

**CL-044R**

**CHEMICAL AGENT MONITORING**  
(GA, GB, GD, GF, HD, Lewisite, HN1, HN3, and VX)  
USING FIELD MINICAMS®

**Revision: 7**

**Date Effective: September 2017**

**Dugway Proving Ground EPA ID Number: UT3750211259**

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## 1.0 Scope and Application

This method provides procedures for safety air monitoring as well as for chemical agent monitoring in headspace from solid waste samples using the CMS Research Corporation (CMS) miniature automatic continuous air monitoring system (MINICAMS®) at US Army Dugway Proving Ground (DPG). This method may also be used for Resource Conservation and Recovery Act (RCRA)-related air monitoring. MINICAMS® are located in various test areas and in other mobile platforms, such as the DPG Mobile Monitoring Trailer (MMT). This method is applicable to all MINICAMS® used to monitor RCRA-related solid-waste samples and air-monitoring functions.

MINICAMS® Operators must be trained and certified in accordance (IAW) with the DPG air monitoring plans and procedures. In addition, operators analyzing samples for RCRA compliance must be familiar with the overall goals and requirements of the DPG Waste Permit, Attachment 1-10, *Central Hazardous Waste Storage Facility (CHWSF) Quality Assurance Program Plan (QAPP)*. A method schematic is provided in Figure 1.

## 2.0 Scientific Basis

MINICAMS® are rugged, portable gas chromatograph (GC) systems and are a primary tool for monitoring chemical agents. MINICAMS® are designed to operate as continuous monitors, but may be used for discrete monitoring.

Sampling solids for chemical agent consists of containerizing solid waste items and allowing the item to off-gas at minimum temperature for a minimum period of time. Following the off-gassing, the air surrounding the item in the container is sampled using MINICAMS® as described in this method. Monitoring air in RCRA-related capacities involves the use of an appropriate sampling train and detector.

For the determination of the G-agents (GA, GB, GD, and GF), VX, and sulfur mustard a MINICAMS® is equipped with a flame photometric detector (FPD) that is sensitive to phosphorus or sulfur-containing compounds. A silver fluoride pad is used in-line when sampling for VX; validation of these pads are done at least annually or per lot, whichever is more frequent. This pad (also called a V-to-G conversion filter) converts VX to its corresponding G-analog, making vapor analysis possible.

Lewisite is first converted (i.e., derivatized) to a chemical compound that can be detected using the MINICAMS®. A compound, 1,2-ethanedithiol (EDT), is reacted with Lewisite to form a thermally stable Lewisite derivative (LD). A halogen selective detector (XSD) is employed to detect LD. The XSD also can be used to detect HN1, HN3, and HD. Using the correct column (DB-1701 or DB-210) all four agents (LD, HN1, HN3, and HD) can be determined in the same analytical run.

MINICAMS® in the FPD configuration are subject to positive interference from volatile organic compounds. These include fuels used to power vehicles and generators that may transport or power the MINICAMS®. Interference may also include other sources of volatile organics such as paints, roofing tars, pesticides, and laboratory contaminants. MINICAMS® in the FPD configuration are also subject to specific interferences because of phosphate-containing detergents (such as JOY® or TIDE®) used for cleaning, as well as caustic alcohol solutions that may be used to de-

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contaminate samples. Such solutions must be allowed to dry completely before sample containerization. MINICAMS® in the XSD configuration are subject to positive interference from volatile halogenated compounds. These may include industrial solvents or Freon. MINICAMS® in the XSD configuration may also be subject to specific interferences because of chemical agent decontamination procedures employing chlorine-containing substances such as bleach. Such solutions must be allowed to dry completely before sample containerization.

### 3.0 Terminology

This section lists in alphabetical order all terms, abbreviations, and acronyms unique to understanding this method.

- %R – percent recovery
- Background Sample – A negative control used to establish that the analytical system is free of interference and contamination. For MINICAMS® monitoring, background or room air is sampled as the background sample.
- Calibration standard (Cal Std) – A calibration standard prepared at a concentration equivalent to the WPL exposure limit or a chemical control limit (CCL) as applicable (assuming a given injection volume, flow rate, and cycle time). For Lewisite a 1 STEL standard is prepared and a volume corresponding to 0.4 STEL is injected. Also known as “initial calibration.”
- CAS® – Chemical Abstracts Service®
- CCL – chemical control limit. A chemical concentration considered a maximum point exposure limit. A CCL is used when a reliable STEL or WPL has not been determined.
- CCV – continuing calibration verification
- CMS – CMS Research Corporation
- DAAMS – Depot Area Air-Monitoring System
- 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.
- DPG – US Army Dugway Proving Ground
- FPD – flame photometric detector
- GA – tabun: ethyl N,N-dimethylphosphoramidocyanidate, CAS® 77-81-6, a nerve agent
- GB – sarin: isopropyl methylphosphonofluoridate, CAS® 107-44-8, a nerve agent
- EDT – 1,2-Ethanedithiol, CAS® 540-63-6
- FPD – flame photometric detector
- GC – gas chromatography
- G- and V- agents – the nerve agents determined by this method, which include: GA, GB, GD, GF, and VX
- GD – soman: pinacolyl methylphosphonofluoridate, CAS® 96-64-0, a nerve agent
- GF – cyclohexyl methylphosphonofluoridate, CAS® 329-99-7, a nerve agent
- HayeSep-D (40/60) – a type of preconcentrator tube used to collect the nerve agents determined by this method (also called G- and V- agents, which include: GA, GB, GD, GF, and VX).
- HD – mustard, distilled: bis-2-chloroethyl sulfide, CAS® 505-60-2, a blister agent
- HN1 – bis (2-chloroethyl) ethylamine, CAS® 538-07-8 [a nitrogen mustard]
- HN3 – tris-2-chloroethylamine, CAS® 555-77-1 [a nitrogen mustard]

