ECBC IOPs AND SOPs

ATTACHMENT 10

Pueblo Chemical Depot
Pueblo, Colorado

Document Type	Document Number/Version	Title	Reference
IOP	IOP MT-2, Rev. 17, 18 July 2013	Operation and Maintenance Procedures for Fixed-Site MINICAMS	Part II, Attachment 3, Attachment 6
IOP	IOP MT-8, Rev. 8, November 2011	Analysis of Chemical Warfare Agents in Extracts Using a Gas Chromatograph/Mass Spectrometer System	Attachment 3
IOP	IOP MT-11, Rev. 25, 6 November 2013	DAAMS Tubes Monitoring Procedures	Part II, Attachment 3, Attachment 6
IOP	IOP MT-13, Rev. 15, 1 November 2013	Analysis of Chemical Warfare Agents and Degradation Products on DAAMS Tubes using Gas Chromatography System Coupled with a Mass Spectrometer Detector (GC/MS)	Part II, Attachment 3, Attachment 6
IOP	IOP MT-60, Rev. 0, March 2010	Analysis of Residual Sulfur Mustard (HD) and HD Breakdown Products in EDS Neutralent/Waste including Monoethanolamine (MEA)	Part II, Attachment 3
SOP	SOP CSC-193 Rev 1	Accountability and Transportation of Chemical Munitions and DOT Bottles in Support of the Pueblo Agent-Destruction Pilot Plant Explosives Destruction System Project at Pueblo Chemical Depot	Attachment 2
SOP	SOP CSC-194 (Draft)	Accountability and Transportation of Hazardous Waste in Support of the Pueblo Agent-Destruction Pilot Plant Explosives Destruction System Project at Pueblo Chemical Depot	Attachment 2

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IOP MT-2 Operation and Maintenance Procedures for Fixed-Site MINICAMS

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Approved by Environmental Monitoring Laboratory Chief	Steven D. Norman	18 Jul 13
	Steven D. Norman	Date

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This IOP meets requirements of Revision 6 of the CASARM QA Plan.

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IOP MT-2 Operation and Maintenance Procedures for Fixed-Site MINICAMS

Record of Change Page

- Revision 17 – Updated all calibration/challenge procedures; updated Headspace monitoring; updated First Entry Monitoring, and made formatting changes.
- Revision 16 – Updated all procedures; updated standard concentrations; added appendixes on First Entry Monitoring, Monitoring for Waste Transport Operations, Heated Sample Line Cleaning, and Heated Sample Line Challenges; removed MBFORM-04 Sample; removed Appendix II-Baseline Study Information; and made formatting changes.
- Revision 15 – Changed minimum height for GF to 70. Added requirement for tracking expiration date of V-G pads and V-G Tubes in Section 5. Added NOTE: Check that the expiration date marked on the container of the V-G pad or V-G Tube lot being used has not expired. If expiration date is not on the container, do not use until expiration date is verified by ECBC personnel, then add expiration date to container and date and initial your entry.
- Revision 14 – Added initial baseline challenge requirement to Appendix 2 & 3 (Daily challenges to 2 – 3 hrs between challemges with minimum of 3/day to conform to LMQAP, December 2007. Added requirement: To re-cap standards vial at end of operational day. Removed "dated" requirement in paragraph 7.13.
- Revision 13 – Deleted note in Section 10 paragraph 4. Added Todd Duff as UPDATE originator.
- Revision 12 – Added archive information for Chrom-Link Operations and changed approval signatures from JAM to TBD. Updated several paragraphs to reflect current operations. Added calculations for Industrial Agent – Calibration. Changed digital flow meter requirement to permit use of any type flow measuring device.
- Revision 11 – Added changes based on CMA comments. Added requirement to cap ends of sample lines not being used. Added reference to SOP# CNG-048 (dilute standard preparation). Added note that flow rate of 400 ml required when monitoring for VX.
- Revision 10 – Changed HSL challenge frequency from 3 to 2 Months for CMA operations. Added Appendix III, Requirements for Special Projects/Personnel Safety/EDS Monitoring.
- Revision 9 – Added/Changed requirements to meet CMA requirements.
- Revision 8 – Added requirement to record Standard vial numbers on MBFORM-04 each week when new standard vials are received and used. Added requirement to use a digital flow meter attached to the distal end of the HSL during the quarterly HSL check and record the actual flow rate on MBFORM-34.
- Revision 7 – Added clarification of requirement for MINICAMS Operator to review previous days MINICAMS printout to determine that there were no errors/alarms or "agent hits" while the MINICAMS was monitoring without the operator being present. Removed APG in header. Removed requirement to check sample line continuity every other week note in paragraph 8.02.

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- Revision 6 – Added minimum height requirements for GB, GD, HD to Section 5.0 and Section 6.0 for HD.
- Revision 5—Changed 6 µl challenge to 4 µl challenge throughout the document to conform to the released Revision 4 of CASARM QA Plan.
- Revision 4—Incorporated 1 µl and 6 µl level challenges IAW CASARM Revision 4 draft.
  Incorporated requirement for separate standards to be used for calibration and Quality Control.
  Incorporated all other changes required by Revision 4 of the CASARM QA Plan.
- Previous revisions of this document did not incorporate a record of change page.

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Signature Page

Operator's Statement: I have read, or have had read to me, the procedures in this IOP. I, by my signature below, indicate that I thoroughly understand and agree to abide by these instructions.

Name (print)	Signature	Date

Supervisor's Statement: I have personally reviewed this IOP and, to the best of my knowledge, believe that the information listed herein is correct.

Supervisor: Steven D. Norman Date: 7/18/13

Steven D. Norman

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IOP MT-2 Operation and Maintenance Procedures for Fixed-Site MINICAMS

1.0 PURPOSE

The purpose of this IOP is to describe the operation and maintenance of the MINICAMS Agent Monitoring System during Fixed Site Operations. The determination of a Fixed Site or a Mobile Site is dependent on movement of the heated sample lines (HSL) and movement of the platform that the MINICAMS are stationed in. This decision will be made by the Air Monitoring Manager and/or the MINICAMS Team Leader.

2.0 MINICAMS OVERVIEW

The MINICAMS provides workers involved in chemical agent-related operations with near real-time (NRT) monitoring for the presence of airborne chemical warfare material in the workplace**.** The MINICAMS monitor at the Short-Term Exposure Limit (STEL) and/or the Vapor Screening Level (VSL). Each system is calibrated and challenged each operational day. Continuous monitoring is achieved through the use of a HSL. The sample is drawn through the HSL and into the MINICAMS unit, which desorbs and analyzes the sample during the analytical period of a MINICAMS cycle. The data collected from each MINICAM is stored on a CD and/or on a hard copy printout. This serves as a historical record of (no) agent release.

A member of the monitoring team monitors the performance of the MINICAMS on a daily basis. The MINICAMS is challenged each workday using certified standards for the agent(s) of interest to assure that the MINICAMS is operating correctly and in control.

During setup/pre-op, the Air Monitoring Manager and/or MINICAMS Team Leader determines the appropriate method to be used.

2.1 Definition of the Presence of Chemical Agent

During fixed site operations, chemical agent is defined as present if detected by the MINICAMS in a concentration greater than or equal to the alarm level set point for three consecutive cycles. The operator shall notify the EML Chief and/or Air Monitoring Manager that agent has been detected during the monitoring operations.

2.2 Calibration Standards and QC Standards

Separate chemical agent standards will be used for calibration and QC check of the MINICAMS. The MINICAMS will be calibrated for the agent(s) of interest with two individual 4 µl (1Z) injections of the Calibration standard. Then a 1Z challenge of the QC standard shall be used to check the calibration. The challenge is acceptable if the analytical result(s) are within ±25% of the target concentration. See Sections 5 or 6 for specific injection requirements. Chemical agent calibration and challenge standards are prepared in accordance with SOP# CNG-048.

Industrial compound calibration and challenge standards are prepared from a single source reference of certified standard reference materials. The challenge is acceptable if the analytical result(s) are within ±25% of the target concentration. See Section 7 or Appendix III for specific injection requirements.

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3.0 CONTROL SAMPLES

A MINICAMS sample is defined as the volume of air that is sampled during one automatic cycle of the instrument. All challenges of dilute chemical agent are injected into the MINICAMS injection port or HSL during the beginning of the sampling period of the MINICAMS cycle. MINICAMS monitoring for VX will have injections made onto the silver fluoride (AgF) conversion pad/tube. MINICAMS monitoring for L will have injections made into the Lewisite probe. MINICAMS equipped with a loop-sampling module will be calibrated and challenged IAW the specific calibration procedures specified in section 7.

A record of the test date, time, calibration peak height, found concentration, and the operator's initials will be maintained for sample challenges on MBFORM-04/MBFORM-38. All QC challenge chromatograms and operating conditions will be printed out on hard copy and the operator will initial next to the flow rate to acknowledge that it is within limits. This record will be maintained with the MINICAMS at the monitoring site. The data is transported back to the EML at the end of each operational month for final documentation review and storage.

Any data that is generated should be neat and free of mistakes. When a mistake does occur, the mistake should have a single line through it, with the correction, operator's initials, and date next to it to correctly document and fix the mistake. If correction cannot be made neatly, cross out the entire line.

Monitoring Operations conducted under the purview of CMA will require Baseline Method Certification and Validation. Appropriate baseline studies will be performed and associated data will be provided as required by the approved site plan.

4.0 PREVENTIVE MAINTENANCE PROCEDURES

4.1 Verify that PM has been performed within one year and/or does not exceed fourteen months. If it is out of PM, remove MINICAMS from service and replace it with one that has a current PM.

4.2 Check that the MINICAMS date/time is set correctly, as well as the CHROM-LINK date/time.

4.3 Verify that the unit is in the correct method, and the appropriate agent(s) are selected. Verify that the serial number/station number is correct.

4.4 Verify that the unit is in the run mode.

4.5 Verify that all operating parameters are correct for the selected method.

4.6 Verify that the alarm lamp and horn come on when the MINICAMS is challenged.

4.7 Verify that there are no errors or alarms, and if MINICAMS is linked to a data acquisition system, verify that the NRT data acquisition system screen displays all required information, e.g., station number, location etc., as shown on the MINICAMS screen.

4.8 Verify that each compressed-gas cylinder contains at least 200 psig. If not, change the cylinder. If monitoring for L, verify that the Ethanedithiol (EDT) is at 30 – 35 psi. If

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using a Hydrogen generator, check the level of deionized (DI) water and add more as necessary. Do not fill DI water past the fill marker on the tank.

4.9 Check that the printed data is dark enough to scan. Replace the printer cartridge if necessary. Check the amount of printer paper remaining. Replenish, if necessary.

4.10 Heated sample lines used for Lewisite (L) monitoring require Line-Cleaning with ~100 mls of acetone followed by ~100 mls of 2-propanol to eliminate L carryover after each week of operation, or as required to keep the instrument in control. See Appendix VI for procedures.

4.11 Replace the Pre-Concentrator Tube (PCT) and reactor tube (if applicable) weekly at a minimum. Replace the V-G pad/tube (if applicable) every two weeks at a minimum.

4.12 Check the external gas lines and fittings for leaks.

4.13 Sample Flow Rates (limits are set to ±25% of the established flow rate)

XSD: 400 ± 100 ml/min
FPD: 400 ± 100 ml/min
FPD: 800 ± 200 ml/min
LOOP: 100 ± 25 ml/min

4.14 Minimum Agent Peak Heights (nanoamps):

VX, GF, L: 70 nA
GA, GB, GD, HD, HN-1, HN-3: 250 nA
CG, PS, CF, CK: 20 nA

4.15 Alarm Set Points:

0.70 Z for all agents except L
0.40 Z for L

*The MINICAMS is only capable of storing a single alarm level, so when monitoring for Lewisite and other agents, the alarm set point will be set to the lowest alarm level.

5.0 CALIBRATION AND CHALLENGE PROCEDURES FOR FPD MINICAMS

The FPD MINICAMS is capable of detecting VX, G-series agents, and HD. HD cannot be calibrated with any of the V or G agents. HD is calibrated by itself after replacement of the Photo-Optical Filter and Sorbent Tube.

5.1 At the beginning of each work week, get a calibration standard vial and a QC standard vial of each agent to be challenged and record the vial numbers in the space provided in upper right hand corner of MBFORM-04.

5.2 These standards should be kept cold except when in use and shall have a new cap installed at the end of each operational day that the vial is used to limit standard degradation.

5.3 Unused standards shall be taken to the lab for decon when no longer required.

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5.4 Change the PCT weekly at a minimum. Operator shall document that weekly replacements have been made in the comments section of MBFORM-04.

5.5 Check that the certification paperwork is located inside the V-G container and that the V-G tubes/pads (if applicable) have not expired. If there is no paperwork in the container, do not use until expiration date is verified by ECBC personnel.

5.6 If the MINICAMS is programmed for VX operation, replace the V-G pad/tube at the distal end of the HSL every two weeks at a minimum. V-G pads/tubes may require changing more frequently during rainy or very humid weather. Operator is to document replacement in the comments section of MBFORM-04.

5.7 The MINICAMS operator shall start a new QC data sheet, MBFORM-04 when the MINICAMS is changed and/or on the first operational day of each month. The operator will make sure that the MBFORM-04 is properly completed with the MINICAMS serial number, agent, HSL serial number, standard vial numbers, and the LMF identification number and expiration date. The data and the QC data sheets (MBFORM-04) for the previous month shall be collected and turned in to the MINICAMS group for review and filing.

5.8 Prior to beginning MINICAMS calibration, the operator will check the data printout from previous 24 hours to determine that there is no evidence of agent presence, alarms or MINICAMS errors. Date and initial the printout to provide evidence of this review. If there were errors and/or maintenance done on the MINICAMS, the operator should provide an explanation and/or annotation to the paperwork and MBFORM-04. If there were 3 consecutive alarms the operator will notify the Air Monitoring Manager or EML Branch Chief immediately.

5.9 Check MINICAMS operational parameters for errors. If there are no errors, press the PARAM button until RUN is flashing in the lower left corner, then press the INCR button until CAL is flashing and press ENTER.

5.10 After the word INJECT appears on the display screen, perform separate 4 µl injection(s) of the CAL standard(s) into the MINICAMS inlet or onto on the surface of the V-G pad/tube (if monitoring for VX). After the MINICAMS completes the sampling cycle, desorbs, and the results are generated, the operator shall label the chromatogram as 4 µl CAL 1 location initials. (An annotation of "4 µl Cal 1 CTF TBD" would show that the injection was a 4 µl calibration at the CTF by the operator TBD). Press ENTER to accept the shot. For any calibration injection, the operator may choose not to accept the calibration injection and repeat the injection.

5.11 For calibration injection 2, repeat the 4 µl calibration injection described in the step above. After the cycle is complete and the results are generated, the operator shall evaluate the results of calibration injection 2. The results of calibration injection 2 are acceptable if the result displayed for all agents is ±25% of the target concentration (0.75 – 1.25 Z). If the results are acceptable press ENTER and label this chromatogram as 4 µl CAL 2 location initials. Print the chromatogram from the second calibration injection. If the results are not 0.75 to 1.25 Z, do not accept the injection. The injection may be repeated, the operator

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may choose to restart the calibration sequence from CAL 1 or the instrument may be judged to require maintenance and/or corrective action.

5.12 If minimum calibration peak height (calH) cannot be achieved, corrective action shall be taken and documented. Corrective action may include increasing the PMTV by increments/multiples of 25 volts until peak height is acceptable. If photomultiplier voltage is already set to the maximum set point of 1250 volts and the minimum height still cannot be achieved, the instrument should be removed from service for repairs and replaced with a working MINICAMS. The instrument must be recalibrated for all agents if instrument parameters that affect the calibration have been changed after the initial calibration has been completed.

5.13 For challenge injections, the HSL should be reconnected to the MINICAMS (when possible) after injections are made into the inlet. This may not be possible if a V-G tube/pad is located at the inlet.

5.14 Press the PARAM button on the MINICAMS until CAL is flashing on the lower left of the screen, press INCR button until RUN is flashing and then press ENTER.

5.15 Using the QC standard, perform 4 µl injections into the MINICAMS inlet or onto the V-G pad/tube (if monitoring for VX), when the screen reads INJECT. Result shall be within ±25% of the target concentration. During the Purge period, label this chromatogram as a challenge and enter the operator's initials and location on the chromatogram, i.e., "4 µl CHALL location initials."

5.16 An audible alarm will sound if a concentration greater than the alarm set point is detected for any of the selected agents. Press any button on the MINICAMS to silence the alarm horn. The alarm light will continue to flash until the concentration falls below the alarm set point.

5.17 Print out the chromatogram and operating conditions from the challenge. The average calibration height results will be found near the bottom of the page of operating conditions. The operator will initial next to the flow printout to indicate that the flow rate is within ±25% of the flow rate set point.

5.18 Record each found concentration on MBFORM-04 under the FC 1 column. Each of the concentrations must be acceptable. The 4 µl concentration must be within ±25% of the Target Concentration (0.75 – 1.25) for each agent. If not, repeat steps 5.9 through 5.15 above and record data under the FC2 column.

5.19 All agents injected must pass this second challenge. On the second challenge, if any of the injected agents are not within the acceptable limits perform corrective actions and/or another calibration. Repeat steps 5.9 through 5.15 again and record found concentration under the FC3 column and second CAL HT under corrective action (CA) column on MBFORM-04. If the corrective action only affects the agent selected (e.g., change conversion pad for VX), the instrument need only be re-calibrated for that agent. However, if MINICAMS parameters are changed, the MINICAMS shall be recalibrated for all agents.

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5.20 If the third challenge was performed after corrective action and is still out of range, this instrument must either be removed from service or additional corrective action must be performed and documented on the QC data sheet. After the corrective action has been taken, the instrument must be re-calibrated and the data recorded on the next line. If the instrument is replaced, a new QC data sheet must be started and calibrations and challenges must be performed as indicated above.

5.21 Print out chromatograms, operating conditions and online/past reports for calibrations and challenges; label as appropriate. The average calibration and height results will be found near the bottom of the page of printed operating conditions. The operator shall copy the "CAL HT" and FC in the appropriate columns of MBFORM-04.

5.22 Operator shall initial the parameters printout next to the printed flow rate to indicate that the flow rate is within ±25% of the flow rate set point.

5.23 Make sure the operator's initials and location, CAL height, heated sample line serial number, standard vial number, LMF number and calibration due date, and challenge data are recorded properly on the QC data sheet (MBFORM-04).

5.24 Plot the 4 µl QC challenge shot on the QC performance chart located on MBFORM-04. The 4 µl QC challenge shots shall be within the acceptance limits listed on the performance chart on MBFORM-04 to be acceptable.

6.0 CALIBRATION AND CHALLENGE PROCEDURES FOR THE XSD MINICAMS

6.1 At the beginning of each workweek, get a calibration standard vial and a QC standard vial of each agent to be challenged and record the vial numbers in the space provided in upper right hand corner of MBFORM-04.

6.2 These standards should be kept cold except when in use and shall have a new cap installed at the end of each operational day that the vial is used to limit standard degradation.

6.3 Unused standards shall be taken to the lab for decon when no longer required.

6.4 Change the PCT weekly at a minimum. Operator shall document that weekly replacements have been made in the comments section of MBFORM-04.

6.5 If monitoring for L, replace the Reactor Tube in the Lewisite sample probe weekly at a minimum. The Reactor Tube may require changing more frequently during rainy or very humid weather. The operator will document replacement in comments Section of MBFORM-04.

6.6 The MINICAMS operator shall start a new QC data sheet, MBFORM-04 when the MINICAMS is changed and/or on the first operational day of each month. The operator will make sure that the MBFORM-04 is properly completed with the MINICAM S serial number, agent, HSL serial number, standard vial numbers, and the LMF identification number and expiration date. The data and QC data sheets (MBFORM0-04) for the previous month shall be collected and turned in to the MINICAMS group for review and filing.

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6.7 Prior to beginning MINICAMS calibration, the operator will check the data printout from previous 24 hours to determine that there is no evidence of agent presence, alarms or MINICAMS errors. Date and initial the printout to provide evidence of this review. If there were errors and/or maintenance done on the MINICAMS, the operator should provide an explanation and/or annotation on the paperwork and MBFORM-04. If there were 3 consecutive alarms the operator will notify the Air Monitoring Manager or EML Branch Chief immediately

6.8 Check MINICAMS operational parameters for errors. If there are no errors, press the PARAM button until RUN is flashing in the lower left corner, then press the INCR button until CAL is flashing and press ENTER.

6.9 After the word INJECT appears on the display screen, perform separate 4 µl injection(s) of the CAL standard(s) into the MINICAMS inlet or into the Lewisite Sample Probe. After the MINICAMS completes the sampling cycle, desorbs, and the result are generated, the operator shall label the chromatogram as 4 µl CAL 1 location initials. (An annotation of "4 µl Cal 1 CTF TBD" would show that the injection was a 4 µl calibration at the CTF by the operator TBD). Press ENTER to accept the shot. For any calibration injection, the operator may choose not to accept the calibration injection and repeat the injection.

6.10 For calibration injection 2, repeat the 4 µl calibration injection described in the stop above. After the cycle is complete and the results are generated, the operator shall evaluate the results of calibration injection. The results of the calibration injection are acceptable if the results for all agents is 0.75 to 1.25 Z. If the results are acceptable, press ENTER and label this chromatogram as 4 µl CAL 2 location initials. Print the chromatogram from the second calibration injection. If the results are not 0.75 to 1.25Z, do not accept the injection. The injection may be repeated, the operator may choose to restart the calibration sequence from CAL 1, or the instrument may be judged to require maintenance and/or corrective action.

6.11 If the minimum calibration peak height(s) (calH) cannot be achieved, corrective action shall be taken and documented. The instrument must be recalibrated for all agents if instrument parameters that affect the calibration have been changed after the initial calibration has been completed.

6.12 The HSL should be reconnected to the MINICAMS (when possible) after injections are made into the inlet. When monitoring for L, the HSL is always connected to the inlet, and injections are made into the sample probe connected to the distal end of the HSL.

6.13 Press the PARAM button on the MINICAMS until CAL is flashing on the lower left of the screen, press INCR button until RUN is flashing and then press ENTER.

6.14 Using the QC standard, perform 4 µl injections into the MINICAMS inlet or into the Lewisite Sample Probe when the screen reads INJECT. Results shall be within ±25% of target concentration. During the Purge period, label this chromatogram as a challenge and enter the operator's initials and location on the chromatogram, i.e., "4 µl CHALL location initials."

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6.15 An audible alarm will sound if a concentration greater than the alarm set point is detected for any of the selected agents. Press any button on the MINICAMS to silence the alarm horn. The alarm light will continue to flash until the concentration falls below the alarm set point.

6.16 6Print out the chromatogram and operating conditions from the challenge. The average calibration height results will be found near the bottom of the page of operating conditions. The operator will initial next to the flow printout to indicate that the flow rate is within ±25% of the flow rate target.

6.17 Record each found concentration on MBFORM-04 under the FC 1 column. The 4 µl concentration must be within ±25% of the target concentration (0.75 – 1.25 Z) for each agent. If not, repeat steps 6.9 through 6.14 above, record data under the FC2 column.

6.18 All agents injected must pass this second challenge. On the second challenge, if any of the injected agents are not within acceptable limits, perform corrective actions and/or another calibration. Repeat steps 6.7 through 6.14 again and record found concentration under the FC3 column and 2nd CAL HT under corrective action (CA) column on MBFORM-04. If the corrective action only affects the agent selected, (e.g., change Reactor Tube for L), the instrument need only be re-calibrated for that agent. However, if MINICAMS parameters are changed, the MINICAMS shall be recalibrated for all agents.

6.19 If a third challenge was performed after corrective action and is still out of range, this instrument must either be removed from service or additional corrective action must be performed and documented on the QC data sheet. After the corrective action has been taken, the instrument must be re-calibrated and the data recorded on the next line. If the instrument is replaced, a new QC data sheet must be started and calibrations and challenges must be performed as indicated above.

6.20 Print chromatograms, operating conditions, and online/past reports for calibrations and challenges; label as appropriate. The average calibration and height results will be found near the bottom of the page of printed operating conditions. The operator shall copy the "CAL HT" and FC in the appropriate columns of MBFORM-04.

6.21 Operator shall initial the parameters printout next to the printed flow rate to indicate that the flow rate is within ±25% of the flow rate target.

6.22 Make sure the operator's initials & location, CAL height, heated sample line serial number, standard vial number, LMF number and calibration due date, and challenge data are recorded properly on the QC data sheet (MBFORM-04)

6.23 Plot the 4 µl QC challenge shot on the QC performance chart located on MBFORM-04. The 4 µl QC challenge shots shall be within the acceptance limits listed on the performance chart on MBFORM-04 to be acceptable.

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7.0 CALIBRATION AND CHALLENGE PROCEDURES FOR THE XSD MINICAMS WITH LOOP SAMPLING MODULE

7.1 Verify CG and CK cylinders have not reached expiration dates and expiration dates are correct on the QC data sheet (MBFORM-38)

7.2 Prior to beginning MINICAMS calibration, the operator will check the data printout from previous 24 hours to determine that there is no evidence of agent presence, alarms or MINICAMS errors. Date and initial the printout to provide evidence of this review. If there were errors and/or maintenance done on the MINICAMS, the operator should provide an explanation and/or annotation on the paperwork and MBFORM-38. If there were 3 consecutive alarms the operator will notify the Air Monitoring Manager or EML Branch Chief immediately

7.3 Each operational day the operator shall get either a new or a previously used Tedlar® "Gas Standard Bag" that is still functional. See Appendix 1 for bag filling procedures.

7.4 The MINICAMS operator shall start a new QC data sheet (MBFORM-38) when a new MINICAMS is installed and/or on the first day of every month. The operator will make sure that the MBFORM-38 is properly completed with the MINICAMS serial number, agent, HSL serial number, standard vial numbers, LMF identification number and expiration date, CK & CG expiration dates. The data and QC Data Sheets (MBFORM-38) for the previous month shall be collected and turned in to the MINICAMS group for final documentation review and filing.

7.5 From the main screen, press the PARAM button until RUN is flashing in the lower left corner. Press the INCR button until CAL is flashing and then press ENTER.

7.6 Check MINICAMS for errors. If no errors are present, close the MINICAMS sample bypass valve, and attach the gas sample bag containing a 1 TWA concentration of the agent(s) of interest to the inlet while in purge and open bag until 2 complete threads are showing on the valve. After the sample cycle is complete and results have been generated, annotate this chromatogram as "1 TWA Cal 1 location initials."

7.7 Allow the MINICAMS to complete another sampling cycle. After the cycle results have been generated, evaluate the results. Results are acceptable if the FC is 0.75 to 1.25 STEL. Annotate this chromatogram as "1 TWA Cal 2 location initials" and press ENTER to accept the second calibration result. If the results are not 0.75 to 1.25 STEL, do not accept the injection. The injection may be repeated, the operator may choose to restart the calibration sequent from CAL 1 or the instrument may be judged to required maintenance and/or corrective action.

7.8 The calibration height must meet minimum height requirements found in section 4.0.

7.9 Press the PARAM button until CAL is flashing on the lower left hand side of the screen, press the INCR button until RUN is flashing then press ENTER.

7.10 Allow the MINICAMS to complete another sampling cycle. After results are generated, label the chromatogram as "1 TWA Chall location initials." The challenge results are acceptable if they are within ±25% of the target concentration (0.75 – 1.25). If the first

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challenge fails for any agent(s), operator shall perform a second challenge of that agent. Reconnect HSL and open MINICAMS sample bypass valve if all results are acceptable.

7.11 All agents must pass this second challenge. On the second challenge, if any of the injected agents are not within acceptable limits, perform corrective actions and/or another calibration. Repeat steps 6.7 through 6.14 again and record found concentration under the FC3 column and 2nd CAL HT under corrective action (CA) column on MBFORM-04. If the corrective action only affects the agent selected, the instrument need only be re-calibrated for that agent. However, if MINICAMS parameters are changed, the MINICAMS shall be recalibrated for all agents.

7.12 If a third challenge was performed after corrective action and is still out of range, this instrument must either be removed from service or additional corrective action must be performed and documented on the QC data sheet. After the corrective action has been taken, the instrument must be re-calibrated and the data recorded on the next line. If the instrument is replaced, a new QC data sheet must be started and calibrations and challenges must be performed as indicated above.

7.13 Print Challenge chromatograms, operating conditions and online/past reports for calibrations and challenges; label as appropriate. Initial the operating conditions next to the flow rate to show that the actual flow rate was within ±25% of the flow rate target (100 ml/min ± 25 ml/min).Typical Chromatographic Conditions

8.0 CHROM-LINK ARCHIVE PROCEDURES

The CHROM-LINK data from each MINICAMS will be archived to a CD(s) each Month and/or at 200% (using the same disk) and returned to EML with the hard copies of the MINICAMS data. For all short term projects (less than one Month duration), the CHROM-LINK data will be archived to a CD at the end of the project and returned to EML along with the hardcopy data.

8.1 Archiving

8.1.1 Double click CHROM-LINK Archive from the desktop.

8.1.2 Click on the “Set Archive Parameters” icon.

8.1.3 Click on the Non-Recurring Archive.

8.1.4 Deselect “Backup Only”, and set the number of days to retain to 1.

8.1.5 Select “Now” and click “OK”. Click “OK” in the pop-up box to begin archive.

8.1.6 Once archive is complete, exit out of the Archive program.

8.2 Creating a Desktop Folder for Archive Data

Once all data is archived and burned to a disc, the operator will save the archived data to the computer’s desktop in case the data needs to be accessed. Data can be deleted only at the discretion of the Air Monitoring Manager and/or MINICAMS Team Leader.

8.2.1 Right click on desktop, select “New,” and select “Folder.”

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8.2.2 A new folder will appear on the desktop. Name this folder with the site, month, and year of the archived data.

8.2.3 On the desktop, double click on the "My Computer" icon. Double click on "Local Disk (C)." Double click on "DATA" folder. Double click on "Archive" folder. This will show the data from the recent archive.

8.2.4 Select all files to be moved to the new folder on the desktop and drag this data to the new folder.

8.3 Burning Archived Data to Disc

8.3.1 Double click on folder on the desktop of the data to be archived.

8.3.2 All Archive data to be burned to disc should be highlighted and dragged down to Roxio CD & DVD Creator, which should be on the bottom-right hand side of the desktop.

8.3.3 Click on the Burn to CD icon. Click OK on the Record Setup pop-up page. Once disc is burned, click Close on the Launch label Creator pop-up. Click NO for save project changes.

8.3.4 Verify that the data has been burned to CD by opening the CD drive and viewing the files on the CD. If the data has been burned correctly, the disc may be ejected from the computer. Exit out of the program.

8.3.5 All Disks should have the following written on the face of the CD in black marker: Site Name, MINICAMS serial number, Agent(s) of Concern, Location and Position, Month and Year of Archive Data, and Station Number.

9.0 HEADSPACE MONITORING

There is no headspace monitoring requirement for industrial agents. Reusable PPE must be monitored at the WPL level.

If for any reason the MINICAMS Operator determines that it is more efficient to run DAAMS on the samples, contact the DAAMS tech. For any headspace monitoring operations a maximum of 10 items can be cleared as part of a single batch. Each batch must be bracketed by acceptable challenges.

9.1 Soil Headspace Monitoring

9.1.1 Contractor/Govt. delivers soil samples to monitoring personnel with proper chain of custody documentation. Operator must not sign chain of custody unless all information has been verified as correct.

9.1.2 Ensure a passing challenge has been completed within the last 4 hours. Place up to ten samples in a heated sample box. Don Mask and Gloves prior to opening bags and remove sample jar lids. Insert temperature probe into a selected soil sample, and ensure soil temperature has reached 90°F + 10. Remove temperature probe, close sample box lid and allow samples to off-gas for 15 minutes.

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9.1.3 Attach the MINICAMS probe (if monitoring for L) or the distal end of the HSL to the heated sample box. If monitoring for VX, the AgF tube/pad must be at the end of the probe where air sample is introduced into HSL. Monitor headspace for two complete cycles on the MINICAMS.

a. If MINICAMS result is below 0.25 VSL for 2 consecutive cycles, perform an ending challenge to bracket these results. If your challenge is successful then go to step 9.1.12. If two consecutive challenges fail, you, the samples are not clear and must be reanalyzed.

b. If MINICAMS results are above 0.25 VSL, go to step 9.1.4.

9.1.4 Don Mask and Gloves.

9.1.5 Open sample box and replace lids on sample containers and close bags.

9.1.6 Allow sample box to re-equilibrate for 15 minutes.

9.1.7 If MINICAMS had exceeded the alarm level, attach MINICAMS probe or distal end of HSL to the heated sample box and monitor headspace. Once the MINICAMS result is below the alarm set point, proceed to next step.

