LABORATORY ACCOUNTABILITY, STORAGE, AND HAZARD COMMUNICATIONS PROCEDURES

FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION

CHEMISTRY PROGRAM

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Appendix A

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List of Acronyms

AM	Agent Manager
AR	Army Regulation
CA	Chemical Agent
CAHO	Chemical Agent Hygiene Officer
CAO	Chemical Agent Operator
CASRM	Chemical Agent Standard Reference Material
CHO	Chemical Hygiene Officer
CHP	Chemical Hygiene Plan
COR	Contract Officer's Representative
CWA	Chemical Warfare Agent
DEP	Department of Environmental Protection
DoD	Department of Defense
DR	Difficulty Report
DSHP	Dilute Solution Hygiene Plan
ECBC	Edgewood Chemical and Biological Center
EPA	Environmental Protection Agency
FDEP	Florida Department of Environmental Protection
IAA	Interagency Agreement
LIMS	Laboratory Information Management System
LLNL	Lawrence Livermore National Laboratory
LM	Laboratory Manager
MSDS	Material Safety Data Sheet
NEMC	National Exposure Measurement Center
OSHA	Occupational Safety and Health Act
PPE	Personnel Protective Equipment
QAO	Quality Assurance Officer
RCRA	Resource Conservation and Recovery Act
RDTE	Research, Development, Test and Evaluation
SRM	Standard Reference Material
UDA	Ultra-Dilute Agent

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1. Scope and Application

The purpose of this SOP is to establish procedures that will be used at the Florida Department of Environmental Protection (FDEP) Central Laboratory to track the preparation, use, and disposal of ultra-dilute Chemical Warfare Agent (CWA) solutions. Because of the need for strict accountability, an electronic tracking system integrated within our Laboratory Information Management System (LIMS) is used to maintain separate accounting for each of the dilute agent solutions.

The dilute solution chemical agent tracking system is designed to ensure accountability of dilute agent solutions from receipt to disposal.

1.1 Chemical Agent Concentrations, Volumes and Amounts

This SOP describes procedures involving standards at the ultra-dilute level as defined in Table 6-1 of AR 50-6 provided by the Edgewood Chemical and Biological Center (ECBC). The definition and limits of ultra-dilute CWA solutions are provided in an Interagency Agreement (IAA) between EPA and DoD dated November 22, 2006. Table 1 summarizes the dilute agent levels.

Table 1. Maximum Concentrations, Volumes, and Amounts for Chemical Agent Solutions

Ultra-Dilute CWA Thresholds		
Agent	**Maximum Concentration[a]**	**Agent Total Mass[b]**
Sarin (GB)	10 mg/L	100 µg
Soman (GD)	10 mg/L	100 µg
Cyclohexyl Sarin (GF)	10 mg/L	100 µg
VX	10 mg/L	100 µg
Sulfur Mustard (HD)	10 mg/L	100 µg
Lewisite (L)	10 mg/L	100 µg
[a] Concentrations are aggregate. If more than one agent is in a single solution, the sum of their concentration is used to determine the classification. [b] Not to exceed 10 mL total volume separated in 1 mL vials.		

Furthermore, the purpose of this SOP is to establish minimum requirements for storage and labeling of dilute chemical agent solutions and to apprise personnel of the hazards of the chemicals in their work area at the time of their initial assignment and whenever a new hazard is introduced. The UDA laboratory augments the general laboratory Hazard Communication Program with several additional measures to underscore hazards unique to the UDA laboratory operations.

This SOP applies to all laboratory personnel, support staff, and all visitors to the laboratory. The communication functions apply to all Ultra-dilute CWA solutions.

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1.2 Applicable FDEP Preparation and Analysis Tests

Water Matrix Tests:

- W-CW-SQFS – Extraction and analysis of CWAs in a water matrix by SOP ERLN-002. Analysis is performed by GC/MS in full-scan mode.
- W-CW-SQSM – Extraction and analysis of CWAs in a water matrix by SOP ERLN-002. Analysis is performed by GC/MS in selected-ion monitoring (SIM) mode.
- W-CW-TF1D – Extraction and analysis of CWAs in a water matrix by SOP ERLN-002. Analysis is performed in full-scan mode using a Time-of-Flight mass spectrometer.
- W-CW-TF2D – Extraction and analysis of CWAs in a water matrix by SOP ERLN-002. Analysis is performed in 2-D full-scan mode using a Time-of-Flight mass spectrometer.

Soil Matrix Tests:

- S-CW-SQFS – Extraction and analysis of CWAs in a soil matrix by SOP ERLN-002. Analysis is performed by GC/MS in full-scan mode.
- S-CW-SQSM – Extraction and analysis of CWAs in a soil matrix by SOP ERLN-002. Analysis is performed by GC/MS in selected-ion monitoring (SIM) mode.
- S-CW-TF1D – Extraction and analysis of CWAs in a soil matrix by SOP ERLN-002. Analysis is performed in full-scan mode using a Time-of-Flight mass spectrometer.
- S-CW-TF2D – Extraction and analysis of CWAs in a soil matrix by SOP ERLN-002. Analysis is performed in 2-D full-scan mode using a Time-of-Flight mass spectrometer.

