

ATTACHMENT 1-10 CHWSF QUALITY ASSURANCE PROGRAM PLAN

1.0 INTRODUCTION

- 1.0.1 The U.S. Army Dugway Proving Ground (DPG) originally submitted this Quality Assurance Program Plan (QAPP) in accordance with State of Utah (State) Solid and Hazardous Waste Control Board Consent Order (CO) Number 9505024. This CO required DPG to prepare and submit a QAPP prepared in accordance with the U.S. Environmental Protection Agency (EPA) Interim Guidelines for Preparing Quality Assurance Project Plans, EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846), and other appropriate and relevant guidance documents. Upon Utah Division of Solid and Hazardous Waste (DSHW) approval, appropriate QAPP information has been incorporated into the Waste Analysis Plan (WAP) of DPG’s hazardous waste storage permit (Permit).
- 1.0.2 This QAPP outlines the policies, requirements, procedures, and responsibilities established to support analysis of chemical agent-related wastes conducted at DPG. Chemical agents are summarized in Table 1. This document also provides specific quality assurance (QA) and quality control (QC) procedures necessary to generate data of acceptable quality and completeness.
- 1.0.3 The purpose of this section is to outline the QAPP policies regarding collection and analysis of chemical agent-related wastes conducted on-site at DPG. Section 1.1 discusses the purpose, as well as the outline and documentation sources, for the QAPP. Sections 1.2 and 1.3 contain DPG’s QAPP quality and ethics policies, respectively.

| Table 1 List of Chemical Agents | | |
|--|--------------------|---|
| Agent | Common Name | Chemical Name |
| CX | Phosgene Oxime | dichloroformoxime |
| GA | Tabun | ethyl N,N-dimethylphosphoramidocyanidate |
| GB | Sarin | isopropyl methylphosphonofluoridate |
| GD | Soman | pinacolyl methylphosphonofluoridate |
| GF | Cyclosarin | cyclohexyl methylphosphonofluoridate |
| H | Mustard | bis-(2-chloroethyl)sulfide |
| HD | Distilled Mustard | bis-(2-chloroethyl)sulfide |
| HL | Mustard/Lewisite | see components |
| HN1 | Nitrogen Mustard | bis-(2-chloroethyl)ethylamine |
| HN2 | Nitrogen Mustard | bis-(2-chloroethyl)methylamine |
| HN3 | Nitrogen Mustard | tris-(2-chloroethyl)amine |
| HT | Mustard/T | see components |
| L | Lewisite | 2-chlorovinyl dichloroarsine |
| T | O-Mustard | bis-[2(2-chloroethylthio)ethyl]ether |
| VX | | o-ethyl-S-(2-diisopropylaminoethyl) methylphosphonothiolate |

1.1 PURPOSE

- 1.1.1 The purpose of the QAPP is to ensure the quality and defensibility of chemical agent-related analytical data. As used in this QAPP, chemical agents include the following compounds listed in R315-2-11(e)(1):
- 1.1.2 The quality systems described in this QAPP have been developed to comply with local and national standards for environmental laboratories producing data for hazardous waste compliance.
- 1.1.3 This QAPP contains 13 sections.
- Section 1.0 is an introduction to the QAPP.
 - Section 2.0 describes the project organization and details the responsibilities of key project personnel.
 - Section 3.0 outlines required personnel qualifications and personnel training.
 - Section 4.0 describes the facilities and equipment used to generate chemical agent-related waste data.
 - Section 5.0 describes the required format, development, approval, and control of methods and other documents related to the QAPP.
 - Section 6.0 outlines the documentation and procedural requirements for sample collection.
 - Section 7.0 describes several general laboratory procedures including sample receiving, sample handling, and labware cleaning.
 - Section 8.0 discusses the calibration requirements for laboratory and field instrumentation.
 - Section 9.0 outlines laboratory QC including the project data quality objectives (DQOs), analytical method performance, method detection limits (MDL), and reporting limits (RLs).
 - Section 10.0 describes analytical data management including recording, reduction, reporting, review, and validation.
 - Section 11.0 identifies the methods for laboratory quality assessment including control charts and control limits, proficiency test samples, audits, and reviews.
 - Section 12.0 outlines the requirements for implementing and documenting corrective action procedures.
 - Section 13.0 defines the terms used in the QAPP.

1.2 QUALITY POLICY

- 1.2.1 DPG is committed to producing high quality analytical data that is technically and legally defensible. As part of DPG's commitment to high quality data, project management will ensure that employees and contractors have sufficient experience and training to perform QAPP-related duties and procedures. Sample collection, sample handling, instrument calibration, sample analysis, and related activities will be conducted and documented as described in this QAPP and related methods. Routine QA samples will be prepared, analyzed, and reviewed according to method-specific procedures, and at specified frequencies. Regular internal and external audits will be conducted and documented to assess compliance with the QAPP and methods. Corrective actions will be initiated and completed to address discrepancies or problems noted at any point in the process.

1.3 ETHICS AND CONFIDENTIALITY POLICY

- 1.3.1 Without exception, DPG requires honest and ethical behavior of its employees and contractors. Employees and contractors are required to fully and accurately represent all aspects of their QAPP-related activities. Personnel must never intentionally report dates, data, or times other than those actually observed. Personnel must never intentionally represent another individual's activities as his/her own or misrepresent any other aspect of the analytical process. Alterations, additions, and/or deletions to data, reports, and other documentation must be made according to scientifically acceptable standards as described in this QAPP. Employees and contractors are required to inform, in a timely manner, DPG or project management of any such unethical behavior observed of other employees.
- 1.3.2 In a similar manner, employees and contractors are required to protect the integrity and confidentiality of sample and data information. Except as permitted in writing, data are released only to the submitting party. Care should also be taken when transmitting data by facsimile or other electronic means. Sample information, results of analyses, and other proprietary and/or sensitive information must not be discussed with, or transmitted to, individuals outside DPG without DPG's authorization. Environmental, safety, or other concerns should be communicated within the chain of command at DPG. Likewise, auditors and other individuals visiting the facility are required to maintain the confidentiality of proprietary and/or sensitive information.

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

- 2.0.1 This section describes the requirements and responsibilities of specific personnel involved with the sampling and analysis of chemical agent-related waste. Section 2.1 describes the DPG and QAPP organizations. Section 2.2 outlines the QAPP-related responsibilities of key project personnel.

2.1 DPG ORGANIZATION

- 2.1.0 Two DPG organizations are jointly responsible for the establishment and implementation of the QAPP. They include the Dugway Environmental Programs (DEP) and the West Desert Test Center (WDTC) as diagramed in Figure 1.
- 2.1.1 Environmental Organization
- 2.1.1.1 DPG Environmental personnel coordinate and manage the environmental conservation, restoration and compliance projects at DPG. They work directly with the State of Utah Division of Solid and Hazardous Waste (DSHW) to ensure compliance with applicable hazardous waste permits and regulations. Environmental personnel and contractors are primarily responsible for sample collection and hazardous waste management as described in this QAPP and other permit documents.
- 2.1.2 Laboratory Organization
- 2.1.2.1 The Combined Chemical Test Facility (CCTF) provides analytical testing in support of DPG's chemical agent defense programs. CCTF personnel and contractors are primarily responsible for chemical testing and air monitoring described in this QAPP.

2.2 PERSONNEL RESPONSIBILITIES

2.2.0 The individuals listed below are responsible for conducting the various activities detailed in this QAPP as well as implementing the methods and operating procedures listed in Section 5.0.

2.2.1 Environmental Laboratory Supervisor

2.2.1.1 The Environmental Laboratory Supervisor (however designated by laboratory management) has the following responsibilities relative to the analysis of chemical agent-related waste:

- Read, understand, and direct the sampling, analysis, documentation, and QC activities described in this QAPP and the QAPP-related methods.
- Ensure that all data reported by the laboratory is of high quality as well as technically and legally defensible.
- Ensure that technical and support personnel have sufficient qualifications and training to perform their assigned functions.
- Promptly review and respond to QC deficiencies and complaints reported by QA/QC personnel, the Compliance Restoration Division (CRD), Resource Conservation and Recovery Act (RCRA) Coordinator, and/or other clients.

2.2.2 QA/QC Personnel

2.2.2.1 The QA/QC personnel have the following responsibilities relative to the analysis of chemical agent-related waste:

- Read, understand, and assess the QC activities described in this QAPP and the related methods.
- Review analytical data and reports to ensure compliance with this QAPP and the QAPP-related methods.
- Conduct annual internal audit of sampling and analysis activities to ensure compliance with this QAPP and the QAPP-related methods.
- Ensure performance of annual MDL studies for QAPP-related methods and analytes.
- Maintain records of ongoing personnel training for QAPP-related activities.
- Maintain a corrective action program to review and respond to QC deficiencies and complaints.

2.2.3 Sample Collection Personnel

2.2.3.1 Personnel collecting chemical agent-related waste samples have the following responsibilities:

- Read, understand, and follow the QC guidelines as described in this QAPP.
- Read, understand, and follow sample collection procedures as described in the QAPP-related sampling methods.
- Accurately and honestly record pertinent information and complete required documentation as described in the QAPP-related sampling methods.
- Promptly deliver samples to the CCTF for analysis.

2.2.4 Analytical Personnel

2.2.4.1 Analytical personnel include DPG and subcontractor chemists and technicians located in the CCTF, as well as MINICAMS[®] operators and other field analytical personnel. They have the following responsibilities relative to the analysis of chemical agent-related waste:

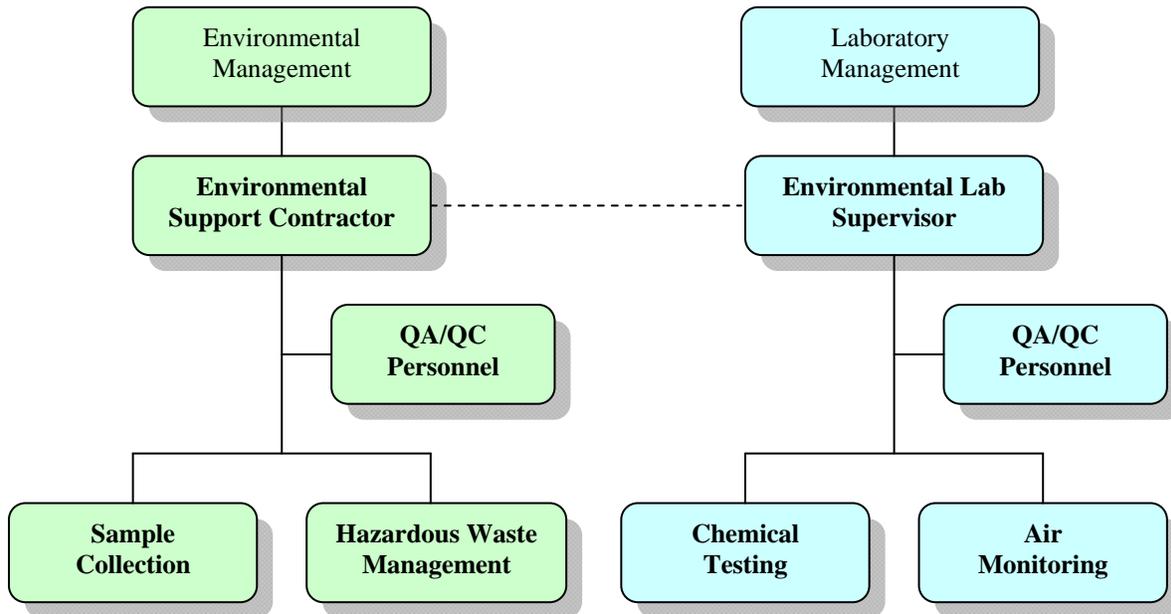
- Read, understand, and follow the QC guidelines as described in this QAPP.
- Read, understand, and follow the procedures as described in the QAPP-related analytical methods.
- Accurately and honestly record pertinent information and complete required documentation as described in the QAPP-related analytical methods.
- Ensure that analytical results are accurate, technically defensible, and meet the QC requirements as described in this QAPP and the QAPP-related analytical methods.
- Complete ongoing training as described in Section 3.0.
- Demonstrate training effectiveness by successful completion of method-required QC such as blanks, calibration verification standards, spikes, and spike duplicates.
- Maintain data quality and client confidentiality for all chemical agent-related results by following the reporting procedures as described in this QAPP and the QAPP-related analytical methods.
- Properly operate and regularly maintain laboratory analytical instrumentation and equipment.
- Report technical and quality problems immediately to QA/QC personnel or Environmental Laboratory Supervisor.