9.1.8 Don mask and gloves and open lid of sample box. Samples will be monitored one at a time.

9.1.9 Open bag and remove lid from a single sample container. Insert temperature probe into soil sample and ensure that the temperature is 90°F ± 10. Remove temperature probe, close sample box lid and allow sample to off-gas for 15 minutes.

9.1.10 Attach the MINICAMS probe or distal end of HSL to the heated sample box. Monitor headspace for two complete cycles on the MINICAMS.

a. If MINICAMS result is below 0.25 VSL, go to step 9.1.8 for next sample container. After completing all samples (maximum 10), perform an ending challenge to bracket these results. If the challenge is successful then go to step 9.1.12. If two consecutive challenges fail, the samples are not clear and must be reanalyzed.

b. If MINICAMS result is above 0.25 VSL, sample must be confirmed with DAAMS tubes. Go to step 9.1.11.

9.1.11 Contact DAAMS technician to collect confirmation samples for the selected soils.

a. If agent is detected from DAAMS tubes analysis, the EML Branch Chief and/or Air Monitoring Manager must be immediately notified. The Site-Safety Officer and/or Site Manager must also be immediately notified.

b. If DAAMS analysis results in a non-detect for agent, go to step 9.1.12.

9.1.12 Give clear samples to the contractor/government personnel for proper disposition and complete any necessary transfer documents.

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9.1.13 Print out the chromatogram from the highest cycle of the 2 cycles. Label it accordingly. Print out online/past reports for those cycles; annotate all cycles. Fill out the corresponding MBFORM-37A (if using FPD), or MBFORM-37B (if using XSD).

9.2 Scrap /Bulk Item Headspace Monitoring.

9.2.1 A sample of scrap or a bulk item must be contained in an environment heated to a minimum of 70° F for 4 hours prior to monitoring with the MINICAMS/DAAMS. However, the use of a heated box may be used to raise the temperature of the items in the case of inclement weather or colder temperatures.

9.2.2 Once the item has been contained for 4 hours and meets the referenced temperatures (70°F), monitoring may proceed following these procedures. It is recommended the DAAMS collection procedures be used for items suspected of CWM contamination.

9.2.3 Samples are delivered by contractor/government to monitoring personnel with proper chain of custody documentation. Operator must not sign chain of custody unless all information has been verified as correct.

9.2.4 Ensure a passing challenge has been completed within the last 4 hours. Don mask and gloves. Connect MINICAMS probe or distal end of HSL to sample port of the heated sample box (if utilizing a hotbox) or puncture the bag containing the item with the HSL or sample probe and run two complete cycles on the MINICAMS.

a. If MINICAMS reading is below set alarm level for 2 consecutive cycles, go to step 9.2.6. After completing all samples (maximum 10), perform an ending challenge to bracket these results. If the challenge is successful then go to step 9.2.7. If two consecutive challenges fail, the samples are not clear and must be reanalyzed

b. If MINICAMS reading is above set alarm level, go to step 9.2.5.

9.2.5 Contact DAAMS technician to collect DAAMS tubes on sample.

9.2.6 Remove HSL from hotbox or bagged item. Place duct tape over puncture on bag.

9.2.7 Give clear samples back to contractor/government personnel for proper disposition and complete any necessary transfer documents.

9.2.8 Print the chromatogram from the highest cycle of the 2 cycles. Label it accordingly. Print out online/past reports for those cycles; annotate all cycles. Fill out the corresponding MBFORM-37A (if using FPD), or MBFORM-37B (if using XSD).

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LIST OF ACRONYMS

Acronym	Definition
AEL	Airborne Exposure Limit
AgF	Silver Fluoride
CASARM	Chemical Agent Standard Analytical Reference Material
CMA	Chemical Materials Agency
DAAMS	Depot Area Air Monitoring Systems
DI	Deionized
ECBC	Edgewood Chemical Biological Center
EDS	Explosive Destruction System
EDT	Ethanedithiol
EML	Environmental Monitoring Laboratory
FC	Found (or measured) concentration
FEM	First Entry Monitoring
FPD	Flame-photometric detector
GC	Gas chromatograph, gas chromatography
HSL	Heated Sample Line
IAW	In accordance with
IOP	Internal Operating Procedure
LVS	Low Volume Sampler
MINICAMS	Miniature Continuous Air Monitoring System
NRT	Near Real-Time
PCT	Pre-Concentrator Tube
QA	Quality Assurance
QC	Quality Control
QP	Quality Point
SOP	Standing Operating Procedure
STEL	Short Term Exposure Limit
TC	Target (or true) concentration
TWA	Time Weighted Average
VSL	Vapor Screening Level
XSD	Halogen Selective Detector
Z	Hazard Level

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APPENDIX I – FILLING PROCEDURE FOR GAS STANDARD BAG

Each operational day the operator shall get either a new or a previously used Tedlar® Gas Standard Bag that is still functional. If previously used, the operator shall visually check the bag and the Septa to assure that there are no leaks, which will dilute the Gas Standard and cause improper calibration results**.** Gas Standard bag size will be selected based on monitoring requirements. Calibration Standard concentrations shall be determined by the team lead and/or Air Monitoring Manager prior to beginning the bag filling operation to assure that the MINICAMS is calibrated and challenged at the correct TWA levels. The proper calibration and QC standard concentrations shall be calculated based on concentration (gas or liquid) in the tank from which the standard is being taken.

Calculations for Industrial Agents Calibration: Based on using a 1.0 liter Tedlar® bag, the following volumes should be used:

Chloroform: 4 µl of 130 ng/µl standard
Chloropicrin: 4 µl of 175 ng/µl standard
Phosgene: 1.25 mLs of 80 ppm phosgene
Cyanogen Chloride: 3.00 mLs of 100 ppm CK

Chloroform target of 0.1 ppm (.49 mg/m3)
Chloropicrin target of 0.1 ppm (0.7 mg/m3)
Phosgene target 0.1 ppm
Cyanogen chloride target 0.3 ppm

For gaseous standards, use a 1.0 liter Tedlar® bag and this formula:
X mls CK = 300 ÷ cylinder ppm
X mls Phosgene = 100 ÷ cylinder ppm

For liquid standards, concentrations are prepared by the lab. Use 4 µl of the standard for a 1.0 liter bag. Each 4 µl sample drawn would then be equivalent to 1 TWA concentration.

1. Purge the 1-liter bag with 500 to 1000 mls of Nitrogen prior to filling the gas standard bag. This is applicable to both new and used bags.
2. Using a gas-tight syringe, fill 1000 ml of Nitrogen into the syringe. Connect the syringe to the Tedlar® bag and fill the bag with the 1000 ml of Nitrogen. Using the pre-determined injection volumes, inject the agent(s) of concern into the bag.
3. During the MINICAMS Purge Cycle, the filled bag shall be attached to the MINICAMS inlet port by placing the protruding end of the valve into the inlet of the MINICAMS and hand tightening the ¼ inch nut with the Teflon® ferrule to secure the bag. The MINICAMS sample bypass valve should be closed.
4. Before the MINICAMS sample cycle begins, the operator shall manually open the valve on the bag until two (2) complete threads are visible. This same procedure shall be used

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when attaching the filled bag to the end of the sample line to permit challenges to be made through the sample line.

5. Bags shall be purged with Nitrogen and "re-filled" for each QP Challenge when challenging every 4 – 5 hours of operation during personnel safety monitoring.
6. The proper concentration of the filled bag is adversely affected by heat, cold and light, so filled bags must be stored and transported inside a closed container that keeps out light and helps maintain a consistent temperature.
7. Ensure that cylinder and vial expiration dates have not been reached. Replace prior to expiration.

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APPENDIX II – SPECIAL PROJECTS/EDS/TDC MONITORING

CMA OPERATIONS: Alternate Baseline Study- Initial 3 day baseline study, One passing challenge at the start of each operational day, then not less than 3 challenge events per station per day with a challenge event every 2 to 3 hrs. Continuing baseline study challenges shall be 4 – 5 hours as indicated below. See Table 13-4 in the CMA LMQAP, DEC 2007.

*These procedures may apply to other types of agent monitoring operations.

NOTE: When performing monitoring operations requiring Continuing Calibration Verification (CCV) every 4 – 5 hours, the following conditions shall apply for the CCV challenges: All heated sample lines not in use shall have both ends capped to preclude improper or inadvertent connection or use of the HSL.

A 1Z CCV challenge shall be performed for every 4 – 5 hours of operations and/or at the end of the operational day. A second challenge is permitted if the first challenge fails to meet requirements. The HSL will be reconnected immediately after the QP verification challenge only if the background being monitored is relatively free of contamination. Monitored areas with high background levels such as EDS area could cause the CCV challenge results to be biased to a degree that results in an incorrect calibration verification. If the background/ contamination is such that the MINICAMS calibration cannot be performed or verified by performing a CCV challenge, the MINICAMS Operator will notify personnel that the worker PPE Level of Protection shall be adjusted accordingly.

RATIONALE: Reconnecting the HSL immediately after performing the QP verification challenge in a highly contaminated area can cause the MINICAMS to indicate incorrectly that the calibration is still valid and the instrument is operating properly. To preclude this biasing of the calibration and/or CCV challenge, the MINICAMS must be calibrated and checked in a relatively clean environment to provide assurance that all results are accurate and reliable within the specified calibration range. After the MINICAMS is operating correctly and the HSL is reconnected, the background level readings are based on the proper calibration criteria since the MINICAMS is calibrated and operating properly.

Worker protection CCV challenges are normally performed during scheduled work breaks so personnel are not in the "Hot Area" during CCV challenges.

When monitoring high contamination areas, the MINICAMS operator may (at the operators discretion) inject a 1Z environment challenge of the agent(s) of interest into the injection port to provide assurance that the agent of interest is still within the agent window. No quantification results will be required, however the operator will label and print chromatogram & parameters, and make a note of this challenge on the QP Challenge sheet in the comments section.

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APPENDIX III – FIRST ENTRY MONITORING (FEM)

First Entry Monitoring will be conducted, prior to personnel entry, to determine the potential contamination of an enclosed area that was previously contaminated or has not been under continuous monitoring. This includes Interim Holding Facilities (IHF), storage bunkers, rooms, etc. First entries require NRT monitoring at the STEL.

To perform FEM, the corresponding MINICAMS will be calibrated for the agent(s) of interest. Following a successful QP challenge, the sample line(s) will be placed for FEM as appropriate. Two complete sampling cycles will be performed. Results of these cycles must be two (2) non-increasing consecutive readings below the alarm level, or three (3) consecutive readings below 0.25 STEL. Following FEM, an ending challenging on the MINICAMS will be performed. If the first ending challenge fails, a second challenge must be completed. If two challenges fail, FEM is unsuccessful and must be repeated. Confirmation monitoring is available upon request.

APPENDIX IV – OFFSITE WASTE SHIPMENT MONITORING

For operations involving offsite waste shipment from Chemical Depots to approved Destruction or Incineration Facilities, monitoring of the shipping vehicle will be performed at the VSL. The vehicles/trailers will be monitored before and after loading at the Chemical Depots, and once again before and after unloading at the receiving facility.

The MINICAMS will be calibrated of the agent(s) of interest. Following a successful QP challenge, monitoring can be performed. Vehicles/trailers will be monitored for 2 complete sampling cycles prior to loading to ensure no interferences are present in the empty trailer. Following loading operations, each trailer will be closed and allowed to sit for at least 15 minutes. Then each trailer will be monitored to establish pre-shipment conditions. Prior to unloading operations, each vehicle/trailer will be sampled to determine the "as found" condition. If the results of the "as found" yield two consecutive, non-increasing results below the alarm level, or three consecutive readings below 0.25 VSL, the trailer is unloaded. If the results are above the alarm level, confirmation monitoring will be performed, and the receiving facility will implement contingency plans. After contingency plans have been implemented, monitoring will continue until acceptable readings have been achieved. During unloading, the MINICAMS sample line will be placed in the workspace area where unloading is taking place. Following unloading, the vehicle/trailer will be closed, and re-monitored. Monitoring results of the empty trailers must yield at least two consecutive, non-increasing results below 0.25 VSL, or three consecutive readings below 0.25 VSL.

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APPENDIX V – HEATED SAMPLE LINE FLUSHING PROCEDURE

Heated Sample Lines must be cleaned with solvent on a periodic basis in order to maintain agent transmission efficiency, and to minimize chromatographic background noise. The solvents commonly used are acetone and isopropyl alcohol.

The frequency for line cleaning is determined by the agents of interest and environmental conditions. When monitoring for L, HN-1, and HN-3, the frequency shall be weekly. For all other agents, the frequency is on an "as needed" basis, which will be determined by the operator, MINICAMS team leader, and/or Air Monitoring Manager.

a. The HSL must be unplugged and allowed to cool down prior to introducing solvents.

b. Connect the vacuum side of a sample pump to the side port of an Ehrlenmeyer flask, and connect the distal end of a HSL to the top port of the flask. It is important to connect the HSL this way to properly flush away from where the HSL connects to the MINICAMS.

c. With the sample pump running, allow ~100 ml of acetone to be pulled into the inlet end of the HSL. Visually inspect the flask to make sure that the acetone is being collected into the flask.

d. When the acetone begins to slowly drip into the flask, plug the HSL back into power and immediately allow ~100 ml of isopropyl alcohol to be pulled into the inlet end of the HSL.

e. Ater the solvent has been entirely pulled through the line, allow a minimum of 15 minutes of vacuum to pull fresh air through the HSL.

f. The solvent waste mixture in the flask must be transferred to an appropriately labeled storage container for future waste disposal. This container should be labeled "50/50 Acetone/IPA Waste." It is not acceptable to store the solvent waste in the flask.

g. The MBFORM-04 should be annotated that the HSL was cleaned.

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APPENDIX VI – LINE CHALLENGE PROCEDURES – FIXED-SITE OPERATIONS ONLY

The frequency of line challenges is specified in section 4.8.5 of the ECBC EML LMQCP. For normal fixed-site operations, lines are challenged every three months (not to exceed 100 working days). For CMA operations, lines are challenged every two months (not to exceed 60 working days). Operators will follow the procedures listed here all HSL challenges. Heated sample lines not in use shall be capped on both ends to prevent inadvertent connections or use.

Heated Sample Lines will have injections made at the distal end on a periodic basis to ensure that challenges at the MINICAMS inlet and at the distal end of a HSL are within acceptable criteria. This is known as a "line challenge", and demonstrates the transmission efficiency of chemical agents through a HSL and into the MINICAMS. When monitoring for Lewisite, all challenges are injected into the probe connected to the HSL, and thus are not injected directly at the MINICAMS. However, operator will still reprint a copy of the challenge shots; label and complete a separate MBFORM-34.

a. Calibrate the MINICAMS as required in this IOP. Perform a successful 1Z challenge to verify acceptable calibration.

b. Make sure that the HSL is connected to the MINICAMS. If using a stream selector, all HSL's will be connected prior to the calibration. Calibration/challenge injections can be made at the auxiliary port or the inlet. When using a stream selector, all "line challenges" will be made at the distal end.

c. Perform a 1Z challenge for all agents of concern at the distal end (point at which the HSL is collecting air sample) of the HSL. While the MINICAMS is in "Sample" mode, connect a calibrated flow meter to the distal end of the HSL and record the flow rate. Acceptable flow rates must be ±25% of the target flow rate. If the flow rate is out of range, check for leaks/restrictions/loose fittings/altitude setting.

d. The line challenge is acceptable if the results are 1Z ± 25% (0.75 – 1.25). If the 1Z challenge fails, perform a second 1Z challenge. If the second challenge fails, operator must perform appropriate corrective actions.

e. Blank cycles can be collected before and after the challenge to verify absence of background interference and/or carryover.

f. Label all line challenge chromatograms with "4 UL HSL CHALL location initials." Insure to include the HSL serial number.

g. Print out chromatograms, Operating Conditions, and online/past reports for corresponding HSL challenges. Label all as such.

h. Fill out MBFORM-34 with the results of the challenge. Record the measured flow rate at the distal end. The distal end flow rate shall also be annotated on the operating conditions from the MINICAMS cycle that the challenge was made. MBFORM34 and corresponding documents will be provided to QA/QC.

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Internal Operating Procedure: Directorate of Program Integration

Environmental Monitoring Laboratory

Title: **Analysis of Chemical Warfare Agents in Extracts using a Gas Chromatograph/Mass Spectrometer System**

Division: Chemical Operations

Branch: Environmental Monitoring Laboratory

Building/Area: E3330 and other areas

This internal operating procedure covers operations, methods, and procedures of a general nature not covered by a standing operating procedure. This procedure will be effective until rescinded or superseded.

Changes to this procedure will be accomplished by submission of revisions or amendments for approval. This IOP meets "Written Method" requirements of Revision 6 of the CASARM QA Plan.

Revised By:	Jill M. Meuser	11/7/2011 Date
Analytical Team Leader	John L. Schwarz	11/7/11 Date
Quality Assurance Coordinator	Paul L. Stewart	11-7-2011 Date
Environmental Monitoring Laboratory Chief	Steven D. Norman	11/7/11 Date

Date: November 2011

IOP Number: MT-8

Revision Number: 8

Revised by: J. M. Meuser
(410) 436-6731

Approved by: S. D. Norman
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Record of Change Page

- Revision 8 – Update CASARM QA Plan version on cover page. Add language in Section 6 to allow deviation from typical sample aliquot size and extraction volumes.
- Revision 7 – Correct section references. Add extraction protocols to provide additional details that support SOP CNG-044.
- Revision 6 – Change QC limits for matrix spikes to the limits calculated for lab control spikes. Change lab control spike recovery criterion to apply to each sample individually, not to the average of the LCS and LCSD. Add table summarizing QC criteria. These changes bring IOP into conformance with DoD Quality Systems Manual Version 3.

  Deletes Appendix I and incorporates requirements into Section 7.7.5.

  TDG removed as target analyte and VR added as target analyte. Added distinction between PQL and LOQ. Added calculations for all matrices. Minor text changes made throughout.
- Revision 5 – Changes the acceptance criterion for Method Blanks from below the PQL to below one-half the PQL. See Section 7.6.1.
- Revision 4 – Add use of OCAD for OPCW projects in paragraph 5.2. Adds paragraph 5.3 for non-OPCW projects.
- Revision 3 – Analytes Q, L1, L2, L3, and CN added to text throughout and to Table 1. Section 3.2.1 and 3.2.2 updated to include other modes of injection and other column phases. Practical Quantitation Limit (PQL) definition added to Section 6.4.4.1 in place of Method Quantitation Limit. Section 6.4.6 Internal Standard Calibration Option added. Appendix II updated to include LCS/LCSD and MS/MSD criteria for analytes where no laboratory QC data currently exists. Minor text changes made throughout.
- Revision 2 – Changes made to amount of BFB injected for tuning and combining BFB tune verification with calibration verification. Surrogate and matrix spike compounds and concentrations added. Extract holding time added. Retention time criteria added. Data qualifiers added. Requirement of CCV every 10 samples removed.
- Revision 1 – Table of Contents, Record of Change added
- Revision 0 – IOP created.

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Signature Page

Title: Analysis of Chemical Warfare Agents in Extracts using a Gas chromatograph/Mass Spectrometer System

Operator's Statement: I have read, or have had read to me, the procedures in this IOP. I, by my signature below, indicate that I thoroughly understand and agree to abide by these instructions.

Name (print)	Signature	Date

Supervisor's Statement: I have personally reviewed this IOP and, to the best of my knowledge, believe that the information listed herein is correct.

Supervisor: _____ Date:_____
Steven D. Norman

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Analysis of Chemical Warfare Agents in Extracts using a Gas Chromatograph/Mass Spectrometer System

1.0 SCOPE AND APPLICATION

1.1 This method is used to determine Chemical Warfare Agents and their breakdown products in a variety of matrices. This method is applicable to nearly all types of samples, regardless of water content, including ground and surface water, aqueous sludges, wipes, soils, lime, and tissue. The following compounds can be determined by this method:

GA, GB, GD, GF, HD, L, HN-1, HN-3, 1,4-Dithiane, 1,4-Thioxane,
VX, VR, CEES, TDP, CN, Q, L1, L2, L3
(For L1, L2, L3 reference ECBC SOP CNG-012 for extraction procedure)

Other compounds and matrices (filter cakes, coupons, spent carbon, spent catalysts, hydrolysate, and sediments) may be determined as long as there is proper demonstration that all quality control criteria can be met. Not all compounds may be able to be determined simultaneously.

1.2 The Practical Quantitation Limit (PQL) for determining an individual compound is approximately 100 – 200 µg/Kg (wet weight) for soil/sediment samples and 100 – 200 µg/L for water samples (10 – 20 µg/Kg or 10 – 20 µg/L for HD). PQLs will be proportionately higher for sample extracts that require dilution to avoid saturation of the detector. Data will be reported to the higher of the PQL or the lowest calibration standard; this value is the Limit of Quantitation (LOQ).

1.3 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatograph/mass spectrometers, and skilled in the interpretation of mass spectra and their use as a quantitative tool.

2.0 ADDITIONAL NON-STANDARD RESPONSIBILITIES

2.1 There are no additional responsibilities for the supervisor, operator, or the supporting activities other than those outlined in Appendix O in the SOP Guidebook.

3.0 APPARATUS AND MATERIALS

3.1 Chemical Reagents and Solvents

3.1.1 Methylene Chloride, CH_2Cl_2 with 0.1 % Betamercaptoethanol (BME)

3.1.2 QC Sand

3.1.3 Distilled Deonized (D.I.) Water, Milli-Q Water (18 MΩ)

3.1.4 Surrogate/Internal Standard Solution: 4-Bromofluorobenzene (BFB), C_6H_4BrF, 5,000 µg/L and Hexachlorobenzene-C^{13} (HCB), C_6Cl_6, 5,000 µg/L

3.1.5 Calibration Standard containing all target analytes, 100, 150, 200, 250, and 500 µg/L except HD. HD at 10, 15, 20, 25, and 50 µg/L.

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3.1.6 Calibration Verification Standard containing all target analytes, made from different source material than used for calibration standard, 250 µg/L except HD. HD at 25 µg/L.

3.1.7 Spiking Standard(s) containing all target analytes, made from different source material than used for calibration standard, 5,000 µg/L except HD. HD at 500 µg/L.

3.1.8 Bleach for Decon

3.2 Extraction Accessories

3.2.1 16 mm x 125 mm round-bottomed centrifuge tubes

3.2.2 Re-pipetter with disposable tips

3.2.3 Serological Pipette, 2 mL

3.2.4 Pipette Bulbs, large and small

3.2.5 Glass Pasteur Pipette, 22 cm

3.2.6 120-Volt MiniVortexer, or equivalent

3.2.7 Milligram Balance

3.2.8 2-mL Amber Autosampler Vials with Crimp Caps

3.2.9 Autosampler Vial Crimper

3.2.10 Autosampler Vial De-crimper

3.2.11 Disposable Spatulas

3.2.12 2-in x 2-in Gauze Wipes

3.2.13 40-mL Amber Vials

3.2.14 3-mL disposable syringes equipped with 0.45 µm PTFE filters

3.2.15 Centrifuge

3.3 Gas Chromatograph/Mass Spectrometer System

3.3.1 Gas chromatograph (GC) - An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. Other modes of injection may be used as appropriate after successful completion of an analyte Method Detection Limit (MDL) study in accordance with 40 CFR 136 Appendix B.

3.3.2 Column - 30 m x 0.25 mm, ID 1 µm film thickness, silicone-coated fused-silica capillary column (J&W DB-1701 or equivalent). Other column phases may be used as appropriate after successful completion of an analyte MDL study in accordance with 40 CFR 136 Appendix B.

3.3.3 The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout the temperature program operation.

3.4 Mass Spectrometer

3.4.1 Mass spectrometer (MS) - Capable of scanning from 35 to 300 amu every 2 seconds or less, using 70 volts (nominal) electron energy in the electron impact ionization mode.

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The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the criteria in Table 1 when 1 – 50 ng of the GC/MS tuning standard (BFB) are injected into the GC. To ensure sufficient precision of mass spectral data, the desirable MS scan rate allows acquisition of at least ten spectra while a sample component elutes from the GC.

3.4.2 GC/MS interface - Any GC-to-MS interface may be used that gives acceptable calibration points at 2ng or less per injection for each compound of interest and achieves acceptable tuning performance criteria. For a narrow-bore capillary column, the interface is usually capillary-direct into the mass spectrometer source.

3.5 Data System

3.5.1 Data system - A computer system should be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer should have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software should also be available that allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

4.0 HAZARDS

4.1 Dilute Chemical Warfare Agents

4.1.1 The Environmental Monitoring Laboratory utilizes near drinking water level dilute chemical warfare agents in preparing quality control samples for the IOP. The Environmental Monitoring Laboratory office contains a reference file of material safety data sheets (MSDS) for the dilute CW agents.

4.2 Solvents and Other materials

4.2.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available which includes, but is not limited to, donning the appropriate laboratory safety attire and working in well-ventilated hoods. The laboratory is responsible for maintaining a current file of Occupational Safety and Health Administrations (OSHA) regulations regarding the safe handling of the chemical specified in this IOP. A reference file of material safety data sheets (MSDS) of materials used in these operations is available at the worksite.

5.0 SUMMARY OF METHOD

5.1 The CWAs are introduced into the GC/MS by injecting the sample extract into a Gas Chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a Mass

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Spectrometer (MS) connected to the gas chromatograph. Analytes eluted from the capillary column are introduced into the mass spectrometer via direct connection.

5.2 For OPCW projects, identification of target analytes is accomplished by comparing the mass spectra from the sample with electron impact mass spectra of standards of target analytes contained in the version of OCAD delivered by the OPCW for the purpose of this verification activity. The comparison is made using one of the following: Agilent ChemStation Data Analysis software, AMDIS or NIST Search. The minimum NET match factor for successful identification is ≥ 80. Retention time of the target analyte in the sample should not differ from the standard more than ±0.1 min. Quantification is accomplished by comparing the response of a major (quantification) ion to a known amount using a calibration curve calculated from a minimum of three standards. The target ions for the list of analytes associated with this method is are shown in Table 2. The responses of the qualifier ions should fall within an acceptance window of 20 % relative to the calibration standard ion ratio response. The software will adjust the upper and lower ratios upon calibration. Page three and following of the ChemStation report contain the ratio information. It is acknowledged that interferences can cause the ion ratio to differ; the analyst will use professional judgment in interpreting data which deviates from the above acceptance criteria.

5.3 For non-OPCW projects, identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion to a known amount using a five-point calibration curve.

6.0 EXTRACTION PROCEDURE

Sample aliquot sizes and extraction solvent volumes provided in the following sections are typical, but may be modified based on sample characteristics or project requirements. Deviations to the extraction procedures described below must be noted in the analytical narrative and/or results report.

Volumes given below for surrogate/internal standard and spiking solutions are based on solution concentrations given in Section 3.1. Changes in solution concentrations will require modification of the volumes given below.

6.1 Water and Other Liquids

6.1.1 For method blank (MB), lab control spike (LCS), and lab control spike duplicate (LCSD), measure 2 mL D.I. water into a 16-mm Centrifuge Tube. Record the exact volume to 3 significant figures. If the sample matrix is not aqueous, clean matrix as similar as possible to the sample should be used.

6.1.2 For matrix spike (MS), matrix spike duplicate (MSD), and samples, measure 2 mL of sample into a 16-mm Centrifuge Tube. Record the exact volume to 3 significant figures.

6.1.3 For LCS, LCSD, MS, and MSD, add 100 µL of each spiking standard to each sample.

6.1.4 Add 100 µL of BFB/HCB surrogate/internal standard to each sample.

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6.1.5 Add 1.9 mL of CH_2Cl_2 with 0.1 % BME to the MB and samples and 1.7 mL to the LCS, LCSD, MS, and MSD.

6.1.6 Vortex each tube for 10 seconds to mix. Allow sufficient time for solvent layer to separate after vortexing. If the layers do not separate (e.g., organic liquid matrix), add 100 µL of BFB/HCB surrogate/internal standard to each tube and 100 µL of each spiking standard to the MS and MSD. Add 100 µL of each spiking solution to the LCS and LCSD if organic liquid was used as the clean matrix.

6.1.7 Transfer the bottom (organic layer) into a 2-mL autosampler vial. Cap the Vial. The sample is now ready for analysis.

6.2 Soil and Other Unknown Solids

6.2.1 For MB, LCS, and LCSD, weigh approximately 2 g of QC sand into a 16-mm Centrifuge Tube. Record the exact weight to 3 significant figures.

6.2.2 For MS, MSD, and samples, weigh approximately 2g of sample into a 16-mm Centrifuge Tube. Record the exact weight to 3 significant figures.

6.2.3 For LCS, LCSD, MS, and MSD, add 100 µL of each spiking standard to each sample.

6.2.4 Add 100 µL of BFB/HCB surrogate/internal standard to each sample.

6.2.5 Add 1.9 mL of CH_2Cl_2 with 0.1 % BME to the MB and samples and 1.7 mL to the LCS, LCSD, MS, and MSD.

6.2.6 Vortex each tube for 10 seconds to mix. Allow sufficient time for solvent layer to separate.

6.2.7 Transfer the liquid into a 3-mL syringe fitted with a 0.45 µm PTFE filter tip. Gently push the barrel of the syringe to pass the liquid through the filter into a 2-mL autosampler vial. Cap the Vial. The sample is now ready for analysis.

6.3 Lime

6.3.1 For MB, LCS, and LCSD, weigh approximately 2 g of clean lime into a 16-mm Centrifuge Tube. Record the exact weight to 3 significant figures.

6.3.2 For MS, MSD, and samples, weigh approximately 2g of sample into a 16-mm Centrifuge Tube. Record the exact weight to 3 significant figures.

6.3.3 For LCS, LCSD, MS, and MSD add 200 µL of each spiking standard to each sample.

6.3.4 Add 200 µL of BFB/HCB surrogate/internal standard to each sample.

6.3.5 Add 3.8 mL of CH_2Cl_2 with 0.1 % BME to the MB and samples and 3.4 mL to the LCS, LCSD, MS, and MSD. [Note: The volume of extraction solvent is twice that used for soils/solids. The LOQ for lime is twice the LOQ for soils/solids.]

6.3.6 Place the tube in a centrifuge and rotate at 3000 RPM for 5 minutes. Allow sufficient time for solvent layer to separate.

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6.3.7 Transfer the liquid into a 3-mL syringe fitted with a 0.45 µm PTFE filter tip. Gently push the barrel of the syringe to pass the liquid through the filter into a 2-mL autosampler vial. Cap the Vial. The sample is now ready for analysis.

6.4 Fish Tissue

6.4.1 Purchase a whole fish from a commercial supermarket to prepare fish tissue as the clean matrix for MB, LCS, and LCSD. After thawing as necessary, remove the fillets from the fish utilizing a filleting knife. Cut the fillets into pieces and then pulverize with a mortar and pestle into a single homogenous sample.

6.4.2 If samples do not arrived prepared, i.e., pulverized and homogenized, prepare the tissue as described in Section 6.4.1 above. If samples arrived at the laboratory frozen, thaw the matrix before starting the extraction procedure.

6.4.3 For MB, LCS, LCSD, weigh approximately 2 g of thawed, homogenized fish tissue into a 16-mm Centrifuge Tube. Record the exact weight to 3 significant figures.

6.4.4 For MS, MSD, and samples weigh approximately 2 g of thawed, homogenized sample to a 16-mm Centrifuge Tube. Record the exact weight to 3 significant figures.

6.4.5 For LCS, LCSD, MS, MSD, add 100 µL of each spiking standard to each sample.

6.4.6 Add 100 µL of BFB/HCB surrogate/internal standard to each sample.

6.4.7 Add 1.9 mL of CH_2Cl_2 with 0.1 % BME to the MB and samples and 1.7 mL to the LCS, LCSD, MS, and MSD.

6.4.8 Vortex each tube for 10 seconds to mix. Allow sufficient time for solvent layer to separate.

6.4.9 Transfer the liquid into a 3-mL syringe fitted with a 0.45 µm PTFE filter tip. Gently push the barrel of the syringe to pass the liquid through the filter into a 2-mL autosampler vial. Cap the Vial. The sample is now ready for analysis.

6.5 Wipes

6.5.1 For MB, LCS, and LCSD, place a clean 2″ x 2″ wipe into a 40-mL Amber Vial.

6.5.2 For the sample, remove the wipe from the receiving container and place it in a 40-mL Amber Vial.

6.5.3 For LCS, LCSD, add 500 µL of each spiking standard to each sample.

6.5.4 Add 500 µL of BFB/HCB surrogate/internal standard to each sample.

6.5.5 Add 9.5 mL of CH_2Cl_2 with 0.1 % BME to the MB and samples and 8.5 mL to the LCS and LCSD.

6.5.6 Shake each tube for 10 seconds followed by vortexing each tube for 20 seconds. Finally, insert a new, clean disposable spatula into the tube and squeeze the liquid out of the wipe by pressing on it.