Cloth Wipe Matrix Tests:

- SA-CW-SQFS – Extraction and analysis of CWAs in a wipe matrix by SOP ERLN-002. Analysis is performed by GC/MS in full-scan mode.
- SA-CW-SQSM – Extraction and analysis of CWAs in a wipe matrix by SOP ERLN-002. Analysis is performed by GC/MS in selected-ion monitoring (SIM) mode.
- SA-CW-TF1D – Extraction and analysis of CWAs in a wipe matrix by SOP ERLN-002. Analysis is performed in full-scan mode using a Time-of-Flight mass spectrometer.
- SA-CW-TF2D – Extraction and analysis of CWAs in a wipe matrix by SOP ERLN-002. Analysis is performed in 2-D full-scan mode using a Time-of-Flight mass spectrometer.

1.3 Supporting Standard Operating Procedures (SOPs)

Refer to the following Standard Operating Procedures (SOPs) for additional procedural information with regard to sample custody, safety, analytical methods, and non-conformance reporting. These SOPs contain critical details not fully described within this Accountability SOP. The chemical agent operators must be completely familiar with all of these SOPs.

1.3.1 Florida Department of Environmental Protection, *Health and Safety Plan*.
1.3.2 Florida Department of Environmental Protection, *Building Security Plan*.
1.3.3 Florida Department of Environmental Protection, *Standard Operating Procedure for Handling Perceived Threat Materials* (SOP **CM-017**)

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1.3.4 Florida Department of Environmental Protection, *Standard Operating Procedure for Receiving Packages and Freight at the Loading Dock (SOP* **RC-017**).
1.3.5 Florida Department of Environmental Protection, *Chemistry Section Receiving Room Standard Operating Procedure for the Receipt and Log-in of Samples by the DEP Central laboratory* (SOP **RC-007**).
1.3.6 Florida Department of Environmental Protection, *Standard Operating Procedure for Documentary Evidence Chain-of-Custody within the DEP* (SOP **RC-009**).
1.3.7 Florida Department of Environmental Protection, *Standard Analytical Protocol for Extractable Chemical Warfare Agents Using Gas Chromatography/Mass Spectrometry* (SOP **ERLN-002**).
1.3.8 Florida Department of Environmental Protection, *Chemical Agent Laboratory Training Module.*
1.3.9 Florida Department of Environmental Protection*, Non-Conformance Reporting System* (SOP **LB-002**).
1.3.10 Florida Department of Environmental Protection, *Standard Operating Procedure for Records Storage and Retention* (SOP **LB-006**)

2. Responsibilities

The following subsections describe specific accountability, safety, and chemical hygiene responsibilities associated with the FDEP Ultra-dilute agent program. It is the responsibility of all laboratory staff and their managers to know and follow the provisions of this plan. Responsibilities are listed by title. Personnel names, management matrix, and contact information are available through the Laboratory Manager (LM). Please refer to the Laboratory Quality Manual for additional details regarding responsibilities and a list of specific staff assigned to these roles. The following roles and responsibilities are emphasized:

2.1 Laboratory Director

The Laboratory Director is responsible for ensuring that this administrative practice is followed by all users of ultra-dilute CWA solutions and that resources and support are provided for the implementation of this plan and the requirements outlined therein. The following tasks are the responsibility of the Laboratory Director:

- Responsible for the health and safety of personnel,
- Responsible to ensure all recognized hazards are promptly addressed,
- Interact with laboratory management and personnel to ensure that DSHP procedures are understood and followed and assistance or resources are provided as needed, and
- Serve as a back up to the Agent Manager (AM) by holding a back-up key (primary lock and key entry system) to the UDA standard storage refrigerator in case of the AM's absence.

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2.2 Laboratory Manager

The Laboratory Manager (LM) is responsible for the daily operation of the CWA laboratory and daily execution of the Dilute Solution Hygiene Plan as it relates to the laboratory's activities. The LM is responsible for the health and safety of the Chemical Agent Operators (CAOs) during all UDA procedures. The LM also shares responsibilities for development, implementation, review, and support of the DSHP with the Chemical Agent Hygiene Officer (CAHO). The following tasks are the responsibility of the Laboratory Manager:

- Ensure that the Ultra-Dilute Agent laboratory has required safety supplies and equipment necessary to handle or store UDA materials safely,
- Ensure that UDA personnel are familiar with the Dilute Solution Hygiene Plan (DSHP) and routinely follow the requirements and procedures,
- Ensure that safety equipment is checked and ready for use,
- Request and coordinate delivery of UDA reference material from the U.S. Army Edgewood Chemical and Biological Center and Engineering Center (ECBC) with Contracting Officer's Representative (COR) or ECBC Chemical UDA Accountability Officer,
- Document all requests, coordination, and communication concerning delivery of Ultra-Dilute Agents in the Accountability Assessment log,
- Appoint a qualified CAO to perform the dry runs with dilute agents to test Standard Operating Procedures (SOPs) and approve staff readiness,
- Routinely observe CAOs performing UDA SOPs and provide recommendations in conjunction with the CAHO to improve UDA laboratory safety,
- Determine staffing, UDA laboratory access and/or training level needs as necessary and ensure identified training levels are met by the individual,
- Conduct regular UDA inspections of housekeeping, personal protective equipment (PPE), and emergency equipment,
- Ensure that current copies of the CHP, DSHP, procedures, and other pertinent documents are readily accessible for use in the Ultra-Dilute Agent laboratory,
- Ensure that security requirements are met as specified in the Security Plan,
- Perform quarterly accountability audits with the Agent Manager as a formal witness,
- Serve as back-up for the Agent Manager for receipt of Standard Reference Materials (SRMs), and
- Serve as a back-up Agent Manager for other functions.