2.2.5 Support Personnel

2.2.5.1 Support personnel include sample custodians, documentation clerks, data package assembly personnel, and others. They have the following responsibilities relative to the analysis of chemical agent-related waste:

- Read, understand, and follow the QC guidelines as described in this QAPP.
- Read, understand, and follow the pertinent analytical procedures as described in the QAPP-related methods.
- Accurately and honestly record pertinent information and complete required documentation as described in the QAPP-related methods.
- Receive samples for analysis as described in the QAPP-related methods.
- Verify field and custody documentation, preservation and holding times as described in the QAPP-related methods.
- Collect and maintain sampling and analytical records as described in the QAPP-related methods.
- Prepare data packages for external validation as described in the QAPP-related methods.
- Maintain a document control system for this QAPP and the QAPP-related methods.
- Report technical and quality problems immediately to QA/QC personnel or Environmental Laboratory Supervisor.

Figure 1. Quality Assurance Program Plan Organization Chart.



3.0 PERSONNEL QUALIFICATIONS AND TRAINING

- 3.0.1 DPG recognizes that well trained and experienced personnel are the laboratory's most important resource. All personnel contributing to the quality of chemical agent-related waste data must have an adequate combination of education and experience to perform their required functions. Individuals who work from the guidance of this QAPP must be familiar with the general QAPP requirements as well as the applicable specific requirements detailed in the QAPP-related methods and procedures. Ongoing training and proficiency demonstration is also required of all personnel who implement the requirements of this QAPP.
- 3.0.2 This section outlines the requirements for personnel qualifications and training. Section 3.1 describes the technical qualifications and experience required of key QAPP personnel. Requirements for continuing education and training are outlined in Section 3.2.

3.1 PERSONNEL QUALIFICATIONS

- 3.1.1 Laboratory management is ultimately responsible for the quality and defensibility of analytical data produced in the laboratory. Laboratory management and the Environmental Laboratory Supervisor determine minimum qualifications for laboratory positions. Qualifications for key personnel can be documented using QAPP Qualifications Summary (Figure 2).
- 3.1.2 The administrative and documentation requirements of environmental analyses are often different from those of the military programs supported by the laboratory. For this reason, specific environmental laboratory experience is indispensable for QAPP personnel. In addition to specific requirements set forth by DPG management, QAPP personnel should have the following minimum qualifications:
- 3.1.3 Environmental Laboratory Supervisor
- 3.1.3.1 The Environmental Laboratory Supervisor should have a minimum of a bachelor's degree in the chemical, environmental, or biological sciences, with a minimum of 24 college semester credit hours in chemistry or equivalent to include familiarity with general statistics. This person should have at least two years of experience performing and at least two years supervising the analysis of environmental or similar samples.
- 3.1.4 QA/QC Personnel
- 3.1.4.1 QA/QC personnel should have sufficient education and experience to accomplish all required duties. A bachelor's degree in science or engineering, with a minimum of 24 college semester credit hours in chemistry or equivalent experience is recommended. These individuals should also be familiar with general statistics and demonstrate a working knowledge of environmental QC methods and procedures.
- 3.1.5 Sample Collection Personnel
- 3.1.5.1 Sample collection personnel performing environmental samples should have sufficient education and experience to accomplish all required duties. These individuals must have adequate experience in environmental sampling and demonstrate competence in that technology. These individuals should also be familiar with EPA/DSHW methods used to obtain representative samples of wastes.

3.1.6 Analytical Personnel

3.1.6.1 Analytical personnel performing environmental analyses should have sufficient education and experience to accomplish all required duties. For more complex analyses (e.g. GC/MS, GC-FPD etc.) a minimum of a bachelor's degree in the chemical, environmental, or biological sciences, with a minimum of 24 college semester credit hours in chemistry is recommended for laboratory chemists. Experience in environmental analyses can be substituted for education. For simple analysis (e.g. MINICAMS®) the analysts must have sufficient experience in the analysis of environmental samples and demonstrate competence in that technology. In addition to the DPG-required experience analyzing chemical agents, analytical chemists should demonstrate a working knowledge of environmental QC methods and procedures.

3.1.7 Other Technical Personnel

3.1.7.1 Where possible, sampling, analytical, and other technical personnel should have formal training in their area(s) of responsibility. Such training could come from in-house or outside sources.

3.2 PERSONNEL TRAINING

3.2.0 The training program will include initial and annual QAPP training, method-proficiency demonstrations, and other training as described below. Training documentation will be maintained, accessible, and up-to-date by the supervising organization. The DPG Civilian Personnel Office (CPO) and Environmental Support Contractors maintain pre-employment information for DPG employees. DPG will ensure that the laboratory staff is adequate to complete the analysis of waste in a timely manner, including cross-training where possible.

3.2.1 Initial and Annual QAPP Training

3.2.1.1 Employees and subcontractors involved in the collection, handling, analysis, and/or processing of chemical agent-related wastes will undergo initial QAPP training. This training will familiarize personnel with QAPP quality and ethics policies, analytical methods, documentation requirements, and other information contained in this QAPP and related methods. An understanding of the information contained in this QAPP will be demonstrated by successful completion of a written examination.

3.2.1.2 QAPP personnel are required to participate in refresher training on an annual or more frequent basis. Annual QAPP training will include a review of general QAPP concepts, methods status, and regulatory changes.

3.2.2 Method Proficiency Demonstration

3.2.2.1 In addition to possessing sufficient qualifications and experience as outlined in Section 3.1, personnel performing QAPP-related methods and procedures must demonstrate annual method-specific proficiency. In order to demonstrate proficiency for an analytical method or procedure, personnel must read and understand the method, perform the method under the direction of a qualified supervisor or mentor, and demonstrate the ability to consistently perform the activity within method required specifications. The supervisor or mentor may include a written test, blind audit samples, or other activities as part of the initial and/or ongoing proficiency assessment. Successful demonstration of method proficiency is approved by the supervisor or mentors and is documented in the training records (Figure 3).

3.2.3 Other Training

3.2.3.1 The DEP RCRA Coordinator encourages ongoing training and continuous improvement for QAPP personnel. Where necessary, formal instruction should be sought from outside sources, such as for instrumentation and/or software operation. Other sources of continuing instruction and education include in-house seminars and training sessions, technical subscriptions, and participation in professional organizations.

3.2.4 Training Documentation

3.2.4.1 Training documentation will be maintained for QAPP personnel. Training Documentation will include: Qualification Summary Form, Method Training Record, Method Proficiency Demonstration Record, written test results (when applicable), and relevant on-the-job training certificates. QA/QC Personnel will ensure that applicable QAPP training documentation is maintained and available for inspection. The DPG CPO in addition to Environmental Support Contractors maintains pre-employment information including employee education, background, previous experience, and copies of relevant certificates and degree(s) for DPG employees.

Figure 2. Example of QAPP Qualifications Summary.

**Dugway Quality Assurance Program Plan
 Personnel Qualifications Summary**

| | |
|-------------------------------|--|
| Name: | |
| Supervisor's Name & Employer: | |
| Comments: | |

Hazardous Waste Management-Related Duties

| | | |
|--|--|---|
| <input type="checkbox"/> Support Personnel | <input type="checkbox"/> Peer Review Personnel | <input type="checkbox"/> Other (specify) ¹ : |
| <input type="checkbox"/> Sample Collection Personnel | <input type="checkbox"/> QA/QC Personnel | |
| <input type="checkbox"/> Analytical Personnel | <input type="checkbox"/> Environmental Lab Supv. | |

¹Examples of "other" include: Laboratory supervisor, Laboratory manager

Relevant Education and Certifications

| Degree/Field | Year Earned | School/Location | Semester Credit Hours in Chemistry |
|--------------|-------------|-----------------|------------------------------------|
| | | | |
| | | | |
| | | | |

Relevant Experience

| Years | Employer | Title and Description |
|-------|----------|-----------------------|
| | | |
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| | | |

I attest that, to the best of my knowledge, the information given herein is true and complete

Signature: _____ Date: _____

Supervisor: _____ Date: _____

Figure 3. Example of QAPP Method Training Record.

QAPP Training
Method CL002R Chemical Agents by Gas Chromatography with Flame-Photometric and/or Mass Selective Detection
Method CL071R Dry Weight for Solids
 Date _____

| <i>Initial (I) Annual (A)</i> | <i>Last Name</i> | <i>First Name</i> | <i>Organization</i> | <i>Personnel Responsibilities</i> | <i>Signature</i> | <i>Demonstration of Knowledge & Performance (Supervisor's Initials & Date)</i> |
|-----------------------------------|------------------|-------------------|---------------------|---------------------------------------|------------------|--|
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|---|--|
| <p>The above listed personnel have read and attended training for methods CL002R and CL071R. They have been determined to be proficient to analyze waste samples by methods CL002R and CL071R.</p> <p>I attest to the best of my knowledge, the information given herein is complete.</p> <p>_____ Supervisor Name (print)</p> <p>_____ Instructor Name</p> | <p>Overview of training content:</p> <p>QAPP Training</p> <ul style="list-style-type: none"> • Quality & Ethics Policy • Analytical & Administrative Methods • Document Requirements • QAPP Overview • Method Status & Regulatory Overview <p>Method</p> <ul style="list-style-type: none"> • CL002R Chemical Agents by Gas Chromatography with Flame-Photometric and/or Mass Selective Detection • CL071R Dry Weight for Solids |
| <p>_____ Supervisor Signature</p> <p>_____ Instructor Name</p> | <p>_____ Initial</p> |

*By signing you attest that you have read and understood methods CL002R and CL071RR discussed in this training.

4.0 FACILITIES AND EQUIPMENT

4.0.1 Buildings 4153, 4156, and Building 4165 are located within the Combined Chemical Test Facility (CCTF). This modern facility supports the testing of protective clothing and masks, detectors, and decontamination systems using chemical agents and simulants as challenge materials. Testers determine agents, simulants, and other analytes in samples, which were collected in laboratory and chamber trials. In addition, the facility supports the analysis of environmental samples from DPG operations to ensure compliance with Federal, State, and local regulations.

4.1 LABORATORY FACILITIES

4.1.1 The CCTF laboratories (Buildings 4156 and 4165) have been specifically designed for the analysis of chemical agents. The laboratories are designed to provide a safe and comfortable working environment. Separate work areas are provided for labware cleaning, sample storage, sample preparation, sample analysis, sample disposal, records retention, and other laboratory activities. Offices are located around the perimeter of the buildings to provide easy access.

4.1.2 Buildings 4156 and 4165 provide a high degree of environmental control. Variables such as temperature, humidity, ventilation, and lighting can be controlled and monitored as necessary. Environmental conditions that are specified in a particular method are monitored and documented.

4.1.3 The laboratories are environment-friendly. A double-wall drain system and a 5,000-gallon holding tank contain contaminated water from any agent spill cleanup or from emergency shower use. The exhaust air from all laboratory areas (not only fume hoods) is charcoal-filtered before it is returned to the atmosphere. In addition, a demand-controlled variable-volume ventilation system minimizes the volume of air requiring heating or cooling. Heat-recovery coils downstream from the fume hood filter units recover energy from exhausted air. Motion sensors automatically turn off lights in unoccupied rooms. Thick wall and roof insulation and heat-reflector windows minimize heat transfer through the building shell.

4.2 LABORATORY EQUIPMENT

4.2.1 The CCTF is equipped to safely test, analyze, and process chemical agent-related wastes. Laboratory analytical capabilities include:

- Automated thermal desorption chromatography systems (analysis of organic compounds).
- Continuous vapor monitoring [MINICAMS[®], which employ gas chromatography (GC) for low-level vapor and infrared spectroscopy for high-level vapor].
- GC (flame ionization, flame photometric, photo ionization and thermal conductivity detectors).
- GC/mass spectrometry (MS).
- High-performance liquid chromatography.
- Infrared spectrometry.
- Spectrofluorometry.
- Ultra Violet (UV)-visible spectrophotometry.