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- IAW – in accordance with
- IPA – Isopropyl Alcohol (2-propanol) CAS® 67-63-0
- ICV – Initial Calibration Verification
- Lewisite – dichloro (2-chlorovinyl) arsine CAS® 541-25-3
- Lewisite derivative (LD) – Compound formed by reaction of Lewisite with EDT
- LLC – low level challenge. A calibration standard injection at 0.40 STEL for Lewisite
- LQAP – Laboratory Quality Assurance Plan
- Method Detection Limit (MDL) – Estimate of the lowest level of an analyte that a method can distinguish from noise.
- MMT – mobile monitoring trailer
- PCT – preconcentrator tube
- PMT – photo-multiplier tube
- ppm – parts per million
- psi – pounds per square inch
- QAPP – Quality Assurance Program Plan
- QC – quality control
- Quality Control Standard (QC Std) – Used as a calibration check (CC) standard. A standard, prepared at the 1 WPL concentration from a source separate from that used for the calibration standards, which verifies that the analytical system is operating as designed and is capable of detecting and quantifying chemical agent at the required concentrations. For Lewisite a 1 STEL standard is prepared and a volume corresponding to 0.4 STEL is injected.
- RCRA – Resource Conservation and Recovery Act
- RSD – relative standard deviation
- STEL – short term exposure limit. The STEL is a 15-minute TWA
- Tenax-TA (35/60) – a type of preconcentrator tube used to collect the blister agents determined by this method, which include HD, HN-1, HN-3, and Lewisite:
- TWA – time weighted average V-to-G conversion pad (or V-to-G conversion filter) converts VX to its corresponding G-analog, making vapor analysis possible.
- VX – o-ethyl S-(2-diisopropylaminoethyl) methylphosphonothiolate, CAS® 50782-69-9, a persistent nerve agent
- WPL – worker population limit, the airborne exposure limit for unprotected workers and general populations. The WPL is an 8-hour TWA
- XSD – halogen specific detector
- XXXX – Four X Level of Decontamination equivalent to the WPL

#### 4.0 Safety

Generally, RCRA waste samples received by the laboratory have been exposed to chemical agent and subsequently decontaminated. Handle all samples with caution and IAW Army toxic chemical safety guidelines until final test results have been released. For all operations involving chemical agents, comply with all laboratory safety rules and procedures. Be familiar with and follow safety guidelines contained in Safety Data Sheets for the chemicals being used or analyzed. Wear appropriate personal protective equipment when performing MINICAMS® operations. Wear protective gloves when directly handling waste bags.

Use extreme caution in dealing with MINICAMS® instrumentation to reduce the potential for burns and electrical shocks. Turn the MINICAMS® off or set photo-multiplier tube (PMT) volt-

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age to 0 when working on the PMT to avoid the high voltage hazard and damage to the PMT from light overflow.

The PCT, reactor chambers, and detector block operate at high temperatures. Avoid contact with these components when the instrument is, or was recently in use. Additional safety recommendations for operating MINICAMS® may be found in the instrument operating manuals.

The reagent gas EDT, which is used to convert the Lewisite to its derivative in the heated sample line, can be harmful in sufficiently high concentrations. It has a low odor threshold and can be detected readily through the sense of smell in the event of a leak. Any leaks noted should be found and repaired.

The Lewisite sampling system is not designed for operation during inclement weather. Conditions that result in contact with or the condensation of water on heated sample lines, or connecting electrical cables should be considered hazardous and, therefore, avoided.

Generally, samples designated for analysis by MINICAMS® have been exposed to or are suspected of being exposed to chemical warfare agent and subsequently decontaminated. Handle all samples with caution. For all operations involving chemical agents, comply with all laboratory safety rules and regulations. Be familiar with and follow safety guidelines contained in Safety Data Sheets for the chemicals being used or analyzed.

## 5.0 Apparatus and Reagents

To perform the procedures in this method, the following apparatus and reagents may be required.

### 5.1 Apparatus

The following equipment will be used to perform this method:

- CMS Field MINICAMS® with an FPD detector or an XSD detector, an appropriate GC column, and an appropriate solid sorbent preconcentrator tube sampling system as described in Table 1.