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2.3 Chemical Agent Hygiene Officer

The Chemical Agent Hygiene Officer (CAHO) is a UDA laboratory specific position and is an extension of the facility Chemical Hygiene Officer (CHO). It may or may not be the actual facility CHO. The CAHO position is held by an individual who has the knowledge and competence to develop and implement this plan, as qualified by appropriate levels of education, training, and experience. The CAHO also must demonstrate the ability to use appropriate equipment and testing procedures to anticipate, identify, and evaluate health, safety, and environmental hazards, as well as the ability to suggest means for reducing those risks. The following tasks are the responsibility of the Chemical Agent Hygiene Officer:

- Development, implementation, review, and technical support of the DSHP in conjunction with the Laboratory Manager,
- Provide technical, environmental, health and safety guidance to the LM in development and implementation of programs and procedures related to the UDA laboratories,
- Advise and assist in the improvement of Ultra-Dilute Agent laboratory safety procedures, and review and update the DSHP on an annual basis as necessary,
- Responsible for the DSHP with full authority to prepare and enforce safety policies,
- Investigate reports or situations of non-conformance with DSHP requirements,
- Prepare and/or approve UDA training materials for required visitor, Chemical Agent Operator (CAO), responder and remediation training levels,
- Provide and/or approve training level certifications for visitors, staff, managers, and others as appropriate for the assigned duties and level of potential exposure,
- Audit UDA laboratory functions for compliance with the DSHP, Occupational Safety and Health (OSHA) regulations, Resource Conservation and Recovery Act (RCRA) regulations and other requirements for laboratory procedures, and
- Determine the level and type of personnel protective equipment (PPE) required for the various UDA procedures, conditions, audits, dry runs, and emergency response for decontamination procedures.

2.4 Agent Manager

The Agent Manager (AM) is responsible to assure the accurate accountability of the UDA agent reference materials, from receipt and storage of the primary materials from ECBC through usage and disposal. The following tasks are the responsibility of the Agent Manager:

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- Serve as the primary contact with the UDA regulatory authorities and is the team leader for compliance surveys, audits, and inspections by EPA, ECBC or other bodies,

- Assure accurate accountability of UDA agent reference materials (primary material as received from ECBC) from receipt through disposal, and is responsible for maintaining the security and integrity of stored and in-use primary UDA solutions at all times,

- Sign courier forms for receipt of CWA material, complete the ECBC chain of custody form, and accompany the UDA solutions until they are secure in the laboratory,

- Perform checks for each working day of accountability records and housekeeping in the UDA laboratories, (Note: A "working day" is defined as any day where UDA materials are removed from secured storage.)

- Prepare monthly agent inventory reports and perform and document quarterly accountability audits,

- Serve as backup for coordinator of UDA primary solution delivery in case the Laboratory Manager is not available, and

- Hold the keys (primary lock and key entry requirement) to the UDA standard storage refrigerator.

2.5 Chemical Agent Operators

A Chemical Agent Operator (CAO) is anyone that works directly with dilute agent solutions or unknown samples potentially containing chemical warfare agents. Chemical Agent Operators must be certified on the SOP under which they will be using CWA dilute solutions. The following tasks are the responsibility of the CAO:

- Perform all UDA operations using only the current and approved SOP for which operator certification has been completed,
- Conduct all operations strictly in accordance with the provisions of the DSHP, SOPs, and all applicable regulations, manuals, and directives. Any improvements or alternations must be formally approved by laboratory management before changes to the original SOP operations or procedures can be implemented,
- Observe receipt of UDA materials at the loading dock, transport of the material to the UDA laboratory, and completion of the initial entry in the UDA Accountability log.
- Document the preparation, transfer, and disposal of dilute agent standards of the Chemical Agent in the LIMS Standard Preparation Module.

- Ensure all chemical solutions are properly labeled and stored; this also includes good housekeeping practices within the work area. Responsible for maintaining the security and integrity of the UDA solutions at all times,

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- Responsible for tracking the current location of all CWA dilute standards they are using. This includes documentation of transfer to other laboratories for analysis or use,
- Responsible for maintaining security for stored and in-use CWA Dilute standards at all times. Hold the numerical combination to the key pad entry system (secondary lock and key entry requirement) on the UDA standard storage refrigerator.
- Receive chemical agent orientation and safety training, and comply with the accountability procedures in addition to responsibilities under the primary CHP.
- Responsible for informing their supervisor of any factors that may compromise the safe operation of the UDA program,
- Report any accidents, incidents, hazardous conditions, or unusual circumstances to the immediate supervisor, LM, AM, or CAHO/CHO. Any event that could potentially cause or resulted in exposure or injury within the designated UDA laboratory areas must be reported.
- Use Personnel Protection Equipment required by the SOPs in the prescribed manner.

2.6 Inventory Witness

A witness is required when auditing the CWA dilute solution inventories. The witness is selected by the Agent Manager from the list of personnel with training appropriate to enter the CWA Dilute Solution Laboratory. The witness must be independent of the dilute agent operations being audited. The physical inventory witness verifies the status of the chemical inventories by signing and dating the inventory logbook.