- 4.2.2 Formal training and experience is required to operate most analytical equipment (see Section 3.2.3). Manuals and instructions for the operation of test equipment are maintained up-to-date in the laboratory.

4.3 EQUIPMENT MAINTENANCE

- 4.3.1 Routine equipment and instrument maintenance minimizes down time and prevents unexpected problems within the laboratory. Routine maintenance is performed on all laboratory instrumentation as required according to the manufacturer's recommendations. Where possible, manufacturer service contracts are maintained on major pieces of equipment. The DPG Calibrations Unit performs calibration of measurement equipment, such as balances and flow meters, at least annually. Routine maintenance for MINICAMS[®] is performed at least annually by trained service personnel.
- 4.3.2 Maintenance of all major laboratory and field instrumentation is recorded in instrument maintenance logbooks. Maintenance logbooks document all routine (change column, pump tubing, etc.) and non-routine (troubleshooting, instrument service, etc.) maintenance operations.

4.4 SAFETY

- 4.4.1 A sophisticated exhaust system, with redundant fans, controls, and alarms, provides the airflow in fume hoods used for all agent operations. The building's pressurization system keeps laboratory rooms at a lower air pressure than corridors, which in turn are kept at a lower pressure than the offices. Emergency generator capacity supports the fume hoods, ventilation system, egress lighting, and other essential equipment in the event of a power loss. Emergency showers and eyewashes are provided in the laboratories and corridors. Epoxy and stainless steel work surfaces and interior finishes are resistant to chemical agents.

4.5 SECURITY

- 4.5.1 A security fence surrounds the CCTF, restricting access to the facilities. Access to the CCTF is controlled by a security gate through which only authorized personnel or escorted visitors are allowed. All agent storage areas have concrete vault construction, high-security hardware and locks, and an intrusion detection system. Archived laboratory data is stored in a locked room equipped with fire suppression capability.

5.0 OPERATING PROCEDURES AND DOCUMENT CONTROL

- 5.0.1 The development and routine use of written operating procedures promotes consistency and reproducibility within the laboratory. Activities related to the sampling and analyses of chemical agent-related wastes are documented in WDTC technical methods. Table 2 lists the sampling and analytical methods associated with this QAPP.
- 5.0.2 This section describes the requirements for technical methods, method development and approval, and document control for methods referenced in this QAPP. Sections 5.1 outlines the requirements for analytical methods. Analytical method development is described in Section 5.2 and document control is described in Section 5.3.

5.1 REQUIREMENTS FOR TECHNICAL METHODS

5.1.1 Technical methods detail the requirements for QAPP-related activities such as sample collection and sample analysis. Each technical method will include, where applicable, the following elements:

- Title/Approval Page.
- Header (method number, title, revision number, etc.).
- Scope and Application.
- Scientific Basis.
- Terminology.
- Safety.
- Apparatus and Reagents.
- Standards and QC.
- Procedure.
- Data Reduction and Assessment.
- References.
- Figures, Tables, and Exhibits.

5.2 METHOD DEVELOPMENT AND APPROVAL

5.2.1 Methods approved for characterizing chemical agent-related wastes are listed in the Waste Analysis Plan (Hazardous Waste Permit Attachment 1-1). Where it is necessary to change existing methods or employ new methods for these analyses, the methods or changes will be subject to agreement between the laboratory, DEP, and the Executive Secretary. New or updated methods must be fully documented and approved before they are implemented. Exceptional departures from approved methods and procedures must be clearly documented and approved by DEP and the Executive Secretary.

5.2.2 During method development, the laboratory must demonstrate that the analytes of interest can be determined in the expected matrices, and that precision, accuracy, and detection limits are adequate for the intended use of the data. Factors to be considered during method development include:

- Sampling and preservation requirements.
- Stability of samples.
- Extraction efficiencies.
- Stability of extracts.
- Analytical matrix effects and interferences.
- MDLs.
- RLs.
- Precision.
- Accuracy.

5.3 DOCUMENT CONTROL

5.3.1 Key documents within the laboratory, such as this QAPP and associated methods, are controlled to ensure that changes are made in a uniform manner and that only the latest revision of each document is being used. The West Desert Document Control Clerk maintains original QAPP

documents and distributes controlled copies to designated personnel. Controlled documents may be electronic or hardcopy. Controlled hardcopy documents are sequentially numbered and designated as controlled documents (See Figure 4).

5.3.2 Technical or administrative personnel may initiate revisions to controlled documents. Revisions to QAPP-related documents must be approved by DEP. The Executive Secretary must also approve significant changes to sampling or analytical methods.

| Table 2 | |
|---|--|
| Methods for Chemical Agent-Related Wastes. | |
| Method Number | Method Title |
| SAMPLING METHODS | |
| CL-022R | Sampling Solid Wastes with DAAMS ^a |
| CL-055R | Sampling Liquid Wastes |
| CL-057R | Sampling Soils |
| ANALYTICAL METHODS | |
| CL-002R | Chemical Agents in Liquid and Solid Wastes by GC/MS ^b |
| CL-044R | Chemical Agent Monitoring using Field MINICAMS [®] |
| CL-052R | Chemical Agents in DAAMS by Gas Chromatography |
| CL-071R | Dry Weight for Solids |
| OTHER METHODS | |
| DP-0000-M-73 | Preparation of Standard Solutions |

^a DAAMS - Depot Area Air Monitoring System

^b GC/MS - Gas Chromatography/Mass Spectroscopy

Figure 4. Example of Controlled Document Stamp.



(Note: Red ink is normally used)

6.0 SAMPLE COLLECTION

- 6.0.1 Proper sample collection is critical to making correct waste disposal and treatment decisions. Sample collection personnel must ensure that samples delivered to the laboratory are representative of the waste in question. Sample collection, preservation, and transportation procedures must minimize sample loss and analyte degradation. Additionally, sample collection personnel must ensure samples and QC samples (blanks, duplicates) are collected in sufficient volume for laboratory analysis.
- 6.0.2 General sample collection protocols, equipment, preservation, storage, and QA/QC procedures are described below. Sections 6.1 and 6.2 describe the requirements for generator and sample collection planning and documentation. Section 6.3 describes the cleaning of sample collection equipment and containers. Sections 6.4 through 6.6 describe collecting liquid, soil, and solid samples. Section 6.7 describes collecting field QC samples. Sections 6.8 and 6.9 describe the requirements for maintaining sample custody and requesting sample analysis. Communicating potential safety concerns and delivering samples to the laboratory are described in Sections 6.10 and 6.11.

6.1 PLANNING AND DOCUMENTING WASTE GENERATION ACTIVITIES

- 6.1.1 In order to minimize unnecessary sample collection and analysis, waste generation activities should be well planned and documented. For chemical agent-related wastes, the waste generator (such as the test officer or building operator) is responsible for clearly documenting and communicating the history of the samples to be tested.

6.2 DOCUMENTING SAMPLE COLLECTION ACTIVITIES

- 6.2.1 Pertinent sample collection information is recorded as it occurs. The following information should be recorded in the field logbook or worksheet, as applicable:
- Name(s) of sample collection personnel.
 - Collection date.
 - Collection time for each sample.
 - Start time, end time, and flow rate for air samples.
 - Location of sample collection.
 - Type of waste (liquid decontamination solution, solid test item, etc.).
 - Sample identification (drum number, barcode number, etc.).
 - Description of sample (color, consistency, tentative identification, historical information, etc.).
 - Number of phases present and description of each phase.
 - Identifying marks or number on container.
 - Sample collection equipment, method, and description.
 - Personal protective equipment used.
 - Environmental conditions (temperature, moisture, etc.).
 - Unusual or hazardous conditions.
 - Other observations.
- 6.2.2 All sample containers (or sample collection devices such as sorbent tubes) must be clearly marked to avoid misidentification. Affix tags or self-adhesive labels to the sample containers

before, or at the time of, sample collection. Sample labels (or accompanying paperwork if samples are small) should include the following information, as applicable:

- Unique field sample identification number.
- Name of collector.
- Date of collection.
- Time of collection.
- Start time, end time, and flow rate for air samples.
- Place of collection.
- Analyses requested.
- Comments.

6.3 CLEANING SAMPLE COLLECTION EQUIPMENT AND CONTAINERS

- 6.3.1 Sample collection equipment and containers must be free of all analytes of interest and potential interferences. Where possible, disposable sample collection equipment and sample containers are used for collecting and transporting chemical agent-related waste samples.
- 6.3.2 Between uses, scoops, shovels or other soil sample collection equipment are cleaned using a soap and water wash followed by a triple rinse with distilled water. Spent cleaning liquids are collected in drums designated and managed as potential chemical agent-related waste. Alternatively, disposable sample collection equipment can be used.

6.4 COLLECTING LIQUID SAMPLES

- 6.4.1 Liquid chemical agent-related wastes may include spent bleach and caustic decontamination solutions, Installation Restoration Program (IRP) wastes, investigation derived waste (IDW), and other miscellaneous liquids. Such wastes may include solid materials associated with testing. Liquid chemical agent-related wastes are typically stored in 55-gallon drums or large storage tanks. Where possible, liquid wastes have been segregated into waste streams based on the source of the waste, chemical agent exposure, and type of decontamination procedure used.
- 6.4.2 Generally, one sample is collected per drum or container of liquid waste. In the case of homogeneous liquid wastes being transferred from a large storage tank (>500 gallons) to multiple 55-gallon drums (a single “batch”), two samples (one at the beginning and another at the end of the transfer process) are considered sufficient. If the waste stream is multiple layers or non-homogeneous, the number of samples needed to be collected must be agreed upon with the Division of Solid and Hazardous Waste. A rinse blank is collected if the sample collection equipment has been previously used.
- 6.4.3 Collecting liquid waste samples is described in WDC-CL-055R “Sampling Liquid Wastes.” A Composite Liquid Waste Sampler (Coliwasa) is commonly used to collect free-flowing liquids and slurries from drums, shallow open tanks, pits, etc. Other acceptable liquid sample collection devices include the glass thief and the bailer. Samples with a distinct solvent layer greater than 10% will be separated and each layer analyzed individually.
- 6.4.4 Liquid samples designated for chemical agent analysis are collected into clean glass containers. Samples are delivered to the laboratory as soon as possible as described in Section 6.11. Sample collection criteria are summarized in Table 3.

6.5 COLLECTING SOIL AND SOLID SAMPLES

- 6.5.1 Soil and solid chemical agent-related wastes may include soils related to spilled materials or any other soil from miscellaneous sources. Soils and solids generated during planned restoration activities are sampled and analyzed as part of the IRP. Soils and solids collected in compliance with a State-approved IRP sampling plan will be acceptable for analysis.
- 6.5.2 The waste generator usually determines the number and location of samples to be collected with input from DEP and sample collection personnel, or the project plan. A rinse blank is collected if re-useable sample collection equipment is used. Collecting soil and solid waste samples is described in WDC-CL-057R “Sampling Soils and solids.” Sample collection equipment must be free of analyte contamination and could include a stainless steel spoon, scoop, auger, and/or shovel.
- 6.5.3 Soil samples designated for chemical agent analyses are collected into clean glass containers. Samples are delivered to the laboratory as soon as possible as described in Section 6.11. Sample collection criteria are summarized in Table 3.