Item	For Use with FPD	For Use with XSD
PCT	HayeSep-D (40/60) for G- and V-agents, or Tenax-TA (35/60) for HD	Tenax-TA (35/60) for Lewisite, HN1, HN3, and HD
Gasses/Regulators	Hydrogen, 99.9% Nitrogen, 99.9% Compressed Air, Breathable Grade	Nitrogen, 99.9% Compressed Air, Breathable Grade EDT, 200 parts per million (ppm) in Nitrogen
Conversion Filters (Pads)	Silver fluoride pads for V-to-G conversion	
GC Columns	DB-1, DB-210, DB-1701	DB-1701, DB-210
Optical filters	Sulfur and phosphorus	

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Item	For Use with FPD	For Use with XSD
Manuals	<ul style="list-style-type: none"> <li>CMS Research Corporation (CMS) Field MINICAMS® Operation and Maintenance Manual</li> </ul>	<ul style="list-style-type: none"> <li>CMS Field MINICAMS® Operation and Maintenance Manual</li> <li>CMS Supplement to the FM-3000 Manual entitled “Detection of Lewisite using a MINICAMS® Equipped with a Halogen-Specific Detector (XSD)” dated October 1995</li> </ul>
Monitoring Options		<ul style="list-style-type: none"> <li>Lewisite Monitoring Option LEW-051 with heated sample line (Temp <math>\geq 60^{\circ}\text{C}/140^{\circ}\text{F}</math>) and probe</li> <li>Lewisite Monitoring Option LEW-051 with modified heated sample line (Temp <math>\geq 60^{\circ}\text{C}/140^{\circ}\text{F}</math>)</li> </ul>

- A sample pump
- Electrical power supply rated for 110 volts, 20 amperes, alternating current (from building or generator)
- Syringes, 10  $\mu\text{L}$ , or other as applicable
- Charcoal filters, CMS
- Dust pads
- Assorted silastic tubing
- Teflon™ tubing, 1/4-in outside diameter, 1/8-in inside diameter
- Assorted tubing connectors
- Gas-powered heater (i.e., Herman-Nelson)
- Assorted plastic bags, tarps, etc., for containerizing samples
- Packing tape

## 5.2 Reagents

The following reagents may be needed to perform this method:

- IPA, pesticide or chromatography grade
- Acetone
- Hexane
- Chemical agent standards, associated vials, and sealed carriers

## 6.0 Standards and Quality Control – Preparing, Storing and Using Standards

This section presents procedures for technical personnel to prepare standards.

Working chemical agent standards are generally prepared by technical personnel from neat agent or stock standard solutions, and are labeled, documented, and handled IAW internal laboratory procedures.

Prepare calibration standards (Cal Stds), and calibration verification standards, (QC Stds), at the WPL concentration IAW Equation 1. Table 2 lists the STELs and WPL action levels for each agent. Note that the action level for Lewisite is 0.4 STEL

Agent	STEL (mg/m <sup>3</sup> )	WPL (mg/m <sup>3</sup> )	WPL Calibration and QC Concentration (ug/mL)	STEL Calibration and QC Concentration (ug/mL)
GA <sup>(1)</sup>	0.0001	0.00003	0.0188	0.0625
GB <sup>(1)</sup>	0.0001	0.00003	0.0188	0.0625
GD <sup>(1)</sup>	0.00005	0.00003	0.0188	0.0312
GF <sup>(1)</sup>	0.00005	0.00003	0.0188	0.0312
HD/HN1/HN3 <sup>(2)</sup>	0.003	0.0004	0.20	1.5
VX <sup>(3)</sup>	0.00001	0.000001	0.00125	0.0125
Lewisite <sup>(4)</sup>	0.003	0.0012 <sup>(5)</sup>	1.5 <sup>(5)</sup>	1.5 <sup>(5)</sup>

1. The recommended standard concentrations assume the following sampling parameters:  
Cycle = 8 minutes, Purge = 3 minutes, Flow = 0.5 L/min, Injection Volume = 4 µL

2. The recommended standard concentrations assume the following sampling parameters:  
Cycle = 7 minutes, Purge = 3 minutes, Flow = 0.5 L/min, Injection Volume = 4 µL

3. The recommended standard concentrations assume the following sampling parameters:  
Cycle = 12 minutes, Purge = 2 minutes, Flow = 0.5 L/min, Injection Volume = 4 µL

4. The recommended standard concentrations assume the following sampling parameters:  
Cycle = 8 minutes, Purge = 4 minutes, Flow = 0.5 L/min, Injection Volume = 4 µL

5. **NOTE:** The 0.4 STEL level for Lewisite (0.0012 mg/m<sup>3</sup>) is achieved by injecting 1.6 µL of the calibration/QC Solution described above made at the 0.003 mg/m<sup>3</sup> level.