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6.5.7 Transfer the liquid into a 3-mL syringe with a 0.45 um PTFE filter tip and. Gently push the barrel of the syringes to pass the liquid through the filter into a 2-mL autosampler vial. Cap the Vial. The sample is now ready for analysis.

7.0 ANALYSIS PROCEDURE

7.1 Typical Chromatographic Conditions

7.1.1 Injector temperature: 250 °C

7.1.2 Carrier gas (He) flow rate: 1.5 mL/min

7.1.3 Column: J&W DB-1701, 30m x 0.25 mm x 1 µm (or equivalent)

7.1.4 Initial temperature: 50 °C, hold for 1 minute

7.1.5 Temperature program: 25 °C/min

7.1.6 Final temperature: 260 °C, hold for 2.0 minutes

7.1.7 Post Time: 2.5 minutes

7.2 Typical MSD Conditions

7.2.1 Mass range: 35 – 260 amu (full scan) or Selected Ion Monitoring (SIM)

7.2.2 Scan time: 0.6 – 2 sec/scan

7.2.3 Source temperature: 230 °C

7.3 Tuning the MSD

7.3.1 Each GC/MS system must be hardware-tuned to meet the criteria in Table 2 for a 1 – 50 ng injection of 4-bromofluorobenzene. Analyses must not begin until these criteria are met.

7.3.1.1 The mass spectrum of BFB must be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak.

7.3.1.2 Use the BFB mass intensity criteria in Table 2 as tuning acceptance criteria.

NOTE: All subsequent standards, samples, MS/MSDs, LCS/LCSDs, and blanks associated with a BFB analysis must use identical mass spectrometer instrument conditions.

7.4 Initial Calibration

7.4.1 Calibration must be performed using the sample introduction technique that will be used for samples.

7.4.2 Proceed with the analysis of the calibration standards following the procedure in the introduction method of choice. For direct injection, inject 1 µL – 2 µL into the GC/MS

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system. The injection volume will depend upon the chromatographic column chosen and the tolerance of the specific GC/MS.

7.4.3 Calibration of an analytical instrument involves the delineation of the relationship between the response of the instrument and the amount, or concentration, of an analyte introduced into the instrument. The graphical depiction of this relationship is often referred to as the calibration curve. In order to perform quantitative measurements, this relationship must be established before the analysis of any samples, and thus, is termed initial calibration. The initial calibration for this chromatographic method involves the analysis of standards containing the target compounds at a minimum of five different concentrations covering the working range of the instrument. In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration. The extrapolation of the calibration to concentrations above or below those of the actual calibration standards is not appropriate and may lead to significant quantitative errors regardless of the calibration model chosen. Analysts are advised that it may be necessary to prepare calibration standards that cover concentration ranges that are appropriate for specific projects or type of analyses.

7.4.4 For each analyte and surrogate of interest, prepare calibration standards at a minimum of five different concentrations by adding volumes of one or more stock standards and diluting to volume with an appropriate solvent. Typical calibration levels range from 100 µg/L to 500 µg/L (10 µg/L to 50 µg/L for HD).

7.4.4.1 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards be stored at minus 4 °C or less, in amber bottles with PTFE-lined screw-caps.

7.4.4.2 The lowest concentration calibration standard that is analyzed during an initial calibration establishes the Limit of Quantitation (LOQ) based on the final volume of extract (or sample) described in the preparative method or employed by the laboratory. In some cases, the LOQ of an analyte may be higher that the lowest concentration calibration standard, if the calculated MDL is greater than 1/3 the concentration of the lowest standard.

7.4.4.3 The other concentrations should define the linear working range of the detector or correspond to the expected range of concentrations found in actual samples that are also within the working range of the detector.

7.4.4.4 For each analyte, at least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project, which may include establishing compliance with a regulatory or action limit.

7.4.5 External standard calibration procedure

7.4.5.1 External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas are compared to peak areas of the standards.

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The ratio of the detector response to the amount (mass) of analyte in the calibration standard is defined as the calibration factor (CF).

7.4.5.2 Tabulate the area response of the characteristic ions (see Table 1) against the concentration for each target analyte.

7.4.5.3 Linear calibration using a least squares regression

7.4.5.3.1 The analyst must employ a regression equation that does not pass through the origin. This is most easily achieved by performing a linear regression of the instrument response versus the concentration of the standards. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). This is a statistical requirement and is not simply a graphical convention. For all instances, an appropriate unweighted least squares method should be used. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = mx + b$$

where:
y = Instrument response (peak area or height)
m = Slope of the line (also called the coefficient of x)
x = Concentration of the calibration standard
b = Y-intercept

The analyst must not force the line through the origin, but have the intercept calculated from the five data points. The use of a linear regression may not be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. The regression calculation will generate a correlation coefficient (r^2) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.000 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.990.

7.4.5.3.2 In calculating sample concentrations by the external standard method, the regression equation is rearranged to solve for the concentration (x), as shown below.

$$x = \frac{y - b}{m}$$

7.4.6 Internal standard calibration procedure (Preferred)

7.4.6.1 Internal standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards as normalized by the sample or standard internal standard response. Sample response factors are compared to response factors of the standards.

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This calibration procedure is acceptable for use after successful completion of an analyte MDL study in accordance with 40 CFR 136 Appendix B.

7.4.6.2 The compound used for internal standard quantitation should be similar to the target analytes in retention time and composition but should not be present in the samples to be analyzed. The purpose of the internal standard is to correct for system sensitivity fluctuations. The internal standard area counts of each sample should fall within the QC limits of +100 % and -50 % of the integrated area counts of the internal standards from the daily check standard. Samples failing this criterion will be reanalyzed if possible. Repeated failures will be qualified as such in the case narrative. All samples and standards will be spiked to the same concentration of internal standard.

7.4.6.3 The Response Factor (RF) is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = peak area of the analyte or surrogate
A_{is} = peak area of the internal standard
C_s = concentration of the analyte or surrogate
C_{is} = concentration of the internal standard

7.4.6.4 Linear calibration using least squares regression

7.4.6.4.1 Construct a calibration curve in accordance with the specifications in Section 7.4.5.3, with the exception that the y-value is the Response Ratio $\left(\frac{A_s}{A_{is}}\right)$ and the x-value is the Amount Ratio $\left(\frac{C_s}{C_{is}}\right)$. The correlation coefficient (r^2) must be at least 0.990 for the curve to be valid.

7.4.6.4.2 In calculating sample concentrations by the internal standard method, the regression equation is rearranged to solve for the concentration (x), as shown below.

$$C_s = \frac{C_{is} \times \left(\frac{A_s}{A_{is}} - b\right)}{m}$$

7.4.7 Initial calibration verification

7.4.7.1 Immediately following the initial calibration, initial calibration verification (ICV) must be performed. This is accomplished by analyzing a calibration standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the calibration standard analysis must meet the verification acceptance criteria in Section 7.5.2.

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7.4.7.2 The calibration verification spiking standard must not be prepared from the same standards as the initial calibration standards. However, the same spiking standard prepared for the MS, MSD, LCS, and LCSD may be used.

7.5 GC/MS Tuning and Calibration Verification – Tuning verification is performed at the beginning of each 12-hour analytical shift. Continuing calibration verification (CCV) consists of analysis of a midpoint calibration standard from a different source than the initial calibration. It is recommended, but not required, that additional CCVs be analyzed throughout the sequence (e.g., every 10 samples).

7.5.1 Before analyzing samples or calibration standards, inject or introduce 1 – 50 ng of the 4-bromofluorobenzene standard into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria in Table 2 before sample analysis begins. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

NOTE: The BFB and calibration verification (CCV or QC Check) standard may be combined into a single standard as long as both tuning and calibration verification acceptance criteria for the project can be met without interferences.

7.5.2 The initial calibration curve (Section 7.4) for each compound of interest should be verified not less than once every 12 hours prior to sample analysis using the introduction technique used for samples. This is accomplished by analyzing a CCV standard at a concentration near the midpoint concentration for the calibrating range of the GC/MS. The results from the CCV standard analysis must meet the following verification acceptance criteria:

7.5.2.1 If the percent difference between found and true amounts for each target agent and surrogate is less than or equal to ±20% (±25% for VX, L, CN, Q, L1, L2, L3), the initial calibration is assumed to be valid.

7.5.2.2 If the criterion is not met (i.e., greater than ±20% difference, ±25% for VX, L, CN, Q, L1, L2, L3), for any one agent, then corrective action must be taken prior to the analysis of samples. If the agent(s) are not included in the list of analytes for a project, no corrective action is required.

NOTE: If the CCV fails, one additional CCV may be analyzed. If the additional CCV is analyzed and the percent difference between found and true amounts for the CCV is greater than -20% (-25% for VX, L, CN, Q, L1, L2, L3), all samples prior to the failing CCV and after the last acceptable CCV must be reanalyzed. If the percent difference between found and true amounts for the CCV is greater than +20% (+25% for VX, L, CN, Q, L1, L2, L3), analysis must stop however previous samples may be reported.

7.5.3 The calibration verification spiking (CCV or QC) standard must not be prepared from the same standards as the initial calibration standards. However, the same spiking standard prepared for the matrix spike and LCS may be used.

7.6 Method Blank – A method blank should be analyzed after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (extraction protocol,

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introduction device, transfer lines, and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Section 8.7.5 for method blank performance criteria.

7.7 GC/MS Analysis of Samples

7.7.1 BFB tuning criteria and GC/MS calibration verification criteria as described in Sections 7.3, 7.4, and 7.5 must be met before analyzing samples.

7.7.2 All samples and standard solutions must be allowed to warm to ambient temperature before analysis. Set up the introduction device as outlined in the method of choice. Extracts must be analyzed within forty (40) days from the date extraction was completed.

7.7.3 Inject a 1 µL – 2 µL aliquot of the sample extract into the GC/MS system, using the same operating conditions that were used for the calibration (Section 7.4). The injection volume must be the same volume used for the calibration standards.

7.7.4 A typical analytical sequence, with and without initial calibration is shown in Appendix I.

7.8 Qualitative Analysis

7.8.1 The qualitative identification of each compound determined by this method is based on retention time, and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. In some cases, a higher mass, more unique ion with an intensity of less than 30% may be used due to interferences in the lower mass range. Compounds are identified as present when the following criteria are met:

7.8.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

7.8.1.2 The relative intensities of the characteristic ions agree within ±30 percentage points of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

7.9 Quantitative Analysis

7.9.1 After a compound has been identified, the quantitation of that compound will be based on the integrated abundance from the EICP of the primary characteristic ion.

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7.9.2 The concentration in the extract is determined from initial calibration data using the calculation in Section 7.4.5.3.2 if external calibration is used; the concentration in the extract is determined using the equation in Section 7.4.6.4.2 if an internal standard is used.

7.9.3 If the concentration of any target analyte is below the current laboratory LOQ but above the laboratory MDL, the agent concentration is flagged with the "J" qualifier. A concentration below the MDL is considered a non-detect.

7.9.4 If the concentration of any target analyte is above the concentration of the highest level of the initial calibration, a dilution must be performed if there is sufficient sample, and the diluted aliquot reanalyzed. If there is not sufficient sample for dilution, the concentration is considered an estimate and the reported result is flagged with the "E" qualifier. Any concentrations in a dilution analysis must be flagged with the "D" qualifier.

7.9.4.1 The sample should be diluted so that the concentration of the highest agent falls within the range of the mid-level to upper level of the calibration. The amount of dilution, the target dilution factor (DF), is calculated as follows:

$$\text{Target DF} = \frac{\text{Highest concentration found}}{\text{Concentration of mid} - \text{level}}$$

(Round down to the nearest whole number. If greater than 5, round down to nearest multiple of 5.)

7.9.4.2 For example:

If an analyte were detected at 1000 µg/L and the upper calibration range for that analyte were 500 µg/L, then the extract must be diluted. If the mid-level calibration standard was 250 µg/L, the TDF would be 4 (1000/250 = 4). If the analyte were detected at 3,000 µg/L the TDF would be 10 (3000/250 = 12; round down to nearest 5 = 10).

7.9.5 Calculate the concentration of target analyte(s) in the original matrix, using the appropriate formula as shown in Section 9.

7.9.6 All concentrations are reported to two significant figures, in terms of the original matrix. Results are generally in units of µg/L or µg/Kg, but may be mg/L, mg/Kg, or %. Units must be consistent within a project.

7.9.7 If an analyte is not detected, the data is reported as "less than the LOQ" ("< [numerical value of LOQ]"). A concentration below the MDL is considered a non-detect.

7.9.8 If the concentration of an analyte is below the current laboratory Limit of Quantitation (LOQ) but above the laboratory Method Detection Limit (MDL), the agent concentration is flagged with the "J" qualifier.

7.9.9 If a target analyte is detected in any sample and was detected in the method blank, all results are flagged with the "B" qualifier.

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8.0 QUALITY CONTROL

8.1 Program Requirement – Each laboratory must maintain a formal quality assurance program. The laboratory must also maintain records to document the quality of the data generated.

8.2 Method Detection Limit – At least annually, the MDL shall be determined in accordance with the protocol in 40 CFR 136 Appendix B. The PQL shall be established at 3 times the MDL. The Limit of Quantitation (LOQ, also referred to as the quantitative reporting limit) shall be established as the PQL or the lowest calibration level, whichever is higher.

8.3 Tuning – The GC/MS system must be tuned to meet the BFB criteria listed in Table 2.

8.4 Initial Calibration – There must be an initial calibration of the GC/MS system as described in Section 7.4.

8.5 Calibration Verification – The GC/MS system must meet the calibration verification acceptance criteria in Section 7.5.

8.5.1 Retention time (RT) windows must be established by the laboratory for each instrument. These windows are determined prior to analysis of samples. The laboratory must calculate new retention time windows for each standard on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory.

8.5.2 A retention time window is calculated for each analyte, surrogate, and internal standard using the initial calibration check standard (CCV) of each sequence. The windows shall be ±0.10 minutes around the absolute retention time for each compound. Analytes are identified when peaks are observed in the RT window for the compound.

8.6 Instrument Blanks

8.6.1 Before processing any samples, the analyst must demonstrate through the analysis of an instrument blank that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, an instrument blank should be analyzed as a safeguard against chronic laboratory contamination. A manual integration for each agent requested by the client will be included with the instrument blank report.

8.6.2 All initial instrument blanks must be free of target analytes to one-half the LOQ.

8.6.3 Instrument blanks analyzed as part of the analytical sequence may not contain target analytes at a concentration greater than one-half the LOQ.

8.6.4 If a mid-run instrument blank contains one or more target analytes at or above one-half the LOQ and that analyte is detected in any unspiked sample run before that blank, corrective action (e.g., repeated analysis of instrument blanks) must be performed and documented. After the system is free of contamination, the sample(s) having detectable target analyte must be re-analyzed. All other tuning and calibration criteria must be met for the re-analysis.

8.7 Sample Quality Control for Preparation and Analysis – The laboratory must also have procedures for documenting the effect of the matrix on method performance (precision,

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accuracy, and detection limit). At a minimum, this includes the analysis of QC samples including a MB, MS, MSD, LCS, and LCSD in each analytical batch and the addition of surrogates to each field sample and QC sample.

8.7.1 Quality Control Limits for surrogate recovery and for LCS/LCSD percent recovery (%R) and relative percent difference (RPD) will be established at the mean of the %R and RPD, respectively, for the specific analyte plus or minus three times the standard deviation of recovery data. These limits shall be based on at least 30 data points generated under the same analytical process and shall not exclude failed data points or statistical outliers unless there is a documented, scientifically-valid reason (e.g., equipment malfunction, bad standard). Limits shall be updated annually or after major changes in the analytical process.

Until a sufficient number of data points are collected, the default limits shall be 50 – 150 %R and 20 % RPD.

If an out-of-statistical-control situation occurs, the analyst must examine the analytical system for unusual conditions and should perform routine maintenance as needed in an attempt to determine the cause of the situation. Recalibration of the analytical system may be required. All findings and corrective actions must be documented.

8.7.2 Great care must be taken to maintain the integrity of all standard solutions. It is recommended all standards be stored at minus 4 °C or less, in amber bottles with PTFE-lined screw-caps. Solutions used for spiking LCS, LCSD, MS, and MSD samples should not be prepared from the same standards as were used for preparing calibration standards. However, the same standards used for preparing CCV and ICV standards may be used in preparation of the LCS, LCSD, MS, and MSD spiking solutions.

8.7.3 Internal Standard (IS)

8.7.3.1 Purpose: The IS used to normalize sample responses and correct for fluctuations in system sensitivity. The recommended internal standard is hexachlorobenzene.

8.7.3.2 Frequency: IS will be added to all samples, QC samples, and standards at the same concentration.

8.7.3.3 Evaluation and Corrective Action: The internal standard area counts of each sample should fall within the QC limits of +100 % and -50 % of the integrated area counts of the internal standards from the daily check standard. Samples failing this criterion will be re-analyzed if possible. Repeated failures will be qualified as such in the case narrative. If re-extraction is not possible, the case narrative will identify the failure and analyte results for the sample will be "J" qualified.

8.7.4 Surrogate Standard (SS)

8.7.4.1 Purpose: The SS is a compound whose analytical characteristics closely resemble those of the target analytes, but which is unlikely to be found in samples. The suggested surrogate is 4-bromofluorobenzene (BFB).

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Additional or different surrogates may be used to conform to project specific requirements. The surrogate should be spiked at a level consistent with the mid-point of the instrument calibration range.

8.7.4.2 Frequency: Surrogate is spiked into all samples and QC samples at the same concentration.

8.7.4.3 Evaluation and Corrective Action: The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory, as described in Section 8.7.1. The surrogate recovery is calculated as:

$$\text{Recovery}\,(\%) = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

If the recovery is not within surrogate recovery limits, the following procedures are necessary:

- Check to be sure that there are no errors in the calculations or surrogate solution standard. If errors are found, recalculate the data accordingly.
- Rerun the extract (to ensure a proper injection). If it fails again, the sample must be re-extracted (in a new batch with new QC) and re-analyzed.
- If the re-extraction passes, then it is assumed there was a spiking error and the initial analysis is not reported. If it fails yet again, then it is assumed there is a matrix effect and both the initial and re-extraction analyses are reported.
- If re-extraction is not possible, all results for the sample with failing surrogate %R shall be reported with a "J" flag.

8.7.5 Method Blank

8.7.5.1 Purpose: The Method Blank is used to assess the preparation batch for possible contamination during the preparation and processing steps. The Method Blank shall be processed along with and under the same conditions as the associated samples to include all steps of the analytical procedure.

8.7.5.2 Frequency: A Method Blank shall be prepared and analyzed at a minimum of one per preparation batch.

8.7.5.3 Composition: The Method Blank shall consist of a quality system matrix that is similar to the samples being analyzed and is known to be free of the analytes of interest. When the sample matrix cannot be mimicked analyte-free distilled water shall be used for the Method Blank.

8.7.5.4 Evaluation and Corrective Action: If the concentration in the Method Blank exceeds one-half the LOQ, the source of the contamination shall be investigated and measures taken to minimize or eliminate the problem. All actions shall be documented.

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If the concentration in the Method Blank exceeds one-half the LOQ and any samples in the batch have one or more target analytes at a concentration greater than or equal to one-half the LOQ, those samples shall be reprocessed and re-analyzed after the source of the problem in eliminated. If the Method Blank contains one or more target analytes in the re-extracted/re-analyzed batch, any sample with a target analyte above the MDL shall be reported with a "B" qualifier. If samples cannot be reprocessed, the results shall be reported with a "B" qualifier.

8.7.6 Laboratory Control Spike (LCS)/ Laboratory Control Spike Duplicate (LCSD)

8.7.6.1 Purpose: The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Results of each sample are compared to established criteria and, if found to be outside these criteria, indicates that the analytical system is "out of control." When the results of the matrix spike analysis indicate a potential problem because of the sample matrix itself, the LCS/LCSD results are used to verify that the laboratory can perform the analysis in a clean matrix.

8.7.6.2 Frequency: One LCS and one LCSD per preparation batch.

8.7.6.3 Composition: It is generally desirable for the matrix to be similar to the matrix being tested, known to be free of analytes of interest. The samples will be spiked with IS/SS and all target analytes at or below the mid-point of the calibration range.

8.7.6.4 Evaluation and Corrective Action: The percent recovery (%R) and relative percent difference (RPD) is calculated for the LCS and LCSD, as follows:

$$\%R_{LCS} = \frac{FC_{LCS}}{TC_{LCS}} \times 100 \qquad \%R_{LCSD} = \frac{FC_{LCSD}}{TC_{LCSD}} \times 100$$

$$RPD = \frac{|FC_{LCS} - FC_{LCSD}|}{\frac{1}{2}(FC_{LCS} + FC_{LCSD})} \times 100$$

where: FC_{LCS} = Found concentration in the LCS
FC_{LCSD} = Found concentration in the LCSD
TC_{LCS} = True spike concentration in the LCS
TC_{LCSD} = True spike concentration in the LCSD

RPD must be calculated as a comparison of measured concentrations, not a comparison of %R.

All recoveries must be within current control limits, established as described in Section 8.7.1, for the system to be considered in control. If any recovery fails to meet its criterion, the failing extract must be analyzed again. If the LCS or LCSD fails yet again, it is deemed out-of-control and the entire batch

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must be re-extracted and re-analyzed after the cause of the exceedance is identified and corrected. Recalibration of the analytical system may be required. If there is insufficient material for re-extraction of any sample in the batch, the results for that sample will be reported with a "Q" qualifier. If the recovery from the LCS or LCSD fails after re-extraction and re-analysis of the batch, all sample results for the failing analyte in that batch will be reported with a "Q" qualifier.

8.7.7 Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

8.7.7.1 Purpose: The MS is used to indicate the effect of the sample matrix on the precision and accuracy of the results. The information from these controls is sample/matrix specific and not used to determine the validity of the entire batch.

8.7.7.2 Frequency: One MS and one MSD per preparation batch. If the known concentration of one or more target analytes is greater than 5 times the LOQ, a matrix duplicate shall be analyzed in place of an MSD. An MS is still required, although the concentration of the spike can be adjusted upward to ensure that the spike can be differentiated from the concentration in the un-spiked sample. For wipe samples, it is not possible to obtain multiple aliquots of the same sample. For this sample type, no MS/MSD is required.

8.7.7.3 Composition: The MS and MSD must be the same matrix as the samples being analyzed in the batch. Generally, these QC samples will be additional aliquots from one of the unknown samples in the batch. If adequate sample material is not available, the lack of MS/MSD shall be noted in the case narrative.

If the native concentration is not known, the samples will be spiked with IS/SS and all target analytes at or below the mid-point of the calibration range, using the Spiking Standard shown in Table 1. If the native concentration is known, the samples will be spiked with IS/SS and target analytes at 1 – 4 times the known concentration.

8.7.7.4 Evaluation and Corrective Action: The percent recovery (%R) and relative percent difference (RPD) is calculated for the MS and MSD, as follows:

$$\%R_{MS} = \frac{FC_{MS} - FC_P}{TC_{MS}} \times 100 \qquad \%R_{MSD} = \frac{FC_{MSD} - FC_P}{TC_{MSD}} \times 100$$

$$RPD = \frac{|FC_{MS} - FC_{MSD}|}{\frac{1}{2}(FC_{MS} + FC_{MSD})} \times 100$$

where: FC_{MS} = Found concentration in the MS
FC_{MSD} = Found concentration in the MSD
FC_P = Found concentration in the un-spiked parent sample

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TC_{MS} = True spike concentration in the MS
TC_{MSD} = True spike concentration in the MSD

Recoveries shall be evaluated against the current LCS/LCSD control limits, established as described in Section 8.7.1. If any recovery does not meet its criteria, the data shall be evaluated to determine the source of the difference and to determine if there is a matrix effect or analytical error. If the analyte is found in the parent sample, the result for that sample and the failing QC sample shall be reported with "J" qualifier.

If the RPD does not meet the statistical control limit, the parent, MS, and MSD results shall be reported with a "J" qualifier.

8.8 The experience of the analyst performing GC/MS analyses is invaluable to the success of the methods. Each day that analysis is performed, the calibration verification standard should be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram can indicate whether the column is still performing acceptably, the injector is leaking, the injector septum needs replacing, etc.

8.9 A summary of all quality control criteria is listed in Appendix II.

9.0 CALCULATIONS

9.1 Dilution Factor (DF). This is the final volume divided by the initial volume of solution, as shown below. When determining the DF, only dilution of the extract is included. Do not include any factors related to extraction of the initial sample aliquot.

$$DF = \frac{V_i + V_b}{V_i} = \frac{V_f}{V_i}$$

where: V_i = volume in mLs of extract used in the dilution
V_b = volume in mLs of blank water used in the dilution
V_f = final volume in mLs of diluted extract

For multiple dilutions, the dilution factor is the product of the dilution factors for each individual dilution. For example if 5 mL were diluted to 100 mL and 2 mL of the resulting solution was diluted to 50 mL, the dilution factor would be:

$$DF = \frac{100}{5} \times \frac{50}{2} = 20 \times 25 = 500$$

If no dilution was performed, the DF = 1 in the equation below.

9.2 Water and Other Liquid Samples (Mass/Volume Results)

9.2.1 Results for liquid samples are generally reported on a mass/volume basis, where the analytical sample aliquot is measured volumetrically. If the analytical aliquot of the liquid is determined by weight, the calculations described in Section 9.3 should be used.

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Results are reported to two significant figures, in terms of the original sample. Results are generally in units of µg/L, but may be mg/L or %. Units must be consistent within a project.

9.2.2 Calculate the sample concentration as follows:

$$\mu g/L = \frac{R_i \times DF \times V_D}{V_S}$$

where: R_i = Instrument result in µg/L
DF = Dilution factor, as described in Section 9.1
V_D = Final digestion volume in liters (L)
V_S = Sample volume in liters (L) used in the digestion

9.2.3 If any of the calculation factors have been included in the instrument software for reporting results, ensure that duplicate calculations do not occur.

9.3 Soil and Other Solid Samples (Mass/Mass Results)

9.3.1 Results are reported to two significant figures, in consistent units on wet weight basis. Units are generally µg/Kg or mg/Kg.

9.3.2 Calculate the sample concentration as follows:

$$\mu g/Kg = \frac{R_i \times DF \times V_D}{W_S}$$

where: R_i = Instrument result in µg/L
DF = Dilution factor, as described in Section 9.1
V_D = Final digestion volume in liters (L)
W_S = Sample weight in kilograms (Kg) used in the digestion

9.3.3 If any of the calculation factors have been included in the instrument software for reporting results, ensure that duplicate calculations do not occur.

9.4 Wipe Samples

9.4.1 Data are reported to two significant figures in units of µg/cm^2 or mg/m^2. If the area wiped is not provided, results are reported on a per sample basis.

9.4.2 Calculating the sample concentration is a multi-step process, as follows:

9.4.2.1 Calculate the µg/sample in the media blank

$$M_b = \frac{R_i \times V_D \times DF}{1000}$$

where: M_b = Total analyte mass in the media blank (µg)
R_i = Instrument result in µg/L
V_D = Final digestion volume in liters (mL)
DF = Dilution factor, as described in Section 9.1

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9.4.2.2 Calculate the µg/sample in the sample

$$M_S = \frac{R_i \text{ x } V_D \text{ x DF}}{1000}$$

where: M_S = Total analyte mass in the sample (µg)
R_i = Instrument result in µg/L
V_D = Final digestion volume in liters (mL)
DF = Dilution factor, as described in Section 9.1

9.4.2.3 Calculate the concentration of the target analyte in the sample:

$$C_S, \mu g/cm^2 = \frac{M_S - M_b}{A}$$

or

$$C_S, mg/m^2 = \frac{M_S - M_b}{0.1 \times A}$$

where: C_S = Sample concentration
M_S = Total analyte mass in the sample (µg)
M_b = Total analyte mass in the media blank (µg)
A = Area sampled (cm^2)

9.5 Percent recovery is reported for ICV, CCV, and LCS/LCSD (calculated as described in Section 8.6.7.4).

9.6 Relative percent difference (RPD) between spiked duplicate determinations is reported for LCS/LCSD and calculated as described in Section 8.6.7.4.

10.0 REPORTING REQUIREMENTS AND ORDER OF DATA DELIVERABLES

10.1 The analyst shall provide reports and other deliverables as specified in this section unless superseded in writing by a client-specified format. The required content and form of each deliverable is described in this section. All reports and documentation must be:

- Complete
- Legible, including handwriting and copies,
- Clearly labeled and completed in accordance with instructions in this section,
- Arranged in the order specified in this section,
- Properly corrected (handwritten corrections must be legible, initialed, and dated), and
- Free of White-out® and Post-It® notes or other items which are not allowed.

10.2 Prior to submission, the analyst shall arrange items and the components of each item in the order listed in these sections.

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10.3 If samples from more than one client are analyzed in one batch, a separate complete package shall be prepared for each client. Information not related to the client receiving the report shall be removed, including sample designations for the MS/MSD pair in the batch.

10.4 If samples from more than one client are analyzed in one batch, all QC results will be copied and included in the package for each client.

10.5 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS system must identify such edits or manual procedures. A hardcopy printout of the Extracted Ion Current Profile (EICP) or the Quantitation ion displaying the manual integration shall be included in the raw data. This applies to all target analytes and surrogates.

10.5.1 If an analyte is Q-deleted in any sample, standard or blank, the full-page report (with EIC, spectrum, and quantification data) for that analyte before deletion shall be included with a notation stating the technical reason why the analyst judged the analyte to be a false positive (e.g., not all ions present or wrong retention time) in such a manner that the judgment can be verified by an independent reviewer. The notation shall be initialed and dated by the analyst.

10.5.2 If an analyte is manually integrated in any sample, standard, or blank, the full-page report (with EIC, spectrum, and quantification data) for that analyte before and after manual integration shall be included. The technical reason why the analyte required manual integration shall be noted (e.g., baseline drawn incorrectly or wrong peak) in such a manner that the judgment can be verified by an independent reviewer. The notation shall be initialed and dated by the analyst.

10.6 The Sample Data Package is divided into the eight major units described in this section. The Sample Data Package shall include data for the analyses of samples from one client, in one or more batches, including field samples, dilutions, re-analyses, instrument blanks, calibration, ICV, CCV, Method Blank(s), LCS/LCSD, and any requested or required MS/MSD. A blank, colored sheet of paper will separate sections.

10.6.1 Section 1

10.6.1.1 Narrative: This document shall be clearly labeled "ECBC Environmental Monitoring Laboratory Analytical Narrative" and shall contain at a minimum:

- Laboratory name
- Client/Project name
- ECBC sample numbers being reported
- Analytical batch number(s)
- Detailed documentation of any quality control, sample, shipment, and/or analytical problems encountered in processing the samples reported in the data package.
- Discussion of any IOP modification/variance.

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10.6.1.2 Injection log covering the entire analytical sequence in chronological order. If more than a single log is necessary, forms shall be arranged in chronological order by instrument.

10.6.1.3 Scratch Log, complete with analyst, instrument, date extracted, date analyzed, and sample results. After supervisor review, this is moved to Section 8 and the Analytical Results (Part 1) Report and Analytical QC Results (Part 2) Report are placed in this section.

10.6.1.4 Surrogate Recovery Form. This form includes all QC, samples, dilution, and re-extractions.

10.6.1.5 Method Blank Summary. This form includes all QC, samples, dilution, and re-extractions associated with a method blank. If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the blank, by instrument.

10.6.2 Section 2

10.6.2.1 GC/MS tune verification. This form is used to report the results of the GC/MS instrument tune verification. For each ion listed on the form, report the percent relative abundance.

NOTE: For BFB, one or more of the high mass ions may exceed the abundance of the ion listed on the form as the nominal base peak, m/z 95 for BFB. Despite this possibility, all ion abundances shall be normalized to the nominal base peak listed.

If more than a single form is necessary, forms shall be arranged in chronological order, by instrument.

10.6.2.2 Initial calibration data, area report, and plots of linear regression shall be included in chronological order by instrument, if more than one instrument is used. Required items are standard(s) quantitation reports and reconstructed ion chromatograms for the initial (five-point) calibration regardless of which day the calibration was performed. Spectra are not required; however, EICPs displaying each manual integration are required (Section 10.3.1).

10.6.2.3 ICV data, including Evaluate Continuing Calibration Report for the ICV, standard quanitation report and reconstructed ion chromatograms, if calibration was not performed on the same day(s) as the sample analyses being reported.

10.6.3 Section 3

Check Standards data and evaluation report shall be included in chronological order by instrument, if more than one instrument is used. The Evaluate Continuing Calibration Report form must be followed by the quantitation report and the reconstructed ion chromatograms. Spectra are not required; EICPs displaying each manual integration are required (Section 10.3.1).