3. Ultra-Dilute Agent Accountability

"Accountability" of the primary and non-primary CWA standards means that the lab must be able to secure, track, document, and audit each of the standards from the time of initial receipt until their disposal.

In case of Laboratory Manager's (LM) and Agent Manager's (AM) absence, delivery of UDA reference material is not to be accepted. Also, unannounced, unplanned or unscheduled shipments of UDA reference material will not be accepted.

If any UDA shipment container is discovered to be damaged or leaking during inspection it must be immediately placed inside one of the CWA hoods and the CAHO and LM are to be notified immediately.

Log-in is the procedure by which submitted UDA materials are introduced into the laboratory. The documentation recorded during log-in of reference materials includes: agent name and ID number, origin and quantity (concentration and volume per vial and number of vials). Upon receipt, the accompanying documentation (chain-of-custody sheets and certificates),

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preservation, and shipping conditions are documented by electronic entry into the LIMS Standard Preparation module.

Both the AM and CAOs must be present to access material in the primary UDA refrigerator. This material is tracked in the UDA Accountability records maintained in the LIMS Standard Preparation Module.

4. Standard and Sample Descriptions

The general analytical standard information provided in this section is designed to give Accountability requirements and not technical procedural instruction. Please refer to the Analytical Method SOPs for handling instructions.

4.1 Primary Standards

A primary standard is the first dilution from Chemical Agent Standard Reference Material (CASRM) chemical agent. This would be the ultra-dilute CWA solution supplied by the Army or Lawrence Livermore National Laboratory (LLNL). As a primary standard, the purity, source, and expiration date should be provided by the supplier. All primary standards are permanently recorded and tracked in the LIMS on a microliter level.

4.2 Non-Primary Standards

A non-primary standard is a second or further dilution from the primary standard solution. This solution may be designated as a Secondary or Working standard. The non-primary standards are permanently recorded and tracked in the LIMS on a microliter level.

4.3 Spikes

Every sample that is created for analysis in the Laboratory (such as Laboratory Fortified Blanks or Matrix Spikes) must be accounted for using Non-primary Standards accountability protocol. The spikes must be entered into the LIMS in the same fashion as the secondary and working standards in Section 4.2.

4.4 Field Samples

Field samples received for CWA analysis are not tracked using this SOP, but must be evident in the laboratory records. Procedures for the receipt and log-in of field samples at the FDEP Central laboratories are provided in DEP SOP **RC-007** "Chemistry Section Receiving Room Standard Operating Procedure for the Receipt and Log-in of Samples by the DEP Central laboratory." For chain-of-custody protocols refer to DEP SOP **RC-009** "Standard Operating Procedure for Documentary Evidence Chain-of-Custody within The DEP Laboratory." As well as **RF-005** "State of Florida Department of Environmental Protection Chain-of-Custody Form"

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4.5 Handling Sample Guidelines

Before beginning any procedures on samples with unknown characteristics or hazards, refer to the Florida Department of Environmental Protection SOP **CM-017** "Standard Operating Procedure for Handling Perceived Threat Materials" for general safety precautions and required guidelines. In addition, the following special safety procedures apply to UDA materials.

4.5.1 Receiving Shipping Containers:

All UDA reference materials shipping containers are to be handled with caution. The UDA materials are hazardous. They can cause non-lethal chemical burns and nervous system disorders. Upon arrival, all shipping containers must be inspected at the loading dock prior to entry into The DEP Central Laboratory. Refer to DEP SOP **RC-012** "Standard Operating Procedure for Receiving Packages and Freight at the Loading Dock". During the inspection of each shipping container, if any of the containers have been damaged or are leaking, they are to be placed promptly in one of the CWA hoods. The CAHO and LM are to be notified immediately.

4.5.2 Moving UDA Material outside of the UDA Laboratory:

The handling and use of UDA material (other than receipt of the samples) is to be restricted to the UDA laboratory. UDA reference material moved outside of the UDA laboratory must be in triple containment at all times.

4.5.3 Storage of UDA Standards:

Each UDA agent must be stored separately in secondary containment in locked refrigerators (plastic containers with lids within trays on each shelf). Primary UDA glass vials are to be stored in the designated UDA refrigerator in closed secondary containers within trays on each shelf.

4.5.4 "Buddy System" Procedures:

All sample receipt procedures and UDA operations are to use the "buddy system" with a second person, trained in the CWA handling procedures. The second person must observe the procedures from a safe location, but still within eyesight. Under no circumstance should laboratory staff work with CWA samples or standards alone.

4.5.5 Before the Work Begins:

Refer to Appendix A for a safety checklist. Prior to beginning any laboratory activities involving an agent, operators must:

- Verify that the passage to all first aid/safety equipment (fire extinguishers, showers, eyewash stations, and emergency exits) is unobstructed,

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- Ensure that the first aid kit is properly stocked, including an appropriate number of nerve agent antidote kits,
- Verify that there is an appropriate quantity of personnel decontamination solution (liquid soap and tap water) and sponges for personnel decontamination,
- Verify that an adequate quantity of spill decontamination materials (5-6% commercial bleach or prepared from granular chlorine and waste container absorbent lab paper/absorbent mats) are available,
- Ensure that sink drain stoppers are in place, and catch pools or drains for emergency showers are available, and
- Ensure that waste disposal containers are available.