6.6 AIR MONITORING OF SOLID SAMPLES

- 6.6.1 Solid chemical agent-related wastes may include decontaminated solid test items, gloves and other project wastes, ventilation system wastes (including chemical agent contaminated pre-filters, high efficiency particulate air filters, plenums, duct work and activated carbon filters), IRP wastes, IDW, and other miscellaneous solid items (not including soils). Where possible, solid wastes are segregated into waste streams based on the source of the waste, chemical agent exposure, and type of decontamination procedure used. Air Monitoring of solids may be used for agent screening purposes only and not for waste determination.
- 6.6.2 Sampling for air monitoring is performed using one of the following methods:
- WDC-CL-022R “Sampling Solid Wastes with DAAMS
 - WDC-CL-044R “Chemical Agent Monitoring using Field MINICAMS®
- 6.6.3 Generally, one sample is collected per container of solid waste (bag or Wrangler). Bagged items are sampled individually before transfer into a barrel or other larger container. Dry solid waste samples are placed in a sealed container and the contents are allowed to equilibrate for at least four hours at a temperature of 21°C (70°F) or higher. Small items may be placed, and heated if necessary, in a plastic bag having a minimum thickness of 4 MIL. Seal the bag such that it contains sufficient air to complete the monitoring task. Larger items may be placed, and heated if necessary, in a roll-off or gondola and sealed with a tarp and packing tape. Following the equilibration period, the air surrounding the item in the container is sampled using a Depot Area Air Monitoring System (DAAMS) tube sampler or MINICAMS® as described in the methods listed above. Sample analysis using MINICAMS® is performed at the location of the solid waste. Three samples are collected from large waste containers such as gondolas and roll-offs – one sample at each end and one in the middle. Following sample collection, DAAMS tubes are sealed, labeled with a unique sample number, and delivered to the laboratory for analysis. Sample collection criteria are summarized in Table 3.
- 6.6.4 Solid samples obtained for subsequent laboratory preparation and analysis must be collected in a representative manner in accordance with a DEP-approved sampling plan. The sampling plan

must outline the sampling objectives, sample collection procedures, number and location of samples, required analyses for each sample, etc. A rinse blank is collected if re-useable sample collection equipment is used. Samples are generally collected into clean glass containers and delivered to the laboratory as soon as possible as described in Section 6.11.

6.7 COLLECTING FIELD QC SAMPLES

- 6.7.1 Field QC samples are intended to provide a measure of the cleanliness and representativeness of the sample collection activities. For chemical agent sample collection activities, field blanks, rinse blanks and/or duplicate samples may be required (see Table 4).
- 6.7.2 Field Blanks are used to detect possible contamination in the sample collection system. They are generally used when off-gas samples are collected using MINICAMS[®] or DAAMS. Generally, one rinse blank is collected per sample collection lot (samples collected from the same waste description at the same time).
- 6.7.3 Rinse blanks are required when sample collection equipment (such as non-disposable coliwassas) is cleaned and reused. Generally, one rinse blank is collected per sample collection lot (samples collected from the same waste description at the same time). Rinse blanks are prepared by running an analyte-free solution through sample collection equipment after cleaning but before sample collection. The rinse blank is analyzed and used to determine the effectiveness of equipment cleaning procedures.
- 6.7.4 Sample duplicates are required for liquids or soils when a new or unknown waste source is collected. Generally, one duplicate is collected per sample collection lot (samples collected from the same waste description at the same time). Sample collection personnel may also collect sample duplicates in order to accurately characterize complex matrices. A sample duplicate is simply a repeat of the sample that is sent to the laboratory to see whether the original sample results can be repeated.
- 6.7.5 Field spike samples (also known as Quality Plant (QP) Samples) are required when air sampling using DAAMS. Generally, two field spike samples are collected per sample collection lot (samples collected from the same waste description at the same time). Field spike samples are prepared in the laboratory by adding a known amount of analyte to a DAAMS tube. The spiked sample is taken to the field, aspirated with the same air as the sample, and returned to the laboratory for analysis.

6.8 MAINTAINING CHAIN-OF-CUSTODY

- 6.8.1 To ensure integrity of compliance samples, sample collection personnel must be able to trace possession and handling of samples from the time of collection through delivery to the laboratory. A sample is considered to be under a person's custody if it is in the individual's physical possession, in the individual's sight, secured in a tamper-proof way by that individual, or secured in an area restricted to authorized personnel.
- 6.8.2 A completed chain-of-custody (COC) record such as the one shown in Figure 5) must accompany each sample or group of samples. To relinquish samples from custody, the sample relinquisher and receiver inspect the samples and review the completeness, accuracy, and legibility of the

accompanying documentation. The relinquisher and receiver sign the COC form and record the date and time of sample transfer.

6.9 REQUESTING LABORATORY ANALYSIS

6.9.1 Analyses to be performed on each sample must be clearly indicated on the COC or other documentation (see Figures 5 and 6). The analysis request documentation should include the following information:

- Type of analysis being requested.
- Name, location, and phone number of sample requestor or contact.
- Project and/or site description.
- Sample identification (must be consistent with the sample containers).
- Sample matrix (liquid, soil, oil, etc.).
- Sample collection date and time.
- Comments.

6.10 NOTIFICATION OF SAFETY CONCERNS

6.10.1 Samples and accompanying paperwork must be adequately labeled to indicate any known or potential hazards such as flammability, corrosivity, toxicity, radioactivity, etc. Collection personnel and laboratory receiving personnel are responsible to communicate safety concerns to laboratory management and to laboratory personnel so that appropriate precautions can be taken during sample handling, storage, and disposal.

6.11 TRANSPORTING SAMPLES

6.11.1 Samples should be delivered to the laboratory as soon as possible after collection to ensure adequate time for analysis. Samples that cannot be delivered immediately to the laboratory must be held securely under documented control until delivery to the laboratory. Samples that cannot be delivered to the laboratory within 30 minutes should be stored and transported on ice to avoid degradation. A completed COC form must accompany samples and analysis request as described in Sections 6.8 and 6.9 above. The laboratory Sample Coordinator (or designated alternate) has the responsibility to reject samples at check-in for improper sample containers, incomplete paperwork, improper temperature preservation at the time of receipt (i.e., the sample was not received on ice), or any other sample problem that may cause an invalid analytical result. The sample requestor will be notified immediately upon recognition of these problems.

| Table 3. Summary of Chemical Agent-Related Waste Sample Collection Criteria. | | | | |
|---|---|---|-------------------------|---|
| Matrix | Waste Streams^a | Sample Collection Devices^b | Sample Container | Collection Frequency |
| Liquid Wastes | <ul style="list-style-type: none"> ▪ Spent decontamination solutions ▪ IRP liquid wastes ▪ IDW liquid wastes ▪ Miscellaneous liquid wastes | <ul style="list-style-type: none"> ▪ Glass Coliwasa ▪ Glass thief ▪ Bailer ▪ Other as appropriate | Glass | 1 per drum 2 per batch |
| Soil Wastes | <ul style="list-style-type: none"> ▪ Spill materials ▪ Miscellaneous soil wastes | <ul style="list-style-type: none"> ▪ Spoon ▪ Scoop ▪ Shovel ▪ Auger ▪ Other as appropriate | Glass | Project specific |
| Solid Wastes (for air monitoring) | <ul style="list-style-type: none"> ▪ Decontaminated test items ▪ Project wastes ▪ Ventilation systems wastes ▪ IRP solid wastes ▪ IDW solid wastes ▪ Miscellaneous solid wastes | <ul style="list-style-type: none"> ▪ MINICAMS[®] ▪ DAAMS apparatus | DAAMS tube | 1 per container ^c 3 per gondola or roll-off |
| Solid Wastes (for extraction) | <ul style="list-style-type: none"> ▪ Miscellaneous solid wastes | <ul style="list-style-type: none"> ▪ Spoon ▪ Scoop ▪ Shovel ▪ Auger ▪ Other as appropriate | Glass | Project specific |

^a IRP – Installation Restoration Program; IDW – Investigative Derived Waste

^b DAAMS – Depot Area Air Monitoring System

^c For MINICAMS[®], one sample consists of three cycles

**Table 4.
 Summary of Field Quality Control Sample Collection Requirements.**

| Matrix | Waste Streams^a | Blank Requirements | Field Duplicate Requirements | Other Field QC Requirements^b |
|----------------------------------|---|--|---|---|
| Liquid Wastes | <ul style="list-style-type: none"> ▪ Spent decontamination solutions ▪ IRP liquid wastes ▪ IDW liquid wastes ▪ Miscellaneous liquid wastes | One rinse blank per sample collection lot when sample collection equipment is reused | One field duplicate per sample collection lot when a new or unknown waste source is collected | None |
| Soil Wastes | <ul style="list-style-type: none"> ▪ Spill materials ▪ Miscellaneous soil wastes | One rinse blank per sample collection lot when sample collection equipment is reused | One field duplicate per sample collection lot when a new or unknown waste source is collected | Consult with the laboratory to determine if extra sample is required for matrix spikes |
| Solid Waste (for air monitoring) | <ul style="list-style-type: none"> ▪ Decontaminated test items ▪ Project wastes ▪ Ventilation systems wastes ▪ IRP solid wastes ▪ IDW solid wastes ▪ Miscellaneous solid wastes | One field blank per sample collection lot | Not applicable | For DAAMS samples: two field spike sample (also known as QP sample) per sample collection lot |
| Solid Waste (for extraction) | <ul style="list-style-type: none"> ▪ Miscellaneous solid wastes | One rinse blank per sample collection lot when sample collection equipment is reused | One field duplicate per sample collection lot when a new or unknown waste source is collected | Consult with the laboratory to determine if extra sample is required for matrix spikes |

^a IRP - Installation Restoration Program; IDW - Investigative Derived Waste

^b DAAMS - Depot Area Air Monitoring System; QP - Quality Plant Sample

Figure 5. Example of Chain of Custody/Analysis Request Form for Liquids and Soils

Chain of Custody ID Number: _____

| | |
|---|---|
| <p align="center">DPG CHEMICAL TEST DIVISION</p> <p align="center">Combined Chemical Test Facility Dugway, UT 84022-5000 Phone: (801) 831-5137</p> | <p>Requestor: _____ Office: _____ Phone: _____ Jono: _____ CC Project: _____ Costs: _____</p> <p>Special Instructions: _____</p> |
|---|---|

| Sample Identification <small>(barrel number, bag number, field number or other unique ID)</small> | Date Sampled | Time Sampled | Sample Matrix ¹ | Volume of Air Sampled ² <small>(Liters)</small> | ANALYSES REQUESTED | | | | | | | | | | | | Laboratory ID <small>(lab use only)</small> | |
|--|--------------|--------------|----------------------------|---|--------------------|--|--|--|--|--|--|--|--|--|--|--|--|--|
| | | | | | | | | | | | | | | | | | | |
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| | | | | | | | | | | | | | | | | | | |
| Comments: | | | Description: | | | | | | | | | | | | | | | |
| | | | Phases: | | | | | | | | | | | | | | | |

(1) Matrix = liquid (Waste), soil, bubbler, DAAMS, MINICAMS, or other (specify) (2) If applicable, volume of air (L) = sampling rate (L/min) X sampling time (min)

| CHAIN-OF-CUSTODY | |
|-------------------------------|--------------|
| 1) Relinquished By (Sampler): | Received By: |
| | Date/Time: |
| 2) Relinquished By: | Received By: |
| | Date/Time: |
| 3) Relinquished By: | Received By: |
| | Date/Time: |

| CHAIN-OF-CUSTODY | |
|-------------------------------|--------------|
| 4) Relinquished By (Sampler): | Received By: |
| | Date/Time: |
| 5) Relinquished By: | Received By: |
| | Date/Time: |
| 6) Relinquished By: | Received By: |
| | Date/Time: |

Reference WD-C Method ADM-018R (Maintaining Chain-of-Custody)

Figure 6. Example of Sampling Request Form.

Sample Request Form

(Please Fill Out All Information with in the Box)

| | |
|--------------------------|-----------------------------|
| Name of Requester: _____ | Date/Time of Request: _____ |
| Telephone #: _____ | Date/Time of Testing: _____ |
| Report Results To: _____ | Project Name: _____ |
| Telephone #: _____ | CWO/JONO# _____ |

Location of Sampling:

CCTF
 MTF
 3445
 IglG
 IRP
 DTC

Type of Sampling:

Safety Monitoring
 VON
 XXXX Hazardous Waste (items for disposal)

XXX
 XXXX (items to stay in place)
 XXXX (items to be removed)

Description of Items Being Sampled:

Special Instructions:

Solid Sorbent Tubes (DAAMS) # of Sets - _____

VX
 HD
 GA
 GB
 GD
 GF
 HN3
 EA4243
 EA6043

| | | |
|---------------------|---------------------------|-----------------|
| # of Samples: _____ | # of Confirmations: _____ | # of QPs: _____ |
|---------------------|---------------------------|-----------------|

Bubblers Sets - _____

L

| | | |
|---------------------|---------------------------|-----------------|
| # of Samples: _____ | # of Confirmations: _____ | # of QPs: _____ |
|---------------------|---------------------------|-----------------|

MINICAMS®

VX
 HD
 GA
 GB
 GD
 GF
 L
 HN3

Issues and/or help with this form please see or call James Westra @ 5988

Dugway Proving Ground
 West Desert Test Center
 Chemical Test Division

WDC-FRM-072
 Revision 0

7.0 GENERAL LABORATORY PROCEDURES

- 7.0.1 When samples are delivered to the laboratory, designated receipt personnel ensure that sample collection operations have been properly conducted and clearly documented. Receiving personnel must correctly document testing requirements and other information (such as required test methods, turnaround time, required sensitivity, and safety concerns) to analytical personnel.
- 7.0.2 This section describes the requirements for receiving and handling chemical agent-related waste samples. Sections 7.1 through 7.3 describe laboratory sample receipt, the storage and distribution of samples within the laboratory, and sample custody respectively. Section 7.4 discusses sample disposal. Section 7.5 discusses cleaning procedures for labware and sample collection equipment. Section 7.6 describes obtaining reagents, supplies, and services.