10.6.4 Section 4

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Matrix Spike/Matrix Spike Duplicate Recovery (MS/MSD) data and evaluation form. If more than a single form is necessary, forms shall be arranged in chronological order by date of analysis of the MS/MSDs, by instrument. The MS/MSD form must be followed by the quantitation report and the reconstructed ion chromatograms. Spectra are not required; EICPs displaying each manual integration are required (Section 10.3.1).

10.6.5 Section 5

Method, reagent, and instrument blanks in chronological order, by instrument. All false positive detections must be Q-deleted. The quantitation report and the reconstructed ion chromatograms plus spectra for all detected and non-detected agents shall be provided. For instrument blanks, reconstructed ion chromatograms and spectra shall be provided for all compounds with a response (area count) greater than zero, with a notation/evaluation of whether the compound is present in the instrument blank.

10.6.6 Section 6

Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD) data and evaluation form. If more than one form is necessary, forms shall be arranged in chronological order by analysis date, by instrument. The LCS/LCSD form must be followed by the quantitation report and the reconstructed ion chromatograms. Spectra are not required; EICPs displaying each manual integration are required (Section 10.3.1).

10.6.7 Section 7

Sample data, including dilutions and re-analyses, shall be arranged in packets with the quantitation report and the reconstructed ion chromatograms plus spectra for all detected and non-detected target agents. The samples shall be placed in increasing EML sample number order, considering both letters and numbers. This may not correspond to the order in which the samples were analyzed.

10.6.8 Section 8

10.6.8.1 Chains of Custody and copies of extraction log book showing all samples in the data package.

10.6.8.2 Any other supporting data. For example, screening data, memos, notations, Scratch Logs used to create Analytical Results Reports.

10.6.8.3 MBFORM 70 showing the numbers for standards used for calibration and spiking. These numbers will be used to append data from the Standards Log to ensure that all standards are traceable.

10.7 Data and Report Formats

10.7.1 In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS system must identify such edits or manual procedures. A hardcopy printout of the Extracted Ion Current Profile (EICP) of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all target agents and surrogates.

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10.7.2 The analyst shall identify all samples, including dilutions and re-analyses, LCS/LCSD, MS/MSD, Method Blanks, and standards with a unique Environmental Monitoring Laboratory sample number.

10.7.2.1 For field samples, the Environmental Monitoring Laboratory sample number is the eleven- or twelve-character unique identifying number assigned at log-in to the MUD tracking system. The first three characters are letters designating the location of the laboratory. The next six characters are numbers assigned by the data system in sequential order. The final two or three characters are letters signifying the type of sample. In order to facilitate data assessment, the analyst shall use the following sample suffixes:

AAAXXXXXX = Environmental Monitoring Laboratory sample number

AAAXXXXXX-MS = Matrix spike sample

AAAXXXXXX-MSD = Matrix spike duplicate sample

AAAXXXXXX-REA = Re-extracted and re-analyzed sample

AAAXXXXXX-DL = Sample analyzed at a dilution

AAAXXXXXX-DL2 = Sample analyzed at a secondary dilution

AAAXXXXXX-DL3 = Sample analyzed at a third dilution

AAAXXXXXX-DUP = Sample duplicate

10.7.2.2 The Environmental Monitoring Laboratory sample number shall be unique for each blank and LCS/LCSD within an SDG. The unique number will be the eight digit extraction batch number (as described in SOP# CNG-44, YYMMDD0X, where X=1, 2, 3, etc.) plus the following suffixes:

YYMMDD0X = extraction batch number

YYMMDD0X-MB = Method blank

YYMMDD0X-LCS = Laboratory control sample

YYMMDD0X-LCSD = Laboratory control sample duplicate

10.7.3 Strike a single line through unused columns and spaces. Initial and date all strike outs.

10.7.4 Do not use paper clips or staple pages together.

10.7.5 A summary of the data format and order can be found in Appendix III.

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Table 1 BFB (4-Bromofluorobenzene) Mass Intensity Criteria

m/z Required Intensity (relative abundance)
50m/z – 8 to 40 % of m/z 95
75m/z – 30 to 66 % of m/z 95
95m/z – Base peak, 100 % relative abundance
174m/z – Greater than 50 % to 120 % of m/z 95
176m/z – Greater than 93 % but less than 101 % of m/z 174

Table 2 Characteristic Masses (m/z) for Chemical Warfare Agents

Compound	Primary Characteristic Ion	Secondary Characteristic Ions
1,4-Dithiane	120	92, 61, 59
1,4-Thioxane	104	61, 74
BFB	174	95, 176
CEES	124	75, 126
CN	91	105, 77, 51
GA	106	117, 133, 162
GB	99	125, 81
GD (1 & 2)	99	126, 82
GF	99	81, 67, 54
HCB	249	284, 142, 107
C^{13}-HCB	290	255, 148, 113
HD	109	111, 158, 160
HN-1	122	120, 92, 56
HN-3	156	154, 63
L (BME Derivative)	212	107, 151, 186
L1 (ET Derivative)	136	145, 107, 258
L2 (ET Derivative)	136	145, 107, 258
L3	136	145, 110, 113
Q	123	182, 109, 218
TDG	104	61, 45
TDP	150	61, 93
VR	86	99, 56
VX	114	127, 167, 72

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Appendix I Typical Analytical Sequence

With initial calibration:

1. tune with 1 – 50ng BFB
2. instrument blank
3. 5-point calibration
4. calibration verification
5. method blank
6. LCS/LCSD
7. samples, MS/MSD
8. CCV (every 10 samples)
9. instrument blank (optional)
10. ending calibration check standard

Without initial calibration:

1. tune with 1 – 50ng BFB
2. instrument blank
3. calibration verification
4. method blank
5. LCS/LCSD
6. samples, MS/MSD
7. CCV (every 10 samples)
8. instrument blank (optional)
9. ending calibration check standard

QC Criteria

- Tune: GC/MS system must meet all criteria before any analysis (Table 2).
- Initial Calibration: linear regression (do not force through origin). Correlation coefficient (r^2) ≥ 0.990 (Section 7.4.5.3.1).
- Calibration verification/check: ±20 % difference (±25 % for VX, L, CN, Q, L1, L2, L3) (Section 7.5.2).
  - If calibration check is less that 20 % (±25 % for VX, L, CN, Q, L1, L2, L3), all samples prior to the calibration check must be reanalyzed. If the calibration check is greater than 20 % (±25 % for VX, L, CN, Q, L1, L2, L3), analysis must stop however previous samples may be reported.
- Surrogate: all surrogate recoveries must be within current lab limits. If not, sample must be re-analyzed. If it fails again, the sample must be re-extracted and re-analyzed (Appendix I).
- LCS/LCSD: Average %Rec. and RPD must be within current lab limits. If not, re-analyze. If either one fails again, the entire batch must be re-extracted. Sample analysis can not begin until the LCS/LCSD meet all criteria (Section 8.7.6). For analytes where no lab limits are established, use an initial recovery limit of ±50 % and an RPD limit of 20%. Establish laboratory limits after 20 data sets are collected and reevaluate yearly.
- MS/MSD: recoveries are evaluated against current laboratory limits; however, if any recovery is outside these limits there is no correction action. All failures will be discussed in the report narrative. For analytes where no lab limits are established, use an initial recovery limit of ±50 % and an RPD limit of 20%. Establish laboratory limits after 20 data sets are collected and reevaluate yearly.

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Appendix II Summary of Quality Control Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Before using any test method and at any time there is a significant change in instrument type, personnel, or test method.	QC acceptance criteria for calibration, blanks, LCS/LCSD and MS/MSD.	Recalculate results; locate and fix problem, then rerun demonstration.	NA	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check samples (e.g., LCS). No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL Study	At initial set-up and subsequently once per 12 months. Quarterly analysis of MDL verification check samples.	See 40 CFR 136 Appendix B. MDL samples must produce a signal at least 3 times the instrument noise level, at a concentration approximately 2 times the reported MDL.	Run MDL verification check at higher level and set MDL higher or reconduct MDL study.	NA	Samples cannot be analyzed without a valid MDL.
Tuning	Tuning verified before calibration and every 12 hours during sample analysis.	Refer to method-specific criteria. For thisi IOP, criteria are shown in Table 1.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Initial Calibration (ICAL): Minimum 5 standards and a calibration blank for all target analytes.	Before sample analysis, at any major change in analytical system, or after failing calibration verification.	$r^2 \geq 0.990$ for linear least squares regression. For non-linear regression, coefficient of determination (COD) $r^2 \geq 0.990$ (6 points shall be used for second order; 7 points shall be used for third order)	Correct problem, documenting findings and actions; repeat initial calibration	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second Source Calibration Verification (ICV)	Once after each initial calibration, before sample analysis.	Value of second source within ± 20 % of expected value (initial source).	Correct problem, document findings and actions; verify second source standards. Rerun ICV. If that fails, correct problem, document findings and actions; repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

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Appendix II Summary of Quality Control Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Establish Retention Time Window	Once per ICAL.	Position for all analytes, surrogate, and/or internal standards set using the midpoint standard of the initial calibration curve.	NA	NA	
Evaluation of Retention Times (RT)	With each sample.	RT of analytes, surrogates, and/or internal standards in standards and samples within ± 0.05 minutes of established retention time.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	
Continuing Calibration Verification (CCV)	Daily, before sample analysis if ICAL not required. Rerun at least every 20 samples or every 12 hours, whichever is sooner and at the end of the analytical sequence.	Analytes between 80 % and 120 % of the expected value.	At start of sequence, rerun CCV. If that fails, correct problem; document findings and actions; run ICAL. Rerun CCV. For mid or end of sequence, if result < 80 % of expected value (<75 % for L, VX, others noted in Section 7.5.2), correct problem, repeat ICAL, and re-analyze all samples since the last passing CCV. Rerun CCV. For mid or end of sequence, if result >120 % of expected value (>125 % for L, VX, others noted in Section 7.5.2), analysis must stop but previous samples may be reported if ND for affected analyte. Correct problem before continuing.	Apply Q flag if no sample material remains and analyte exceeds criteria.	No samples may be analyzed without a passing CCV at the beginning of the analytical sequence.

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Appendix II Summary of Quality Control Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Internal Standards (ISTD) Verification	In all samples and standards	Retention time ± 3 seconds from retention time of the midpoint standard in the ICAL. EICP area within -50 % and +100 % of ICAL midpoint standard.	Inspect GCMS for malfunctions. Re-analysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q flag to analytes in sample with non-compliant IS. Flagging not appropriate for standards.	
Method Blank	One per preparatory batch, matching sample matrix.	No analytes detected > ½ LOQ.	Correct problem, documenting findings and actions. If blank contamination exceeds 1/10 the level found in any sample, all samples with detectable results will be reprocessed in a subsequent preparation batch.	If no sample remains for reprocessing, apply B-flag to all detectable concentrations in all samples in the preparatory batch.	
Instrument (or Calibration) Blank	Before beginning a sample run.	No analyte detected > 2 times the MDL.	Correct problem, documenting findings and actions. Reprepare and re-analyze calibration blank and samples since the previous acceptable blank.	Apply B-flag to all results for samples associated with the blank that cannot be re-analyzed.	

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Appendix II Summary of Quality Control Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS/LCSD Containing all target analytes and QC compounds	One LCS and one LCSD per preparatory batch, matching sample matrix.	Limits established at ± 3σ around the mean %R, based on at least 30 points. Default: 50 – 150 % recovery with RPD ≤ 20 %, until sufficient points collected.	For % Recovery: Re-analyze. At second failure, correct problem, documenting findings and actions. Re-extract and re-analyze all samples in a new preparatory batch with new batch QC, if sufficient sample available. For RPD: Evaluate source of difference. Re-analyze, as appropriate.	For % Recovery: If corrective action fails, apply Q-flag to all samples in the associated preparatory batch.	
MS/MSD	One MS and one MSD per preparatory batch. If sample is known to contain analyte greater than 5 times LOQ, a sample duplicate may be used in place of MSD. An MS is always required, but should be spiked at 1 – 4 times the known concentration.	For matrix evaluation. Acceptance criteria same as LCS/LCSD.	Advisory limits only. Corrective action only as specified by client.	If detectable concentration in parent, apply J-flag if acceptance criteria not met for %R or RPD.	For matrix evaluation only. If MS or MSD results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

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Appendix II Summary of Quality Control Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All samples, including QC.	Limits established at ± 3σ around the mean %R, based on at least 30 points. Default: 50 – 150 % recovery until sufficient points collected.	Re-analyze failed low samples. On second failure, correct problem then re-extract and re-analyze all samples with failed surrogates in the preparatory batch, if sufficient sample material is available. If fails high in LCS and/or LCSD and internal standard response is acceptable and target compound recoveries are in control, review spiking procedures, look for trends,.	For sample failures, report surrogate results with a J flag for the affected sample(s). For QC sample failure (where matrix mimics field samples), report surrogate results with a Q flag for all samples in the associated preparatory batch.	
Results Reported Between MDL and LOQ	NA	NA	NA	Apply J-flag to all results between MDL and LOQ.	

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Appendix III Data Package Format

Section 1

- Narrative
- Scratch log
- Injection log
- QC Charts (if requested by client)
- Surrogate summary
- Method Blank summary

Section 2 (Calibration)

- Tune verification data in chronological order
- Initial Calibration data in chronological order, by instrument
  - Area report and plots
  - Raw data(from day that a calibration is analyzed – no spectra except for manual integrations or deletions, with notations)
  - ICV from day that a calibration is analyzed

Section 3 (Check Standards)

- Check standards in chronological order
  - Check standard evaluation report
  - Quant report/chromatogram(no spectra except for manual integrations or deletions, with notations)

Section 4 (MS/MSD)

- MS/MSDs in chronological order, by instrument
  - MS/MSD evaluation report
  - Quant report/chromatogram(no spectra except for manual integrations or deletions, with notations)

Section 5 (Blanks)

- Blanks in chronological order
  - Detailed report for each blank
  - Spectra for manual deletions – with notations
  - Spectra for compounds with areas counts greater than zero in instrument blanks

Section 6 (LCS/LCSD)

- LCS/LCSDs in chronological order, by instrument
  - LCS/LCSD evaluation report
  - Quant report/chromatogram(no spectra except for manual integrations or deletions, with notations)

Section 7 (Analyses – in EML Sample # Order)

- Quant report/Chromatogram
- Final quant report (after Qdeleting false hits)
  - Manual spectra/blowups of target agents for manual integrations and Q-deletes, with notations.

Section 8 (Support Data)

- COCs
- Extraction logbook pages
- Any other information (i.e., memos, notes, support data)
- MBFORM-70

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IOP MT-11 DAAMS Tubes Monitoring Procedures

Prepared By	Jill M. Meuser	11/5/13 Date
Reviewed By Sample Team Leader	for J.A. Snyder Juanita A. Snyder	11/5/13 Date
Reviewed By Air Monitoring Manager	Michael E. Knudsen	11/5/13 Date
Reviewed By Quality Assurance Coordinator	Jill M. Meuser	11/5/13 Date
Approved By Environmental Monitoring Laboratory Chief	Steven D. Norman	11/5/13 Date

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This IOP meets requirements of Revision 6 of the CASARM QA Plan.

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IOP MT-11 DAAMS Tubes Monitoring Procedures

Record of Change Page

- Revision 24 – Typographical errors corrected on pages 9, 12, 15, 19, 20, 21, and 23. Discussion of NO_x filter expiration and VG tubes added to Section 13 Special Note. Additional explanation of VG-conversion tubes and pads added to Section 13.2. Sections 15 and 20 revised to indicate that an alternate authorized vehicle (e.g., a rental car at travel sites) may be used to transport samples. Tedlar® bag procedure added as a technical section. Appendices deleted.
- Revision 23 – Monitoring amendment for MINICAMS alarms in locations without continual MCCF monitoring; modification of outer perimeter pumps distances from source location. Typographical corrections. Update to current IOP format.
- Revision 22 – Grammatical corrections and table updates.
- Revision 21 – Addition of new sampling profiles and additional GPL sampling methods. Inclusion of a new Appendix D for Umatilla site. Update IOP format.
- Revision 20 – Addition of GPL monitoring and utilization of Glass bead VX tubes. Removal of the 3-Ion method references; change in AEL level, MCCF flow rate changes; inclusion of form for recording broken tubes. Addition of the requirement to retain a copy of any chain-of-custody documents received from clients as part of the monitoring history.
- Revision 19 – Reorganization of monitoring types and setup procedures. The addition of one and three day baseline monitoring procedures. Addition of training and certification process for sample technicians.
- Revision 18 – Include procedures for resampling an item that contained multiple individual items that could not be cleared. Procedure explains labeling process and how to enter collected samples in t LIMS so they can be referenced back to the original sample. Updated TAP monitoring locations and clarified vague procedures.
- Revision 17 – Include establishment of a minimal sampling time needed during sample collection interval. Include the procedure for identifying QPs with corresponding samples/locations. Make sure that VGPAD containers contain an expiration date. Make ST aware to verify that expiration dates are written on the field tags for QPs. Correction of typographical errors found throughout the document. Addition of first entry monitoring and STEL BKGD procedures. Remove references to obsolete sampling pumps no longer used.
- Revision 16 – Clarification of DAAMS tube positioning during SOP operations. Added: 2-hour monitoring time for SOP operations. Special 1-hour monitoring time for conducting WPL background monitoring due to an interference or confirmation result for an SOP. Identified the requirement to use safety glasses in the laboratories when conducting monitoring operations. Made grammatical and spelling corrections throughout the document. Deleted appendixes referencing to closed sites.
- Revision 15 – Added requirement to install new V-G pads at the beginning of the work week, if possible. Used V-G pads are to be disposed of as hazardous waste in accordance with established procedures

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- Revision 14 – Addition of procedures for DAAMS sample tube collection in the event of a MINICAMS alarm.
- Revision 13 – Addition of STEL background monitoring. Establish the flow rates and time intervals for DAAMS tubes that are to be used while monitoring using the Direct VX and 3-Ion sampling methods. The 48-minute sampling interval used for monitoring 1X items is being removed throughout document. Update of site diagrams.
- Revision 12 – Remove references to TAG program, include WPL requirements. Change BME spike amounts to 5 µls instead of 10 µls. Add additional requirement for inclusion of hood numbers in sample data during SOP operations. Inclusion of the client's monitoring request and the item identification for XXX/TAPC or Labwaste items along with copy of Scratchlog. Update additional monitoring requirements at the PBA worksite.
- Revision 11 – Tedlar® bag procedures added for EDS operation.
- Revision 10 – Corrected typo's and removed conflicting requirements on pages 13, 14, and 15. Changes requested by Mr. T. Rosso.
- Revision 9 – Document format updated. Perimeter pump arrangement revised to allow for better overall sample coverage. Sampling procedures amended and updated. Addition of Amendment D (Pine Bluff Monitoring Procedures).
- Revision 8 – IOP is being revised to include sampling procedure changes and equipment usage clarification. IOP is also being revised in a manner that will allow for one primary IOP concerning DAAMS monitoring procedures. Individual appendices shall contain the specific requirements that pertain to a particular site. Requirements can include site layouts, special equipment needs as well as flow rates, time interval, and sample types specific to that site.
- Previous revisions of this document did not incorporate a record of change page.

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Signature Page

Operator's Statement: I have read, or have had read to me, the procedures in this IOP. I, by my signature below, indicate that I thoroughly understand and agree to abide by these instructions.

Name (print)	Signature	Date

Supervisor's Statement: I have personally reviewed this IOP and, to the best of my knowledge, believe that the information listed herein is correct.

Supervisor: Steven D. Norman Date: 11/5/13

Steven D. Norman

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IOP MT-11 DAAMS Tubes Monitoring Procedures

1.0 INTRODUCTION

This document presents the air sampling equipment and current procedures used by the Environmental Monitoring Laboratory, Sample Collection Team to collect chemical warfare material (CWM) air samples at Edgewood, MD and other locations.

2.0 PURPOSE

The purpose of this plan is to establish monitoring procedures that shall be used by the Environmental Monitoring Laboratory (EML), Sample Collection Team's technicians to set up sampling equipment and monitor the airborne concentrations of CWM and specific metals during the various types of monitoring procedures.

3.0 SCOPE

To establish the procedures and responsibilities for the sample technicians setting up and conducting air monitoring operations both at Edgewood, Maryland and at off-site locations. This plan describes the rationale for monitoring strategies for historical and confirmation air monitoring and choice of monitoring equipment.

4.0 MONITORING TYPES AND PLACEMENT PROCEDURES

4.1 Background Monitoring

4.1.1 Background monitoring can be performed at the Worker Population Limit (WPL) or Short-Term Exposure Limit (STEL).

4.1.2 EML personnel shall pay special attention to calibration, status of equipment, and accurately filling out forms and chain of custody documentation.

4.1.3 Allowable times, flows, and limitations for background monitoring are noted in Figure 1. Do Not Exceed noted flow or time limitations.

Figure 1

Sample Type	Description Dual tubes shall be used for ALL samples	Time Interval	Target Flow	Flow Limits
Background Monitoring WPL	Facilities/areas where personnel enter/work and CWM is or was present (IHF, Labs, Agent Storage rooms).	2 hours 4 hours 5 hours 8 hours	200 mls/min 100 mls/min 80 mls/min 50 mls/min	190 – 210 mls/min 100 – 110 mls/min 80 – 88 mls/min 50 – 55 mls/min
Background Monitoring GPL	For facilities being released to general public	1 hour	400 mls/min	380 – 420 mls/min

4.2 Background Monitoring Setups

4.2.1 Outdoors – The Sample Technician (ST) shall assess the area and setup a minimum of two positions (upwind & downwind). Positions may be placed up to

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50 feet from the working area/source. More positions may be set up if deemed necessary in the judgment of the ST.

4.2.2 Facility monitoring for turn-in of the structure requires that all areas of the facility be sampled to WPL for 24 hours. Monitoring can be conducted for three consecutive 8-hours periods or over the course of three days (8-hours each day) for a total of 24 hours.

a. All rooms (hallways, staircases, bathrooms, etc.) will be sampled with a minimum of 2 sampling points in each area.

b. For extremely small areas (10 ft x 10 ft or less) a single sample point may be utilized. More positions may be set if the sample technician determines the size of the area indicates the need for additional sample points.

c. Positions will be labeled using the building number followed by the room number and finally pump positions labeled clockwise with an alpha character (e.g., 3344.5A; 3344.5B; 3344.5C). The first position will be at the entrance to the area/room.

d. Multiple floored facilities shall be monitored one floor at a time with the floor level identified in the sample location ID (e.g., 3344.1st FLR.HALL.A).

4.2.3 Room background monitoring equipment will be determined based on the size of the room/building. Typically two pumps are placed in each room being monitored (one at rear and other at front of room near entrance) based on the air flow within the room. More positions may be set if the sample technician determines the size of the area indicates the need for additional sample points. Positions will be identified by the room number(s) followed by location (e.g., 5REAR; 5FRONT).

4.2.4 Perimeter monitoring shall consist of 4-5 positions. This will allow for fluctuating winds, without having to reposition sample pumps due to wind shifts. Time and flow settings shall be based on project requirements.

a. When performing open air operations, position “A” shall be located within the exclusion area as close to the source as possible without interfering with the workers or equipment being used. The “A” position will be set using the WPL and MCCF flow settings indicated in Figure 2. If the MINICAMS heated sample line (HSL) is not used, the flow rates will coincide with the rest of the perimeter flow rates. MCCF will only be used to confirm an alarm from MINICAMS heated sample line located at that position.

b. The four outside perimeter positions shall be situated in a clockwise pattern with the “B” position placed on the corner to the left of the entrance into the exclusion zone. Place the remaining pumps in the appropriate positions.

c. Perimeter pumps can be placed up to 100 feet from the source based on the site situation. Pumps can be set no closer than 10 feet from the source.

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d. For unusual site obstacles (e.g., walls, streams, ditches) – the sample technician can make adjustments to the perimeter setup to accommodate the obstacle.

4.2.5 Filterbank Procedures.

a. DAAMS tubes located in WPL positions on the filterbank should run for the duration of the sampling interval. Sites working 8-hours have the option of using two 4-hour intervals or one single 8-hour interval. Sites working 10-hours will use two 5-hour sampling setups. Time intervals for perimeter WPL positions are noted in Figure 2. The longest time interval should be used whenever possible.

b. DAAMS tubes located in MCCF positions on filterbanks shall be set to run for a time interval NTE 12 hours. The flow rate for all MCCF positions will be 400 mls/min. In the event of a MINICAMS alarm the tubes (**from the alarming position only**) will normally have ending flows taken and the tubes replaced after the third consecutive alarm. Tubes can be pulled prior to three consecutive alarms if needed. Removed tubes will be logged into LIMS for analysis.

c. For filterbanks that are routinely operated without DAAMS tubes in place for continual MCCF monitoring (e.g., 2390, 3938) – in the event of a MINICAMS alarm, a one hour WPL BKGD at 400 mls/min will be collected.

d. Special monitoring requirements will be dealt with as they arise.

e. Allowable times and flows must not exceed noted limitations. Variations for flow and times intervals require the approval of the team leader or supervisor. See Figure 2.

Figure 2

Sample Type	Description Dual tubes shall be used for ALL samples	Time Interval	Target Flow	Flow Limits
Background Monitoring WPL	Hoods – no activity or equipment inside hood. Rooms – no activity during monitoring	2 hours 4 hours 8 hours	200 mls/min 100 mls/min 50 mls/min	190 – 210 mls/min 100 – 110 mls/min 50 – 55 mls/min
Confirmation Monitoring (MCCF)	DAAMS tubes co-located with MINICAMS HSL to confirm alarms on MINICAMS	NTE 12 hours	400 mls/min	380 – 420 mls/min
Background Monitoring WPL	DAAMS collected on filterbank without co-located MCCF tubes to confirm MINICAMS alarms	1 hour	400 mls/min	380 – 420 mls/min

5.0 STANDARD OPERATING PROCEDURES (SOP) MONITORING

5.1 SOP monitoring is done to verify there is no personnel exposure while performing operations conducted with CWM. It is also used to verify the engineering controls used during the SOP operation perform properly. SOPs are always monitored at WPL levels.

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IOP MT-11 DAAMS Tubes Monitoring Procedures

5.2 SOP Monitoring Requirements

5.2.1 Initial monitoring for new SOP operations consists of the first 5 days the operation is run. The days do not need to be consecutive. Initial monitoring is complete when a single agent has been monitored for five days (e.g., 5 days of HD).

5.2.2 Once initial monitoring is complete, if the agent is changed and the volatility of the agent is higher than the one used for the initial 5-days, then Day-1 will begin again (e.g., 5 days of HD, switch to GB).

5.2.3 Quarterly SOP monitoring is conducted once the 5 days of initial monitoring is complete as long as the agent remains the same or is a lesser volatility.

5.2.4 Initial monitoring shall be re-initiated in the event that monitoring is not conducted within 365 days of the last monitoring performed.

5.2.5 Sample equipment will be placed as close to the worker's breathing zone as possible without interfering with their operation. NO sample equipment or tubes monitoring SOPs will be situated within engineering controls.

5.2.6 Samples collected that are used to monitor SOP processes (e.g., incubators, chambers) within the engineering controls require the following steps:

a. Sample equipment will be place in a ziploc bag prior to placing in the hood. Do not seal the bag.

b. Two pumps may be required depending on the operation. First is a 10 minute SCREENING (SCRN) sample, the second will match the SOP pump's time interval but will be logged as a PROCESS sample. The SCRN sample will be analyzed first – if the SCRN sample is not clear the process sample will not be analyzed and the sampling equipment will need to be sampled to XXX status. If the SCRN sample is okay, then the process sample will be logged in and analyzed.

5.2.7 SOP information will include the following:

a. Building and room number
b. Hood number or location where the SOP operation will take place
c. SOP number and agents used during the actual operation
d. POC and phone number

5.2.8 SOP QPs will be formatted to include a room number along with the hood number or location followed by the letter Q (e.g., 19Hood7Q; 19Bench.Q). In the event that the ID ends with a letter, a period will be placed between the last letter of the ID and the Q designation (19Bench.Q). Each hood/location requires its own set of sample and QP tubes.

5.2.9 Sample technician will annotate the Laboratory Monitoring Record maintained in the room where the SOP is conducted after equipment is placed and running.

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5.2.10 Sample equipment will not be set in calibrate mode for SOP monitoring.

Figure 3

Sample Type	Description Dual tubes shall be used for ALL samples	Time Interval	Target Flow	Flow Limits
SOP Monitoring (WPL)	Laboratory agent operations performing testing with CWM	2 hours 4 hours 8 hours	200 mls/min 100 mls/min 50 mls/min	190 – 210 mls/min 100 – 110 mls/min 50 – 55 mls/min

6.0 FIRST ENTRY MONITORING (FEM)

6.1 FEM monitoring equipment is set for one hour. **Sample Techs will be masked during FEM monitoring.** See Figure 4 for applicable flow rates and limits.

6.2 Agents to be monitored will be indicated on the Monitoring Request Form.

6.3 Two positions will be used in rooms requiring FEM monitoring. One position at the rear of the room, the other near the entry door.

6.4 Always use the FEM profile when entering samples.

6.5 Call lab and inform them that FEM samples will be coming. These samples have high priority over other samples.

Figure 4

Sample Type	Description Dual tubes shall be used for ALL samples	Time Interval	Target Flow	Flow Limits
FEM Monitoring (STEL)	Done when engineering controls fail; CWM incident or spill occurs	1 hour	400 mls/min	380 – 420 mls/min

7.0 FILTER CHANGES

7.1 Sample pumps will be set to sample for all agents (GA, GB, GD, GF, HD, HN-1, HN-3, L, and VX) for filter change operations.

7.2 Two sampling positions (POS.A & POS.B) will be used for change outs, along with the associated QPs.

7.3 Sample times and flow rates are listed in Figure 5 below.

7.4 Sample type will be STEL BKGD for all filter changes.

7.5 Filter system CBR unit ID will be entered into LIMS when available.

Figure 5

Sample Type	Description Dual tubes shall be used for ALL samples	Time Interval	Target Flow	Flow Limits
Filter Changes (STEL)	Background monitoring during change out of filters in filter systems	1 hour	400 mls/min	380 – 420 mls/min

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8.0 LABWASTE/XXX/TAPC/XXXX AND RESAMPLE ITEMS

8.1 Labwaste is any trash generated during agent operations that requires monitoring before disposal. Labwaste is always sampled using STEL levels.

8.2 XXX monitoring is for equipment or tools that have been used or potentially exposed to agent during agent operations that need clearance prior to being used again for the same operation or are to be disposed. These items will be sampled to STEL levels.

8.3 XXXX monitoring is used to sample items that have potentially been exposed to agent during operations that require maintenance/repairs or will be used for other operations. The items are sampled to WPL levels.

8.4 XXXXX monitoring is for items that will be released to the general public.

8.5 TAPC is clothing or items that will be worn again. All items designated as TAPC will be sampled using WPL levels. This includes reusable boots, gloves, coveralls, masks respirators, uniforms, SCBA, and associated equipment/harnesses. It does not include disposable coveralls, booties, or gloves. Bagged items shall be taken to building 2390, bay 4 by the Application Integration personnel for monitoring. A mask must be donned when placing and retrieving pumps (2 positions plus QP) inside the bay. Monitoring will done for the agents of interest noted for an 8-hour period. See Figure 6 for applicable flow rates.

8.6 All XXX/Labwaste/XXXX and resample items must be double-bagged, dry and sealed for a minimum of 4 hours at an ambient temperature of at least 70 degrees Fahrenheit. Samples with noticeable liquids inside bags will not be sampled. Notify the client so they may take corrective action.

8.7 Gloves will be worn when sampling any of the items listed. Dispose of gloves appropriately at the sampling location when sampling is complete. DO NOT bring gloves back.

8.8 Client requests for XXX and LABW will be setup, completed and samples turned into the lab for analysis prior to beginning another request.

8.9 Item identification:

8.9.1 The last six digits of the Hazardous Waste Barcode shall be used to identify XXX/LABW/XXXX items requiring monitoring if available.

8.9.2 If no barcode is present, the ST will assign an item identification number for XXX/LABW/XXXX in the following format (ymmdd###; example 20201001). The format indicates the year, month, day, and numerical sequence of the items. Serial number for client equipment shall be listed n the SOP field if requested.

8.9.3 Off-site item IDs shall contain 2 identifying characters from the site. Examples: Lowery site – LW20201001; Spring Valley site – SP20201001). Avoid using vowels whenever possible as some can be mistaken for numbers instead of letters (Zero and letter O).

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8.9.4 EDS item identification numbers will contain EDS followed by the barcode or assigned sample ID (e.g., EDS116329; EDS91104001). A general description of bagged contents shall be listed in the field designated for comments in LIMS for tracking purposes.