Equation 1:

$$\text{Standard Concentration} = \frac{\text{Conc} \times (\text{Cycle} - \text{Purge}) \times 1,000 \times \text{Flow}}{\text{Volume}}$$

where:

- Conc – the hazard level (mg/m<sup>3</sup>) (WPL or 0.4 STEL for Lewisite)
- Cycle – the MINICAMS® cycle time in minutes
- Purge – the duration of the purge period in minutes
- Flow – the sample flow rate in liters per minute (L/min)
- Volume – the volume of standard solution to be injected (µL)
- 1000 – the conversion factor with units of [(m<sup>3</sup> µL µg)/(mL mg L)]

For example, injecting a 4 µL volume of a 1 Z (0.003 mg/m<sup>3</sup>) Lewisite standard using a 8-minute

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cycle time, 4-minute purge time, and a flow rate of 0.5 L/min would require a standard concentration of:

Example

$$\text{Req. Std. Conc.} = \frac{0.003 \text{ mg/m}^3 \times (8 \text{ min} - 4 \text{ min}) \times (1,000 \text{ m}^3 \text{ } \mu\text{L } \mu\text{g/mL mg L}) \times 0.5 \text{ L/min}}{4 \text{ } \mu\text{L}} = 1.5 \text{ } \mu\text{g/mL}$$

- Prepare QC standards from a different stock solution than that used to prepare the initial calibration or by a different analyst using the same standard solution.
- Document the standard preparation in the Analyst's notebook. Include the following information:
  - Material source and lot number
  - Mass or volume taken
  - Final volume
  - Solvent type and lot number
  - Analysts initials
  - Date prepared
  - Expiration date
- Label, document, and handle standards in accordance with laboratory operating procedures.
- Store chemical agent standards in a refrigerator at or below 10°C.
- Allow solutions to warm up to room temperature before being opened for use.
- Return solutions to the refrigerator immediately after use.
- Single component working standards may be used for up to 30 days after preparation. Multi-component standards may also be used for up to 30 days if VX and HD are prepared in separate solutions.

**7.0 Procedure**

Technical personnel trained in MINICAMS® operations perform the following procedures to monitor chemical agents in air samples:

- Prepare Samples
- Receive Sample Request
- System Setup
- Establish an Initial Calibration
- Verify Calibration
- Analyze Background Samples
- Analyze Samples
- Shut Down MINICAMS®
- Troubleshoot

**7.1 Prepare Samples**

To prepare regulatory compliance solid waste samples, requestors are responsible for the following tasks:

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1. Ensure that chemical agent-related waste samples have been thoroughly decontaminated before analysis IAW operating procedures. To the extent possible, disassemble items before decontamination.
2. Ensure that solid waste is dry following decontamination operations. Several decontamination products (such as bleach or industrial solvents) may interfere with the analysis if not thoroughly rinsed and dried.
3. Place the dry solid waste in a sealed container and allow the contents to equilibrate for at least 4 hours at a temperature of 21°C or higher. Place small items in a plastic bag having a minimum thickness of 4 mil (~50 µm), and heat if necessary. Place larger items in a roll-off or gondola sealed with a tarp and packing tape, and heat if necessary.

## 7.2 Receive Sample Request

Requestors are responsible to complete the sample request form and ensure that the MINICAMS® operator receives the form from his or her supervisor. The MINICAMS® operator will review the form for completeness and verify with the Requestor what type sampling will be performed and which chemical agents will be determined.

## 7.3 System Setup

### 7.3.1 System Setup for monitoring G-Agents, VX, and HD.

To prepare and operate the MINICAMS® for calibration or analysis of G-agents, VX, or HD the MINICAMS® Operator performs the following tasks:

1. When analyzing for the agent VX, install a V-to-G conversion pad at the distal end of the MINICAMS® sampling line. The V-to-G pad degrades when exposed to light or moisture and should be replaced weekly at a minimum.
2. For G-agents, VX, or HD, completely open the air, hydrogen, and nitrogen gas cylinders. The cylinder pressure for each should be at least 500 pounds per square inch (psi) for a full day of monitoring.
3. Select the pre-concentrator tube and column type using Table 3.
4. Turn on the power to the MINICAMS® by placing the on/off switch to the "on" position.

Pre-Concentrator/Column	GA	GB	GD	GF	HD	VX
Pre-concentrator Type	HayeSep-D	HayeSep-D	HayeSep-D	HayeSep-D	Tenax-TA	HayeSep-D
GC Column	(1)	(1)	(1)	(1)	(1)	(1)

(1) DB-1, DB-210, DB-1701 or equivalent. Second column confirmation requires a different phase column than the primary column.