In addition, the CAHO shall conduct spot checks to reinforce that the safety equipment listed above is utilized in accordance with this procedure.

4.5.6 Opening UDA Glass Ampules:

UDA glass ampules shall only be opened in UDA laboratory fume hoods. When opened, UDA vials shall be supported by a vial rack or other physical support to prevent tipping. At no time should the CAO's face/head break the plane of the hood sash while handling UDAs.

4.5.7 In Case of Direct Exposure:

If there is suspected or actual direct contact or exposure to UDA, wash the affected area with liquid soap and rinse with tap water. This wash and rinse cycle is to be repeated three times using the sink or safety shower. An eye wash should also be available.

4.5.8 Labels on Agent Vials:

Labels on agent vials must not be removed or defaced until the container is empty, decontaminated and ready for disposal. If labels are accidentally removed, this is to be noted in the UDA Accountability Notebook and the AM notified.

5. Records and Notebooks

All of the CWA standards and reagents received into the laboratory must be recorded into the LIMS Standard Preparation module. Subsequent standard preparation procedures using the primary or secondary standards must also be recorded in an auditable electronic form. CWA dilute solution users are required to complete the appropriate records for all the reagents they use. This recordkeeping must be completed by the end of each day of use. The FDEP Laboratory Information Management System (LIMS) is used to enter, track, and permanently record all of the data entries. The LIMS maintains separate records for each Primary and Secondary solution.

5.1. CWA Dilute Solution Primary Standard Record

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The following information must be entered into the LIMS for each Primary Standard.

- Primary Solution ID
- Agent Name
- Concentration
- Solvent
- Date of Receipt
- Storage Location
- Storage Conditions
- Usage of the Primary Standard to Prepare Secondary or Working Standards
  - Date of Operation
  - Operation Type
  - Amount Only Used to Perform Dilution
  - Total Used, including Amount used for Dilution and Waste (to nearest microliter)
  - Remaining Amount
  - Diluted Solution ID
  - Storage Location of new Secondary or Working Standard
  - Operator Approval (Name)
  - Custodian Approval (Name)
- Expiration Date
- Disposal of Primary Solution
  - Disposal Date
  - Disposal Operator Approval (Name)
  - Disposal Custodian Approval (Name)

5.2. CWA Dilute Solution Non-Primary Standard Record

The following information must be entered into the LIMS for each Secondary, Working or Spike Standard.

- Preparation Date of Secondary, Working, or Spike Standard
- Primary Solution ID
- New Secondary or Working Solution ID
- Chemical Agent Name (s)
- Concentration(s)
- Solvent Used
- Volume of Non-Primary Solution
- Storage Location
- Storage Conditions
- Expiration Date
- Operator Approval (Name)
- Custodian Approval (Name)
- Disposal of Secondary, Working, or Spike Solution

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- Disposal Date
- Disposal Operator Approval (Name)
- Disposal Custodian Approval (Name)

5.3 Preparation of a Dilution from a Primary Standard:

The original UDA solutions received by the laboratory may require dilution in order to be used. This section describes the first dilution performed on the original UDA solutions.

- A CAO removes the Primary Standard from storage and performs the necessary operations using the appropriate SOP.
- Agent information includes: preparation date, solution ID, agent present, concentration, solvent type, expiration date, operation type, amount used, amount remaining, new standard solution ID, and storage location of new solution. All of this information must be entered and recorded into the LIMS Standard Preparation module.
- The Dilute Primary Standard Record is started with receipt of the Primary Standard from ECBC or LLNL. A separate CWA Dilute Solution Primary Standard Record is generated for each stock standard solution.
- When the balance remaining in the container is determined, this number will be recorded in the appropriate Primary Standard LIMS record.
- The container is returned to its designated storage when finished.
- The Agent Manager must provide custodial approval of the entered information concerning Primary Standard usage. This approval is used as verification that the record is complete and accurate.
- The operator must complete the LIMS Primary Standard Record. An ID number is automatically generated once a LIMS standard record is complete. The Primary Standard ID must be printed on a label from the LIMS and placed on each standard solution.
- All further uses of the Primary Standard are tracked within the LIMS.
- Note: Primary standard usage will be recorded to the nearest µL (0.001 mL). Usage that is measured in the fractions of a µL (e.g., 0.2 µL) will be recorded at the next highest whole µL.

5.4 Preparation of a Dilution from a Non-Primary Standard:

Most working standards will be prepared directly from the original primary standards as described in section 5.3. However, if it becomes necessary to prepare a working solution from a secondary stock solution, then the following procedure is to be used.

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- A CAO removes the non-primary standard from storage and performs the necessary operations using the appropriate SOP.
- Agent information includes: preparation date, original solution ID, new solution ID, agents present, concentration of agent(s), solvent used, initial volume (mL), expiration date, storage location of new solution, and operator name. All of this information must be entered and recorded into the LIMS Standard Preparation module.
- A separate Dilute Standard Tracking Record is generated for each standard prepared.
- The original container is then returned to storage.
- Disposal of the non-primary standards is tracked in the LIMS standard preparation module.
- Custodial approval is not required for non-primary standards.
- An ID number is automatically generated once a LIMS standard record is complete. The standard ID must be printed on a label from the LIMS and placed on each standard solution.