7.1 LABORATORY SAMPLE RECEIPT

- 7.1.1 Samples should be transported to the laboratory as soon as possible after sampling (see Section 6.0, Sample Collection). Chemical agent-related waste samples are received by the laboratory sample coordinator or designated alternate coordinators if the sample coordinator is unavailable.
- 7.1.2 At sample receipt, the sample coordinator will ensure that:
- COC, analysis request, and other receiving documentation is accurate and complete (see Section 6.8).
 - Samples have been transported to the laboratory on ice (see Table 5).
 - Sample containers are of an acceptable material, in good condition, and properly labeled (See Table 5).
 - The proper number of field QC samples have been submitted (see Section 6.7).
- 7.1.3 The sample coordinator logs the samples into the sample tracking system including the following information:
- The unique laboratory number for each sample.
 - Date and time of sample receipt.
 - Requester's name and contact information.
 - Project name.
 - Person delivering the samples.
 - Person receiving the samples.
 - Number of containers for each sample.
 - Field sample identification number.
 - Date of sample collection.
 - Sample matrix (liquid, soil, DAAMS, etc.).
 - Requested analyses.
 - Preservation method for samples (regular ice or cold blue ice).
 - Temperature upon receipt.
- 7.1.4 The sample coordinator places a laboratory number on each sample container. All samples, sub-samples, extracts, digestates, or other fractions derived from a sample will be labeled with the unique sample number assigned during sample receipt.

7.2 SAMPLE STORAGE AND DISTRIBUTION

- 7.2.1 Although no specific maximum holding time has been determined for chemical agent-related wastes, recommended holding times are listed in Table 5. Analysis of sample extracts usually occurs within 7 days of sample preparation. Waste samples are stored at a temperature for samples with a specified storage temperature of 4°C. Sample storage at a temperature above the freezing point to 6°C shall be acceptable while awaiting analysis. Samples removed from the refrigerator for analysis are returned as soon as possible.
- 7.2.2 The sample coordinator distributes samples to the analysts. Analytical personnel are alerted, in writing, of any special analytical or handling requirements as well as turnaround requirements.

7.3 SAMPLE CUSTODY

- 7.3.1 The security and integrity of each sample is very important. Access to the facility is limited to approved employees, contractors and vendors. Samples are to remain in designated storage areas except when analysts are preparing samples for analysis. Laboratory records (such as sample request forms, login forms, bench sheets, etc.) are sufficient to track the procedures a sample is subject to while in the laboratory's possession.
- 7.3.2 Samples are collected and transported to the laboratory under COC (see Section 6.8). All documentation that is transmitted to the laboratory by the sample requestor, including memos, transmittal forms, and COC forms, will be maintained as described in Section 9.

7.4 SAMPLE DISPOSAL

- 7.4.1 Unless other arrangements have been made with the sample coordinator, all samples will be disposed of after analysis and review of the Analytical Report. Sample disposal is performed in accordance with applicable safety and environmental regulation as described in Attachment 1-1, CHWSF Waste Analysis Plan.

7.5 LABWARE CLEANING AND MAINTENANCE

- 7.5.1 All glass and reusable plastic labware is thoroughly cleaned before use to avoid contamination. The cleanliness of reusable labware is evaluated using method blanks (MB). Labware should be rinsed, decontaminated, and placed in a suitable soaking solution (such as a mild soap solution) immediately after emptying so that residues are not allowed to dry onto the glassware. All containers or washtubs should be clearly marked to indicate their contents and, if applicable, the return location.
- 7.5.2 After cleaning, borosilicate and other glass products should be inspected for chipping, cracking, or other abnormalities. Glass labware, which is excessively contaminated or exhibits signs of damage, will be removed from service until repaired or discarded. Labware should also be inspected to determine if unusual cleaning might be required.
- 7.5.3 In general, labware washing procedures should include the following steps:
- Presoak labware as necessary
 - Wash labware with phosphate-free detergent mixed with hot water
 - Manual or automated washing is appropriate

- Ensure that all surfaces are thoroughly cleaned
- After cleaning, triple rinse the labware with tap water then de-ionized water
- Dry labware at approximately 150°C

7.6 SUPPLIES & SERVICES

7.6.1 The laboratory relies on many outside sources for supplies and services which impact analytical quality. Laboratory equipment and supplies are purchased to meet or exceed the requirements of the analytical methods. Standard solutions, reagents and other chemicals must meet or exceed the quality and purity standards specified in the QAPP methods. The preparation of all reagent and standards solutions must be clearly documented and provide traceability to the materials and procedures used.

| Table 5. Recommended Analytical Methods, Containers and Sample Holding Times | | | | |
|---|-------------------------------------|------------------------------|---|---|
| Determination^a | Method Reference^b | Container^c | Preservative for Samples^d | Recommended Maximum Holding Time |
| Chemical Agents - GC or GC/MS ^d | CL-002R | G | <6°C but above freezing | Prepare: 14 days Analyze: 7 days |
| Chemical Agents - MINICAMS [®] | CL-044R | NA | NA | Field Analysis |
| Chemical Agents - DAAMS | CL-052R | DAAMS Tube | <6°C but above freezing | Prepare/Analyze: 7 days |

^a GC or GC/MS Gas chromatography; Mass spectroscopy

^b Equivalent methods may be used if approved by the Executive Secretary.

^c Container for solid samples is generally 4-6 ounce clear wide-mouth glass jar or plastic bag. G – Glass; NA – not applicable; DAAMS – Depot area air monitoring system

^d Preservation for solid samples is generally cooling to <6°C but above freezing

8.0 CALIBRATION PROCEDURES AND FREQUENCIES

8.0.1 Calibration is accomplished through the use, when available, of reference materials supplied by the Chemical Agent Standard Analytical Reference Materiel (CASARM) Program of the Soldier and Biological Chemical Command (SBCCOM). The reference materials are stringently analyzed and certified by the CASARM Program. The program includes ongoing validation to ensure that reference material degradation does not occur. These reference materials are used throughout the military chemical-defense complex. Solutions derived from these reference materials are prepared at DPG and used to calibrate instrumentation. This system ensures that all measurements within the military complex are comparable and traceable to an accepted standard. Testing at DPG may involve chemical agents for which CASARM does not supply standard analytical reference materials. In such situations, DPG will establish the purity of the standard and document it by preparing a Statement of Purity.

8.0.2 The sections below detail the calibration procedures used in the DPG chemical agent-related Hazardous Waste Analysis Program. Additional calibration information is found in the individual analytical methods listed in Section 2.0. Section 8.1 describes the handling of reference materials. Section 8.2 describes the calibration requirements for laboratory instrumentation, and Section 8.3 describes the calibration requirements for MINICAMS[®].

8.1 HANDLING REFERENCE MATERIALS

8.1.1 Where available, standards are prepared from CASARM in accordance with the requirements of WDTC Standing Operating Procedure DP-0000-M-73. Generally, two analysts independently prepare two stock solutions. One solution is used to prepare working standards and the other is used to prepare verification standards. All manipulations and dilutions are recorded. All solutions must be traceable to the CASARM. All uses of solutions are recorded to ensure traceability.

8.2 CALIBRATING LABORATORY INSTRUMENTS

8.2.1 This section describes the calibration of laboratory instrumentation such as stationary GCs. Detailed calibration instructions are given in individual analytical methods.

8.2.2 Initial calibration is required for laboratory instrumentation within a method-specified time period if significant changes are made to the instrument, or if the calibration verification fails. In general, initial calibration is performed for each analyte with a minimum of four concentrations. The linear or second order regression analysis of the calibration curve must result in an r^2 value (where r is the correlation coefficient) of at least 0.990. The calibration curve is verified with an initial calibration verification solution that must be recovered within $\pm 15\%$ of true value, unless specified otherwise in the analytical method.

8.2.3 Continuing calibration is performed by analyzing calibration check (CC) standards within each analytical run to ensure that the initial calibration is still valid. At a minimum, a CC is analyzed after every ten or fewer waste samples and/or after any standby period or other period of disuse or within 12 hours whichever is more frequent. CC standards must be recovered within $\pm 20\%$ of true value, unless specified otherwise in the analytical method. If the CC fails, it is repeated. If it fails a second time, then an initial calibration must be performed or corrective action must be taken. Samples with a failed low CC will be reanalyzed. If the CC fails high for a particular analyte and that analyte is not detected in the sample, the non-detected value may be reported. The high bias must be documented and narrated.

8.3 CALIBRATING FIELD MINICAMS[®]

8.3.1 The calibration procedures for field MINICAMS[®] are detailed in WD-C METHOD CL-044R "Chemical Agent Monitoring (GA, GB, GD, GF, HD, HN1, HN3, VX, and Lewisite) using Field MINICAMS[®]". Calibration verification is required each time the MINICAMS[®] is moved to a new location (such as a new building) or if significant changes are made to the instrument. Calibration verification may also be performed as part of troubleshooting as described in the operating procedure. Generally, the field MINICAMS[®] is initially calibrated for the analyte(s) of interest by first placing it in the calibration mode. A known amount [at the Worker Population Limit (WPL)] of standard is injected into the instrument during two successive cycles. The MINICAMS[®] will automatically calculate the average response factor from the three injections and store the new calibration. Initial calibration is verified by injecting a known standard prepared at two times the regulatory level. A result of between 0.75 and 1.25 times the known value (+25%) is considered satisfactory.

8.3.2 Continuing calibration is required after initial calibration, at the beginning and end of each run, and after every 10 hazardous waste samples. To perform a continuing calibration, a QC standard (prepared at or near the regulatory level) is injected into the instrument during the sampling

period of the MINICAMS[®] cycle. A result of between 0.75 and 1.25 times the known value ($\pm 25\%$) is considered satisfactory. If the first QC fails, a second is injected. If the second QC also fails, corrective action should be taken as described in the method. If, following corrective action, a third QC fails, the MINICAMS[®] should be removed from service for repair or refurbishment. If the CC fails high for a particular analyte and that analyte is not detected in the sample, the non-detected value may be reported. The high bias must be documented and narrated.

9.0 LABORATORY QC

9.0.1 Method-specific laboratory QC measures are used to assure that the analytical process is in control. QC parameters may include rinse and MBs (used to evaluate cleanliness), method blank spike (MBS) samples (used to evaluate accuracy), and method blank spike duplicate (MBSD) samples (used to evaluate precision). DQOs for cleanliness, accuracy, and precision (Sections 9.1 through 9.3) are established to ensure that the data will support the objectives of the DPG waste analysis and management programs. Section 9.4 outlines the determination of analytical method performance. Sections 9.5 and 9.6 discuss determination of the MDL and RL.

9.1 OBJECTIVES FOR CLEANLINESS

- 9.1.1 Cleanliness is defined as the absence of contamination in the field and laboratory. Field contamination is evaluated using field blanks and rinse blanks (Section 6.7). In general, field blanks are collected when off-gas samples are collected using MINICAMS[®] or DAAMS. Rinse blanks are required for liquid and soil matrices when sampling equipment is being re-used. The concentration of all target analytes in the rinse blank should be less than the RL. Specific requirements for the sample collection are found in Section 6.0 and the individual sampling methods.
- 9.1.2 Laboratory contamination is evaluated using the MB. In general, the concentration of all target analytes in the MB should be less than the RL. Specific requirements for the preparation and evaluation of MBs are found in the individual analytical methods. Results that do not meet the DQO for cleanliness require corrective action as described in Section 12.0. Cleanliness DQOs for chemical agent-related waste analysis are provided in Table 6.