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PARAMETER	GA	GB	GD	GF	HD	VX
<u>Temperatures, °C</u>						
Ambient (AMB)	40	40	40	40	40	40
Inlet (INL)	75	50	75	75	75	50
FPD block (FPD)	150	150	150	150	150	150
FPD flame (FLA)	275	275	275	275	275	275
Low column (LCOL)	50	40	50	50	50	40
High column (HCOL)	200	175	200	200	200	175
Low PCT (LPCT)	40	40	40	40	40	40
High PCT (HPCT)	250	230	250	250	235	230
<u>Times, sec</u>						
Purge (PUR)	0-120	0-120	0-120	0-130	0-140	0-120
Sample (SAM)	120-300	120-300	120-300	130-310	140-320	120-600
Desorb (DES)	5-55	5-55	5-55	5-55	5-55	5-45
Column (COL)	30-100	30-90	30-100	30-100	30-130	30-75
Inject (INJ)	120-130	120-130	120-130	120-130	140-150	120-130
Zero (FPDZ)	ON	ON	ON	ON	ON	ON
<u>Pressures, psi</u>						
Hydrogen (H2PR)	25	25	25	25	25	25
Air (AIRP)	25	25	25	25	25	25
Nitrogen (N2PR)	40	40	40	40	40	40
PMT voltage (PMTV)	900	900	900	900	700	1000
Sample flow (SAMF)	500	500	500	500	500	500
<u>Temperature error limits, °C</u>						
Ambient (AMB)	15	15	15	15	15	15
Inlet (INL)	15	15	15	15	15	15
FPD block (FPD)	15	15	15	15	15	15
FPD flame (FLA)	99	99	99	99	99	99
Low column (LCOL)	15	15	15	15	15	15
High column (HCOL)	15	15	15	15	15	15
Low PCT (LPCT)	15	15	15	15	15	15
High PCT (HPCT)	20	20	20	20	20	20
<u>Other error limits</u>						
H <sub>2</sub> pressure (H2PR)	5	5	5	5	5	5
Air pressure (AIRP)	5	5	5	5	5	5
N <sub>2</sub> pressure (N2PR)	5	5	5	5	5	5
Samp. flow rate (SAMF)	99	99	99	99	99	99
PMT voltage (PMTV)	50	50	50	50	50	50
Col heat rate (COLR)	20	20	20	20	20	20
PCT heat rate (PCTR)	20	20	20	20	20	20
Peak width (PKW)	2	2	2	2	2	2

- Set instrument parameters as in accordance with four (4)-day method certification, Table 5 lists the suggested starting points for Lewisite, HN1, HN3, and HD.
- Allow the MINICAMS® to warm up for at least 30 minutes.
- Conduct all MINICAMS® operations at temperatures of 21°C (70°F) or higher. If necessary, heat transfer lines (using a thermal wrap) and samples (using gas or electric heaters) to this temperature. Document the sample collection temperature and any use of heat tape on the MINICAMS® Report.
- Ensure that the airflow through the MINICAMS® sample line is 0.5 (±25%) L/min. If the flow rate is out of tolerance, adjust flow as needed.

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9. If a heated sample line is used, verify that it is functioning properly.
10. Ensure that the MINICAMS® are in the correct mode.
11. Print out the parameters list.

### 7.3.2 System Setup for Monitoring Lewisite, HN1, HN3, and HD

The basic operation of the MINICAMS® when monitoring for Lewisite, HN1, HN3, or HD is the same as when monitoring for other chemical agents. However, during the MINICAMS® sampling period the sample stream and a small flow of EDT reagent are allowed to flow into the sample probe and heated sample line, or the modified heated sample line. The Lewisite and the EDT react to form the derivative LD. The LD is collected through the MINICAMS® inlet onto the PCT. The analysis of LD then proceeds as for any other chemical agent collected on a solid-sorbent bed inside the MINICAMS®. About 60 seconds before the end of the sample period, the flow of EDT is halted to allow excess EDT to be swept from the sorbent bed before the MINICAMS® purge period. EDT does not negatively impact the recovery of HD, HN1, or HN3 if the MINICAMS® is set up to sample these agents with Lewisite.

To prepare and operate the MINICAMS® for calibration or analysis of Lewisite, HN1, HN3, and HD, the MINICAMS® Operator performs the following tasks:

1. Ensure the instrument has the proper column installed. If monitoring for Lewisite and HN1 or HN3 at the same time a DB-1701 is required.
2. Completely open the air, nitrogen, and EDT cylinders. The pressure for the air and nitrogen cylinders should be at least 500 psi and the pressure for the EDT cylinder should be at least 50 psi for a full day of monitoring.
3. Turn on the power to the MINICAMS® by placing the on/off switch to the on position.

Table 5: Typical Operating Parameters for Lewisite, HN1, HN3, and HD  
[CMS Research Corporation (CMS) MINICAMS®]

PARAMETER	Lewisite, HN1, HN3, HD
<u>Temperatures (°C)</u>	
Ambient (AMB)	50
Inlet (INL)	100
XSD block (XSD)	150
Low column (LCOL)	70
High column (HCOL)	200
Column Rate (°C/min)	230
Low PCT (LPCT)	50
High PCT (HPCT)	250
<u>Times (sec)</u>	
Purge (PUR)	0-240
Sample (SAM)	240-480
Desorb (DES)	20-70
Column <sup>(1)</sup> (COL)	70-225
Inject <sup>(2)</sup> (INJ)	240-250
Zero (XSDZ)	ON
<u>Pressures (psi)</u>	
Hydrogen <sup>(3)</sup> (AIR2)	15
Air <sup>(3)</sup> (AIR1)	15
Nitrogen (N2PR)	40