8.9.5 Identification for the associated QP can be the room/area followed by the letter Q (e.g., 2390.5Q).

8.9.6 Allowable times and flows must not exceed limitations noted in Figure 6.

Figure 6

Sample Type	Description Dual tubes shall be used for ALL samples	Time Interval	Target Flow	Flow Limits
XXXX/Labwaste or XXX items	PPE or trash generated during agent operation or field operations	1 hours	400 mls/min	380 – 420 mls/min
TAP Clothing WPL (TAPC)	All TAPC is monitored at WPL levels	8 hours	50 mls/min	50 – 55 mls/min

9.0 QPs

A QP is a set of DAAMS tubes spiked with a known concentration of agent and run for a specific time and at a specific flow rate. Tubes are spiked by the laboratory based on the type of sample. QP sets must be used within 10 calendar days of being spiked, as recorded on MBFORM-66. If the tubes are not used within that time they must be reconditioned before being used again. GPL QPs are prepared the day they are needed and shall be spiked with 1.0 GPL of the standard for the agent of interest. Tubes to be used for HD GPL QPs are to be spiked with NO_x filters in place. Tubes must be kept in the freezer portion of the sample refrigerator. QPs should run concurrently with actual samples for the same times and flows used for the samples (e.g., SOP operation run 4 hours @ 100mls/min – QP will be setup identically and run concurrently). QP field tags will be annotated with the agent(s) of interest, highlighted along with an expiration date for easy recognition by lab personnel. A set of QP tubes will be run for every 10 bagged or wrapped items (e.g., 22 labwaste would generate 3 QP sets). QPs will not be placed inside the bags or wrapping with the actual sample, they shall be placed near the items being monitored. SOP operations will have 1 QP for each setup. All QPs will be identified with a building or room number followed by the letter "Q" (e.g., 3938BAY = 3938BAY.Q). For SOP operations, a hood number or pump location shall be identified (e.g., 19HOOD12Q or 19BENCH.Q). Laboratory personnel will prepare the appropriate number of QPs based on the sample technician's request.

10.0 INITIAL AND CONTINUING BASELINE MONITORING

10.1 Baseline studies shall be conducted for CMA projects. The number of days of initial baseline monitoring required will be determined by the length of the project and documented in writing.

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a. One-day initial baseline studies will require collecting up to 2 days of QP samples from WPL and MCCF positions. See Figure 7. Grayed section (day 1-2) for flow rates, time intervals and flow limitations.

b. Three-day initial baseline studies for projects lasting more than one month. This will require collecting up to 4 days of QP samples from WPL and MCCF positions. See Figure 7 for flow rates, time intervals and flow limits for methods being collected for the 3-day baseline study.

c. WPL positions will be setup with pre-spiked QP tubes from the lab. Field tags will be annotated QP Baseline day#.

d. MCCF tubes setup for the baseline study will be post-spiked by the lab when received. Field tags will be annotated MCCF Baseline Day# and POST-SPIKE.

e. Background samples are not required to be collected during baseline studies.

10.2 During continuing baseline studies, QP samples will be collected at a minimum of one location per method (2 WPL & 2 MCCF) per day. Locations will rotate daily whenever possible.

10.3 Baseline location will start with dual method locations (WPL & MCCF co-located), then rotate to a new station for each subsequent baseline day. The secondary set of baseline tubes collected may be from independent WPL and MCCF positions (e.g., 1EDS.PRE & 1EDS. MID2) in the event there are limited dual positions available.

Figure 7

Day	Sample Location	QP Type	Profile type	sample time (min)	Flow rate (mls/min)	Flow rate Limits
1	1EDS.EXH.Q	WPL	QPBASE	480	50	50 – 55
1	1EDS.EXH	MCCF	MCCF	NTE 12 hrs	500	380 – 420
2	EDS.VESSELDOOR.Q	WPL	QPBASE	480	50	50 – 55
2	EDS.VESSELDOOR	MCCF	MCCF	NTE 12 hrs	500	380 – 420
3	2EDS.EXH.Q	WPL	QPBASE	480	50	50 – 55
3	2EDS.EXH.	MCCF	MCCF	NTE 12 hrs	500	380 – 420
4	EDS.PRE.Q	WPL	QPBASE	480	50	50 – 55
4	1EDS.MID2	MCCF	MCCF	NTE 12 hrs	500	380 – 420

11.0 TEDLAR® BAG SAMPLING

1. A mask and gloves will be donned for this procedure.
2. The filled Tedlar® bag is placed on sampling table by EDS personnel.
3. The sample pump is set up with a single DAAMS tube set at a flow rate of 400 mls/min per the procedure identified in Section 14 of this IOP. **NOTE: Tedlar® bag samples do not need to equilibrate to 70 degrees**.
4. Place a clean Teflon® fitting (normally used with NO_x filters) on the frit end of the DAAMS tube. **DO NOT use fitting that was used with VG conversion pads.**

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5. Attach the other end of the Teflon® fitting to the fill port on the Tedlar® bag.
6. Open the valve on the Tedlar® bag and turn on the sampling pump.
7. Sample bag until all the contents have been collected onto the DAAMS tube. (Depending on the volume of the bag, collection time will vary from 5 to 30 minutes).
8. When notified by operators that the bag is empty, don mask and gloves prior to reentry into facility.
9. Close the valve on the Tedlar® bag and remove Teflon® fitting from the DAAMS tube and bag. Store the Teflon® fitting for reuse.
10. Take the ending flows for the sample and turn off the sample pump.
11. Exit the facility, record sample information and enter into the LIMS as a XXX sample.

12.0 HOT BOX PROCEDURES FOR HEADSPACING ITEMS

Below are the procedures for monitoring bagged, wrapped, or boxed items when weather or site conditions do not allow items to reach the minimum 70° Fahrenheit ambient temperature requirement. This procedure is to monitor labwaste, XXX and TAPC items generated during off-site field operations to the STEL level. Sample equipment is set up following the procedures in Section 13.0. In the event items are sampled with MINICAMS, a completed copy of MBFORM 37A/B is to be supplied to the DAAMS technician for filing. A copy of Chain-of-Custody documents will be retained with monitoring documents.

12.1 Items shall be wrapped/double-bagged prior to acceptance and placing inside hotbox.

12.2 Item identification

a. If available, the last six digits of the Hazardous Waste Barcode shall be used to identify XXX/soils/XXXX items requiring monitoring.

b. If no barcode is present, the ST will assign an items identification number for XXX/soils/XXXX in the following format (ymmdd###; example 20201001). The format indicates the year, month, day, and numerical sequence of the items. Serial number for client equipment shall be listed n the SOP field if requested.

c. Off-site item IDs shall contain 2 identifying characters from the site. Examples: Lowery site – LW20201001; Spring Valley site – SP20201001). Avoid using vowels whenever possible as some can be mistaken for numbers instead of letters (Zero and letter O).

d. EDS item identification will contain EDS followed by the barcode or assigned sample ID (e.g., EDS116329; EDS91104001). A general description of bagged contents shall be listed in the field designated for comments in LIMS for tracking purposes.

e. Identification for the associated QP can be the room/area followed by the letter Q (e.g., 2390.5Q).

f. Allowable times and flows must not exceed limitations noted in Figure 8.

12.3 DON MASK and GLOVES.

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12.4 Place items in heated hotbox. If items are drums/jars, the lids must be taken off in order for the contents to off-gas into the bags. Make sure not to place items near the heat source.

12.5 Items are heated to greater than 70° Fahrenheit for 4 hours.

12.6 Sample equipment should be set according to procedures in Sections 13.2 and 13.3. Insert DAAMS tubes through all layers of wrapping/bags. Use the appropriate time interval and flow rates for the sample type indicated in Figure 8 and begin sampling.

12.7 Allowable times and flows must not exceed noted flow or time limitations in Figure 8.

Figure 8

Sample Type	Description Dual tubes shall be used for ALL samples	Time Interval	Target Flow	Flow Limits
XXXX/Labwaste or XXX items	PPE or trash generated during agent operation or field operations	1 hours	400 mls/min	380 – 420 mls/min

12.8 At the conclusion of the sample period, remove DAAMS tubes from the items and seal the hole with tape.

12.9 Use procedures in Section 16 at the conclusion of the sampling period. Do not make adjustments to the ending flow rates. If flows cannot be obtained, an explanation is needed (e.g., broken tube). Identify extreme changes in ending flow rated form beginning flows. Proceed until all items are completed.

12.10 Remove Mask and gloves. Dispose of gloves appropriately on location. Turn off heat source.

13.0 SOIL HEADSPACE ANALYSIS PROCEDURES

Soil sample headspace monitoring can be conducted using MINICAMS or DAAMS tubes. If DAAMS tubes are used in place of MINICAMS, start the following sequence at step 2. DAAMS tubes must be used to confirm all positive MINICAMS results above 0.25 STEL. This monitoring shall be used to screen soil samples for GVHL contamination. Headspace monitoring for CG, CK or chloropicrin is not necessary due to the extreme volatility of these compounds. Headspace monitoring procedures of soil samples using the MINICAMS and DAAMS are as follows. In the event items are sampled with MINICAMS, a completed copy of MBFORM 37-A/B is to be supplied to the DAAMS technician for filing.

13.1 Contractor delivers soil sample(s) to monitoring personnel with proper chain of custody (COC) documentation. A copy of COC will be maintained with official monitoring records.

13.2 Always don protective gloves. Don a mask when soil sample(s) are collected by personnel wearing EPA/OSHA Level C PPE or above.

13.3 Place up to six samples in a heated sample box. Open bags and remove sample jar lids. Close sample box lid and allow samples to equilibrate to 90° ± 10 degrees Fahrenheit for 15 minutes.

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13.4 Insert temperature probes into a selected soil sample. Allow samples to off-gas an additional 15 minutes.

13.5 Use procedures outlined in Section 13.0 to setup equipment, set flow and appropriate time intervals indicated in Figure 9 to collect samples. Make sure that frit of DAAMS tubes are not inserted into the soil sample.

Figure 9

Sample Type	Description Dual tubes shall be used for ALL samples	Time Interval	Target Flow	Flow Limits
Soil Samples	Soil, stone, concrete samples generated from a CWM location	1 hour	400 mls/min	380 – 420 mls/min

13.6 Transport DAAMS tubes to the MAP/lab for analysis.

13.7 Soil headspace data notification: The on-site Safety Specialist will be notified of all confirmed detections. This includes concentration levels below the AEL value.

13.8 Soil resample procedures: In the event that soil has been decontaminated, the soil cannot be re-sampled using the headspace analysis procedure. ECBC may request a sample of the decontaminated soil for extraction and subsequent analysis for the agent(s) of interest by the lab to confirm complete decontamination.

13.9 Copies of all soil headspace air sampling records and results will be maintained by the ECBC EML.

14.0 SAMPLE EQUIPMENT

This section addresses sampling equipment used during monitoring and the procedures for setting it up prior to taking the actual sample(s). See Section 15.0 for the procedures to follow at the end of the sampling period.

Special Notes:

1. NO_x filters shall be used in conjunction with DAAMS tubes as deemed appropriate for collecting GPL samples for HD. NO_x filters have a six-week shelf life which is noted on the package. Check the expiration date before use. Expired filters can be disposed as general trash.
2. NO_x filters must be in place when preparing QPs for HD GPL levels.
3. NO_x filters may be used with WPL/MCCF samples if required by site conditions.
4. NO_x filters are to be changed weekly, but can be changed more frequently if deemed necessary.
5. VGPADs and VG-conversion tubes are to be changed weekly. Annotate MBFORM-92 with date and initials. When not in use, the fittings containing the VGPADs/tubes should be sheltered from light. VGPADs/tubes are to be disposed of appropriately in a

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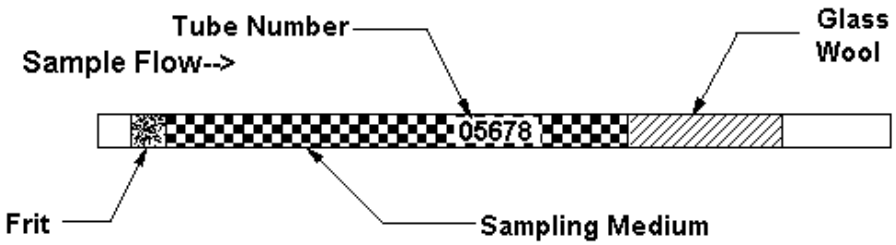
container to be turned in as hazardous waste. DO NOT throw spent pads/tubes in the trash.

6. VGPAD containers shall be identified with the batch number, manufacture date, and expiration date. Pads are good for one year from manufacturing date. If expiration date is not on the container, do not use until the expiration date is verified by ECBC personnel, then add expiration date to container and date and initial your entry.
7. Technicians shall don safety glasses and lab coats (as required) when setting up or conducting monitoring operation in laboratories.
8. Standard QPs are good for a maximum of 10 calendar days from the time of spiking, as recorded on MBFORM-66. Sets are marked by the lab with expiration dates.
9. Samples must run a minimum of ½ of the set time interval in order to collect an adequate amount of sample to analyze (4 hours sample must run at least 2 hours; 1 hour sample must run at least 30 minutes).

14.1 Depot Agent Area Monitoring System (DAAMS) tubes

The components of the DAAMS tubes are identified in Figure 10. The DAAMS tube is always situated to draw the sample though the frit and onto the sampling medium. Newer DAAMS tubes contain alpha characters that are part of the tube identification. Note that specific tube types are used for sampling certain agents. If the DAAMS tube number is illegible – **do not use it**.

Figure 10



DAAMS Tube Components

14.2 Portable Battery Powered Pumps

14.2.1 Set timer to the required sampling interval (see Figure 12 for time and flow rates).

14.2.2 Set delay time if required. If pump is not on electric power – delay time is limited to 10 minutes.

14.2.3 For battery-powered sample pumps, attach a dual- or quad-port sample port harness to the sample pump. Two, four, or six tubes can be setup on a sampling port harness. For BIOS Surveyor pumps, tubes are inserted directly into the two tube ports located on the pump. Pumps can be configured to collect four tubes.

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14.2.4 Remove DAAMS tubes form protective carriers. PRIOR TO USE – inspect tubes for flaws (e.g., chips, cracks or dirt in the frits). Do not use tubes if any flaws are found. Verify tube numbers are legible before using. If any defects are found, record the tube number on the broken tube log and place the defective tube in the broken glass container.

14.2.5 Insert the GLASS WOOL side of the DAAMS tube (see Figure 10) into the dual port sampler. The lowest numbered tube is placed into the port with the rubber band on it. If the rubber band breaks or is missing – replace it with a new one. Dual DAAMS tubes will be used concurrently during the monitoring procedure. The analysis of the DAAMS tube during desorption is a one-shot occurrence. If for some inexplicable reason the desorbed sample is not analyzed as anticipated, it cannot be recovered. Therefore, a second tube is used as a backup.

14.2.6 Adjust flow according to the sampling interval with a calibrated calibrator. Verify the calibrator has not expired. Flows must be within the specified range indicated in Figure 11. Before recording the three beginning flow rates, they must be within 10% of each other. Write the three flows on the field tag (see Figure 11).

14.2.7 Clear pump's timer. If pump is not being used immediately, turn off the pump and place it on charging station.

14.2.8 If VX is being sampled make sure to use the appropriate tubes. Two methods are available for sampling VX. Black dot tubes are for use with VGPADs. Verify that pads have not expired. Glass Bead tubes are for VX monitoring only – DO NOT use VGPADs.

14.2.9 Pumps being used to sample for Lewisite are to be setup as follows:

14.2.9.1 **DON GLOVES.**

14.2.9.2 Set up according to the steps described in Section 14.2.3

14.2.9.3 After the pump is set up, turn it on and spike tubes with 5 µl of beta-mercaptoethanol (BME). Tubes may be spiked the night before, but not more than 24 hours in advance.

14.2.9.4 Clear pump timer and remove gloves.

14.2.10 Pump placement should be as close to the sample or areas as possible without interfering with the operation or operators.

14.2.11 Secure equipment so it cannot inadvertently be knocked over.

14.2.12 XXX/labwaste/XXXX samples are to be checked every 15-20 minutes to ensure the pumps are still running until sampling period is complete.

14.3 Sampling Harness Setup

14.3.1 Items identification

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14.3.1.1 Items that have a Hazardous Waste Barcode affixed to them shall be identified using the last six digits of the barcode.

14.3.1.2 The ST will assign an items identification number for any bagged/wrapped item not having a barcode affixed. Sample ID number shall be in the following format (ymmdd###; example 20201001). The format indicates the year, month day, and the numerical sequence of the items. Serial number of client equipment shall be listed in the SOP field of LIMS.

14.3.1.3 Off-site item ID shall contain 2 identifying characters from the site. Examples: Lowery site = LW20201001; Spring Valley site = SP20201001. Avoid using vowels whenever possible as some can be mistaken for numbers instead of letters (Zero and letter O).

14.3.1.4 EDS items identification will contain EDS followed a barcode or assigned a sample ID in the proper format (e.g., EDS91104001). A general description of bag contents shall be entered into the field designated for comments in LIMS for tracking purposes.

14.3.2 Place one set of DAAMS tubes by each item to be sampled.

14.3.3 DON GLOVES.

14.3.4 Write the building number items ID and the agent(s) being monitored on the field tag attached to the DAAMS tube carriers.

14.3.5 Remove tubes from carriers and insert into sampling ports (frit out).

14.3.6 Make a hole through all layers of the wrapping/bag of the items large enough for the DAAMS tubes to be inserted into and insert the DAAMS tubes. Make sure that the DAAMS tube ends are not positioned in any liquid or loosed debris inside the wrapping/bag.

14.3.7 Verify tube numbers being inserted into item match those on the field tag. Make sure item number matches the number written on the field tag. Repeat until all items have been identified on field tags and DAAMS tubes have been inserted into all items to be monitored.

14.3.8 Turn on vacuum pump. Annotate the start time on the field tag. See Figure 12 for target flows and range limitations.

14.3.9 Starting with the first item – remove tubes from the item and adjust the flow to the appropriate flow rate on both DAAMS tubes. Put tubes back in the item being sampled.

14.3.10 Write the flows for both tubes on the field tag and proceed to the next item. Recorded flow rates should be within 10% of each other. After flows for all items have been adjusted, go back and check flow rates on the first 3 items to verify they did not fluctuate out of the acceptable limitations. Adjust as necessary.

14.3.11 If BME is needed, spike the appropriate DAAMS tubes at this time with 5 µls.

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14.3.12 For samples being taken using VG-conversion tubes/pads or NO_x filters, place the appropriate fitting on the DAAMS tubes. Tubes/pads/filters must be in place before setting the starting flow rate.

14.3.13 Ten minutes prior to the end of the sample period – remove the DAAMS tubes from the first item and record the ending flows (DO NOT ADJUST flows). Replace the tubes in the first item and proceed to the next item, repeating the process until all flows are recorded for all items.

14.3.14 At the end of the sampling period – turn off the vacuum source and pull the DAAMS tubes inserted into the first item out. Seal the hole in the bag with tape. Verify the DAAMS tubes pulled from the sample match the tube numbers written on the field tag.

14.3.15 Verify the sample ID written on the field tag and the item match. Remove DAAMS tubes from the dual port sampler and place them into the carriers, frit side down. Proceed until all samples are collected.

14.4 Field Tags

Field tags are hand-written tags that contain pertinent information about the sample being collected. Tags are to be completed in ink.

14.4.1 DAAMS tubes number are written on the upper right hand corner of the field tag. The lowest numbered tube is listed first, with the second tube being listed below the first (see Figure 11). DAAMS tube numbers can consist of numeric only or alpha and numeric characters. When listing tube IDs on field tags – numeric numbers shall be listed first and IDs containing alpha character next (e.g., tubes C08234 & 10754 – 10754 would be listed as the low tube number followed by C08234). Visually verify tube numbers written on the field tag match actual tubes.

14.4.2 Three beginning flow rates written on the field tag (flows must be within the specified range indicated in Figure 11 and within 10% of each other) prior to collection of a sample.

14.4.3 Three ending flow rates written on the field tag for each tube at the conclusion of the sampling period. DO NOT adjust ending flow rates. If flows cannot be obtained, an explanation is needed (e.g., broken tube, failed pump). Identify large changes in flow rates from beginning flows.

14.4.4 Location where sample was obtained (e.g., building and room number or site location).

14.4.5 Sample identification number; position placement or room number. For SOP operations the field tag will contain the hood number where the operation was conducted (example 19Hood7). QPs shall include a "Q" at the end of the sample identification (e.g., sample is POS.A, QP would be POS.AQ; sample is 19Hood7, QP would be 19Hood7Q). When sampling multiple items the room/building number may be used in the QP identification (e.g., 29A or 3938BQ).

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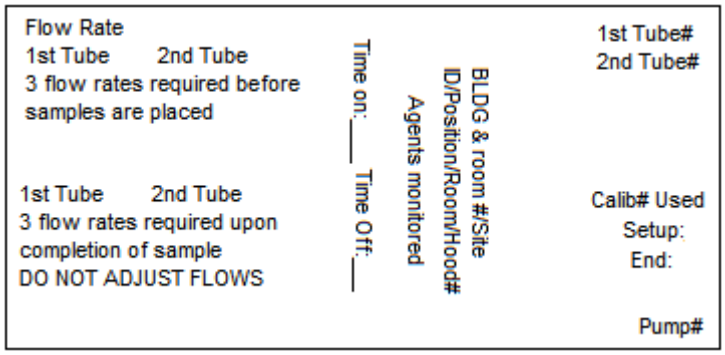
14.4.6 Time the sample started and ended. If the sample did not run the complete time interval, time should reflect actual time ran and an explanation.

14.4.7 Pump serial number should be written on lower right-hand corner of the field tag

14.4.8 Calibrator ID used to setup beginning flow and take ending flows should be written on the lower right side of the field tag. If more than one calibrator is used to set or record ending flows then two calibrator numbers should be entered on the field tag.

Figure 11 (Field Tag Example)



15.0 SAMPLE TYPES, TIMES, AND FLOW RATE TABLE

This section identifies the various sample types, the allowable time intervals, corresponding flow rates and limitations (see Figure 12). Detailed information for individual sample types is located in the designated paragraph in this section. **All samples must run a minimum of half of the sampling interval in order to obtain an adequate sample for analyzing.**

Figure 12

Sample Type	Description Dual tubes shall be used for ALL samples	Time	Target Flow	Flow Range Limits
1X items STEL (XXX)	Items used during agent operations to be disposed or destroyed. For extremely large items 2 samples should be taken at opposite ends.	1 hour	400 mls/min	380 – 420 mls/min
1X items (small) WPL (XXXX)	Items used during agent operations that will be reused, have maintenance done. For extremely large items 2 samples should be taken at opposite ends.	1 hour	400 mls/min	380 – 420 mls/min
Soil Samples	Sample jar of soil shall be double-bagged, with both bags sealed.	1 hour	400 mls/min	380 – 420 mls/min
TAP Clothing WPL (TAPC)	See Section 2.6 for monitoring requirements	8 hours	50 mls/min	50 - 55 mls/min
Monitoring identified in this section cannot be resampled.				
SOP Monitoring WPL (SOP)	Used to monitor personnel exposure during SOP agent operations. No sample equipment should be inside the hood during operations.	2 hours 4 hours 8 hours	200 mls/min 100 mls/min 50 mls/min	190 – 210 mls/min 100 – 110 mls/min 50 – 55 mls/min

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IOP MT-11 DAAMS Tubes Monitoring Procedures

Sample Type	Description Dual tubes shall be used for ALL samples	Time	Target Flow	Flow Range Limits
SOP WPL Background	Used only for verifying positive results reported for SOP operations	1 hour	400 mls/min	380 – 420 mls/min **For SOPs only**
Screening Sample	Used during agent operations with high potential to overload EML laboratory instruments	10 minutes	400 mls/min	380 – 420 mls/min
Process Sample	Used in conjunction with screening sample to protect EML laboratory equipment from potential saturation. Time interval will be determined based on SOP time interval.	2 hours 4 hours 8 hours	200 mls/min 100 mls/min 50 mls/min	190 – 210 mls/min 100 – 110 mls/min 50 – 55 mls/min
Background Monitoring (Historical) WPL (BKGD)	A minimum of 2 positions shall be placed within a room. Hoods will require 1 pump.	2 hours 4 hours 8 hours	200 mls/min 100 mls/min 50 mls/min	190 – 210 mls/min 100 – 110 mls/min 50 – 55 mls/min
GPL Monitoring	Used when facility/equipment will be released to the general public.	1 hour	400 mls/min	380 – 420 mls/min
Background Monitoring Filter Changes (SBKG)	For Filter change out operations a minimum of 2 positions shall be placed. One pump upwind of the operation and the other downwind	2 hours 4 hours	200 mls/min 100 mls/min	190 – 210 mls/min 100 – 110 mls/min
Perimeter Monitoring WPL (BKGD)	A minimum of 4 positions shall be set up (POS.B, C, D and E) 5 hour time interval for sites working 10 hours days	2 hours 4 hours 5 hours 8 hours	200 mls/min 100 mls/min 80 mls/min 50 mls/min	190 – 210 mls/min 100 – 110 mls/min 80 – 88 mls/min 50 – 55 mls/min
Confirmation Monitoring (MCCF)	DAAMS tubes co-located next to the MINICAMS HSL. Used for confirmation of MINICAMS alarms	**NTE 12 hours**	400 mls/min	380 – 420 mls/min
3X Facility WPL (BKGD)	A minimum of 2 positions placed in each room/hallway/egress area of the facility. Facilities for turn-in shall be monitored for a total of 24 hours	4 hours 8 hours	100 mls/min 50 mls/min	100 – 110 mls/min 50 – 55 mls/min
First Entry Monitoring (FEM) (STEL)	Conducted after filter change out; loss of engineering controls; after a chemical incident prior to personnel entering the area/room	1 hour	400 mls/min	380 – 420 mls/min
Tedlar® Bag (XXX)	A single DAAMS tube is used for this sample process. NOTE: sampling time will vary from 5 to 30 minutes based on the actual contents of each bag.	1 hour	400 mls/min	380-420 mls/min
QP	A minimum of 2 per day. A set of QP DAAMS will run concurrently with designated samples. Times and flows will correspond with actual sample types identified in this table. All flow limitations noted shall also apply to QPs taken. GPL QPs shall be prepared the date they are needed.			

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16.0 COMPLETED DAAMS TUBES SAMPLE COLLECTION

BEFORE disconnecting the DAAMS tubes samples from the pumps or harness:

16.1 DON GLOVES prior to collection of samples of all XXX, TAPC, and labwaste items.

16.2 If portable pumps were used, verify the sampling time on the pump. In the event the sample pump fails, if the sample ran a minimum of half of the required sampling interval the sample can be analyzed. If the sample failed to meet the minimum sampling requirement, the sample must be rerun.

16.3 Verify field tag sample ID matches ID on actual items before removing DAAMS tubes from the item.

16.4 Verify DAAMS tubes numbers written on the upper right hand corner of the field tag match the number on the DAAMS tubes extracted from the sampled item.

16.5 Verify that pertinent sample information for each sample is written on the appropriate field tag (e.g., sample ID, bldg number, beginning & ending times, agent(s) of interest, calibrator used).

16.6 Ensure actual sample identification is legible (use permanent marker and make legible).

16.7 Ensure ending flows were taken and recorded on the field tag. Recorded ending flows should be <u>within 10% of each other</u>. Do not make adjustments to the ending flows. Make note of any notable increases/decreases in flow or sample discrepancies on the field tag.

16.8 Return the DAAMS tubes (frit end first) to the protective carrier, cap carrier and place with appropriate field tag.

16.9 Place completed sample(s) in Ziploc bag along with associated QP sample(s).

16.10 A government or other authorized vehicle is used to transport finished samples to be logged into LIMS.

16.11 Prepare Air Monitoring Worksheet, MBFORM-39A (flow sheets). Verify all information was transposed correctly from the field tag to the flow sheets. Make sure to select the correct client and profile. Any broken/compromised tubes are recorded on MBFORM-91 Broken tube log.

16.12 Make sure to include any discrepancies, unusual monitoring/sampling conditions (e.g., exceeded times, bad weather, wet tubes) on the flow sheets, in LIMS and on Scratchlogs.

16.13 Sign into LIMS and enter the sample information. QPs are always entered first, followed by the associated samples. QP information is entered on MBFORM-39B, but is logged in with the samples they were collected with. Verify that the lab ID number assigned by the computer matches on the Scratchlog, TAGS and flow sheets. The LIMS program generates all necessary forms and tags for samples. DO NOT use any other forms.

16.14 Completed samples are to be logged onto LIMS the day they are collected, unless extenuating circumstances prevent it. An explanation must be provided on the Scratchlog. Samples should be turned over to the lab within 1-1/2 hours of completion. Exceptions will be made for samples obtained from remote locations.

16.15 ST entering samples into LIMS signs the Scratchlog. STL or designate reviews and signs Scratchlog before the sample packet goes to the laboratory/MAP. If no one is available to accept samples and sign the Chain-of-Custody on the Scratchlog – DO NOT LEAVE

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THEM. In the event the sample cannot be turned over to the lab/MAP, make a note on the flow sheet and store the samples in the refrigerator located in E3344.

16.16 Sample packet shall contain:

16.16.1 QP tubes.

16.16.2 Samples.

16.16.3 Tags.

16.16.4 Flow sheets MBFORM-37A/B if items sampled with MINICAMS with appropriate signatures.

16.16.5 Scratchlog with all the appropriate signatures dates and times.

16.16.6 Client monitoring request annotated with any sample IDs.

17.0 SAMPLE DOCUMENTS

17.1 All pertinent data shall be recorded in LIMS. Scratchlog generated by LIMS should match information on Air Sample Workorder and sample tags. Data will be collected in Bldg E3344. All data from off-site operations will be collected on a LIMS computer at the site. Upon the return of the LIMS computer the sample information will be down loaded to the EML data serve by the LIMS administrator.

17.2 Sample labels are generated from a PC database program. The sample labels are attached to the field tag and assigned a set of DAAMS tubes. The label is removed by the analyst and affixed to the corresponding chromatogram.

17.3 The ST is responsible for verifying all signatures are in place and printing Scratchlogs and tags.

18.0 SAMPLE TECHNICIAN CERTIFICATION

18.1 Trainees will be required to read and sign all SOPs and IOPs pertinent to sample collection. Auxiliary technicians will read and sign documents bi-annually.

18.2 LIMS system administrator will meet with the trainee and go over the LIMS program. Trainees will receive a copy of the LIMS instructional manual to aid them with learning to enter sample data into the program.

18.3 Trainees will be given up to three weeks of hands on training by a senior technician on how to select and setup sampling equipment based on the request receive from clients. During the first week of training the trainee will received assistance from the senior technicians.

18.4 Trainees will be shown sample documents required during the sampling process and how to properly fill them out.

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18.5 The last week of the training process the trainee must demonstrate that he/she can select a request for a client, select and setup all necessary equipment, collect the samples per IOP MT-11 and enter the sample information into LIMS unassisted.

18.6 The trainee's progress will be entered into the Sample Team Checklist database. New trainees will receive an initial evaluation at the end of the 3-week training period. The trainee will be evaluated to one of three certification levels.

18.6.1 Level 1 supervised Work – the technicians could not demonstrate that they were able to accomplish the tasks required to setup equipment, collect samples, or enter sample data into LIMS without assistance from a senior technician.

18.6.2 Level 2 Assisted Supervision – the technician was able to perform sampling task adequately, but still had problems with some areas or procedures.

18.6.3 Level 3 Minimal Supervision – the technician was able to perform the majority of the sample tasks with little to no assistance.

18.6.4 Auxiliary technicians must work a minimum of 1 day each month to demonstrate their competency and maintain their certifications. Certification will expire after 60 days if the technician does not meet this requirement.

18.7 Trainees rated at Level 1 will be re-evaluated in three weeks. If there is improvement in the trainee's skills, they will be re-evaluated in three months when their certification level will be determined.

18.8 Trainees rated at Level 2 will be certified as a sample tech for six months. At the end of the six months technicians will be re-evaluated and be certified annually after that unless issues with sample collection are observed during the calendar year warranting a midpoint evaluation.

18.9 Trainees rated at Level 3 will be certified through the end of the calendar year and annually after that unless issues with sample collection are observed during the calendar year warranting a midpoint evaluation. Auxiliary personnel will receive 6-month certifications as long as certification requirements are met.

18.10 Sample technicians must generate sample documents/data based on their certification level that is discernibly their own (e.g., your handwriting, LIMS entries) to maintain certifications.

19.0 AIR SAMPLING RECORDS

Maintain all sampling records per AR 40-5 requirements.

20.0 TRANSPORT

Transport of completed DAAMS samples will be accomplished in a government or other authorized vehicle.

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IOP MT-13 Analysis of Chemical Warfare Agents and Degradation Products on DAAMS Tubes using Gas Chromatography System Coupled with a Mass Spectrometer Detector (GC/MS)

Prepared By:	John L. Schwarz	10/31/13 Date
Reviewed by Analytical Team Leader	John L. Schwarz	10/31/13 Date
Reviewed by Quality Manager	Jill M. Meuser	10/31/13 Date
Approved by Environmental Monitoring Laboratory Chief	Steven D. Norman	10/31/13 Date

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This IOP meets requirements of Revision 6 of the CASARM QA Plan.