5.5 Removal of a Primary or Non-Primary Standard From Storage:

- When the standard is removed from the storage location, but is still in the same laboratory complex, the move is called “checkout” and is recorded in the LIMS Standard tracker. Information recorded in the LIMS standard tracker includes Standard ID, location, the reason for checkout, name of person performing checkout, date of checkout, name of person performing check-in, and check-in date.
- When the standard is to be permanently moved to another laboratory complex the move is called “transfer” and the appropriate Dilute Standard Tracking Worksheet row must be moved to the new storage location’s worksheet. All transfers within the laboratory require secondary containment.
- Unique identification numbers are given to each primary and subsequent solution.

6. Chemical Agent Inventory

A blind inventory of all primary UDA solutions must be performed on a quarterly basis. The inventory is recorded in a designated “Inventory logbook”, providing a line item name, description (agent type), volume (of primary solutions), location, signature of the person performing inventory, and a witness signature. The blind inventory must be performed without prior knowledge of the recorded inventory. Thus, the inventory document must not include any information about the number of containers, custody numbers, or volumes prior to opening the storage unit containing the Chemical Agents. The Agent Manager will physically remove all agent containers from their storage locations and the witness will observe and record the volumes of the primary standards with solution IDs and the number of vials of non-primary standards on the inventory sheet. The volumes are measured based on permanent graduations on the vials, but

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if there is uncertainty about the precise volume then a syringe may be used to draw-up the reagent to determine the volume.

The physical inventory is corroborated and certified by the Agent Manager to verify information in the UDA Primary Standard and Dilute Standard Tracking Records. Any discrepancies in the actual inventory and records must be rectified. Any agent shortages must be reported to the Laboratory Manager. Notification to the Laboratory Manager should be using the Difficulty Reporting System (DRS). The DRS report will, in turn, be submitted to the Contract Office's Representative.

7. Chemical Agent Reports

The Agent Manager prepares a Quarterly UDA Standard Summary of neat agent equivalents and number of vials present at the FDEP Central Laboratory and a semi-annual Dilute Solution Standard Report. The AM will include these reports in the appropriate agent consumption report to the COR (Contract Office's Representative). The reports will include information on all UDA solutions and separately, all ultra-dilute agent solutions listed in Table 1.

7.1 Quarterly UDA Standard Summary

The primary tool for accomplishing the Quarterly UDA Standard Summary is the LIMS Standard Preparation module, which contains all the pertinent information as provided in the "CA Dilute Solutions Primary Standard Record" (Figure 1) and the "CA Dilute Standard Tracking Worksheets" (Figure 2) in Appendix A. These records include information about the preparation, use, tracking, and disposal of all solutions at or below Dilute Agent Solution thresholds, as defined in Table 1.

The AM will prepare separate summary reports for the Ultra-Dilute CWA Solution Consumption/Inventory on a quarterly basis. The summaries will include neat agent equivalents and number of ultra-dilute CWA solution vials present at the FDEP facility at that time. Quarterly UDA Solution Reports will be completed by the Agent Manager (AM) by the second day of the following month. The AM will include the quarterly UDA Solution summary in the appropriate quarterly agent consumption/inventory reports to the COR by the fifth day of the following month.

7.2 Semiannual Dilute Solution Standard Report

The semiannual Ultra-Dilute Solution Standard Report, with cut-off dates at close of business (COB) June 30 and December 31, is prepared by the AM by the second day of the following month. The AM will provide the semiannual report to the COR by the fifth day of the following month and follow the guidelines provided by the ECBC Accountability Officer.

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8. Laboratory Audits

An audit of inventory files for the ultra-dilute CWA solutions with concentrations listed in Table 1 will be conducted at least annually by the FDEP Quality Assurance Officer. The audit reports will be provided to the appropriate staff for review and corrective action. The completed report is subsequently routed to the AM, the Laboratory Director, the LM and finally to the QA Officer for final review, approval, and archiving.

9. UDA Solution Destruction

Chemical Agent Operators are authorized to dispose of small quantities of ultra-dilute solutions which are routine waste (e.g., syringe rinses, test item decontamination, etc.). Disposal requires annotation in the LIMS Standard Preparation Tracker.

CAOs are also authorized to dispose of expired standards, non-primary standards, and non-primary working standards. Disposal requires annotation in the LIMS Standard Preparation Tracker. The notebook page must indicate the words “Disposed of by,” the name of the disposer, name of witness, and the date.

10. Storage of Dilute Agents Solutions

10.1 UDA Standard and Sample Refrigerators

Refrigerators used to store UDA standards or unknown samples are equipped with alarms designed to sound when the refrigerator temperature set points are exceeded. Loss of refrigerator temperature control may create a vapor hazard. In a situation where the standards need to be moved to another refrigerator, only trained staff must move the standards. All UDA standards and unknown samples will be stored in double containment. Packaging will be selected to provide containment of dilute solution liquid and vapor. CASARM-certified dilute solutions are stored under refrigeration, or in accordance with instructions supplied by the CASARM Quality Assurance, Quality Directorate or provider. Dilute agent solutions with similar physiological effects should, when possible, be stored as a group, separate from other agents (e.g., nerve agents separated from blister agents). Each UDA agent must be stored separately in secondary containment in locked refrigerators (plastic containers with lids within trays on each shelf).