Table 5: Typical Operating Parameters for Lewisite, HN1, HN3, and HD  
[CMS Research Corporation (CMS) MINICAMS®]

PARAMETER	Lewisite, HN1, HN3, HD
Sample flow (SAMF)	500
<u>Temperature error limits (°C)</u>	
Ambient (AMB)	15
Inlet (INL)	15
XSD block (XSD)	15
Low column (LCOL)	15
High column (HCOL)	15
Low PCT (LPCT)	20
High PCT (HPCT)	30
<u>Other error limits</u>	
H <sub>2</sub> pressure (AIR2)	5
Air pressure (AIR1)	3
N <sub>2</sub> pressure (N2PR)	5
Samp. flow rate (SAMF)	99
Column heat rate (COLR)	30
Peak width (PKW)	2

- 15-m DB-210 or DB-1701 fused silica capillary column.
- Set automatically during the first 10 sec of the sample period.
- Hydrogen is not used in this configuration. Compressed air is fed into both the air and hydrogen feeds on the MINICAMS®.
- Turn on the power to the MINICAMS® detector controller.
- Set instrument parameters IAW four-day method certification. Table 5 lists the suggested starting points for Lewisite, HN1, HN3, and HD.
- Allow the MINICAMS® to warm-up for at least 30 minutes.
- Conduct all MINICAMS® operations at temperatures of 21°C (70°F) or higher. If necessary, heat samples (using gas or electric heaters) to this temperature.
- Ensure that air is flowing through the MINICAMS® heated sampling line at 0.5 L/min (±25%). For Lewisite the flow should be 0.525 L/min (±25%). If the flow rate is out of tolerance, adjust flow as needed.
- Ensure that the EDT flow is approximately 25 mL/min. Adjust flow as needed.
- Verify that the heated sample line is functioning properly.
- Ensure that the MINICAMS® is in the correct mode.
- Print out the parameters list.

### 7.3.3 Flow meter calibration

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

### 7.4 Establish an Initial Calibration

Initial calibration is required, if significant changes are made to the instrument, or if the calibration verification (QC standard) fails. To calibrate the MINICAMS®, the MINICAMS® Operator performs the following steps:

- If necessary, inject a known concentration of the calibration standard to verify column and detector performance and to establish the agent retention time.

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2. Place the MINICAMS® in the calibration mode.
3. Inject a known amount of the Cal Std (see Paragraph 6.1). The calibration standards will be injected at the instrument inlet. When analyzing for VX, make calibration injections onto an inline V-to-G conversion pad.
4. The instrument will automatically calculate the average response factor from the three injections and store the new calibration
5. After the calibration is complete, return the instrument to RUN mode.
6. Maintain records of the initial calibration and calibration verification in the MINICAMS® instrument logbook by recording the following information regarding the calibration of MINICAMS®:
  - Date calibrated.
  - Monitor identification.
  - Operator name.
  - Identification of standard solution(s) used.
  - Results of each injection.
  - Results of calibration.

## 7.5 Verify Calibration

Calibration verification using the QC Standard is required after initial calibration, at the beginning and end of each hazardous waste run, and after every 10 waste samples.

**NOTE:** Non-hazardous waste samples must not be analyzed in a hazardous waste analytical sequence. If the MINICAMS® is in error during sampling and requires corrective action, the data is invalid and the samples must be re-analyzed.

To perform calibration verification, the MINICAMS® Operator performs the following steps:

1. Place the MINICAMS® in Check mode.
2. Inject a QC standard prepared at the 1 WPL concentration (1 STEL for Lewisite) into the sampling inlet as described in Paragraph 7.4 during the sampling period of the instrument cycle. For systems with heated sample lines, such as those used in field monitoring, the injection is made at the end of the sample line. A 4 µL injection is used except for a 1.6 µL injection made to achieve the 0.4 STEL QC for Lewisite. Determine if the results are within ( $\pm 25\%$ ) of 1 WPL ( $\pm 50\%$  for 0.4 STEL for Lewisite).
3. If results are not within criteria, conduct one or more of the following steps.
  - a. Inject a second QC standard.
  - b. Recalibrate.
  - c. Perform routine maintenance.
  - d. Troubleshoot.
  - e. Remove the MINICAMS® from service for further troubleshooting, repair or refurbishment.
4. If corrective actions were required (other than calibration) for one agent and not the other agents selected on the MINICAMS®, QC will need to be performed for the other agents. If the results are  $\pm 25\%$  of 1 WPL ( $\pm 50\%$  0.4 STEL for Lewisite) optional QC may be performed at the STEL level
5. Optional STEL level QC: Inject 4 µL of the STEL QC standard into the MINICAMS®.
  - a. For GA, GB, acceptable recoveries are between 2.5 WPL and 4.16 WPL.
  - b. For GD and GF acceptable recoveries are between 1.25 WPL and 2.1 WPL.
  - c. For HD acceptable recoveries are between 5.63 WPL and 9.38 WPL.