9.2 OBJECTIVES FOR ACCURACY

- 9.2.1 Accuracy is a measure of the ability of the analytical method to achieve a known analytical result. For chemical agent-related wastes, accuracy is usually evaluated by analyzing a clean matrix sample (MB) that has been spiked with known amounts of the target compounds. In some cases, matrix spike samples may also be indicative of method accuracy. Details on the preparation of MBS samples are found in the individual analytical methods.
- 9.2.2 Percent recovery (%R) for each MBS compound is calculated as:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

where: SSR = spiked sample result
SR = unspiked sample result (usually zero)
SA = spike amount added to the sample

- 9.2.3 The %R for each compound, method, and matrix is compared with previous data using statistical QC charts or method defined control limits. The result must be within the 99% confidence limits or control limits. In the absence of adequate statistical data for %R, an acceptance range of 70-130% will be used as a guide. Results that do not meet the DQO for accuracy require corrective action as described in Section 12.0. Accuracy DQOs for chemical agent-related waste analysis are provided in Table 6.

9.3 OBJECTIVES FOR PRECISION

- 9.3.1 Precision is a measure of the variability of the analytical method. For chemical agent-related wastes, precision is most often evaluated by comparing the results of the MBS and MBSD recoveries using the range (R) or the relative percent difference (RPD). In some cases, matrix spike duplicates may also be used to evaluate precision. R and RPD are calculated as:

$$R = |MBSR - MBSDR|$$
$$RPD = \left| \frac{2(MBSR - MBSDR)}{MBSR + MBSDR} \right| \times 100$$

where: MBSR = MBS percent recovery
MBSDR = MBSD percent recovery

- 9.3.2 Either R or RPD for each compound, method, and matrix is compared with control limits. The result must be within the control limits. In the absence of adequate statistical data for RPD, an acceptance limit of 20% will be used as a guide. Results that do not meet the control limits for precision require corrective action as described in Section 12.0. Precision control limits for chemical agent-related waste analysis are provided in Table 6.

9.4 ANALYTICAL METHOD PERFORMANCE

9.4.1 Analytical method performance is defined in terms of accuracy and precision. Method accuracy and precision are determined during method development by preparing and analyzing at least eight mid-level (approximately 10-20 times the estimated MDL) replicate samples. Method performance is often determined in conjunction with the DPG Safety Air Monitoring precision and accuracy study.

Accuracy (percent recovery, %R) and Precision [relative standard deviation (RSD)] are calculated using the following formulas:

◆ Method Accuracy

$$\%R = \frac{\text{average}}{\text{expected}} \times 100$$

where: average = average result
expected = true value

◆ Method Precision

$$RSD = \frac{s}{\text{average}} \times 100$$

where: s = standard deviation of replicate results
average = average result

Results of the method performance studies provide a basis for ongoing QC requirements as described above.

9.5 LIMIT OF QUANTITATION (LOQ), LIMIT OF DETECTION (LOD) AND MDL

9.5.1 The LOD is an estimate of the lowest level of an analyte that can be distinguished from noise. For chemical agent-related analyses, the LOD is experimentally determined initially using the MDL determination defined in 40 CFR Part 136, and, where applicable, verified or determined at least annually. The initial MDL is determined by preparing and analyzing seven or more low level (1-5 times the estimated MDL) interference-free replicate samples. When applicable, the validity of the LOD shall be confirmed on an annual basis by qualitative identification of the analyte(s) in a QC sample in each quality system matrix containing the analyte at no more than 2-3X the LOD for single analyte tests and 1-4X the LOD for multiple analyte tests. This verification must be performed on every instrument that is to be used for analysis of samples and reporting of data. Alternatively, a new MDL study can be performed annually. For GC/MS analyses using the selected ion-monitoring mode, the MDL is calculated using the primary calibration ion. Positive identification is confirmed when the secondary ions are present at their normal abundances.

An LOD/MDL study is not required when test results are not to be reported to the LOD. Where an LOD study is not performed, the laboratory may not report a value below the LOQ.

If the analysis includes standards at or below the action levels in Table 7 then an MDL/LOD study does not have to be performed. In these cases the LOQ must be verified annually as described in Section 9.5.2. In the case where the action level is defined as the MDL/LOD, then an LOD verification or MDL study must be performed annually. For example, Table 7 sets hard limits for liquid samples and samples requiring air sampling. These matrices would not require an annual MDL/LOD study, but would require the verification of the LOQ. In the case of soils/solids, Table 7 defines the action levels as the MDL. In this case, an annual MDL/LOD study would be required.

The MDL is calculated using the formula:

$$MDL = s \times t$$

Where: s = standard deviation

t = student t value from the table below

| Tables of Students' t Values at the 99 Percent Confidence Level | | |
|--|---------------------------------|-------------------|
| Number of Replicates | Degrees of Freedom (n-1) | Ten-1, .99 |
| 7 | 6 | 3.143 |
| 8 | 7 | 2.998 |
| 9 | 8 | 2.896 |
| 10 | 9 | 2.821 |

9.5.2 The LOQ must be at or below the required action level in Table 7. The validity of the LOQ shall be confirmed at least annually by successful analysis of three QC samples containing the analytes of concern in each quality system matrix 1-2.5 times the claimed LOQ. In some cases this is done in each analytical batch (as is the case in air monitoring samples and some liquid samples). In those cases the annual verification requirement is met each analysis batch. A successful analysis is one where the recovery of each analyte is within the established test method acceptance criteria. Where the test method does not have defined criteria for the LOQ verification, the laboratory will use control charts to establish fixed control limits. Until control limits can be established default limits of ±50% recovery of the true value will be used. The LOQ will be prepared in the same matrix as the Method Blank Spikes (i.e., brine solution for aqueous samples and air for air samples).

9.6 REPORTING LIMITS AND ACTION LEVELS

9.6.1 Unlike the interference-free standards prepared for determination of the MDL, field samples often contribute significant noise to the analytical procedure and the instrument response. The RL is defined as the lowest reportable analyte concentration for a particular sample given the MDL, matrix, extraction and dilution effects, interferences, analytical noise, and other relevant factors. The RL is usually a factor of 2 to 20 times the MDL. Given the hazardous nature of chemical agents, RLs should be conservatively chosen to eliminate the chance for false negative results (a non-detect at the RL when analyte is actually present above that level). Analyte levels between the MDL and RL are reported with a J qualifier, estimated value. Hazardous wastes are not transported to the CHWSF if analytical results indicate that chemical agents are present above the action levels listed in Table 7.

| Table 6. Data Quality Objectives for Chemical Agent-Related Waste Analyses. | | | | | | | |
|--|--------------------|--------------|-------------------------------|----------------------------|----------|------------------------------|----------|
| Analytical Method | Matrix | Cleanliness | | Accuracy (%R) ^a | | Precision (RPD) ^b | |
| | | Parameter | Criteria ^c | Parameter | Criteria | Parameter ^d | Criteria |
| CL-002R | Liquids and Soils | Method Blank | all target compounds <RL | Method Blank Spike | 60-140% | MBS/MBSD | <25% |
| CL-044R | Solids | Method Blank | all target compounds <0.5 WPL | Quality Control Sample | 75-125% | NA | NA |
| CL-052R | DAAMS ^e | Method Blank | all target compounds <0.5 WPL | Quality Plant Sample | 75-125% | NA | NA |

^a %R - Percent Recovery

^b RPD - Relative Percent Difference

^c RL - Reporting Limit; WPL - Worker Population Limit

^d MBS - Method Blank Spike; MBSD – Method Blank Spike Duplicate; NA - Not Applicable

^e DAAMS - Depot Area Air Monitoring System

| Table 7. Action Levels for Waste Characterization. | | | | |
|---|---|--|-----------------------|-------------------|
| Matrix | Analytical Methods^a | Analyte^b | Action Level | Units |
| Liquid | CL-002R (GC, GC/MS) | GA, GB, GD, GF, VX | 0.02 | mg/L |
| | | HD, HN1, HN3, HT, Lewisite and T | 0.2 | mg/L |
| Soils/Solids | CL-002R (GC, GC/MS) | All Agents | MDL ^c | mg/kg |
| Air Monitoring | CL-044R (MINICAMS [®]) CL-022R/CL-052R (DAAMS) | GA, GB, GD, GF | 0.00003 ^d | mg/m ³ |
| | | HD, HN1, HN3, HT, and T | 0.0004 ^d | mg/m ³ |
| | | VX | 0.000001 ^d | mg/m ³ |
| | | Lewisite | 0.0012 ^d | mg/m ³ |

^a GC – Gas Chromatography; MS – Mass Spectroscopy; DAAMS – Depot Area Air Monitoring System

^b GA – Tabun; GB – Sarin; GD – Soman; GF – Cyclosarin; VX - o-ethyl S-(2-diisopropylaminoethyl) methylphosphonothiolate; HD - Distilled Mustard; HN1 - bis-(2-chloroethyl)ethylamine; HN3 - tris-(2-chloroethyl)amine; HT - Mustard/T; T - O-Mustard

^c Risk-based action levels have not been determined for soils and solids. The Method Detection Limit is specific to an analytical instrument (such as GC/MS-Selected Ion Monitoring SIM, GC/Flame Ionization Detector FID, and GC/Flame Photometric Detector FPD). The MDL will be used for the action level until action levels are promulgated by Utah Division of Solid and Hazardous Waste. The Central Hazardous Waste Storage Facility may accept F999 and P999 wastes only if associated chemical agent MDL studies are up to date (see Section 9.5). Validation procedures for each Chemical Agent and associated MDL data must be approved by the Executive Secretary before implementation.

^d The air action levels are the Worker Population Limits (WPLs) implemented by the Army for safety air monitoring. These levels apply when air monitoring is the primary analysis method for hazardous waste acceptance to the CHWSF (such as for solid test-related debris, ventilation filters, etc.).

10.0 ANALYTICAL DATA MANAGEMENT

10.0.1 The purpose of the QA program described in this QAPP is to ensure that only valid, reliable data are reported. In order to be reported, analytical data must meet the applicable QC requirements (see Section 9.0), and then be correctly recorded, reduced, reviewed, and reported. In addition, a subset of all reported data is subject to independent validation as described below. The process of generating valid and defensible analytical data includes the following:

- Data Recording (Section 10.1).
- Data Reduction (Section 10.2).
- Data Reporting (Section 10.3).
- QC Review (Section 10.4).
- Final Approval (Section 10.5).
- Data Validation (Section 10.6).

10.1 DATA RECORDING

10.1.1 The laboratory record system must produce unequivocal, accurate records that document all laboratory activities. The laboratory retains on record all original observations, calculations, derived data, calibration records, and a copy of the test report for at least five years. The laboratory also maintains all hardware and software necessary for the historical reconstruction of data for five years. The record-keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes.

10.1.2 The history of the sample within the laboratory must be readily understood through the laboratory documentation. The records for each test must contain sufficient information to allow the historical reconstruction of all laboratory activities related to analytical data produced. Where possible, checklists and/or forms (electronic or printed) are used to ensure that data are recorded and presented accurately and consistently. Records must be legible and give sufficient detail to enable an independent reviewer to:

- Reconstruct the sequence of events.
- Reconstruct calculations.
- Establish that key steps were completed.
- Establish that method specified recording requirements were met.
- Establish that the record is complete.

10.1.3 Data generated within the laboratory must be documented according to scientifically acceptable standards. These include:

- Checklists and/or forms are used, where possible, to ensure that data are recorded and presented accurately and consistently.
- Data must be recorded at the time it is generated.
- Data must be recorded by the generator of the data, or a direct observer.
- Errors are crossed out with a single straight line.
- Corrected data is entered, initialed, and dated.
- No erasures or correction/fluid is allowed.
- Hand-entered data are recorded with permanent ink pen.
- Data recorded or generated electronically shall be printed out, signed, and dated by the operator (on the cover page if the report is stapled or bound).
- Each page of a multi-page record or report must be numbered to show the page number and the first page must state the total number of pages in the record or report.
- Fields in forms that are not used are lined through with a single diagonal line or noted as not applicable (NA).
- If electronic data are to be included in a logbook, the printout is secured (taped, stapled, or pasted) in the logbook, then signed. The signature and date must cross both the print-out and the page to which it is secured.