A record of each entry into the Chemical Agent refrigerator and the daily refrigerator temperature data must be recorded in the Refrigerator Entry & Temperature Log by the Chemical Agent Operator. If the temperature of the UDA materials is outside of the 4±2°C range, the refrigerator is not to be opened and the LM and the CAHO are to be notified. The temperature charts will be changed by the CAOs each week. The charts will be retained in the Refrigerator Entry & Temperature log.

10.2 Storage of Non-CWA Hazardous Materials

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Incompatible hazardous materials must be stored in separate areas, or at least segregated within secondary containment. Labels for all incoming hazardous materials are maintained on the container, and are not removed or defaced until the container is empty or submitted for disposal. It is preferable that labels on secondary (transport and storage) containers be removed when the hazardous material has been removed. Emptied containers are decontaminated and marked as empty.

All hazardous material containers, including waste containers, must be labeled as to content. Containers that are too small to label in this fashion are placed in a secondary container, and the outer container labeled to show the contents of the inner container.

11. Labeling of Containers for CWA Hazardous Materials

All UDA standards, reagents, samples, and waste containers will be clearly labeled identifying their contents. Equipment released for washing will be labeled as 'Decontaminated' prior to removal from the UDA laboratory.

Each inner container and the outer container label for CWA dilute solutions must be distinctive (e.g., borders) from other non-CWA agent labels used in the laboratory, and must be large enough to be easily identifiable. Each inner container and the outer container of dilute solutions and agent candidates must be labeled with its agent and/or code name to properly identify the contents. The label will have a red border (or other distinguishable marking). Containers too small for complete information must have the name or code name (e.g., xGA, xVX, xHD, XL, etc.), and the custody number of the chemical agent clearly marked. The "x" prefix used in conjunction with the agent code name indicates it is an UDA agent solution (for example, xVX or xGB).

Containers with non-primary solutions of the ultra-dilute CWAs are also identified with a different border label and prefix code "UD" to distinguish these from primary UDA dilute solutions.

The mandatory outer container label information for dilute agent solutions includes:

- TOXIC CHEMICAL (in bold red letters),
- The original issue quantity of agent in the container stated in metric terms and the concentration,
- Type of chemical agent involved (name or code name),
- Accountability document number (lab book, unique identification (ID), number for the individual solution),
- The refrigerator ID where the material is stored,
- The name and telephone number of the Agent Manager (AM) of the material,
- The date when the material was first placed in storage,
- Special instructions or notes regarding use or removal of the contents, and

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- Expiration date of the solution.

The current volume of Primary dilute solution is tracked electronically using the CA Dilute Solution Primary Standard Record in the LIMS.

12. Hazard Communication

12.1 Material Safety Data Sheets

Material Safety Data Sheets (MSDSs) from ECBC for all the UDA reference materials and other reagents are available from the CAHO and retained in the MSDS SOP **ERLN-003**. If the chemical composition of a substance produced for use in the facility is known, the hazardous nature of this chemical must be listed on the label. If the composition of a chemical is unknown, it is assumed hazardous. Training in handling chemicals with known and unknown hazards is provided to staff by the CAHO. MSDS that are provided with the material by an outside vendor are provided to staff and kept on file within the laboratory.

12.2 Laboratory Signage

The entrances to Chemical Agent laboratories are posted with signs that indicate the presence of dilute agent(s). Rooms defined as UDA laboratories will bear signage identifying them as a Chemical Agent Laboratory. Only authorized personnel are permitted in these spaces. Access to the CA laboratories will be controlled by locked rooms and key pads.

Refrigerators containing UDA standards or unknown samples will be clearly marked with a sign identifying their contents as hazardous. The refrigerator contents will be protected with a security lock on the UDA refrigerators.

Instrumentation used for the analysis of UDA materials will be identified with signage clearly identifying them as such. The signage will also indicate the current operation type in progress on the instrumentation (e.g. 'UDA Analysis' or 'Clear for Routine' analysis).

The following signs shall be posted in the areas of UDA analyses:

In the hallway directly outside of UDA storage and analyses area/s:

- "Authorized Personnel Only,"
- An egress route from the facility, and
- "Stop" sign – to be posted when UDA material is being utilized.

Inside UDA Laboratories:

- "Emergency procedures for spills, hood failures and power failures: Evacuate Immediately and Notify the CAHO,"
- Information on the symptoms of nerve agent exposure (GB,GD,GF, VX) and immediate treatment,

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- Personnel decontamination procedures,
- Maximum height that the lab hood sash is allowed to be opened,
- Results of monthly lab hood tests (posted outside hood). Hood reports are retained in a log book,
- UDA spill cleanup procedures,
- On Refrigerator (s): "UDA Primary Solutions and Secondary Solutions" and a sign identifying their contents as hazardous, and
- Inside of lab door: "Remove PPE & Wash Your Hands".

12.3 Alarms

In addition to the building emergency/fire alarm, the UDA laboratories utilize equipment which may alarm when certain hazards exist. All fume hoods are equipped with a low flow indicator and a low flow alarm. Each fume hood is tested and certified annually. An engineering control set point is established at the time of the annual certification. If the fume hood flow rate is +/- 10 cubic feet per minute of the control set point, then the fume hood alarm is activated.