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IOP MT-13 Analysis of Chemical Warfare Agents and Degradation Products on DAAMS Tubes using Gas Chromatography System Coupled with a Mass Spectrometer Detector (GC/MS)

Record of Change

- Revision 15 – In Section 3.5.6, added description of how calibration challenges are evaluated. Clarified that calibration linearity requirement is for coefficient of determination (r^2).
- Revision 14 – Added requirement in Section 3.5.6 and Appendix III that all calibration challenges must be within ±15% of the target concentration for successful calibration.
- Revision 13 – Record of Change page formatted to put most recent change first. Typographical errors corrected throughout the document. Chart of QC requirements added as Appendix III. Section 8 "G-analog analysis of VX" removed. This analysis is covered in IOP MT-19.
- Revision 12 -- Typo's corrected and changes made throughout document to reflect current requirements and laboratory operations, e. g., Appendix IV sample data sheet deleted, Table 1 updated to reflect WPL requirements, etc.
- Revision 11 – Added CN (2-Chloroacetophenone) to Table 2A. Changed AEL requirements for GD & GF from 0.00003 to 0.00005 mg/m^3 in Table 2.
- Revision 10—Added: NOTE: Verbal/preliminary Sample Results shall only be provided to clients by the Branch Chief, Lab manager or designee, after successful completion of QC and batch review, e.g., all samples must be bracketed by successful QLs. Added note in paragraph 6.1.4: Flow and Minutes to be adjusted for required volume.
- Revision 9—Changed QP limits to ± 40 percent of TC when operations are under the purview of CMA requirements.
- Revision 8 -Incorporates requirements mandated by CMA LMQAP. Changes QL volume from 3 µl to 4 µl, added WPL & STEL standard concentrations and tables. Added numerous changes to conform to the CMA LMQAP requirements.
- Revision 7 – Added Sections 9.4.1 (reporting criteria for SOP detections), 7.1.3.1 (plotting of QL results), and 6.1.5.3 (Post spiking of QPs for certain samples). Modified Section 10.1 to clarify how QC limits are generated. Added Table 5, Characteristic Ions.
- Revision 6 – IOP was altered by removing references to GCFPD DAAMS analysis. For GCFPD analysis, see IOP MT-19.
- Revision 6 – Incorporated changes required by CASARM QA Plan, Revision 4.
- Revision 6 – Incorporated the following laboratory changes: surrogate added to all samples and QC analyses, removed requirement of a QL every 10 samples, removed requirement of a new calibration every fourteen days, instrument tuning via injection of tuning solution (BFB).
- Previous revisions of this document did not incorporate a record of change page.

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Signature Page

Operator's Statement: I have read, or have had read to me, the procedures in this IOP. I, by my signature below, indicate that I thoroughly understand and agree to abide by these instructions.

Name (print)	Signature	Date

Supervisor's Statement: I have personally reviewed this IOP and, to the best of my knowledge, believe that the information listed herein is correct.

Supervisor: Steven D. Norman Date: 10/31/13

Steven D. Norman

All Deviations from IOP requirements must be authorized by the Analytical Team Leader or designee, and/or Branch Chief.

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IOP MT-13 Analysis of Chemical Warfare Agents and Degradation Products on DAAMS Tubes using Gas Chromatography System Coupled with a Mass Spectrometer Detector (GC/MS)

1.0 GENERAL INFORMATION

One of the main objectives of the Environmental Monitoring Laboratory (EML) is to quantitatively determine the presence of chemical agent vapor contamination in ambient air. This function is provided to ensure worker, public, and environmental safety.

The airborne exposure limits have been revised. The existing Short Term Exposure Limits (STEL) and the revised Worker Population Limits (WPL) are shown in Tables 1 and 2. Items that are processed through the laundry system, Personal Protective Equipment (PPE), re-usable items, items to be released outside government control, and areas where people will not be wearing PPE that could possibly be contaminated must be monitored to the lower WPL limits. Updated guidance allows debris and lab waste to continue to be monitored to the STEL.

1.1 The Gas Chromatography (GC) System coupled with a Mass Selective Detector (MSD) can detect chemical warfare agents (CWA) GA, GB, GD, GF, HD, HN-1, HN-3, CN, L (derivatized with beta mercaptoethanol (BME)), and VX as G-analog (This analysis is covered in IOP MT-19). The CWAs are introduced into the GC system by use of the Depot Area Agent Monitoring System (DAAMS) solid sorbent tubes. The thermal desorption system will desorb the agents from the DAAMS tubes onto an analytical column in the GC that separates the agents and then these agents pass to the MSD.

CN, when analyzed by the current CWA GC/MS method, elutes 0.1-0.2 minutes before Lewisite. During Proficiency and Accuracy (P&A) testing, standard concentrations were approximately 10, 20, and 40 ng/µL (2, 4, and 8 µL).

1.2 The MSD may be used for confirmation or initial analysis. The MSD is a very specific detector that breaks each compound into ion fragments specific to that compound. This allows both qualitative and quantitative analyses. Peak identification is based upon retention time comparison with external standard calibration and by evaluation of the unique spectrum. Analyses are routinely performed in the Selective Ion Mode (SIM) for maximum sensitivity; however, there are occasions where a full scan analysis is desired. The need for full scan analysis shall be determined and/or sanctioned by the Branch Chief or the Laboratory Manager.

1.3 The DAAMS tube is filled with sorbent material, which traps the agents from air flowing through the tube. The DAAMS tube is designed to collect and trap semi-volatile organic compounds, which are released during thermal desorption using an Automated Concentrator for Environmental Matrices (ACEM) desorber. A specific flow rate and sampling time are used to collect each DAAMS sample and each sampling location consists of two DAAMS tubes, one for initial analysis and one for confirmation, as needed.

1.4 The desorber goes through tube dry, tube heat, tube cool, trap heat, and system recycle steps. During the tube heat step, the tube is heated to transfer the collected sample to a focusing trap in the desorber. During the trap heat step the focusing trap is heated to inject the collected sample onto the GC column. At the completion of the system recycle step the desorber is ready to accept the next tube for analysis.

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2.0 DATA COLLECTION AND STORAGE

2.1 The data collected from each GC are stored electronically on a Laboratory Information Management System (LIMS) and/or ChemStation. These electronic data serve as historical records and are stored in a safe place. Paper hardcopies are scanned and preserved on CDs (IOP MT-27). After validating the CDs, the paper hardcopies are destroyed. The chromatogram consists of peak area, retention time, and other related information. Results of analyses are checked for validity. All data are reviewed by the Laboratory Manager and/or designated Senior Staff.

2.2 The electronic systems report the results in nanograms (ng). These values are evaluated with reference to sampling time and rate of airflow during sampling (e.g., STEL level for GB is 7.2 ng if sampled for 120 minutes at 0.6 Liters per minute airflow). Results are transferred to the Daily Run Log (MB-FORM 36) containing information about sampling site, flow rate, sampling time, etc. Analytical results are conveyed to the Point of Contact (POC).

3.0 CALIBRATION PROCEDURES

3.1 The GCs shall be recalibrated in the following events: the GCs or individual components have undergone extensive repair, the instrument is out of control, or at other times when the operators deem it necessary. Instructions listed in Section 3.5 shall be followed for recalibration.

3.2 Separate standards will be prepared for calibration and Quality Control (QC) checks. Calibration standards shall be used to calibrate the instrument and QC standards shall be used (via Quality Lab (QL) and Quality Process (QP) samples) to check instrument operation.

NOTE: All calibration standards, QLs, QPs, and samples analyzed shall be fortified with a surrogate compound and evaluated in accordance with Appendix I.

3.3 Preparation of calibration tubes

3.3.1 Remove agent calibration standards (CAL), and the surrogates standards 4-Bromofluorobenzene (BFB) and Hexachlorobenzene with 0.1% BME (used for Lewisite derivitization) from cold storage and place in fume hood. Allow standard to reach room temperature. The typical CWA standards used are Air Combo (AC) in hexane and Lewisite in methanol.

3.3.2 Using a 10 µL syringe spike a clean, conditioned tube with the appropriate amount of CAL standards, BFB, and HCB/BME by inserting the syringe needle into the frit end of the DAAMS tube and depressing the plunger on the syringe.

3.3.3 Place the DAAMS tube into the ACEM tube desorber for analysis.

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3.4 Tune Verification

3.4.1 At the beginning of each operational day, the analyst will perform tune verification on the GC/MSD in accordance with requirements specified in Appendix II. Criteria for autotune relative abundances and BFB tune criteria are shown in Tables 3 through 6.

3.4.2 If operations exceed 12 hours, tune verification shall be performed and evaluated at least every 12 hours. Upon completion of the tune verification a QL shall be analyzed to ensure that instrument is in control.

3.5 Initial Calibration

3.5.1 Calibration must be performed using the sample introduction technique that will be used for samples.

3.5.2 Calibration of an analytical instrument involves the delineation of the relationship between the response of the instrument and the amount or concentration of an analyte introduced into the instrument. The graphical depiction of this relationship is often referred to as the calibration curve. In order to perform quantitative measurements, this relationship must be established prior to the analysis of any samples, and thus, is termed initial calibration. The initial calibration for this chromatographic method involves the analysis of standards containing the target compounds at a minimum of three different concentrations covering the working range of the instrument.

3.5.3 At a minimum, a three-point calibration curve is required. Additional points may be analyzed if necessary. Typical concentrations of the standards are listed in the Table 7. Each standard is spiked separately onto the DAAMS tube. Typical volumes for calibration levels 1, 2, and 3 are 2 µL, 4 µL, and 8 µL of each CAL solution, while 5 µL of 0.1% BME are used at each level. (These amounts are equivalent to 0.5, 1.0, and 2.0 Airborne Exposure Limit (AEL), except for the vesicants which have a considerable higher AEL).

3.5.4 Tabulate the area response against the concentration for each target analyte. This can be done using the ChemStation Data Analysis Software.

3.5.5 Linear calibration using a least squares regression must be used. For all surrogates and agents, the analyst must employ a linear regression equation that is not forced through the origin. This is most easily achieved by performing a linear regression of the instrument response versus the concentration of the standards. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). This is a statistical requirement and is not simply a graphical convention. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = mx + b$$

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where:
y = instrument response (peak area)
x = concentration of the calibration standard
m = slope of the line (also called the coefficient of x)
b = the y-intercept

The regression calculation will generate a coefficient of determination (r^2) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.000 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.990.

3.5.6 All of the calibration challenges must be within ± 15% of the target concentration for the calibration to be valid. Evaluate the calibration challenges using MBFORM-141. The form will indicate whether all aspects of calibration are acceptable, including the coefficient of determination, the calibration challenges, and the results of the QL. If any factor does not meet acceptance criteria, the instrument must be recalibrated and evaluated again.

3.6 Initial Calibration Verification (ICV)

3.6.1 Immediately following calibration, an initial calibration verification QL must be analyzed. This is accomplished by analyzing a 4ul QL. The results from the calibration standard analysis should meet the verification acceptance criteria provided in Section 3.7.1.

3.6.2 Preparation of QL tubes

3.6.2.1 Remove agent QC standards and the surrogate standards BFB and HCB with 0.1% BME from cold storage and place in fume hood. Allow standard to reach room temperature. For the majority of the analyses there will be two separate QC vials. One containing the Air Combo (AC), and the other the Lewisite (L) standard.

3.6.2.2 Using a 10 µL syringe spike a clean, conditioned tube with the appropriate amount of QC standards, BFB, and HCB/BME by inserting the syringe needle into the frit end of the DAAMS tube and depressing the plunger on the syringe.

3.6.2.3 Place the DAAMS tube into the ACEM tube desorber for analysis.

3.6.3 The calibration verification spiking standard must not be prepared from the same stock solutions as those used for the initial calibration standards.

3.6.4 If the ICV QL fails, a second QL may be analyzed. If the second QL also fails, corrective actions shall be performed on the instrument and documented on the Excel® Data Review Checklist (MB FORM-50) comments section; recalibration will be noted on the Excel® QC Chart (MBFORM-10). The instrument must be recalibrated as described in Section 3.5.

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3.6.5 Instrument maintenance and changes to system parameters as part of the corrective action must be recorded in the instrument logbook and initialed and dated.

3.7 Continuing Calibration Verification (CCV)

3.7.1 The calibration curve (Section 3.5) for each compound of interest must be verified at least once every 12 hours or for every twenty samples analyzed, whichever comes first, using the introduction technique used for samples. This is accomplished by analyzing a 4 µL QL prepared with the QC standards. All samples must be bracketed by passing QLs. The results from the QC standard analysis must meet the following verification acceptance criteria:

If the percent difference between the found and true amount for any target agent is less than or equal to ±15%, the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than 15% difference between the found and the true amount), for any one agent, a second QL challenge may be performed. If the second challenge also fails, then corrective action must be taken prior to the analysis of samples. If the agent(s) not meeting the ±15% criterion are not included in the list of analytes for a sampling event, no corrective action is required. If the first and second QL of the day fails this ±15% test, corrective actions shall be performed on the instrument and documented on the Excel® Data Review Checklist (MB FORM-50) comments section; recalibration will be noted on the Excel® QC Chart (MBFORM-10). The instrument must be recalibrated as described in Section 3.5. Any time an instrument is calibrated, new QLs must be analyzed and quantitated against the new calibration curve. If any target agent in an ongoing or ending QL exceeds the ±15% criterion, then the system is considered out-of-control and analysis must cease. See Section 7.0 for out-of-control situations.

NOTE: If the percent recovery for the second QL is greater than 115%, the samples analyzed before the failing QLs may be reported (see Section 7.1), but analysis must cease and corrective action (recalibration) must be performed before continuing.

3.7.2 The calibration verification QC standards must not be prepared from the same stock solutions as those used for the initial calibration standards. Occasionally, clients will request analyses for one or more non-CASARM analytes. Standard will be supplied by the client if not available commercially. In this case, there may only be one available standard stock.

3.7.3 Instrument maintenance and changes to system parameters as part of the corrective action must be recorded in the instrument logbook and initialed and dated.

4.0 BLANK ANALYSIS PROCCEDURE

Place a clean, pre-conditioned DAAMS tube into the ACEM desorber and depress the "Start" button. Review the chromatogram for instrument contamination. If the blank contains interference peaks, run additional blanks until no interferences are detected. A blank should be

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analyzed immediately before the analysis of QPs and samples, or at any other time during the analytical shift, to ensure that the total system (introduction device, transfer lines, and GC system) is free of contaminants. If the blank indicates contamination, then it may be appropriate to bake the system for an extended period of time or perform other instrument cleaning.

5.0 PROCEDURES

5.1 Receipt of Samples

5.1.1 DAAMS samples are received from the Sample Collection Team (SCT) after they have been logged into the Horizon LIMS. The scratch log is signed to demonstrate chain-of-custody. A copy of the signed scratch log is returned to the SCT technician and another copy is kept with the samples and becomes part of the data results packet. Samples not analyzed the same day as collected are stored in the sample refrigerator at or below 4 degrees Celsius until analyzed.

5.1.2 For field operations, DAAMS samples are logged into the LIMS program that generates the scratch log. The samples are then delivered to the Mobile Analytical Platform (MAP) or appropriate on-site vehicle for analysis. The scratch log is signed by the analyst to demonstrate chain-of-custody.

5.2 Batch Assignment in Horizon LIMS

The analyst assigns samples to a batch in the LIMS before or during instrument analysis. Samples and QPs assigned to a batch shall be sampling event specific (see Section 6.1.3). A batch is a group of samples unique to a sampling event and analyzed on a given instrument on a given day that includes QP samples collected with the samples being analyzed. ICV or CCV QL samples shall be unique to the instrument, agent, and date of operation (i.e., each instrument shall be under operational control as demonstrated by acceptable QLs that bracket samples for all batches analyzed on a given day). The analyst adds QPs and samples into the LIMS batch. The analyst adds QLs to the batch as they are analyzed. There may be batches containing only calibration and/or QL samples. At a minimum, there must be a QL analyzed for each twenty samples. All samples shall be bracketed by passing QLs and there must be one acceptable QP per batch. All calibration analyses shall be added to the batch when the instrument is calibrated. The analytical batch size is limited to twenty samples and their associated QC samples. Only one acceptable QP can be contained in a LIMS batch. If more than one QP was analyzed before an acceptable QP is achieved, the failing QP(s) must be flagged in LIMS as "XX."

5.3 Sample Analysis

NOTE: All deviations from specified requirements shall have written authorization (initials & date) from the Laboratory Manager and/or the Branch Chief. All pertinent information regarding deviations from specified requirements shall be documented and included with the analytical results.

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5.3.1 Initial Analysis

The DAAMS tube will be placed in the ACEM for thermal desorption and subsequent analysis by GC/MS. The analyst will review the data and determine whether the sample is clear or not clear for the agents of interest by evaluating the extracted ion chromatograms.

*NOTE***:** *Verbal/Preliminary Sample Results shall only be provided to clients by the Branch Chief, Lab manager, or designee, after successful completion of QC and batch review. All samples must be bracketed by successful QLs.*

5.3.2 Confirmation Analyses

5.3.2.1 GC/MS is considered to be a confirmation technique. Unique situations or analytical anomalies may require specific samples to undergo sample confirmation when requested.

5.3.2.2 If a MINICAMS alarm needs to be confirmed, the analysis is qualitative and no QP is required, as follows:

5.3.2.2.1 Analyze Tube 1 under normal conditions (See Section 5.3.1). If agent is detected then MINICAMS alarm is confirmed and no further analysis is required.

5.3.2.2.2 If no agent is detected, analyze Tube 2 under the following conditions:

5.3.2.2.2.1 Spike Tube 2 with a mass equivalent to the alarm (4 µL or as determined from the Class 3 P&A).

5.3.2.2.2.2 If agent is detected, the qualitative method is satisfactory and the MINICAMS alarm is considered to be false positive.

5.3.2.2.2.3 If no agent is detected, the method is unsatisfactory and the MINIMCAMS alarm is considered to be a positive alarm

5.3.2.2.3 All analytical results are entered into LIMS with a notation for Tube 2 that the sample was post-spiked.

5.4 Re-conditioning of DAAMS Tubes

All DAAMS tubes analyzed and tubes not analyzed will be re-conditioned after analysis is completed, documented, and results reported.

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6.0 QUALITY CONTROL

A summary of all quality control requirements is shown in Appendix III.

6.1 QP Preparation and Analysis

6.1.1 QPs are collected with every batch or sampling event to as closely as possible represent the sampling environment. For background/SOPs samples, the QP should be co-located with the samples. For vapor screening sampling of multiple items with similar characteristics, only a single set of QPs is required for batch sizes of 20 or less. The QP ensures that the analyte of interest can be detected in the sampling environment. It is also used to correct positive detections based on recoveries less than 100%. QP results greater than 100% are not used for sample correction.

6.1.2 The QP DAAMS tubes are aspirated at the appropriate sampling site for the required amount of time and subsequently returned to the laboratory or MAP for analysis. A minimum of two QPs shall accompany every sample set. More QPs may be included to assure a minimum of one acceptable QP sample per 20 samples.

6.1.3 A sample set is defined as samples of the same sampling event analyzed each day of operation (e.g., samples from J-Field, 5100, or SPT).

NOTE: *For purposes of this document, lab waste samples in a defined area will be considered a specific sampling event. For example, if 13 lab waste bags are located in Building 5100, a QP is necessary for those 13 samples, not each individual bag. Also, if there are two or more locations with lab waste samples, each location will have an associated QP. For any QP monitoring for lewisite, spike 5 µL of 0.1% BME.*

6.1.4 If there is a need to prepare QPs in the laboratory, connect the glass frit end of the tube to the spiking assembly. Spike the tube in the spiking block with 4 µL of the appropriate QC standards. If monitoring for lewisite, spike 5 µL of 0.10% BME. Attach the glass wool end of the spiked tube to the vacuum pump. Set the vacuum flow to be 0.25 liters per minute or a sample volume equal to the volume of the collected samples. Record the date, on time, and flow rate on MBFORM-66. Run the system for required minutes at this flow rate. After the required minutes have elapsed, measure the flow through the tubes and record off time and ending flow rate on MBFORM-66. Remove the tubes and transfer to the ACEM for analysis.

NOTE: *Flow and Minutes may be adjusted for required volume.*

6.1.5 QP samples will be introduced during the analysis routine at one QP per 20 samples. This QP sample validates the ability to detect the agent in field samples. QPs will be spiked using the QC standards and will be of the same sampling event as the samples being analyzed. A QP for waste or XXX evaluation is considered to be acceptable if a minimum percent recovery of 10% is achieved and the instrument is shown to be operating properly as shown by acceptable QLs. However, when

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samples are under the purview of CMA, the QP recovery range shall be 60 to 140%. An initial QP with a recovery greater than 150% shall be considered to have an interference and shall require analysis of the second QP. If both QPs have a recovery greater than 150%, the location shall require resampling. All QPs will be entered in the LIMS batch with the QPs not being used flagged as "XX."

*NOTE***:** *QPs that fail due to high recovery because of interference will be reviewed by the Laboratory Manager or designee, and/or Branch Chief. Clearance of these type samples shall be authorized only by the Laboratory Manager or designee, and/or the Branch Chief. Initials of person authorizing the clearance, plus date authorized, will be included with the documented analytical results.*

6.1.5.1 If a QP response falls below the lower established laboratory limit, a second QP must be analyzed. If the second QP response is within specified limits, analysis may proceed of the samples associated with that QP. If the second QP response falls below the lower established laboratory QC limit, analyze the remaining QPs associated with the samples until one is within established laboratory QC limits. If one QP falls within established laboratory QC limits, analysis may proceed of the samples associated with that QP. If none of the associated QPs are within the established laboratory QC limits, all analyses must cease for all samples associated with those QPs. The location associated with the failing samples will need to be re-sampled.

*NOTE***:** *In case of a unique sample, one that can-not be re-sampled, analysis may proceed without a passing QP. This will be determined on a case-by-case basis by the Laboratory Manager or Branch Chief. If the samples are analyzed, it must be noted on the Daily Run Log, entered in LIMS, and communicated to the client that the QP failed and results are not defensible. The second sample tube must be post-spiked with 4 µL of the analytes of interest and recoveries reported.*

6.1.5.2 In the event of a QP failure for a sample that can't be repeated (i.e. Background, SOP), the Branch Chief or Laboratory Manager will be notified immediately. The second sample tube will be spiked with the agents of interest (post spiked) and analyzed. Results will be reported to the Branch Chief, Laboratory Manager or designee for interpretation and disposition.

6.1.6 Only one sampling event will be put in a LIMS batch. For samples received without QPs, a laboratory prepared QP shall be used. Samples received without QPs will be treated as a single sampling event, therefore samples from different sites may be associated with the same laboratory QP. Different sampling events with QPs must not be entered into the same LIMS batch.

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7.0 OUT-OF-CONTROL SITUATIONS

7.1 Instrument Control

7.1.1 If one or more agents of multiple agent CCV QLs (a second QL permitted) is less than the -15% requirement for both QLs, (i.e., recovery is low for both QLs) the instrument is out of control. In addition, if the average percent recovery of two consecutive out of control QL (CCV) samples is ≤ 50%, implement corrective action, analyze duplicate samples, or use appropriate data qualification for all samples. Samples analyzed after the last in-control QL are considered suspect and must be reanalyzed for the failing agent(s). Corrective action must be performed and documented before continuing any analyses. Sample analysis shall not continue until the system is shown to be in control.

7.1.2 If one or more agents of multiple agent CCV QLs (a second QL permitted) is greater than the +15% requirement (i.e., recovery is high for both QLs) but less than +50% (150% recovery), the instrument is out of control. The analytical results of all samples analyzed since the last in-control QL that do not exceed 1 AEL can be cleared. If any sample has a target agent(s) detected above 1 AEL and that agent(s) fails the QC criterion, the second tube for that sample shall be analyzed and/or the location(s) re-sampled for the failing agent(s). Sample analysis shall not continue until the system is shown to be in control.

7.1.3 If the QL response is greater than the +50% criterion (150% recovery) of the target concentration for both QLs, the instrument is out of control. All samples back to the last acceptable QL are suspect and the second tube for these samples shall be analyzed and/or the location(s) re-sampled for the failing agent(s). Corrective action must be performed and documented before continuing any analyses. Sample analysis shall not continue until the system is shown to be in control.

7.1.4 The average result of the daily QLs shall be plotted on the Excel® QC Chart (MBFORM-10) and included as part of the data package for review by specified personnel (see Appendix IV).

7.1.5 All actions shall be documented on the Excel® Data Review Checklist (MB FORM-50) comments section and recalibration on the Excel® QC Chart (MBFORM-10) comments section.

7.2 Statistical Control

NOTE: *Pre-control procedures are normally used to maintain control of laboratory analytical instruments (i.e., ICV and CCV QL recoveries shall be within ± 15% of the target concentration for all analytes of interest to provide assurance that the instrument is in control prior to analyzing samples and/or during sample analysis). Control charting and statistical control techniques may also be used by management for evaluating/controlling instrument stability, but is not a requirement of CASARM or the*

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CMA LMQAP. The following section defines statistical control requirements that may be used by management.

7.2.1 Out-of-statistical-control situations are defined by the placement and trends of daily average and range QL results on the control charts. An out-of-statistical-control situation does not call for an immediate cessation of analysis. It is an indication that the system could go out of control. The criteria for out-of-statistical-control situations are as follows:

7.2.1.1 A daily QL average that is outside the control limits on the x-bar (average) chart.

7.2.1.2 A point that is above the upper limit on the R (range) chart.

7.2.1.3 Eight (8) or more successive points on the same side of the central line on the x-bar (average) or R chart.

7.2.2 If an out-of-statistical-control situation occurs, the analyst shall examine the system for unusual conditions and may perform several routine maintenance procedures in an attempt to determine the cause of the out-of-statistical-control situation. Re-calibration may be required.

7.2.3 All actions shall be documented on the Excel® Data Review Checklist (MB FORM-50) comments section and recalibration on the Excel® QC Chart (MBFORM-10) comments section. All instrument maintenance shall be documented in the instrument logbook.

8.0 DATA REVIEW

8.1 At the end of each day, the analyst reviews his/her batches, prints the Excel® QC charts (MBFORM-10), completes the checklist (MBFORM-50), initials and dates the daily report (MBFORM-36) to indicate that the data are correct, and completes MBFORM-65. The data are reviewed by the Laboratory Manager or designated senior staff.

8.2 The Laboratory Manager or designated senior staff will review the data, initial the daily report (MBFORM-36) as being acceptable and then will perform the management review function in the LIMS system.

8.3 The QA officer will review the daily report and perform the QA review function in the LIMS system.

8.4 The EML Chief and/or the Laboratory Manager will be informed as soon as possible after a confirmed result above AEL (TWA) (Z) is noted. The EML Chief or the Laboratory Manager will contact the POC and/or the Safety Office as deemed necessary. Information that must be given to the Safety Office includes: The results are expressed in nanograms, percent WPL/STEL, and mg/m^3. Sampling time and flow will also be provided.

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For SOP samples, any positive result must be communicated to the EML Chief and/or the Laboratory Manager as soon as possible. The Laboratory Manager will notify lab personnel of what, if any, action is to be taken. This may include analysis of the second tube or analysis of a QP/QL.

8.5 The required action level calculations are listed in the EML Quality Control Plan and have been included in this document for convenience (see Appendix V and Tables 1 and 2).

9.0 QL AND QP LIMITS

9.1 QL control chart limits are not a requirement, but may be generated from historic DAAMS data for use in monitoring instrument performance. When used, QL control chart limits will be generated using the first twenty data points for each agent and instrument. These limits will be changed as required when the process changes. QP limits are 10% to 150% **except as noted below,** and are not changed.

*NOTE***:** *When under the purview of CMA the QP limits will be 60 to 140%.*

9.2 The QL limits are calculated using the following statistical formulae:

Upper Control Limit $= UCL_{\overline{X}} = \overline{X} + \overline{R}(1.880)$ or X-Bar + 3 Std Dev.

Central Line $= \overline{X}$

Lower Control Limit $= LCL_{\overline{X}} = \overline{X} - \overline{R}(1.880)$ or X-Bar – 3 Std Dev.

10.0 HAZARDS

10.1 Dilute Chemical Warfare Agents

The EML utilizes near drinking water level dilute chemical warfare agents in preparing quality control samples for the IOP. The EML office contains a reference file of material safety data sheets (MSDS) for the dilute CW agents.

10.2 Solvents and Other Materials

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available that includes, but is not limited to, donning the appropriate laboratory safety attire and working in well-ventilated hoods. The laboratory is responsible for maintaining a current file of Occupational Safety and Health Administrations (OSHA) regulations regarding the safe handling of the chemicals specified in this IOP. An MSDS reference file of materials used in these operations is available at the worksite.

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11.0 APPARATUS AND MATERIALS

11.1 Typical Dynatherm Conditions

Carrier gas (He) flow rate: 35-40 mL/min

Tube Dry = 1 min

Tube Heat = 3 min @ 300 °C

Trap Heat = 2 min @ 300 °C

11.2 Gas Chromatograph (GC)

11.2.1 GC – An analytical system complete with a temperature-programmable gas chromatograph suitable for splitless injection and all required accessories, including syringes, analytical columns, and gases. The capillary column should be directly coupled to MSD.

11.2.2 The GC should be equipped with variable constant differential flow controllers so that the column flow rate will remain constant throughout desorption and temperature program operation.

11.2.3 Typical Chromatographic Conditions

Carrier gas (He) flow rate: 1.5 mL/min

Column: 30 m x 0.25 mm ID, 1 µm film thickness, silicone-coated fused-silica capillary column (J&W DB-1701 or equivalent)

Initial temperature: 50 °C, hold for 1 minute

Ramp 1 = 25 °C/min to 150 °C

Ramp 2 = 35 °C/min to 260 °C

Final temperature: 260 °C, hold for 2.0 minutes

11.3 Mass Spectrometer Detector (MSD)

11.3.1 MSD –The MSD creates charged fragments of each compound and then uses quadrupoles with an applied electrical current to separate the fragments. The mass/charge particles and their ratios are unique to a compound. This technique allows for the generation of a spectrum, a "fingerprint," of each compound thus enabling a quick and reliable identification and quantification. Characteristic masses (m/z) for identifying CWA are shown in Table 8.

11.3.2 GC/MSD interface - Any GC-to-MSD interface may be used that gives acceptable calibration points at 2ng or less per injection for each compound of interest and achieves acceptable tuning performance criteria. For a narrow-bore capillary column, the interface is usually capillary-direct into the mass spectrometer source.

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11.3.3 Typical MSD Conditions

Mass range: 35 – 260 amu (full scan) or Selected Ion Monitoring (SIM)

Scan time: 0.6 – 2 sec/scan

Source temperature: 230 °C

11.4 Data system

A computer system should be interfaced to the MSD. The system must allow the continuous acquisition and storage on machine-readable media of all data obtained throughout the duration of the chromatographic program.

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TABLES

Table 1. Required Action Levels (STEL)
For Air Monitoring Program DAAMS Tubes for STEL (Based on Specified Sampling Parameters)

Agent	Allowable AEL* (mg/m^3)	Sample Volume (liters)	Required Action Level (ng on tube)	Concentration of Agent in the Calibration Standard (µg/ml)
GA, GB	0.0001	24	2.4	0.6
GD, GF	0.00005	24	1.2	0.3
VX	0.00001	24	0.24	0.06
HD, L	0.003	24	72	18
HN-1, HN-3	0.003	24	72	18
***CN	0.3	24	20	***5

* Time Weighted Average for 40-hour workweek.
CN AEL 0.3 mg/m^3 (0.05 ppm) = 7200ng *** Concentration in Standard for P&A: 1µl = 5 µg/ml.

Table 2. Required Action Levels (WPL)
For Air Monitoring Program DAAMS Tubes for the WPL (Based on Specified Sampling Parameters)

Agent	Allowable AEL* (mg/m^3)	Sample Volume (liters)	Required Action Level (ng on tube)	Concentration of Agent in the Calibration Standard (µg/ml)
GA, GB	0.00003	24	0.72	0.18
GD, GF	0.00003	24	0.72	0.18
VX	0.000001	24	0.024	0.006
HD	0.0004	24	9.6	2.4

* Time Weighted Average for an 8 hour day.