10.1.4 Records that are stored on computers will have hard copy or write-protected backup copies. Archived records are protected against fire, theft, loss, environmental deterioration, and in the case of electronic records, electronic or magnetic sources.

10.2 DATA REDUCTION

- 10.2.1 Data reduction is the process of converting an analytical signal or response to a reportable result. Depending upon the test and instrumentation involved, data are reduced and reported using both manual and automated procedures. If the data are manually processed and reported by an analyst, all steps in the computation are recorded for review including equations used and the source of input parameters such as response factors, dilution factors, and calibration data. The analyst signs and dates each page of calculations and data in a bound logbook for review and verification purposes.
- 10.2.2 Where computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data, the laboratory will ensure that computer software is documented and adequate for use. Before releasing the data to the reporting system, the analyst must verify that information such as the sample numbers, calibration information, dilution information, and detection limits have been correctly entered. A hard copy printout of all computer-generated data is obtained for data review and verification purposes.
- 10.2.3 Conversion of analyte signal to analyte concentration is performed by comparison to a calibration standard or calibration curve. The resulting concentration must be corrected for digestion, extraction, dilution, and/or concentration before analysis.
- 10.2.4 All soil sample results will be reported on a dry weight basis. The percent moisture used to calculate the correction factor will be determined using a separate portion of soil from the sample container (Method CL-071R Dry Weight for Solids).

10.3 DATA REPORTING

- 10.3.1 Final analytical results are clearly annotated on the analytical data or data summary. An analysis report is prepared using the final results from associated analytical data. Where applicable, the following information should be included on the analysis report:
- Requestor information.
 - Sample information.
 - Project description.
 - Sample description including matrix.
 - Unique sample identifier (see Section 7.1).
 - Name of sample collector.
 - Sample collection date.
 - Sample receipt date.
 - Analytical results.
 - Analyte.
 - Results (indicate < RL if none detected).
 - Round results to 2 significant digits.
 - Units of measure (ug/L, mg/kg, mg/m³, etc.).
 - RL.
 - Method used.
 - Date analyzed.
 - Data qualifiers, if any (such as J for estimated value).
 - Approval signatures.

10.3.2 A case narrative is prepared for each data package which includes a detailed explanation of any QC exceptions noted, any confirmation analyses performed, any variations of the method required, and any other information that would assist a client or regulator in understanding how the analysis was performed and why the analyst considers the results acceptable.

10.3.3 Key analytical information is assembled in an analytical file for easy retrieval and review. Data packages should include the following, where applicable:

- Case Narrative.
- A copy of the Analysis Report.
- COC/Analysis Request form.
- Relevant sample collection information.
- Other sample-related information.
- Analytical summaries.
- A QC summary.
- A copy of run log or sequence summary.
- The calibration curve raw data (including chromatograms).
- A graph of the calibration curve.
- The raw data for samples and QC samples (including chromatograms).
- A photocopy or reference to standard preparation logbook page(s).
- A photocopy or reference to reagent preparation logbook page(s).
- A photocopy or reference to other applicable laboratory records.
- Other associated analytical information.

10.3.4 Analysts should review their own data packages for completeness and accuracy. When the data packages are complete, the analysts signs and dates the final report and submits the packages for peer review (see Section 10.4).

10.4 QC REVIEW

10.4.1 100% of the assembled analytical files are submitted to QA/QC personnel for QC review. During this review, the reviewer checks the data packages for completeness and ensures that the resulting data comply with method requirements. Data package requirements are detailed in Section 10.3. Analytical and QC requirements are detailed in this QA Plan as well as the sample collection (see Table 3) and analytical operating procedures.

10.4.2 QC reviewers should ensure that all documentation is complete and that all analytical and QC requirements have been met, specifically that:

- COC and other documentation was accurate and complete.
- Samples were collected in the proper containers, with correct preservation, and delivered to the laboratory under the proper conditions (see Section 7.1).
- Analysts have documented method and QAPP training.
- Correct analytical methods were used.
- Detection limits are lower than the Action Levels.
- Samples were run within the holding times specified in Table 5.
- Calculations were correctly performed.
- Instruments were properly calibrated.
- QC samples were properly run at the proper frequency.

- QC results were within method-specified limits
- The potential for matrix interferences, peak misidentification, co-eluting peaks, or other potential problems has been considered
- All positive chemical agent peaks have been confirmed or overruled properly.

10.4.3 If problems such as incomplete data, unsigned reports, failing QC, etc. are discovered during peer review, corrective action should be taken as described in Section 12. When all items are acceptable, the peer reviewer signs and dates the final report and submits the data package for QC review (see Section 10.5).

10.5 FINAL APPROVAL

10.5.1 Data packages that have been QC reviewed are submitted to the Environmental Laboratory Supervisor, or designee, for final approval.

10.5.2 The Environmental Laboratory Supervisor should ensure that the data package is complete, the final report is complete, and required signatures have been obtained, and:

- Ensure that sample dates, analysis dates, analytical results, etc. are sensible and reasonable (e.g., analysis dates are after sample dates).
- Compares results with those of previously analyzed similar samples.
- Compares QC data, as necessary, with historical data using control charts or method defined control limits or other means (see Section 11.1).

10.5.3 As necessary, the QC reviewer may independently verify the data package documentation, as well as the sampling, analytical, and QC procedures used. If problems are discovered during QC review, corrective action should be taken as described in Section 12. When all items are acceptable, the QC reviewer signs and dates the final report. The original report is submitted to the requester. A copy of the signed report is placed with the data package and filed in a secure location (see Section 4.5).

10.5.4 Issued Analysis Reports will remain unchanged. Amendments to an Analysis Report after issuance are made only in the form of a further document that clearly states *AMENDED REPORT*. A cover letter should indicate the date and purpose of the amendment and be signed by all original signatories to the original report.

10.6 DATA VALIDATION

10.6.1 At least 10% of the chemical agent-related waste analyses are independently validated. This is accomplished by validating every tenth hazardous waste sample. Validation is performed under the direction of DEP personnel.

10.6.2 During the validation process, analytical records are checked for completeness as well as compliance with this QAPP and applicable methods. Validation personnel will ensure that all computer calculations and manipulations are appropriate and correct. In addition to those items listed under QC Review (Section 10.4) and Final Approval (Section 10.5), the data validator should ensure that:

- The data package is complete and consistent with the original request documentation.

- Sample custody was maintained from the time of sample collection until laboratory sample receipt.
- Sampling and analysis dates on the analytical report are consistent with the field and laboratory documentation.
- Analytical results match those in the raw data.
- Sample dilutions and other manipulations were properly accounted for in the final report.
- QC samples were run at the required frequency and results met method requirements.
- Calculations, computations, and transcriptions leading to the analytical result are correct.
- Calibrations were verifiable and correct.
- Compounds were correctly identified and quantitated.

Items to review in chromatograms include:

- Baseline anomalies such as peak shifts, noise, etc.
- Retention time shifts.
- Extraneous peaks.
- Matrix interferences.
- Peak misidentification.
- Low Resolution.
- Peak anomalies such as shoulders, poor shape, etc.
- Correlation of peaks.

A formal data validation report should be prepared that outlines the reviews performed and the resulting comments or suggestions. Problems discovered during data validation should usually result in formal corrective action as described in Section 12. In cases where the review finding casts doubt on the correctness or validity of reported analytical results, the Environmental Laboratory Supervisor will be notified immediately.

11.0 LABORATORY QUALITY ASSESSMENT

11.0.1 Quality assessment is the process of using internal and external measures to determine the quality of the data produced by the laboratory. Laboratory quality assessment is accomplished using control charts or method defined control limits and proficiency test samples, as well as internal and external audits and reviews. Sections 11.1 and 11.2 describe the use of control charts and method defined control limits and proficiency test samples to assess laboratory performance. Sections 11.3 and 11.4 describe internal and external audits. Section 11.5 describes the management's annual system review.

11.1 CONTROL LIMITS

11.1.1 Control limits derived from control charts or method defined control limits are statistical tools for monitoring the performance of laboratory QC parameters such as CC standards, MBS samples, and MBSD samples. Generally, control limits are used internally to evaluate and improve system quality. Where available, method-defined QC acceptance limits may be used to determine data acceptability for reporting purposes.

11.1.2 Two types of control limits are commonly used in the laboratory: accuracy limits for CC and MBS %R and precision limits for MBS/MBSD RPD. The control limits are set at ± 3 standard deviations from the mean (99 percent confidence limits) for accuracy and precision. When used

to evaluate method performance (see Section 9), control limits are updated at least annually. Control limits also may be based on actual method performance and set by agreement with State regulators.

11.2 PROFICIENCY TEST SAMPLES

11.2.1 The SBCCOM's CASARM QA Team provides a Proficiency Testing Program. DPG participates in all available rounds of this program. QA/QC personnel review proficiency test reports. Corrective actions are undertaken for any missed analytes (see Section 12.0).

11.3 INTERNAL AUDITS

11.3.1 QA/QC personnel perform or arrange for audits to verify that waste-related analytical activities continue to comply with the requirements of the quality system. Persons who are trained and qualified as auditors carry out these audits on at least an annual basis. Auditors must be organizationally independent of the activity to be audited.

11.3.2 During the internal audits, sample collection, handling, analysis, and reporting activities are evaluated according to the requirements of the quality system and methods. Internal quality system audits should include the following areas:

- Sample collection procedures and documentation.
- COC procedures and documentation, including sample identification.
- Laboratory sample receiving procedures and documentation.
- Analytical procedures and documentation, including sample preparation, instrument calibration, and data reduction.
- QC procedures and documentation.
- Data review procedures.
- Method validation for any new procedures.
- Sample storage.
- Data package preparation and reporting procedures.
- Standard preparation and traceability.

11.3.3 The goal of the audit is to detect any deviations from acceptable practices and procedures so that corrective action can be taken. When an audit finding casts doubt upon the correctness or validity of any test results, the laboratory will take immediate corrective action and immediately notify any client whose work may have been affected. Audit-related findings will be addressed through the corrective action system (Section 12.0).

11.4 EXTERNAL AUDITS

11.4.1 From time to time, data users (such as DEP and other sample requesters) and regulators (such as the State of Utah DSHW) will desire to audit the chemical agent-related laboratory activities at CCTF. The laboratory will cooperate, to the fullest extent possible, in assisting with these audits.

11.4.2 All audit-related activities will be coordinated through the Environmental Laboratory Supervisor. While in the laboratory, auditors will be accompanied by CCTF staff to maintain confidentiality and security. Audit-related findings will be addressed through the corrective action system (Section 12.0).

11.5 MANAGEMENT REVIEW

11.5.1 The Environmental Laboratory Supervisor will lead and coordinate an annual management review and evaluation of this QAPP to verify its suitability and effectiveness. The review team will include the Environmental Supervisor and the QA/QC personnel, as well as management representatives from environmental and laboratory management. Results of the review will be documented. Changes implemented based upon the review will be documented and verified.

11.5.2 The management review will include, but not be limited to, the following:

- Review and evaluation of the records of internal and external audits of the laboratory quality system
- Evaluation of external influences such as additional work, new technology, changing or new regulations, organizational changes, etc.
- Evaluation of the adequacy of personnel, facilities, and equipment
- Review of recommended courses of action

11.5.3 QA/QC personnel are responsible for evaluating and responding to the recommendations generated by the management review. Audit-related findings will be addressed through the corrective action system (Section 12.0).

12.0 CORRECTIVE ACTION

12.0.1 The laboratory has a formal system for initiating and implementing corrective action. Corrective action and follow-up are powerful tools for continuous improvement within the laboratory. Specific corrective action procedures depend on the nature of the discrepancy or out-of-control situation. Ultimately, QA/QC personnel are responsible for identifying and correcting systemic quality problems within the laboratory. Individuals working in the laboratory, however, must be familiar with all QC policies and procedures and bring discrepancies to the attention of the QA/QC or management personnel.

12.0.2 For guidance purposes, two types of analytical problems have been identified in Sections 12.1 and 12.2; bench analytical problems and administrative or systemic problems. The chemist or supervisor often will solve bench analytical problems immediately without initiating a formal corrective action report (CAR). Administrative or systematic corrective action usually requires the use of a formal CAR.

12.1 BENCH ANALYTICAL PROBLEMS

12.1.1 Bench analytical problems are those that may occur during sample analysis. These types of errors include failed calibration, failed continuing calibration, failed method spike recovery, etc. Many of these problems can and should be corrected at the time of analysis and do not require external documentation using the CAR.