12.4 Emergency Intercoms and Phones

The phone intercom or phone system may be used to communicate hazardous conditions to Emergency Personnel. In the event of a medical (non-agent related) emergency, operators will notify the facility by broadcasting the message 'UDA Room #' over the phone intercom as soon as feasible.

In the event of a chemical (agent related) emergency, operators will notify the facility by broadcasting the message 'UDA Chemical Room #' over the intercom as soon as feasible.

12.5 Laboratory Sign-in Boards

The laboratory sign-in boards are used to define more specifically where and what is being done within the laboratory. This information is posted on a dry-erase board or signage at the laboratory entrance. This information is vital in the event of a chemical incident and must be clearly filled out. When multiple activities are occurring sequentially in one laboratory, all activities should be listed initially, and the board should be updated as soon as possible for clarity. The posted information should indicate:

- Specific laboratory fume hood or instrument being used.
- Agent in use.
- General level of concentration (UDA or UDA+unknown).
- Volume of agent.
- Activity being performed.
- Identities of the operator and buddy.
- Date.
- Start time.
- Completion time.
- Safety checklist.

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13. References

13.1 "Accountability Procedures for Ultra-dilute Chemical Agent Solutions and RDTE Dilute solutions." NEMC standard operating procedure, NEMC-09, July 2007 Draft.
13.2 Florida Department of Environmental Protection, *Emergency Action Plan- Bob Martinez Center (BMC),* August 2021.
13.3 Army Regulation 50-6 Chemical Surety.
13.4 Army, Marine Corps, Navy, Air Force "Potential Military Chemical/Biological Agents and Compounds" January 2005.
13.5 "Storage Requirements and Hazard Communication for Chemical Agent Dilute Solution Laboratories" NEMC standard operating procedure, NEMC-06, July 2007 Draft.
13.6 Florida Department of Environmental Protection Comprehensive Quality Assurance Plan, Chemistry Section, Bureau of Labs, 2022.
13.7 Florida Department of Environmental Protection, Standard Operating Procedure **CM-017** *Standard Operating Procedure for Handling Perceived Threat Materials.*
13.8 Florida Department of Environmental Protection, Standard Operating Procedure **RC-017** *Standard Operating Procedure for Receiving Packages and Freight at the Loading Dock*
13.9 Florida Department of Environmental Protection, Standard Operating Procedure **RC-007** *Chemistry Section Receiving Room Standard Operating Procedure for the Receipt and Log-in of Samples by the DEP Central laboratory.*
13.10 Florida Department of Environmental Protection, Standard Operating Procedure **RC-009** *Standard Operating Procedure for Documentary Evidence Chain-of-Custody within the DEP.*
13.11 Florida Department of Environmental Protection, Standard Operating Procedure **LB-002** *Non-Conformance Reporting System*.
13.12 Florida Department of Environmental Protection, Standard Operating Procedure **LB-006** *Standard Operating Procedure for Records Storage and Retention.*
13.13 Florida Department of Environmental Protection, *Chemical Agent Laboratory Training Module, May 2010.*
13.14 Florida Department of Environmental Protection, Standard Operating Procedure **ERLN-002***, Standard Analytical Protocol for Extractable Chemical Warfare Agents Using Gas Chromatography/Mass Spectrometry*

14. SOP Update Revision Summary:

The following revisions were made to this SOP from the prior version:

- Incremented revision number and date.
- Updated references

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Chemical Agent Dilute Solution Safety Checklist

Check	Checklist Item
	Verify that the passage to all first aid/safety equipment (fire extinguishers, showers, eyewash stations, and emergency exits) is unobstructed.
	Ensure that the first aid kit is properly stocked, including an appropriate number of nerve agent antidote kits.
	Verify that there is an appropriate quantity of personnel decontamination solution (liquid soap and tap water) and sponges for personnel decontamination.
	Verify that adequate quantities of spill decontamination materials (5-6% commercial bleach or prepared from granular chlorine and waste container absorbent lab paper/absorbent mats) are available.
	Ensure that sink drain stoppers are in place and catch pools or drains for emergency showers are available.
	Ensure that waste disposal containers are available.
	In addition, the CAHO shall conduct spot checks to reinforce that the safety equipment listed above is utilized in accordance with this procedure.
	Document non-conformance by issuing a Non-Conformance Report (NCR) in the LIMS and notify the client.
	Document corrective actions that may be required. Otherwise, notify the client if an NCR is issued.

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Chemical Agent Dilute Solution Primary Standard Record

Solution ID: _____ Agent: _____ Concentration: _____ Solvent: _____
Storage location: _____ Storage conditions: □ < 4°C □ Other: _____ Expiration date: _____

Line #	Date	Operation Type	Used (mL) round to nearest 0.001 mL				Remaining Amount (mL)	Diluted Solution ID	Transfer Location	Signature	
			Dilution	Other Use	Decon	Total				Operator	DSC Custodian
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											
16											
17											
18											

Disposed of on: _____(date) by: _____(name)

Comments (please refer to line number in comment):

_____

_____

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Chemical Agent Dilute Solution Standard Tracking Worksheet

											Disposal	
Preparation Date	**Source Solution ID**	**New Solution ID**	**Chemical Agent(s)**	**Conc. of Agent(s)**	**Solvent**	**New Solution Initial Volume (mL)**	**New Solution Expiration Date**	**Storage Location**	**Operator**	**Project ID**	**Date**	**Initials**

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