Table 3. Example of EI Autotune Relative Abundances (CMA)

Mass (AMU)	Relative Abundance (%)	Isotope Ratio (%)
69	100	Mass 70: 0.54 – 1.6
219	70 – 150	Mass 220: 3.2 – 5.4
502	≥ 2.5	Mass 503: 7.9 – 12.3

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Table 4. Example of EI Autotune Relative Abundances (Agilent)

Mass (AMU)	Relative Abundance (%)	Isotope Ratio (%)
69	100	Mass 70: 0.54 – 1.6
219	> 30	Mass 220: 3.2 – 5.4
502	> 1	Mass 503: 7.9 – 12.3

Table 5. Example of BFB Target Tune Relative Abundances

Mass (AMU)	Relative Abundance (%)	Isotope Ratio (%)
69	100	Mass 70: 0.54 - 1.6
131	≥ 35	None
219	≥ 30	Mass 220: 3.2 – 5.4
502	0.8 – 1.0	Mass 503: 7.9 – 12.3

Table 6. BFB Tune Criteria

m/z	Ion Abundance Criteria
50	8.0 – 40.0% of mass 95
75	30.0 – 66.0% of mass 95
95	Base peak, 100% relative abundance
174	50.0 – 120.0% of mass 95
176	93.0 – 101.0% of mass 174

Table 7. Calibration Levels and Typical Concentrations

		GA/GB/GD/GF	HD	L/ HN-1/HN-3	BFB
CAL Level	µL (TWA)	Amount(ng)	Amount(ng)	Amount(ng)	Amount(ng)
1	2 (0.50)	0.36	4.8	10	0.5
2	4 (1.0)	0.72	9.6	20	1.0
3	8 (2.0)	1.44	19.2	40	2.0

NOTE: For CN Calibration Levels and Typical Concentrations see Paragraph 1.1.
At each calibration level 5 µL of HCB with 0.1% BME is spiked.

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Table 8. Characteristic Masses (m/z) for Chemical Warfare Agents

Compound	Primary Characteristic Ion	Secondary Characteristic Ions
GA	106	117, 133, 162
GB	125	81, 99
GD (1 & 2)	126	82, 99
GF	99	54, 67, 81
HD	109	111, 158, 160
HN-1	120 / 122	56, 92, 120 / 122
HN-3	63	154, 156, 158
L (BME Derivative)	212	107, 151,186
2-Chloroacetophenone (CN)	91	105, 77, 51
BFB	174	95, 176
HCB	249	284, 142, 107

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Appendix I Surrogate Standard

Surrogate Standard – The recommended surrogate is 4-bromofluorobenzene (BFB). Other compounds may be used as surrogates, depending upon the analysis requirements.

Surrogate Recoveries – It is necessary that the laboratory evaluate surrogate recovery data from individual samples. The analyst must evaluate the recovery to determine if acceptable system performance was maintained for each analysis. Generally, a 50% or higher recovery is considered acceptable. However, the analyst should base his observation on instrument behavior that day.

Surrogate recovery is calculated as:

$$\text{Recovery (\%)} = \frac{\text{Concentration (or Amount) Found}}{\text{Concentration (or Amount) Added}} \text{ x } 100$$

If the recovery is not within normal surrogate recovery limits, the following procedures are necessary:

- Check to be sure that there are no errors in the calculations or surrogate solution standard. If errors are found, recalculate the data accordingly.
- Examine chromatograms for interfering peaks and integrated peak areas.
- Check instrument performance.

If an instrument performance problem is identified, correct the problem and re-analyze the second DAAMS tube. If no instrument problem is found, the sample should be re-analyzed. If, upon re-analysis, the recovery is again not acceptable, report the data as an "estimated concentration." If the recovery is acceptable in the re-analysis, provide the re-analysis data to the data user.

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Appendix II GC/MS Tune Evaluation

1.0 MSD Tune Criteria

1.1 Prior to the analysis of any samples, blanks, or calibration standards, the analyst must establish that the GC/MS system meets the mass spectral ion abundance criteria. PFTBA tunes shall be used for (EI) and PFDTD tunes shall be used for (CI). Tunes shall be performed monthly or as needed to maintain relative abundances. The laboratory may also utilize a mid-mass tune or BFB target tune. Tune verification should occur every twelve hours.

2.0 Technical Acceptance Criteria for GC/MS Performance Check

2.1 PFTBA tunes are performed by the instrument software. There are several types of PFTBA tunes that may be utilized.

2.2 The mass spectrum of BFB must be acquired in the following manner.

2.2.1 Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scan prior to the elution of BFB. Do not background subtract part of the BFB peak.

NOTE: *All subsequent standards, samples, QPs, QLs, and blanks associated with a new tune must use identical mass spectrometer instrument conditions.*

2.2.2 Tune verifications must meet the ion abundance criteria given in Table 6.

3.0 Corrective Action for GC/MS Performance Check

3.1 If the technical acceptance criteria are not met, retune the GC/MS system. It may also be necessary to clean the ion source or take other corrective actions to achieve the technical acceptance criteria.

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Appendix III Summary of Quality Control Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability by performance of Analyst P&A or certification test.	The Analyst shall perform an acceptable P&A or Certification Test IAW Section 4.17 of the EML QC Plan. To maintain certification, the analyst shall perform sample analysis for each certified method once each 60 calendar days.	The Analyst Certification or P&A Test shall meet the requirements of Section 4.17 of the EML QC Plan before the analyst is permitted to analyze samples.	Any time that an Analyst becomes "uncertified" he/she shall perform a re-certification test IAW the requirements of the EML QC Plan.	*NA*	This is a demonstration of analyst ability to generate acceptable analytical data IAW specified requirements. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
Tuning	Tuning verified before calibration and every 12 hours during sample analysis.	Refer to method-specific criteria. For this IOP, criteria are shown in Table 7.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Initial Calibration (ICAL): Minimum 3 standards for all target analytes.	Before sample analysis, at any major change in analytical system, or after failing calibration verification.	$r^2 \geq 0.990$ for linear least squares regression. All calibration challenges within ± 15% of target concentration.	Correct problem, documenting findings and actions; repeat initial calibration	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until CAL has passed.

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Appendix III Summary of Quality Control Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second Source Calibration Verification Quality Lab (QL)	Once after each initial calibration, before sample analysis. Rerun at least every 20 samples or every 12 hours, whichever is sooner, and at the end of the analytical sequence.	Value of second source within ± 15% of expected value (initial source).	For ICAL, Rerun QL. If that fails, correct problem, document findings and actions; verify second source standards. Repeat initial calibration. For mid or end of sequence, if result < 85% of expected value, rerun QL. If that fails, correct problem, repeat ICAL, and re-analyze all samples since the last passing QL. For mid or end of sequence, if result >115% and < 150% of expected value, analysis must stop but previous samples may be reported if they do not exceed 1AEL for affected analyte. Correct problem before continuing. A second QL may be analyzed. For mid or end of sequence, if result > 150% of expected value, analysis must stop. Re-run QL and if that fails repeat ICAL, and re-analyze all samples since the last passing QL	Samples failing QL criteria will be reported with a "LF" qualifier. Failing QLs will be given a "XX" qualifier.	Problem must be corrected. No samples may be run/reported until calibration has been verified.

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Appendix III Summary of Quality Control Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Sample Quality Process (QP)	One for each batch (20 samples maximum).	Value of QP must be between 10% and 150% of expected value (initial source). When under purview of CMA QP limits are 60% to 140%.	Run second QP and third if available. If all these fail items must be re-sampled. In case of a unique sample, one that can-not be re-sampled (BKGD, SOP, etc.), analysis may proceed without a passing QP. This will be determined on a case-by-case basis by the Laboratory Manager or Branch Chief. If the samples are analyzed, it must be noted on the Daily Run Log, entered in LIMS, and communicated to the client that the QP failed and results are not defensible. The second sample tube must be post-spiked with 4 µL of the analytes of interest and recoveries reported.	Samples failing QP criteria will be reported with a "PF" qualifier. Failing QPs will be given an 'XX" qualifier.	
Establish Retention Time Window	Once per ICAL.	Position for all analytes, surrogate, and/or internal standards set using the midpoint standard of the initial calibration curve.	NA	NA	
Evaluation of Retention Times (RT)	With each sample.	RT of analytes, surrogates, and/or internal standards in standards and samples within ± 0.05 minutes of established retention time.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	

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Appendix III Summary of Quality Control Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Instrument Blank	Before beginning an analytical sequence or after an extremely contaminated sample.	No analyte detected	Analyze additional blanks. Set the GC oven temp to 250 °C to "bake out" the instrument.	Flagging criteria are not appropriate.	
Surrogate Standard	All samples, including QC.	Generally, a 50% or higher recovery is considered acceptable. However, the analyst should base observations on instrument behavior that day.	Check to be sure that there are no errors in the calculations or surrogate solution standard. If errors are found, recalculate the data accordingly. Examine chromatograms for interfering peaks and integrated peak areas. Check instrument performance. If an instrument performance problem is identified, correct the problem and re-analyze the second DAAMS tube. If no instrument problem is found, the sample should be re-analyzed. If, upon re-analysis, the recovery is again not acceptable, report the data as an "estimated concentration." If the recovery is acceptable in the re-analysis, provide the reanalysis data to the data user.	Flagging criteria are not appropriate.	

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Appendix IV Data Package Format

The Final Data Package shall include the following in the order listed:

1. Package Separator Sheet (MB FORM-65)
2. EML DAAMS Analysis Technical Review Checklist (MB FORM 86)
3. Sample Clearance Reports Generated by LIMS
4. GCFPD/GCMSD/LCQQQ Data Review Checklist (MB FORM 50)
5. GCMSD Daily Run Log (MB FORM 36)
6. QC Charts (MB FORM 10)
7. Calibration Table (generated from Instrument Data Analysis)
8. Calibration Curves (generated from Instrument Data Analysis)
9. Instrument Quantitation Reports including Manual spectra/blowups of target agents, Q-deletes and manual integrations (generated from Instrument Data Analysis). Reports shall be presented in numerical/chronological order.

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Appendix V Required Action Level

GB is used as an example for calculating the Required Action Level.

Allowable Time Weighted Average (TWA) for GB for 40-hour workweek: 0.0001 mg/cubic meter

Required Action Level Calculation:

$$D\,(\text{ng}) = \frac{A\,(\text{mg})}{\text{m}^3} \; x \; \frac{\text{m}^3}{1000\,L} \; x \; \frac{1000\,(\mu\text{g})}{(\text{mg})} \; x \; \frac{B\,(L)}{(\text{min})} \; x \; C\,(\text{min}) \; x \; \frac{1000\,(\text{ng})}{(\mu\text{g})}$$

Where:
A = TWA, AEL, Z (0.0001 mg/cubic meter for GB)
B = Flow Rate
C = Sampling Interval
D = Required Action Level in µg

For example:

Flow rate in the portable pump: 0.5 L/min
Sampling time: 120 min

Plugging in these values in the formula above:

TWA, AEL for GB: 0.006 µg

Note that there are 1000 ng in 1 µg, therefore 0.006 µg = 6.0 ng.

Results showing 6.0 ng and higher for GB, under conditions listed above, are to be reported to the Chief, EML and to the POC involved.

The same calculations are used in determining the action levels for HD, VX, and GD except that the allowable TWAs are different for these agents.

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IOP MT-60 Analysis of Residual Mustard and Breakdown Products in EDS Neutralent and Waste using Gas Chromatography/Mass Spectrometry

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Environmental Monitoring Laboratory

U.S. Army
Edgewood Chemical Biological Center
Directorate of Program Integration
Chemical Operations

IOP MT-60 Analysis of Residual Mustard and Breakdown Products in EDS Neutralent and Waste using Gas Chromatography/Mass Spectrometry

Record of Change

Revision 1 – IOP format updated IAW EML QAQC Plan and redundancies in the previous version removed.

Revision 0 – IOP created.

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IOP MT-60 Analysis of Residual Mustard and Breakdown Products in EDS Neutralent and Waste using Gas Chromatography/Mass Spectrometry

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Signature Page

Statement: I have read, or have had read to me, the procedures in this IOP. I, by my signature below, indicate that I thoroughly understand and agree to abide by these instructions.

Name (print)	Signature	Date

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IOP MT-60 Analysis of Residual Mustard and Breakdown Products in EDS Neutralent and Waste using Gas Chromatography/Mass Spectrometry

1.0 PURPOSE AND APPLICATIONS

The procedures and parameters within this Internal Operating Procedure (IOP) describe the method to determine Sulfur Mustard (HD), Nitrogen Mustard (HN-3), and the breakdown products 1,4-Dithiane and 1,4-Thioxane in liquid samples where neutralization was effected with 90% Monoethanolamine (MEA) and water.

This method is not within the scope of the Environmental Monitoring Laboratory (EML) accreditation under the Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP). Requirements of the EML Laboratory and Monitoring Quality Assurance/Quality Control Plan for Compliance with ISO 17025 and the DoD Environmental Laboratory Accreditation Program (ELAP) specific to methods within the scope of accreditation do not apply to this method.

Samples are extracted with an organic solvent and the extract analyzed using a Gas Chromatograph interfaced with a Mass Spectrometer.

1.1 Analyte Concentration Range

The concentration range for each analyte of interest varies depending on the data available from the most recent Method Detection Limit (MDL) study and the lowest concentration calibration standard analyzed during initial calibration. Calibration levels range from 500 µg/L to 10,000 µg/L unless otherwise noted. Refer to 40 CFR 136 Appendix B for more information on MDL studies and MDL criteria.

1.2 Sample Matrices and Interferences

Liquid matrices of water or 90% Monoethanolamine (MEA) are typical. However, other matrices may be used provided all quality requirements are achieved.

MEA is a decontamination agent (neutralent) for HD and HN-3. This sample matrix, therefore, is destructive of any HD or HN-3 spiked into quality control (QC) samples. Low recoveries of HD and HN-3 are expected from the active matrix. 1,4-Dithiane and 1,4-Thioxane are target analytes because they are degradation products of HD and will be indicative of HD destruction.

Water samples are usually from waste collected during the neutralization process and may exhibit interferences or HD destruction.

1.3 Throughput

Approximately 20 samples and associated QC can be analyzed in a standard 8-hour workday.

2.0 RISK AND SAFETY ASSESSMENT

Chemical Agent Standard Analytical Reference Material (CASARM) Research Development Test and Evaluation (RDTE) Chemical Warfare Agent (CWA) and commercially-available reference materials for other analyte types are used for preparing standard solutions and QC samples for this IOP.

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this

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viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available including, but is not limited to, donning the appropriate laboratory safety attire and working in well-ventilated hoods.

The laboratory is responsible for maintaining a current file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this IOP. A Material Safety Data Sheets (MSDS) reference file of materials used in these operations is available in the EML administrative office and at the worksite.

During the extraction process, a required safety measure is pointing the needle away from crew members when relieving the pressure in the sample bottle. As much as 100 PSI pressure could be contained in the sample bottle assembly. Failure to comply may result in injury.

3.0 SCIENTIFIC BASIS

3.1 Analysis

Liquid samples are extracted using hexane prior to analysis. The target analyte(s) are introduced into the Gas Chromatograph/Mass Spectrometer (GC/MS) system by injecting the sample extract into a gas chromatograph (GC) equipped with a narrow-bore fused-silica capillary column. The GC Column is temperature programmed to separate the analytes after which the eluents are introduced into the mass spectrometer (MS) via direct connection where detection occurs.

Identification of target analytes [including the internal standard (IS) and surrogate standard (SS)] results from comparing their mass spectra with the electron impact, or electron impact-like, spectra of reference standards. Quantitation occurs via comparison of the response of major – quantitation – ions to known responses using a five-point calibration curve.

Characteristic Masses for Agents of Interest		
Agent of Interest	Primary Characteristic Ion	Secondary Characteristic Ion
1,4-Dithiane	120	59, 61, 92
1,4-Thioxane	104	61, 74
Sulfur Mustard (HD)	109	111, 158, 160
Nitrogen Mustard (HN-3)	156	63, 154
C^{13}-HCB (IS)	290	113, 148, 255
BFB (SS)	174	75, 95, 176

3.2 Calibration

3.2.2 Calibration of an analytical instrument involves the delineation of the relationship between the response of the instrument and the amount or concentration of an analyte introduced into the instrument. The graphical depiction of this relationship is often referred to as the calibration curve. In order to perform quantitative measurements, this relationship must be established before the analysis of any samples, and thus, is

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termed initial calibration. The initial calibration for this chromatographic method involves the analysis of standards containing the target compounds at a minimum of five different concentrations covering the working range of the instrument. In order to produce acceptable sample results, the response of the instrument must be within the working range established by the initial calibration. The extrapolation of the calibration to concentrations above or below those of the actual calibration standards is not appropriate and may lead to significant quantitative errors regardless of the calibration model chosen. Analysts are advised that it may be necessary to prepare calibration standards that cover concentration ranges that are appropriate for specific projects or type of analyses.

3.2.2 For each analyte and surrogate of interest, prepare calibration standards at a minimum of five different concentrations by adding volumes of one or more stock standards and diluting to volume with an appropriate solvent.

3.2.3 The lowest concentration calibration standard analyzed during an initial calibration establishes the Limit of Quantitation (LOQ) based on the final volume of extract (or sample) described in the preparative method or employed by the laboratory. In some cases, the LOQ of an analyte may be higher than the lowest concentration calibration standard, if the Practical Quantitation Limit (PQL) calculated during the MDL study is greater than the concentration of the lowest standard.

3.2.4 The other concentrations should define the linear working range of the detector or correspond to the expected range of concentrations found in actual samples that are also within the working range of the detector.

4.0 TRAINING REQUIREMENTS, QUALIFICATIONS, AND RESPONSIBILITIES

Use of this method is restricted to, or under the supervision of, analysts experienced in the use of gas chromatography and mass spectrometers, who are skilled in the interpretation of mass spectra and their use as a quantitative tool.

5.0 APPARATUS

5.1 Instrumentation

5.1.1 Gas Chromatograph

5.1.1.1 Temperature-programmable and suitable for splitless injection and equipped with variable constant differential flow controllers.

5.1.1.2 Column – 30 m x 0.25 mm, ID 1 µm film thickness, silicone-coated fused-silica capillary column 1701, or equivalent.

5.1.2 Mass Spectrometer

5.1.2.1 Capable of scanning from 35 to 300 amu every 2 seconds or less, using 70 volts (nominal) electron energy in the electron impact ionization mode.

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5.1.2.2 Capable of producing a mass spectrum for BFB which meets all of criteria when between 1 and 50 ng of BFB tuning standard is injected into the system.

5.1.2.3 MS scan rate allowing acquisition of at least 10 spectra while a sample component elutes from the gas chromatograph.

5.1.3 GC/MS Interface

5.1.3.1 Any GC-to-MS interface that gives acceptable calibration points for 2 µL injections for each compound of interest and achieves acceptable tuning performance criteria may be used.

5.1.3.2 Capillary-direct into the mass spectrometer interface is typical for narrow-bore capillary columns.

5.1.4 Data System

5.1.4.1 A computer system interfaced to the mass spectrometer allowing continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

5.1.4.2 Software that can search any GC/MS data file for ions of a specific mass plot such ions abundances versus time or scan number – Extracted Ion Current Profile (EICP). Software should also be available that allows integrating the abundances in any EICP between specified time or scan number limits. The most recent version of the EPA/NIST Mass Spectral Library should also be available.

5.2 Glassware, Miscellaneous Equipment, and Supplies

5.2.1 16 mm x 125 mm round bottom culture tubes

5.2.2 Re-pipetter with disposable tips

5.2.3 Serological Pipette, 2 mL

5.2.4 Pipette bulbs, large and small

5.2.5 Glass Pasteur Pipette, 22cm

5.2.6 Gas-tight syringe or micropipette and disposable tips

5.2.7 120-Volt Minivortexer, or equivalent

5.2.8 2-mL Amber Autosampler Vials with Crimp caps

5.2.9 Autosampler Vial crimper and de-crimper

5.2.10 2 Large Cylinder Wrenches

5.2.11 Vent assembly kit, including variable compression ratio (VCR) union, septa and syringe

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5.3 Chemicals

5.3.1 Hexane, HPLC grade ≥ 95%

5.3.2 Distilled, deionized (DI) water, Milli-Q Water (18 MΩ)

5.3.3 Clean monoethanolamine (MEA), for preparing 90 % MEA for method blank (MB), Laboratory Control Spike (LCS), and Laboratory Control Spike Duplicate (LCSD)

5.3.4 Tune verification standard:

5.3.4.1 4-Bromofluorabenzene (BFB), C_6H_4BrF, 25 µg/mL

-or-

5.3.4.2 Decafluorotriphenylphosphine (DFTPP), C6H5P(C6F5)2, 25 µg/mL

5.3.5 Instrument solvent blank, hexane with 5,000 µg/L Hexachlorobenzene-C^{13} (HCB), C_6Cl_6

5.3.6 Surrogate/Internal Standard Spiking Solution

5.3.6.1 4-Bromofluorabenzene (BFB), C_6H_4BrF, 100,000 µg/L

5.3.6.2 Hexachlorobenzene-C^{13} (HCB), C_6Cl_6, 100,000 µg/L

5.3.7 Calibration Standards containing BFB, and all target analytes for the project, any of HD, HN-3, 1,4-Dithiane, and/or 1,4-Thioxane.

5.3.7.1 5 standards covering the following concentrations: 500 µg/L, 1,000 µg/L, 2,500 µg/L, 5,000 µg/L, and 10,000 µg/L

5.3.7.2 Each standard containing 5,000 µg/L HCB

5.3.8 Calibration Verification Standard made from a different source material than calibration standard, containing 5,000 µg/L each HCB, BFB, and all target analytes for the project, any of HD, HN-3, 1,4-Dithiane, and/or 1,4-Thioxane

5.3.9 Spiking Standard Solution containing 100,000 µg/L of each target analyte for the project, any of HD, HN-3, 1,4-Dithiane, and/or 1,4-Thioxane

6.0 PROCEDURE

6.1 Operating Parameters

6.1.1 Chromatographic Conditions

6.1.1.1 Injector Temperature: 250 °C

6.1.1.2 Helium carrier gas flow rate: 1.5 mL/min

6.1.1.3 Column: DB-1701, 30 m x 0.25 mm I.D. x 1 µm Film

6.1.1.4 Initial Temperature: 50 °C, hold for 1 minute

6.1.1.5 Temperature Program: 25 °C/min

6.1.1.6 Final Temperature: 260 °C hold for 4 minutes

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6.1.2 MSD Conditions

6.1.2.1 Mass Range: 35-260 amu (full scan) or Selected Ion Monitoring (SIM)

6.1.2.2 Scan Time: 0.6 -2 sec/scan

6.1.2.3 Source Temperature: 230 °C

6.2 Sample Preparation and Collection

NOTE: Complete the appropriate pages in the extraction logbook (MBFORM-62) during the extraction process. Complete the Batch Standards, Prepared Reagents, Reagent Lot Numbers,Supply Lot Numbers, and Spectral Interpretation Codes (MBFORM-70), Calibrated Weights Record Log (MBFORM-119), Daily Pipette and Dispenser Performance Check Log (MBFORM-99), and Pipette Lot Performance Check (MBFORM-158), as appropriate, if calibrated weights and balance are available.

6.2.1 MEA Subsampling

6.2.1.1 Use a serological pipette to transfer 2 mL of clean 90% MEA into 3, 16-mm culture tubes, 1 each for the MB, LCS, and LCSD.

6.2.1.2 Inside fume hood, clamp the sample bottle assembly into a ring stand, vise, or other available sturdy clamping device, or hold the sample bottle firmly on the bench surface.

6.2.1.3 As necessary, install a new gas chromatograph septum into one side of the VCR union.

6.2.1.4 Remove the VCR cap from the sample bottle assembly using the two wrenches. Replace the metal gasket after completing subsampling.

6.2.1.5 Install the VCR union with the septum onto the sample bottle assembly.

NOTE: finger-tighten only. DO NOT tighten with a wrench.

6.2.1.6 Insert the needle into the septum. You may wish to position a toxic vent near the union for the venting process and a plastic bag to catch any liquid that is expelled.

6.2.1.7 Carefully and slowly, open the sample-bottle assembly valve to relieve the pressure in the valve. Refer to Section 2.0 for safety and risk assessment.

6.2.1.8 Close the sample-bottle assembly valve.

6.2.1.9 Using the two wrenches remove the sample bottle from the valve body.

6.2.1.10 Remove the contaminated valve body.

6.2.1.11 Transfer contents of the valve body to a sample jar.

6.2.1.12 Reassemble the sample bottle assembly and return to EDS operators for disposal.

6.2.1.13 Use a serological pipette to measure 2 mL of liquid sample into a 16-mm culture tube.

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6.2.1.14 Use a serological pipette to measure 2 mL of the sample into 2 16-mm culture tubes, 1 each for the Matrix Spike (MS) and Matrix Spike Duplicate (MSD).

6.2.1.15 Repeat steps 6.2.1.2 through 6.2.1.12 for each additional sample bottle.

6.2.2 Water Subsampling

6.2.2.1 Use a serological pipette to transfer 2 mL of DI water into 3, 16-mm culture tubes, 1 each for the MB, LCS, and LCSD.

6.2.2.2 Inside fume hood, open the sample bottle.

6.2.2.3 Use a serological pipette to measure 2 mL of liquid sample into a 16-mm culture tube.

6.2.2.4 Use a serological pipette to measure 2 mL of the sample into 2 16-mm culture tubes, 1 each for the MS and MSD.

6.2.2.5 Repeat steps 6.2.2.2 through 6.2.2.3 for each additional sample bottle.

6.2.3 Sample Preparation and Extraction

6.2.3.1 Use a gas-tight syringe or repeater pipette to add 100 µL of Spiking Standard Solution to each MS, MSD and LCS, LCSD.

6.2.3.2 Use a gas-tight syringe or repeater pipette to add 100 µL of the Surrogate/Internal Standard Spiking Solution to the MB, LCS, LCSD, MS, MSD, and samples.

6.2.3.3 Add 1.9 mL of hexane to the MB and each sample.

6.2.3.4 Add 1.8 mL of hexane to the LCS, LCSD, MS, and MSD.

6.2.3.5 Vortex each tube for 10 seconds to mix each thoroughly.

6.2.3.6 Using a Pasteur pipette, transfer the top layer of each tube, after allowing sufficient time for separation, into separate 2 mL autosampler vials then cap each vial.

6.2.3.7 Store sample extracts in cold storage until analysis. Extracts must be analyzed within forty (40) days from the date of extraction.

6.3 Sample Analysis

6.3.1 Before analyzing samples or calibration standards, inject or introduce 1-50 ng of BFB tuning verification standard into the GC/MS system. Decafluorotriphenylphosphine (DFTPP) is also acceptable as a tuning and tune verification compound. The resultant mass spectra for the tuning compound must meet the following criteria before sample analysis.

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BFB Key Ions and Abundance Criteria		DFTPP Key Ions and Abundance Criteria (Alternate Tune Verification Compounds)	
Mass	Ion Abundance	Mass	Ion Abundance
50	8-40% of mass 95	51	10-80% of Base Peak
75	30-66% of mass 95	68	< 2% of mass 69
95	Base Peak	70	< 2% of mass 69
174	50-120% of mass 95	127	10-80% of Base Peak
176	93-101% of mass174	197	<2% of mass 198
		198	Base Peak
		199	5-9% of mass 198
		275	10-60% of Base Peak

6.3.2 Setup an analytical sequence in the instrument software. Refer to Appendix I for typical analytical sequences.

6.3.3 Tuning criteria and GC/MS calibration verification criteria (Appendix II) must be met before analyzing samples.

6.3.4 MB, LCS, and LCSD QC samples may be run up to four hours before sample extracts are analyzed.

6.3.5 Remove sample extracts from cold storage and allow to warm to ambient temperature before analysis.

6.3.6 Inject 2 µL of the sample extract into the GC/MS system using the same operating conditions, including injection volume, used for calibration.

6.4 Corrective Procedures

6.4.1 Manual Integration and Q-Deletions

6.4.1.1 Identify and note the technical reason why the analyte requires manual integration or Q-deletion in such a manner so that an independent reviewer can verify the judgment. Acceptable reasons include, but are not limited to the following: missing ions, wrong peak identified, or baseline drawn incorrectly.

6.4.1.2 Initial and date the notation.

6.4.1.3 Include the full-page report with EICP, spectrum, and quantification data for each manual integration event and Q-deletion before and after the manual integration in the data package.

6.4.2 Refer to Appendix II for all QC Requirements and additional corrective procedures.

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6.5 Qualitative Analysis

6.5.1 Select – or allow the data-system target compound search routine to select – a peak based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound specific retention time.

6.5.2 Compare the relative intensities of the characteristic ions for each compound to the relative intensities of each ion in the reference spectrum. The relative abundances must be within ± 30 percentage points of the reference ions abundances.

6.6 Quantitative Analysis

6.6.1 Calculate the extract concentration using the calculations in Section 7.1 for either external calibration or internal standard. Refer to Section 8.0 for interpretations of quantitative analysis.

6.6.2 If the concentration of any target analyte is above the concentration of the highest level of the initial calibration perform a dilution and reanalyze the diluted aliquot. If consistent with client requirements, the result may be reported with an E qualifier indicating the result exceeded the upper calibration range. If the sample is diluted, the results are reported with a D qualifier.

7.0 CALCULATION, CALIBRATION, AND DOCUMENTATION

7.1 Calculation

7.1.1 If sample dilution is required, calculate the target dilution factor (DF) so that the concentration of the analyte falls within the mid-level to upper level of the calibration range, as follows.

$$\text{Target DF} = \frac{\text{Highest concentration found}}{\text{Concentration of mid} - \text{level calibration}}$$

(Round down to the nearest whole number. If the DF is greater than 5, round down to nearest multiple of 5.)

7.1.2 Calculate the concentration of target analyte(s) in the original matrix, as follows:

7.1.2.1 Calculate the Actual Dilution Factor (DF). This is the final volume divided by the initial volume of solution, as shown below. When determining the DF, only dilution of the extract is included. Do not include any factors related to extraction of the initial sample aliquot.

$$DF = \frac{V_i + V_b}{V_i} = \frac{V_f}{V_i}$$

Where:

V_i = volume in mLs of extract used in the dilution

V_b = volume in mLs of blank solvent used in the dilution

V_f = final volume in mLs of diluted extract

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7.1.2.2 Calculate the sample concentration as follows:

$$\mu g/L = \frac{R_i \times DF \times V_D}{V_S}$$

Where:

R_i = Extract concentration (µg/L)

DF = Actual dilution factor

V_D = Final volume (mL)

V_S = Sample aliquot volume used in the extraction (mL)

7.1.2.3 All concentrations are reported to two significant figures, in terms of the original matrix. Results are generally in units of µg/L, but may be µg/Kg, mg/L, mg/Kg, or %. Units must be consistent within a project.

7.1.3 Surrogate Recovery Calculation

$$\text{Recovery}\,(\%) = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}}\, x\, 100$$

7.2 Blank Determination and Calibrations

7.2.1 Method Blank

7.2.1.1 Analyze a method blank after the calibration standard, or at any other time during the analytical shift, to ensure that the total system (extraction protocol, introduction device, transfer lines, and GC/MS system) is free of contaminants. If the method blank indicates contamination, then it may be appropriate to analyze a solvent blank to demonstrate that the contamination is not a result of carryover from standards or samples. See Appendix II for method blank performance criteria.

7.2.1.2 A Method Blank shall be prepared and analyzed at a minimum of one per preparation batch.

7.2.2 Instrument Blanks

7.2.2.1 Before processing any samples, the analyst must demonstrate through the analysis of an instrument blank that interferences from the analytical system, glassware, and reagents are under control. Each time a set of samples is analyzed or there is a change in reagents, an instrument blank should be analyzed as a safeguard against chronic laboratory contamination.

7.2.2.2 All instrument blanks, initial or as part of the analytical sequence, must be free of target analytes to one-half the LOQ. See Appendix II for all QC requirements.

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7.2.3 Calibration

7.2.3.1 Calibration must be performed using the same introduction technique used for samples.

7.2.3.2 Proceed with the analysis of the calibration standards following the procedure using the introduction method of choice. For direct injection, inject 2 µL into the GC/MS system.

7.2.3.3 For each target analyte and surrogate, prepare calibration standards in accordance with SOP CNG-048 at a minimum of five different concentrations by adding volumes of one or more stock standards and diluting to volume with an appropriate solvent. Typical calibration levels are shown in Section 5.3.7.1.

7.2.3.4 For each analyte, at least one of the calibration standards should correspond to a sample concentration at or below that necessary to meet the data quality objectives of the project, which may include establishing compliance with a regulatory or action limit.

7.2.3.5 Great care must be taken to maintain the integrity of all standard solutions. Standards shall be stored at minus 4 °C or below in amber bottles with PTFE-lined screw-caps or crimp caps.

7.2.4 External Calibration

7.2.4.1 External standard calibration involves comparison of instrument responses from samples to the responses from the target compounds in the calibration standards. Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount (mass) of analyte in the calibration standard is defined as the calibration factor (CF).

7.2.4.2 Tabulate the area response of the characteristic ions (see Section 3.1) against the concentration for each target analyte.