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- d. For VX acceptable recoveries are between 7.50 WPL and 12.50 WPL.
- e. Troubleshoot.

## 7.6 Analyze Background Samples

The MINICAMS® Operator analyzes at least one background sample with each run. To analyze the background sample, perform the following tasks:

1. Ensure that the MINICAMS® is in the run mode.
2. Place the sample line in background room air.
3. Collect one clean sweep cycle.
  - a. If the response is less than WPL (0.4 STEL for Lewisite), proceed with analysis.
  - b. If the response is greater than WPL (0.4 STEL for Lewisite), collect two more clean sweep cycles. If both responses are less than WPL (0.4 STEL for Lewisite), proceed with analysis. If either response is greater than WPL (0.4 STEL for Lewisite) take corrective action.

## 7.7 Analyze Samples

To analyze samples, the analyst ensures that the initial calibration and QC standards have been successfully analyzed, and then performs the following:

1. After the 4-hour equilibration period, carefully cut a small hole in the plastic bag or tarp and insert the MINICAMS® sample line into the container as far as possible. Re-seal the bag or tarp around the sample line and begin monitoring operations.
2. Ensure that the MINICAMS® is in run mode or service mode as appropriate.
3. Collect at least *three* cycles for each drum or bag sample. Collect at least three cycles at each end and in the middle of a roll-off (total of nine cycles per roll-off).
4. If the sample readings are below the 0.4 STEL for Lewisite or 1 WPL for all other agents, the sample is considered clean.
5. If a reading above 0.4 STEL for Lewisite or 1 WPL for all other agents or if significant interference is suspected during the chemical agent monitoring process, use another calibrated MINICAMS® or an alternative analytical method to verify the initial reading. If a positive reading is verified, return the bagged material to the requestor for further decontamination.
6. Analyze a background sample (according to Paragraph 7.6) before analyzing each XXXX sample.
7. If the ending continuing calibration verification (CCV) is outside the required limits, resample the container (e.g., plastic bag or tarp). If the ending CCV 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 explained in the case narrative.

## 7.8 Shut Down MINICAMS®

### 7.8.1 Standby

This procedure is used to minimize the warm-up time required when subsequent analytical runs are expected. To initiate the standby mode:

1. If EDT is being used, turn off the EDT at the source (compressed gas cylinder).
2. Turn off recorders and printers.

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### 7.8.2 Complete Shut Down

This procedure is used when subsequent analytical runs are not expected:

1. Turn off the EDT at the source (compressed gas cylinder).
2. Because the entire sample path is exposed to EDT when sampling for Lewisite, the system should be allowed to sample air only for several instrument cycles before shutdown.
3. Turn off the hydrogen, nitrogen, and air at the source (compressed gas cylinders).
4. Turn off accessories (i.e., recorders, printers, sample pumps, heated sample lines).
5. Shut down the portable power generators.

### 7.9 Preventive Maintenance and Troubleshooting

On a regularly scheduled basis, factory-trained service personnel perform routine preventative maintenance on each MINICAMS®. In addition, MINICAMS® Operators must be able to recognize and troubleshoot instrument problems that may cause low QC standard recovery or reduced sensitivity. Record all maintenance performed on the MINICAMS® in the instrument logbook. Refer to Section D of the CMS MINICAMS® Operation and Maintenance Manual for troubleshooting hints regarding the normal operation of a MINICAMS® equipped with a plug-in GC module, PCT sampling system, GC column and detector. Refer to Section 8 of the CMS MINICAMS® supplement for troubleshooting hints regarding the Lewisite configured MINICAMS®.

MINICAMS® Operators may use one or more of the following troubleshooting steps to improve instrument performance:

1. If they are present, replace the V-to-G conversion pad and its backup dust pad at the end of the heated sample line. If a sample line is not used to sample VX, replace the dust filter at the end of the heated sample line.
2. Replace the PCT in the MINICAMS® monitor as needed at the rate of approximately one tube per 5 days of continuous operation.
3. For the FPD, ensure that the GC column extends to at least ½ inch but no more than ¾ inch beyond the ferrule. For the XSD ensure that the GC column at the bottom of the inlet fitting just clears the ferrule used to make a gas-tight connection. That is, do not insert the GC column too far inside the inlet fitting.
4. If the check valve is mechanical, clean it by removing it from the MINICAMS® inlet (with the nitrogen turned off) and pulling approximately 30 to 50 mL of reagent-grade IPA through the valve. Allow air to be pulled through the check valve for approximately 10 to 15 minutes before reinstalling it on the MINICAMS® inlet. Make sure that the 100-mesh screen in the nylon fitting is also reinstalled after cleaning the check valve. If the check valve is electric, it may not be removed. If applicable, check and replace the spider gear.
5. Verify the absence of hydrogen, nitrogen, EDT, and air leaks.
6. Verify that all operating parameters are set correctly for the desired agent.
7. Verify that the flow rates of hydrogen, nitrogen, EDT, and air into the MINICAMS® and out of the detector vent are in the specified ranges.
8. Flush the inlet system and the GC column with acetone as described in the maintenance manual.
9. Lewisite may be prederivatized by injecting a few microliters of EDT stock solution into a vial of Lewisite standard so that the final concentration of EDT in the standard is about 100