12.1.2 All laboratory personnel should be aware of the specific QC requirements associated with their analytical responsibilities. Under no circumstances should data be released from the bench unless: (1) All QC results are within acceptable limits, or (2) The suspect data have been clearly qualified as to the nature of the discrepancy, the corrective actions which have been taken, and the results of the corrective actions.

12.1.3 Corrective action is a function of the type or error encountered. Experienced analysts and supervisors should be consulted when trouble-shooting these types of problems. Possible corrective actions for bench analytical problems may include:

- Re-run failed QC sample and/or calibration standards.
- Re-prepare and re-run QC sample and/or calibration standards and field samples.
- Re-prepare and re-run field sample(s) (if feasible) associated with the failed calibration.
- Perform routine instrument maintenance.

12.2 ADMINISTRATIVE OR SYSTEMIC PROBLEMS

12.2.1 Administrative or systemic problems may include errors in sample receipt, holding time, sample preservation, data transcription, data reporting, performance evaluation results, etc. These types of errors are usually discovered during data review, internal audits, or external performance evaluation audits. They may also be brought to the attention of the laboratory by clients (i.e., customer complaints) or external auditors.

12.2.2 Administrative and systemic problems may be very significant and corrective actions must identify the root cause of the problem (insufficient resources, lack of training, no internal checks, etc.) and recommend possible solutions (improve resources, provide training, increase internal checks, etc.). This process is documented using a Corrective Action Form (see Figure 7). Every effort will be made to identify and resolve quality problems in an equitable and timely manner. As part of the corrective action process, QA/QC personnel and laboratory management will review and recommend changes to the QAPP and methods, if necessary, to avoid similar problems in the future. When completed, CARs are signed and maintained by QA/QC personnel.

Figure 7. Example of a Corrective Action Request Form.

CORRECTIVE ACTION REQUEST

Date: _____ Audit No.: _____ CAR #: _____

Dept. /Process under Review: **Command Group**

Responsible Dept. Manager: _____ From Auditor(s): _____

State Requirement(s): *(of ISO standard, Quality Manual, Procedures or Work Instructions)*

Nonconformity Description: *(provide details)*

Audited by: _____ Lead Auditor: _____ Responsible Dept. Mgr. Acknowledgment: _____
Date: _____ Date: _____ Date: _____

Corrective Action/Prevention Plan: Response Due Date: _____ Implementation Due Date: _____
(Include root cause and means to evaluate effectiveness.)

Proposed by: _____ Date: _____ Approved by: _____ Date: _____

Follow-Up Verification: Not Required Required Corrective action is implemented and effective.
Verification Observations:

Signature: _____ Title: _____ Closed Date: _____

13.0 ACRONYMS AND DEFINITIONS

°C - degree(s) Celsius

Calibration Check Standard - Analytical standard run in a specified sequence or time interval to verify that the calibration of the analytical system remains in control.

CAR - Corrective Action Report

CASARM - Chemical Agent Standard Analytical Reference Materiel

CC - Calibration Check

CCTF - Combined Chemical Test Facility (Buildings 4153, 4156, and 4165)

Chemical Agent - Any of several highly toxic chemical compounds (including CX, GA, GB, GD,H, HD, HL, HN1, HN2, HN3, HT, L, T and VX) that are intended for use in military operations

Cleanliness - The absence of contamination in the laboratory as measured by blanks.

CO - Consent Order

COC - Chain-of-Custody

Coliwasa - Composite Liquid Waste Sampler

Comparability - The degree an analysis performed by one laboratory agrees with an analysis performed on a similar sample by another laboratory.

Completeness - The degree to which an analysis or batch of analyses has met all other DQO.

Controlled Document - A document that is issued to personnel with a document tracking number.

CPO - Civilian Personnel Office

CRD - Compliance and Restoration Division

CX – Phosgene Oxime, CAS 1794-86-1

DAAMS - Depot Area Air Monitoring System

Data Package - A set of records describing the complete history of a defined set of events (records) pertaining to a single laboratory sample lot.

Data Quality Objectives (DQO) - Standards which the laboratory strives to maintain. They establish a goal or benchmark for laboratory performance.

Data Validation - An independent evaluation of an analyses' adherence to the analytical methods and QA procedures.

Decontamination - The process of decreasing the amount of chemical agent on any person, object, or area by absorbing, neutralizing, destroying, ventilating, or removing chemical agents.

DEP - Directorate of Environmental Programs

Depot Area Air Monitoring System (DAAMS) - Various solid sorbent tubes used on DPG to collect safety air monitoring samples from the headspace surrounding solids

Dilution Factor - The volume-to-volume ratio of a sample extract to a dilution of that extract which is analyzed

DPG - U.S. Army Dugway Proving Ground

DQO - Data Quality Objectives

DSHW - Division of Solid and Hazardous Waste

EPA - U. S. Environmental Protection Agency

°F - degree(s) Fahrenheit

Field Duplicate - Duplicate samples collected in the field to establish the overall precision of the sampling and analytical process. Duplicates are handled like routine samples in the laboratory.

Field QC Samples - Samples that provide a measure of the quality of the sampling activities.

Field Sample Lot - Twenty or fewer samples collected from the same waste description at one time (shift) by a single team of Sampling Personnel. Each field sample lot for liquid is accompanied by field QC samples including an Field Duplicate and an equipment Rinse Blank when using non-disposable equipment.

Field Spike Sample - See QP Sample

GA - Tabun: Ethyl N,N-dimethylphosphoramidocyanidate, Chemical Abstracts Service (CAS) 77-81-6, a nerve agent

GB - Sarin: Isopropyl Methylphosphonofluoridate, CAS 107-44-8, a nerve agent

GC - Gas Chromatography

GD - Soman: Pinacolyl Methylphosphonofluoridate, CAS 96-64-0, a nerve agent

GF - Cyclohexyl Methylphosphonofluoridate, CAS 329-99-7, a nerve agent

H – Mustard, Bis-(2-chloroethyl) sulfide, CAS 505-60-2

HD - Mustard, Distilled: Bis-(2-chloroethyl) sulfide, CAS 505-60-2, a blister agent

HL - Mustard/Lewisite mixture

HN1 – bis-(2-chloroethyl)ethylamine, CAS 538-07-8 – Nitrogen Mustard

HN2 - bis-(2-chloroethyl)methylamine, CAS 571-75-2 – Nitrogen Mustard

HN3 - tris-(2-chloroethyl)amine, CAS 555-77-1 - Nitrogen Mustard

HT – Mustard/T

IDW - Investigation Derived Waste

Initial Calibration Verification Standard - A standard material, prepared independently from the calibration standards, which is used to verify a new set of calibration standards

IRP - Installation Restoration Program

Issuing - Distributing and controlling master copies of controlled documents

Laboratory Sample Lot - A laboratory sample lot consists of 20 or fewer samples. It is the maximum number of samples, up to 20, that can be manually processed through the method during a single time period, not to exceed 24 hours.

Lewisite - 2-chlorovinyl dichloroarsine, CAS 541-25-3

LOD - Limit of Detection

Lot Number - Each laboratory sample lot receives a unique lot number for data tracking purposes. Lot numbers are assigned sequentially at the time a laboratory sample lot is established.

Matrix Spike - Positive control prepared in the laboratory to establish that the overall analytical system is performing within expected tolerances with respect to the analytical system's ability to accurately measure target concentrations in the absence of undue matrix effects.

Matrix Spike Duplicate - Positive control prepared in the laboratory to establish that the overall analytical system is performing within expected tolerances with respect to the analytical system's ability to precisely measure target concentrations in the absence of undue matrix effects.

MB - Method Blank

MBS - Method Blank Spike

MBSD - Method Blank Spike Duplicate

MDL - Method Detection Limit

Method Blank - Negative control prepared in the laboratory to establish that the overall analytical system is not causing significant interference with target analyte detection and quantitation

Method Blank Spike - Positive control prepared in the laboratory to establish that the overall analytical system is performing within expected tolerances with respect to the analytical system's ability to accurately measure target concentrations in the absence of undue matrix effects.

Method Blank Spike Duplicate - Positive control prepared in the laboratory to establish that the overall

analytical system is performing within expected tolerances with respect to the analytical system's ability to precisely measure target concentrations in the absence of undue matrix effects.

Method Detection Limit (MDL) - Estimate of the lowest level of an analyte that a method can distinguish from noise.

mg/kg – milligrams per kilogram

mg/L – milligrams per liter

mg/m³ - milligrams per cubic meter

MINICAMS[®] - Miniature Continuous Air Monitoring System

MS - Mass Spectroscopy or Matrix Spike

NA - Not Applicable

Performance Evaluation - The analysis of blind samples that are usually part of a study or performance of a group.

Precision - A measure of an analytical system's agreement between duplicate measurements of the same material. Precision is stated as relative percent difference (RPD). When associated with replicate precision determinations on the same material, precision may be stated as mean D and a confidence level.

QA - Quality Assurance

QAPP - Quality Assurance Program Plan

QC - Quality Control

Quality Assurance (QA) - The overall system of planning, QC, and management activities, which assure quality.

Quality Control (QC) - The specific activities designed to measure quality, including check samples, check sample assessment, audits, reports to management, etc.

Quality Control (QC) Standard - Used as a CC standard. A standard, prepared at the HL concentration, which verifies the analytical system is operating as designed and is capable of detecting and quantifying chemical agent at the required concentrations.

Quality Laboratory (QL) Standard - A QC sample used to verify the initial calibration. QLs are prepared in the laboratory by spiking unexposed DAAMS tubes with a solution of dilute chemical agent and, if necessary, aspirating with laboratory air to remove solvent. QLs are not aspirated with installation air.

Quality Plant (QP) Sample - A quality control (QC) sample used to establish method accuracy and precision. QPs are prepared (in duplicate) in the laboratory by spiking unexposed DAAMS tubes with a solution of dilute chemical agent and, if necessary aspirating with laboratory air to remove residual solvent. QPs are sent into the field with the sample tubes and aspirated with background air.

R – Range

%R - Percent Recovery

RCRA - Resource Conservation and Recovery Act

Receiver - During a transfer of custody, the person who is accepting custody of the sample

Recording - Assigning and documenting method numbers and method revision numbers

Relinquisher - During a transfer of custody, the person who is relieved of the sample custody

Reporting Limit - Lowest reportable analyte concentration for a particular sample, usually a factor of 2 to 20 times the MDL

Representativeness - The degree the sample analyzed represents the waste from which it was derived, as measured by field duplicates.

Rinse Blank - A sample collected in the field to demonstrate that no cross-contamination has occurred during sampling. For liquid and soil samples, one rinse blank per field sample lot is needed when non-disposable sampling equipment is used. Rinse blanks are not required when sampling equipment is used.

RL - Reporting Limit

RPD - Relative Percent Difference

RSD - Relative Standard Deviation

Sample Collection Lot - Twenty or fewer samples collected from the same waste description at one time (shift) by a single team of sampling personnel.

Sample File - Sample collection information associated with a single sample or a sequential group of samples that share the same sample collection information. The sample file consists of an Analysis Report, COC/Analysis Request form, Login Checklist, etc.

SBCCOM - Soldier and Biological Chemical Command

Sequence - The order of standards and samples in the analytical run

Significant Figures - The number of digits required to express the uncertainty of reported data. The following digits are always significant: 1) the non-zero numbers, 2) zeroes between non-zero numbers, 3) zeroes which are to the right of the decimal point and at the end of the number, and 4) zeroes which are to the left of a written decimal point when the number is ≥ 10 .

Submitting Laboratory - Any laboratory generating labware for submission to the washroom

Support Services Personnel - Personnel responsible for recording and issuing controlled documents

SW-846 - EPA Test Methods for Evaluating Solid Waste

T - bis[2-(2-chloroethylthio)ethyl]ether, CAS 63918-89-8

Technical Personnel - Analytical, support, or management personnel responsible for the subject matter of the document

UV – Ultra Violet

VX - O-ethyl-S-(2-diisopropylaminoethyl) methylphosphonothiolate, CAS 50782-69-9, a persistent nerve agent

WAP - Waste Analysis Plan

WDTC - West Desert Test Center

WPL – Worker Population Limit