseous mole volume at 20°C is 24.1 L/mole. The monitoring level is the TWA, which is 5.0 mg/m^3. If the AC monitor reads 10 ppm, a 2.2 TWA concentration should be recorded.

$$10\ \text{ppm} \times \left[\frac{27.03\ \text{g/mole}}{24.1\ \text{L/mole}}\right] = 11.2\ \text{mg/m}^3$$

and

$$\frac{11.2\ \text{mg/m}^3}{5.0\ \text{mg/m}^3 = 2.2\ \text{TWA}}$$

For chloropicrin (PS), the molecular weight is 164.38 g/mole, and the TWA, or Z, is equivalent to 0.7 mg/m^3. A 10 ppm alarm for PS would indicate presence of PS at 97 TWA:

$$10\ \text{ppm} \times \left[\frac{164.38\ \text{g/mole}}{24.1\ \text{L/mole}}\right] = 68\ \text{mg/m}^3$$

and

$$\frac{68\ \text{mg/m}^3}{0.7\ \text{mg/m}^3} = 97\text{Z} = 97\ \text{TWA PS present}$$

D.4 Calculation of Predicted Found Mass on Depot Area Air Monitoring System (DAAMS) Tubes

The following calculations will be used to determine the predicted sorbent tube chemical materiel concentration. The equation starts with a known mass in nanograms and provides the found concentration in TWA, ASC, etc.

$$Z = N \times B \times C \times 1/F \times 1/T \times 1/M$$

where

N	=	known mass in nanograms
B	=	1,000 liters per 1 cubic meter
C	=	1 milligram per 1,000,000 nanograms
F	=	flow rate, in liters per minute

T = actual time sampled (in minutes) during which the collocated NRT gas chromatograph (GC) systems had response(s) greater than 0.2Z for NRT GC systems/sorbent tube station; actual time sampled (minutes) for DAAMS-only stations

M = mg/m^3 equivalency of Z (for GB M=0.0001 mg/m^3).

This equation becomes:

$$Z = \left[\frac{NBC}{FTM}\right]$$

Confirmed chemical materiel readings necessitate notification to the appropriate Field Office. If the Field Office concludes chemical materiel has been confirmed outside of engineering controls, the appropriate Field Office will adhere to approved notification protocol and requirements identified in the site-specific environmental permit(s).

If evidence is present that chemical warfare materiel (CWM) may be below alarm level/reportable limit (for example, presence of peak above the chromatographic baseline in the agent's retention time window, etc.), then a longer aspiration period may be used and shall be documented.

The following calculations can be used to calculate the predicted DAAMS found concentration, in monitoring level units, when an NRT alarm is encountered:

$$\text{Predicted found mass in nanograms} = (X) = F \times T \times B \times C \times M \times D$$

where

F = actual flow rate in liters per minute; lowest flow used

T = actual time sampled in minutes, during which the collocated NRT had a response

B = 1 cubic meter per 1,000 liters

C = 1,000,000 nanograms per milligram

M = average ACAMS reading over time (T)

X = predicted found mass, in nanograms

D = stack dilution ratio, if applicable.

Found concentration in monitoring level units $(Y) = \frac{X}{N}$

where

N = number of nanograms associated with one monitoring level

X = found mass in nanograms

Y = found concentration.

D.5 Statistical Response Rate at the Alarm Level

The statistical response rate at the alarm level (SRR_{AL}) is the probability that a 1.0Z first challenge to the NRT monitor will generate a response greater than or equal to the alarm level.

$$SRRAL = P(Y \geq AL) = P\left(Y \geq \frac{AL - \overline{X}}{SD}\right)$$

where

$P(Y \geq AL)$ = the probability that the normally distributed random variable Y (as percent recovery) is greater than or equal to the AL

AL = alarm level (alarm setpoint)

$\overline{X}$ = average response to 1.0Z first challenges

SD = standard deviation of the 1.0Z first challenges.

D.6 NRT Quantitative Confirmation Sample Conversion to mg/m^3 Detected

When an NRT alarm is encountered and a quantitative confirmation sample is collected to confirm the NRT alarm, the following equations can be used to convert the confirmation sample to mg/m^3 equivalents.

Step 1. Determine vapor concentration in nanograms per liter (ng/L) using the following equation:

$$FM \div (T \times F) = ng/L$$

where

FM	=	found mass detected by the confirmation sample (ng)
T	=	sample time period NRT was in alarm (minutes)
F	=	flow rate of confirmation sample (liters).

Step 2. Convert ng/L to mg/m^3 using the following equation:

$$ng/L \times (1 \div 1000) = mg/m^3$$

For example, a GB ACAMS monitoring at the short-term exposure limit (STEL) concentration (0.0001 mg/m^3) is in alarm for 10 minutes and a quantitative DAAMS confirmation sample is collocated and aspirating at 0.50 liters per minute. The DAAMS tube is pulled and analyzed in the laboratory and provides a found mass of 1 nanogram (ng). The DAAMS confirmation calculation would be as follows:

$$1ng \div (10\text{-minutes} \times 0.50 \text{ liters/min}) = 0.20 \text{ ng/L}$$

Converting ng/L to mg/m^3 is as follows:

$$0.20 \text{ ng/L} \times (1 \div 1{,}000) = 0.0002 \text{ mg/m}^3$$

Knowing that the GB STEL concentration is 0.0001 mg/m^3, the STEL portion detected by the confirmation DAAMS can be determined by dividing the detected vapor concentration in mg/m^3 by the STEL vapor concentration in mg/m^3, as follows:

$$0.0002 \text{ mg/m}^3 \div 0.0001 \text{ mg/m}^3 = 2 \times \text{the STEL concentration}$$

APPENDIX E

PRESSURE DROP CRITERIA

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PRESSURE DROP CRITERIA

Type of Tube	Distance From Bottom to Packing (mm)	Width of Packing (mm)	Highest Acceptable Pressure Drop (inches of Hg)
DAAMS - 6mm Chromasorb® 106	Packing should be equal distance from top and bottom	15	15.0
DAAMS - 6mm Tenax® TA	Packing should be equal distance from top and bottom	15	15.0
DAAMS - 8mm Chromasorb 106	Packing should be equal distance from top and bottom	30	8.0
DAAMS - 8mm Tenax TA	Packing should be equal distance from top and bottom	30	8.0
DAAMS - 15mm HayeSep® D	Packing should be equal distance from top and bottom	12.25	15.0
Transfer Chromasorb 106	72 ±1	15	4.5
Transfer Tenax TA	72 ±1	15	7.0