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times the Lewisite concentration. After a relatively short period of time, all of the Lewisite in the vial of standard will be converted to its derivative (LD). The standard solution of LD may then be used to troubleshoot the operation of the MINICAMS® independently of the EDT reagent source and independently of the sample probe and heated sample line. That is, the LD may be injected directly into the MINICAMS® sample inlet during the MINICAMS® sample period. An HD or HN3 standard may be used for troubleshooting the GC module.

## 8.0 Prepare Data Package

This section presents the following procedures performed by the analyst to properly prepare a MINICAMS® data package.

- Perform data reduction.
- Assess quality control data.
- Assess sample results.
- Assemble complete data package.

### 8.1 Perform Data Reduction

Obtain a printout of sample results from the MINICAMS®. If properly configured and calibrated, the MINICAMS® will report sample results directly in WPL-equivalent units (1.00 = 1.00 times the WPL 0.400 STEL for Lewisite).

**NOTE:** Data packages should be completed using the MINICAMS® database software.

### 8.2 Assess Quality Control Data

To assess QC data, technical personnel ensure that the QC samples listed were analyzed and that QC sample results meet the listed criteria in Table 6. The criteria listed in Table 6 must be met for the sample results to be considered acceptable.

QC Sample	Criteria	Action
ICV	1 WPL ( $\pm 25\%$ ), 0.4 STEL ( $\pm 50\%$ ) for Lewisite	Troubleshoot as necessary, then recalibrate instrument
Accuracy/CCV	1 WPL ( $\pm 25\%$ ), 0.4 STEL ( $\pm 50\%$ ) for Lewisite For waste analyses, every batch of 10 field samples must be bracketed by valid CCVs.	Reanalyze samples analyzed after the last valid CCV. If the ending CCV 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.
Cleanliness	Background air sample result must be less than the WPL, 0.4 STEL for Lewisite.	Initiate corrective action, and reanalyze background air.

### 8.3 Assess Sample Results

To assess sample results technical personnel ensure that the sample results meet the criteria listed in Table 7.

Number of cycles for air monitoring for different types of containers needs to be discussed in details such as a roll-off should have over 10 cycles based on the headspace. This is addressed in Paragraph 7.7.3.

Sample	Criteria	Action
Safety air monitoring samples for all agents except Lewisite.	Two cycles $\geq 1.0$ WPL	Initiate alarm confirmation via DAAMS or alternate MINICAMS® with a different column. Generate alarm report.
Safety Air Monitoring samples for Lewisite.	Two cycles $\geq 0.4$ STEL	Initiate alarm confirmation via bubbler sampling or alternate MINICAMS® with a different column. Generate alarm report.
XXXX Sample.	One of three cycles $\geq 1.0$ WPL or $\geq 0.4$ STEL for Lewisite	Inform requester and suggest further decontamination. Generate alarm report.
RCRA-related air monitoring for all agents except Lewisite.	$\geq 1.0$ WPL	Initiate alarm confirmation via DAAMS or alternate MINICAMS® with a different column. Generate alarm report.
RCRA-related air monitoring for Lewisite.	$\geq 0.40$ STEL	Initiate alarm confirmation via bubbler sampling or alternate MINICAMS® with a different column. Generate alarm report.

### 8.4 Assemble Complete Data Package

- Verify that all of the following items are included in the data package:
  - Data Package Review Form.
  - Request Form.
  - Parameter list and station locations.
  - Results printouts for XXXX and waste analysis.
  - Corrective Action Form to include operator case narrative and alarm report, if needed.
  - QC Summary Form.
  - Report Summary.
- Include the following information for each analyte on the MINICAMS® data package for each monitoring operation:

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- Name of operator
  - Date of analysis
  - MINICAMS® identification number
  - Location of analysis
  - Start and end times
  - Start and end flow rates
  - Start and end QC result(s)
  - Results of blank analysis
  - Indication if the concentration was above the alarm setpoint
  - Parameter list and results printouts
  - Operator's initials and date
  - One to three clean sweeps as required after initial QC and one clean sweep between each sample
  - QC standard identification number
  - Highest sample result
3. Perform peer review.
  4. Deliver data package to the QC Chemist responsible for MINICAMS® data.

## 9.0 References

US Army Dugway Proving Ground (DPG), Utah, Waste Permit, Attachment 1-10, *Central Hazardous Waste Storage Facility (CHWSF) Quality Assurance Program Plan*.

CMS Supplement to Field Manual (FM)-3000, *Detection of Lewisite Using a MINICAMS® Equipped with a Halogen Selective Detector (XSD)*, October, 1995

**Figure 1**  
**Method Schematic**