7.2.4.3 Linear Calibration using a least squares regression

7.2.4.3.1 The analyst must employ a regression equation that does not pass through the origin. This is most easily achieved by performing a linear regression of the instrument response versus the concentration of the standards. Make certain that the instrument response is treated as the dependent variable (y) and the concentration as the independent variable (x). This is a statistical requirement and is not simply a graphical convention. For all instances, an appropriate un-weighed least squares method should be used. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = mx + b$$

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Where:

y = Instrument response (peak area or height)

m = Slope of the line (also called the coefficient of x)

x = Concentration of the calibration standard

b = Y-intercept

7.2.4.3.2 The analyst must not force the line through the origin, but have the intercept calculated from the five data points. The use of a linear regression may not be used as a rationale for reporting results below the calibration range demonstrated by the analysis of the standards. The regression calculation will generate a coefficient of determination (r^2) that is a measure of the "goodness of fit" of the regression line to the data. (If the software calculated a correlation coefficient (r), the value must be squared to obtain r^2 before comparing the value to the performance requirement.) A value of 1.000 indicates a perfect fit. In order to be used for quantitative purposes, r^2 must be greater than or equal to 0.990. In calculating sample concentrations by the external standard method, the regression equation is rearranged to solve for the concentration (x), as shown below.

$$x = \frac{y - b}{m}$$

7.2.5 Internal Standard Calibration (Preferred)

7.2.5.1 Internal standard calibration involves comparison of instrument responses from samples to the responses from the target compounds in the calibration standards as normalized by the sample or standard internal standard response. Sample response factors are compared to response factors of the standards. This calibration procedure is acceptable for use after successful completion of an analyte MDL study in accordance with 40 CFR 136 Appendix B.

7.2.5.2 The compound used for internal standard quantitation should be similar to the target analytes in retention time and composition but should not be present in the samples to be analyzed. The purpose of the internal standard is to correct for system sensitivity fluctuations. The internal standard area counts of each sample should fall within the QC limits of +100% and –50% of the integrated area counts of the internal standards from the daily check standard. Samples failing this criterion will be reanalyzed if possible. Repeated failures will be qualified as such in the case narrative. All samples and standards will be spiked to the same concentration of internal standard.

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7.2.5.3 The Response Factor (RF) is calculated as follows:

$$RF = \frac{A_s \text{ x } C_{is}}{A_{is} \text{ x } C_s}$$

Where:

A_s = peak area of the analyte or surrogate

A_{is} = peak area of the internal standard

C_s = concentration of the analyte or surrogate

C_{is} = concentration of the internal standard

7.2.5.4 Linear Calibration using Least Squares Regression

7.2.4.3.3 Construct a calibration curve in accordance with the specifications in Section 7.2.4.3, with the exception that the y-value is the Response Ratio ($\frac{A_s}{A_{is}}$) and the x-value is the Amount Ratio ($\frac{C_s}{C_{is}}$). The coefficient of determination (r2) must be at least 0.990 for the curve to be valid.

7.2.4.3.4 In calculating sample concentrations by the internal standard method, the regression equation is rearranged to solve for the concentration (C_s), as shown below.

$$C_s = \frac{C_{is} \text{ x } \left(\frac{A_s}{A_{is}} - b\right)}{m}$$

7.2.6 Initial Calibration Verification

7.2.6.1 Immediately following initial calibration, initial calibration verification (ICV) must be performed by analyzing a calibration standard at a concentration near the midpoint concentration of the calibrating range. The results from the calibration standard analysis must meet the verification acceptance criteria in Appendix II. If the ICV does not meet acceptance criteria, a second ICV may be analyzed. Analysis may not proceed for any analyte with a failing ICV.

7.2.6.2 The calibration verification spiking standard must not be prepared from the same standards as the initial calibration standards. However, the same spiking standard prepared for the matrix spike and LCS may be used.

7.2.7 Continuing Calibration Verification

7.2.7.1 Continuing calibration verification (CCV) consists of analysis of a midpoint standard from a different source than the initial calibration standard. It is required that a calibration verification standard be analyzed before sample

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analysis, after no more than every 10 field samples, and at the end of the analytical sequence.

7.2.7.2 The results from the analysis of the CCV must meet the acceptance criteria shown in Appendix II. If the verification analyses do not meet the criteria, the corrective actions shown in Appendix II must be followed.

7.3 Standards, Controls, Spikes, and Validation Programs

7.3.1 GC/MS Tuning

7.3.1.1 GC/MS tuning verification must be performed at the beginning of each 12-hour analytical shift.

7.3.1.2 A valid tune verification must be performed at least every 12 hours during sample analysis. Refer to Appendix II for all QC requirements.

7.3.2 Method Detection Limit

7.3.2.1 The Detection Limit (DL) shall be determined in accordance with the protocol in 40 CFR 136 Appendix B for determining the MDL. The Practical Quantitation Limit (PQL) shall be established at 10 times of standard deviation of the individual analytical results in the MDL study. The reporting limit (Limit of Quantitation or LOQ) shall be established as the PQL or the lowest calibration level, whichever is higher.

7.3.2.2 After a successful MDL study, the LOQ will be verified as shown in Appendix II.

7.3.3 Calibration

Calibration, initial calibration verification, and continuing calibration verification must meet the criteria shown in Appendix II.

7.3.4 Retention time (RT)

Retention time windows for each analyte, surrogate, and internal standard must be established and evaluated in accordance with the criteria shown in Appendix II. The laboratory must calculate new retention time windows for each standard on each GC column and whenever a new GC column is installed. The data must be retained by the laboratory. Analytes are identified when peaks are observed in the RT window for the compound.

7.3.5 Internal Standard (IS)

7.3.5.1 Purpose: The IS used to normalize sample responses and correct for fluctuations in system sensitivity. The recommended internal standard is C^{13}-hexachlorobenzene.

7.3.5.2 Frequency: IS will be added to all samples, QC samples, and standards at the same concentration.

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7.3.5.3 Evaluation and Corrective Action: Refer to Appendix II for QC requirements.

7.3.6 Surrogate Standard (SS)

7.3.6.1 Purpose: The SS is a compound whose analytical characteristics closely resemble those of the target analytes, but which is unlikely to be found in samples. The suggested surrogate is 4-bromofluorobenzene (BFB). Additional or different surrogates may be used to conform to project specific requirements. The surrogate should be spiked at a level consistent with the mid-point of the instrument calibration range.

7.3.6.2 Frequency: Surrogate is spiked into all samples and QC samples at the same concentration.

7.3.6.3 Evaluation and Corrective Action: Refer to Appendix II for QC requirements.

7.3.7 Laboratory Control Spike (LCS) and Duplicate (LCSD)

7.3.7.1 Purpose: The LCS is used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Results of each sample are compared to established criteria and, if found to be outside these criteria, indicates that the analytical system is "out of control." When the results of the matrix spike analysis indicate a potential problem because of the sample matrix itself, the LCS/LCSD results are used to verify that the laboratory can perform the analysis in a clean matrix.

7.3.7.2 Frequency: One LCS and one LCSD per preparation batch.

7.3.7.3 Composition: It is generally desirable for the matrix to be similar to the matrix being tested, known to be free of analytes of interest. LCS and LCSD samples will be spiked with IS/SS and all target analytes at or below the mid-point of the calibration range.

7.3.7.4 Evaluation and Corrective Action: Refer to Appendix II for all QC requirements. The percent recovery (%R) and relative percent difference (RPD) are calculated for the LCS and LCSD, as follows:

$$\%R_{LCS} = \frac{FC_{LCS}}{TC_{LCS}} \times 100 \qquad \%R_{LCSD} = \frac{FC_{LCSD}}{TC_{LCSD}} \times 100$$

$$RPD = \frac{|FC_{LCS} - FC_{LCSD}|}{\frac{1}{2}(FC_{LCS} + FC_{LCSD})} \times 100$$

Where:

FC_{LCS} = Found concentration in the LCS

FC_{LCSD} = Found concentration in the LCSD

TC_{LCS} = True spike concentration in the LCS

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TC_{LCSD} = True spike concentration in the LCSD

RPD must be calculated as a comparison of measured concentrations, as reported in the sample matrix, not as measured at the instrument, and not a comparison of %R.

7.3.8 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

7.3.8.1 Purpose: The MS is used to indicate the effect of the sample matrix on the precision and accuracy of the results. The information from these controls is sample/matrix specific and not used to determine whether the analytical system is in control or the validity of the entire batch.

7.3.8.2 Frequency: One MS and one MSD per preparation batch.

7.3.8.3 Composition: The MS and MSD must be the same matrix as the samples being analyzed in the batch. Generally, these QC samples will be additional aliquots from one of the unknown samples in the batch. If adequate sample material is not available, the lack of MS/MSD shall be noted in the case narrative. If the native concentration is not known, the samples will be spiked with IS/SS and all target analytes at or below the mid-point of the calibration range. If the native concentration is known, the samples will be spiked with IS/SS and all target analytes at 1 – 4 times the known concentration.

7.3.8.4 Evaluation and Corrective Action: Refer to Appendix II for all QC Requirements. The percent recovery (%R) and relative percent difference (RPD) is calculated for the MS and MSD, as follows:

$$\%R_{MS} = \frac{FC_{MS} - FC_P}{TC_{MS}} \times 100 \qquad \%R_{MSD} = \frac{FC_{MSD} - FC_P}{TC_{MSD}} \times 100$$

$$RPD = \frac{|FC_{MS} - FC_{MSD}|}{\frac{1}{2}(FC_{MS} + FC_{MSD})} \times 100$$

Where:

FC_{MS} = Found concentration in the MS

FC_{MSD} = Found concentration in the MSD

FC_P = Found concentration in the un-spiked parent sample

TC_{MS} = True spike concentration in the MS

TC_{MSD} = True spike concentration in the MSD

RPD must be calculated as a comparison of measured concentrations, as reported in the sample matrix, not as measured at the instrument, and not a comparison of %R.

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8.0 STATEMENT OF THE ANALYTICAL RESULT

The analyst shall provide reports and other deliverables as specified in this section unless superseded in writing by a client-specified format. The required content and form of each deliverable is described in this section. All reports and documentation must be:

- Complete,
- Legible, including handwriting and copies,
- Clearly labeled and completed in accordance with instructions in this section,
- Arranged in the order specified in this section,
- Properly corrected (handwritten corrections must be legible, initialed, and dated), and
- Free of White-out® and Post-It® notes or other items which are not allowed.

Verbal results are provided to indicate the treatment goal for HD has been met. Because the EDS is a continuous operation with fixed tasks to complete, verbal results allow the waste to be drained from the main EDS vessel. Written reports are delivered prior to remove of waste containers from the site.

If samples from more than one client are analyzed in one batch, a separate complete package shall be prepared for each client. Information not related to the client receiving the report shall be removed, including sample designations for the MS/MSD pair in the batch.

If samples from more than one client are analyzed in one batch, all QC results will be copied and included in the package for each client.

The Sample Data Package is divided into the eight major units (see Section 9.3). The Sample Data Package shall include data for the analyses of samples from one client, in one or more batches, including field samples, dilutions, re-analyses, instrument blanks, calibration, ICV, CCV, Method Blank(s), LCS/LCSD, and any requested or required MS/MSD. A blank, colored sheet of paper will separate sections.

In all instances where the data system report has been edited, or where manual integration or quantitation has been performed, the GC/MS system must identify such edits or manual procedures. A hardcopy printout of the EICP of the quantitation ion displaying the manual integration shall be included in the raw data. This applies to all target agents and surrogates.

The analyst shall identify all samples, including dilutions and re-analyses, LCS/LCSD, MS/MSD, Method Blanks, and standards with a unique EML sample number, as recorded on the Extraction Log (MBFORM-62).

For field samples, the EML sample number is assigned when the sample is logged into the Laboratory Information Management System (LIMS), either the Oracle-based Horizon system or the Microsoft Access®-based MUD system. For MS and MSD samples, the EML sample number is the sample number assigned by the data system with –MS or –MSD appended to the end of the number.

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For QC samples, the EML sample number shall be unique for each blank, LCS/LCSD, and LOQ within a sample delivery group or workorder. The unique number is the extraction batch number followed by an appropriate suffix as described below.

The basic format of the extraction batch number is YYMMDDXX, where YYMMDD is the day that sample preparation was performed and XX is the sequential number representing each batch analyzed on that date. The MB, LCS, LCSD, and LOQ number will be the batch number plus the appropriate suffix, as defined below:

YYMMDDXX = extraction batch number

YYMMDDXX-MB = Method blank

YYMMDDXX-LCS = Laboratory control sample

YYMMDDXX-LCSD = Laboratory control sample duplicate

YYMMDDXX-LOQ = LOQ verification sample

For analyses conducted at field or project sites, the extraction batch number is preceded by a 3 or 4 character alphabetic code that indicates the field or project location where the work was conducted. Batches for analyses conducted at the fixed EML laboratory in Edgewood, MD do not have a alphabetic prefix.

9.0 DATA AND RECORDS MANAGEMENT

9.1 Additional Protocols

9.1.1 Refer to IOP MT-97 Control of Records for information regarding the handling of quality and technical records.

9.1.2 Refer to IOP MT-27 Scanning and Archiving of Monitoring Branch Hardcopy and Electronic Data.

9.2 Data Package Format

The following sections describe the order in which the analyst assembles the report. There are differences between the two databases used for Laboratory Information Management. Some items are moved or inserted during technical review or QA review; the final package order will differ slightly from the section contents described below.

9.2.1 Section 1

9.2.1.1 Narrative

9.2.1.2 Data Report – from MUD database. Report inserted by technical reviewer when Horizon® database is used. Not applicable for LOQ verification study when Horizon® database is used.

9.2.1.3 Quality Control Review Report – available only from Horizon® database. Serves as Data Report for LOQ verification study. Except for LOQ verification, moved to Section 8 (immediately before MBFORM-70) after QA review.

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9.2.1.4 Data evaluation spreadsheet – if MDL or LOQ verification study.

9.2.1.5 EML Environmental Analysis Data Review Checklist (MBFORM-123) – moved to Section 8 (immediately after Extraction Log) after QA review.

9.2.1.6 EML Internal Reviewer Signature Page (MBFORM-138) – moved to Section 8 (immediately after MBFORM-123) after QA review.

9.2.1.7 ECBC Instrument Injection Report – chronologically by analysis date if more than one.

9.2.1.8 GC/MS QA-QC Check Report – surrogate/internal standard summary – chronologically by analysis date if more than one.

9.2.1.9 ECBC Method Blank Summary Report– chronologically by analysis date if more than one.

9.2.2 Section 2 – Tune Verification and Calibration

Assemble in chronological order by instrument, grouping the following parts together by instrument.

9.2.2.1 Tune Verification Data. If analysis date differs from calibration date, place tune verification for analysis date in chronological order with tune verification from calibration date last.

9.2.2.2 Initial Calibration Data

9.2.2.2.1 ECBC Calibration Report

9.2.2.2.2 Calibration Plot Report(s)

9.2.2.2.3 ECBC Instrument Injection Report – for day of calibration

9.2.2.2.4 GC/MS QA-QC Check Report – surrogate summary for day of calibration

9.2.2.2.5 Raw Data/Quantitation Reports for instrument blank(s) and calibration standards – no spectra except manual integrations or deletions; chronological order

9.2.2.2.6 Evaluate Continuing Calibration Report for ICV from day calibration analyzed

9.2.2.2.7 Raw Data/Quantitation Report for ICV – no spectra except manual integrations or deletions

9.2.3 Section 3 – Check Standards

In chronological order by instrument, with runs on the same instrument in chronological order.

9.2.3.1 Evaluate Continuing Calibration Report

9.2.3.2 Quantitation Report with Chromatogram – no spectra except for manual integrations or deletions

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9.2.4 Section 4 – MS/MSD

In chronological order by instrument, with runs on the same instrument in chronological order.

9.2.4.1 MS/MSD Evaluation Report – only if results are being reported using the MUD database

9.2.4.2 Quantitation Report with Chromatogram – no spectra except for manual integrations, deletions, or when an analyte is not detected.

9.2.5 Section 5 – Blanks

In chronological order by instrument, with runs on the same instrument in chronological order.

9.2.5.1 Detailed report for each blank

9.2.5.2 Spectra for manual deletions

9.2.6 Section 6 – LCS/LCSD in chronological order by instrument

In chronological order by instrument, with runs on the same instrument in chronological order.

9.2.6.1 LCS/LCSD evaluation report – only if results are being reported using the MUD database

9.2.6.2 Quantitation Report with Chromatogram – no spectra except for manual integrations or deletions

9.2.7 Section 7 – Analyses in EML sample number order irrespective of instrument number or date analyzed

9.2.7.1 Final Quantitation Report with Chromatogram, after manual integration and Q-deleting false positives.

9.2.7.2 Full-page EICP of target analytes for manual integrations and Q-deletions, annotated as specified in Section 6.4.1.

9.2.8 Section 8 – Support Data

9.2.8.1 Chain of Custody Records

9.2.8.2 Correspondence between client and EML concerning samples

9.2.8.3 Sample log-in report from database, after any necessary corrections

9.2.8.4 Calibrated Weights Record Log (MBFORM-119) for every day associated with pipette/dispense performance checks and sample analysis

9.2.8.5 Daily Pipette and Dispenser Performance Check Log (MBFORM-99)

9.2.8.6 Pipette Lot Performance Check (MBFORM-158)

9.2.8.7 Printed Instrument Method

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9.2.8.7.1 Method Control Parameters

9.2.8.7.2 Instrument Control Parameters

9.2.8.7.3 Data Analysis Parameters

9.2.8.8 Extraction Logbook pages

9.2.8.9 Any additional information

9.2.8.10 MBFORM-70 current revision

10.0 KEYWORDS AND DEFINITIONS

Accuracy: Degree of agreement between an individual measurement and an accepted reference value.

Calibration: The set of operations that establish, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system, and the corresponding known values. The result of a calibration is sometimes expressed as a calibration factor, or as a series of calibration factors in the form of a calibration curve.

Calibration Range: The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve.

Coefficient of Determination (r^2): A statistical measure of how well the regression line approximates the real data points. An r^2 of 1 indicates that the regression line perfectly fits the data.

Continuing Calibration Verification: The verification of the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

Correlation Coefficient (r): A statistical measure of the strength and direction of a linear relationship between two variables. The square root of the coefficient of determination (r^2).

Detection Limit (DL): In a specified matrix analyzed by a specified procedure, the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type I error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence. Determined using the protocol for Method Detection Limit as described in 40 CRF 136 Appendix B.

EDS: Explosive Disposal System

Initial Calibration Verification (ICV): Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration.

Instrument Blank: Blank sample used to verify that the instrument is free of contamination.

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Internal Standard (IS): A standard added to samples in known amount and carried through the procedure as a reference for calibration and controlling instrumental and analytical precision and bias. The internal standard is also used to verify instrument response and retention time stability.

Laboratory Control Spike (LCS): A known, clean matrix, similar to the field samples in the batch, spiked with compound(s) representative of the target analyte(s), used to document laboratory performance.

Laboratory Control Spike Duplicate (LCSD): A second sample, prepared identically to the LCS, used to evaluate precision of a method.

Laboratory Information Management Systems (LIMS): The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents.

Limits of Quantitation (LOQ): The smallest concentration that produces a quantitative result with known and recorded precision and bias. The LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.

Matrix: The component or substrate (e.g., neutralent, soil, water) which contains the analyte of interest.

Matrix Spike (MS): An aliquot of sample spiked with a known concentration of target analyte(s). The spiking occurs before sample preparation and analysis. A matrix spike is used to document the bias of a method in a given sample matrix.

Matrix Spike Duplicate (MSD): A second sample, prepared identically to the MS, used to evaluate precision of a method in a given sample matrix.

Method Blank: An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank should be carried through the complete sample preparation and analytical procedure. The method blank is used to document contamination resulting from the analytical process.

Percent Recovery (%R): Recovery of analyte spike added to sample before sample preparation. Determination of recovery is based on results provided by spiked and unspiked samples, used to estimate matrix effects and sample preparation losses.

Precision: The degree of agreement among independent replicate analyses of a sample, usually expressed as standard deviation (or relative percent difference for two measurements).

Quantitation Range: The range of values (concentrations) in a calibration curve between the LOQ and the highest successfully analyzed initial calibration standard. The quantitation range lies within the calibration range.

Relative Percent Difference (RPD): A calculation of the precision from duplicate measurements.

Surrogate: A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for QC purposes.

Target Analytes: Analytes or chemicals of primary concern, identified by the customer on a project-specific basis.

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11.0 REFERENCES

40 CFR 136 Appendix B

Environmental Monitoring Laboratory, Laboratory and Monitoring Quality Assurance/Quality Control Plan for Compliance with ISO 17025 and the DoD Environmental Laboratory Accreditation Program (ELAP)

IOP MT-27 Scanning and Archiving of Monitoring Branch Hardcopy and Electronic Data

IOP MT-28 Environmental Monitoring Laboratory (EML) Quality Document Management

IOP MT-97 Control of Records

SOP CNG-048 Preparation of Working Standards from Chemical Stock Material

12.0 APPENDICES

Appendix I Typical Analytical Sequences

With Initial Calibration
1. Tune Verification
2. Instrument Blank
3. 5-Point Calibration
4. Initial Calibration Verification
5. Method Blank
6. LCS/LCSD
7. MS/MSD and Sample
8. CCV (every 10 samples)
9. Instrument Blank (optional)
10. Ending Calibration Check Standard

Without Initial Calibration
1. Tune Verification
2. Instrument Blank
3. Calibration Verification
4. Method Blank
5. LCS/LCSD
6. MS/MSD and Sample
7. CCV (every 10 samples)
8. Instrument Blank (optional)
9. Ending Calibration Check Standard

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Appendix II Quality Control Requirements and Corrective Actions

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analyst capability	Before using any test method and at any time there is a significant change in instrument type, personnel, or test method.	Criteria prescribed in IOP MT-78.	Recalculate results; locate and fix problem, then rerun demonstration.	N/A	This is a demonstration of analyst ability to generate acceptable accuracy and precision using four replicate analyses of a QC check samples (e.g., LCS) and to prepare a complete, accurate data package. No analysis shall be allowed by analyst until successful demonstration of capability (DoC) is complete.
MDL Study	At initial set-up and after any major change in the analytical system or test method.	See 40 CFR 136 Appendix B. MDL samples must produce a signal at least 3 times the instrument noise level, at a concentration approximately 2 times the reported MDL.	Run MDL verification check at higher level and set MDL higher or re-conduct MDL study.	N/A	Samples cannot be analyzed without a valid MDL.
LOQ Verification	Semi-annually, when EDS projects are being supported, with verification conducted at the EML fixed laboratory	Meet criteria described in IOP MT-78.	Reanalyze. At second failure, repeat verification process at a higher concentration and establish the LOQ at the higher concentration.	N/A	
Tuning	Before calibration and not less frequently than every 12 hours during sample analysis.	Criteria are shown in Section 6.3.1.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Initial Calibration (ICAL): Minimum 5 standards and a calibration blank for all target analytes and surrogate	Before sample analysis, at any major change in the analytical system, or after failing calibration verification.	$r^2 \geq 0.990$ for linear least squares regression.	Correct problem, documenting findings and actions; repeat initial calibration	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second Source Initial Calibration Verification (ICV)	Once after each initial calibration, before sample analysis.	All reported analytes and surrogate within ±25% of true value.	Correct problem, documenting findings and actions; verify second source standards. Rerun ICV. If that fails, correct problem, document findings and actions; repeat initial calibration.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified with a second source.
Establish Retention Time Window	Once per ICAL and at the beginning of the analytical sequence.	Position for target analytes, surrogate, and internal standard set using the midpoint standard of the initial calibration curve on days when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	N/A	N/A	Calculated for each analyte and surrogate.
Evaluation of Retention Times (RT)	With each sample.	RT of target analytes in standards and samples within ± 0.10 minutes of established retention time.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second Source Continuing Calibration Verification (CCV)	Daily, before sample analysis if ICAL not performed. Rerun at least every 10 samples or every 12 hours, whichever is sooner and at the end of the analytical sequence.	All reported analytes and surrogates within ± 25 % of true value for initial and mid-sequence CCV. All reported analytes and surrogates within ± 50 % of true value for end of sequence CCV.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs, within 1 hour of the failure). If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	If reanalysis is not possible, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification	No samples may be analyzed without a passing CCV at the beginning of the analytical sequence.
Internal Standards (IS) Verification	In all samples and standards	Retention time ± 0.1 minutes from retention time of the midpoint standard in the ICAL. EICP area within -50% and +100% of ICAL midpoint standard.	Inspect GCMS for malfunctions and correct problem(s). Re-analysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q flag to analytes in sample with con-compliant IS. Flagging not appropriate for standards.	
Method Blank	One per preparatory batch, matching sample matrix.	No target analyte detected > ½ LOQ.	Correct problem, documenting findings and actions. If blank contamination exceeds 1/10 the level found in any sample, all samples with detectable results will be reprocessed in a subsequent preparation batch.	If no sample remains for reprocessing, apply B-flag to all detectable concentrations in all samples in the preparatory batch.	

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Instrument (or Calibration) Blank	Before beginning a sample run and at the end of the analysis sequence.	Same as method blank.	Correct problem, documenting findings and actions. Re-prep and re-analyze calibration blank and samples since the previous acceptable blank.	Apply B-flag to all results for samples associated with the blank that cannot be re-analyzed.	
LCS/LCSD Containing all target analytes, IS, and SS	One LCS and one LCSD per preparatory batch, matching sample matrix.	Limits established at ± 3σ around the mean %R. For RPD the limit is the 99% confidence limit for a one-sided distribution, based on at least 30 points. Default: 50 – 150 % recovery with RPD ≤ 20%, until sufficient points collected.	For % Recovery: Re-analyze. At second failure, correct problem, documenting findings and actions. Re-extract and re-analyze all samples in a new preparatory batch with new batch QC, if sufficient sample available. For RPD: Evaluate source of difference. Re-analyze, as appropriate.	For % Recovery: If corrective action fails, apply Q-flag to all samples in the associated preparatory batch.	
MS/MSD	One MS and one MSD per preparatory batch, if sufficient sample is available. If sample is known to contain analyte greater than 5 times PQL, a sample duplicate may be used in place of MSD. For known concentration less 5 times the PQL, spike at 1 – 4 times the known concentration.	For matrix evaluation. Acceptance criteria same as LCS/LCSD.	Advisory limits only. Corrective action only as specified by client.	If detectable concentration in parent, apply J-flag if acceptance criteria not met for %R or RPD.	For matrix evaluation only. If MS or MSD results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.

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QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate Spike	All samples, including QC.	Limits established at ± 3σ around the mean %R. For RPD the limit is the 99% confidence limit for a one-sided distribution, based on at least 30 points. Default: 50 – 150 % recovery with RPD ≤ 20%, until sufficient points collected.	Re-analyze failed samples. On second failure, correct problem then re-extract and re-analyze all samples with failed surrogates in the preparatory batch, if sufficient sample material is available. Reanalysis may not be necessary if there is obvious chromatographic interference.	Apply Q-flag to all associated analytes if acceptance criteria are not met. Explain in the report narrative.	Identify an alternate, project-specific surrogate in the event of consistent interference.
Results Reported Between DL and LOQ	N/A	N/A	N/A	Apply J-flag to all results between DL and LOQ.	

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Appendix 2-5

SOP#CSC-193

STANDING OPERATING PROCEDURE

Location: Pueblo Chemical Depot
ECBC, EDS Site

(Check all that apply)
__X_ Chemical Agent
____ Biological/Toxin
____ NTA
____ Radioactive Material, Radiation, and/or Lasers
____ Industrial Chemicals
_X_ Pyrotechnics/Explosives
____ other (specify: ____)

ECBC

DIRECTORATE OF PROGRAM INTEGRATION

Title: Accountability and Transportation of Chemical Munitions and DOT Bottles in Support of the Pueblo Agent-Destruction Pilot Plant Explosives Destruction System Project at Pueblo Chemical Depot

SOP#: CSC-193

Submitted by:

_____
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Approved by:

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Timothy A. Blades, Deputy Director, CB Operations
Directorate of Program Integration

Approval Date: _____

Prepared by: RDCB-DPO-FT/Frank Evans, (410) 436-4260/4372

Draft Final

SOP TITLE: Accountability and Transportation of Chemical Munitions and DOT Bottles in Support of the Pueblo Agent-Destruction Pilot Plant Explosives Destruction System Project at Pueblo Chemical Depot

Operator's Statement: I have read and understand, or have had read to me and understand, the general and specific safety and environmental requirements, personnel and hazardous material limits, work description, and inspection requirements described in this SOP, the Hazard Analysis, and Preparation of Standing Operating Procedures.

Print Name	Signature	Date

Supervisor's Statement: I have read and understand, or have had read to me and understand, the general and specific safety and environmental requirements, personnel and hazardous material limits, work description, and inspection requirements described in this SOP, the Hazard Analysis, and Preparation of Standing Operating Procedures. I have verified that the operators are trained in and understand this SOP, and that the tasks can be executed in a safe and efficient manner.

Supervisor's Name	Signature	Date
Supervisor's Name	Signature	Date

Draft Final

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1.0 STATEMENT OF WORK

This Standing Operating Procedure (SOP) outlines the accountability and transportation of Chemical Munitions and DOT Bottles in support of the Pueblo Agent-Destruction Pilot Plant Explosives Destruction System (EDS) Project at Pueblo Chemical Depot.

2.0 EQUIPMENT, TOOLS, AND SUPPLIES

ITEM:	QUANTITY:
Pallet Jack	1 each
Fire symbol, #1, #2, #3, #4	as required
Chemical Symbol	as required
Gloves, Safety	1 per operator
Safety Glasses	1 per operator
Safety Shoes	1 per operator
Chocks	2 per carrier
Fire Extinguisher 10 BC	2 per carrier
DOT Placards	4 per carrier
Munitions Crate	as required
Dunnage	as required
Plastic sheet, 6mil	as required
Tape	as required
Hammer	as required
Bar, wrecking carpenter	as required
Pliers, common	as required
Forklift, 6000 lb or higher	as required
Pick-up truck, 626 qualified	as required
MSDS, IHC, CA's	as required
PDS Equipment	as required

3.0 GENERAL SAFETY REQUIREMENTS

(1) Whenever an electrical storm approached the installation operational personnel will cease ammunition/explosive operations and evacuate from magazine locations.

(2) Employees will not tamper with any safety devises or protective equipment.

(3) Any defect or unusual condition noted that is not covered by this Standing Operating Procedure (SOP) will be reported immediately to the supervisor.

(4) Appropriate fire symbol and chemical hazard symbol shall be displayed in such a manner as to be easily visible from all roads of approach.

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(5) Care will be taken to limit exposure to a minimum number of personnel for a minimum amount of time. This will minimize exposure to the amount of hazardous material consistent with safe and efficient operations.

(6) Any location containing a fire symbol of “1” shall evacuate immediately when threatened by fire or explosion.

(7) Work locations will be maintained in a neat and orderly condition.

(8) All hand tools shall be maintained in a good state of repair.

(9) Personnel in proximity to steel banding operations will wear a face shield or safety eye wear. Operators handling metal banding will also wear protective gloves.

(10) Operators lifting materials will use proper hand tools, assume proper lifting position, and avoid twisting when lifting or carrying.

(11) Each Material Handling Equipment (MHE) and/or vehicle operator will have in his possession a valid operator’s permit for the particular piece of equipment to be operated.

(12) MHE and other lifting devices will have the load rating and the date of next inspection marked on them In Accordance With (IAW) TB 43-0142, Safety Inspection and Testing of lifting Devices. The load rating will not be exceeded and the equipment will not be used without a current inspection date.

(13) Steel-toed safety shoes will be worn by all personnel engaged in material handling operations.

(14) Protective gloves will be worn when handling wooden boxes or sharp objects.

(15) Chemical munitions and chemically filled Department of Transportation (DOT) bottles shall not be handled roughly. These items will be packaged in DOT approved containers designed to permit dragging, rolling or towing, may be so moved when necessary during handling for storage and transportation.

(16) Any items determined to be dangerous to be handled will be reported immediately to supervisory personnel. Operations will be suspended and if warranted, personnel will be evacuated pending further instruction. Doors of operating buildings will not be bolted or locked when operations are being conducted. Posted personnel and explosive limits must not be exceeded at any time. Transient personnel must comply with the same safety requirements as operating personnel.

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(17) DOT placards will be displayed on vehicles used in transporting explosive donor charges intra-depot.

(18) Compatibility will be maintained IAW DOT regulations when hauling explosives.

(19) Movement of items within the storage igloo (H1102) will be made by manual pallet jacks.

(20) All operators shall have a current Commercial Drivers License with a Hazardous Materials Endorsement, DOT Medical Card and a U.S. Government Motor Vehicle Operators Identification Card.

(21) Appropriate housekeeping must be observed at all times.

(22) All dunnage and packing materials must be removed when not in use.

4.0 HAZARDS INVOLVED

All hazard information is held in the MSDS file LOCATED IN THE Command Post (CP). Information such as toxicity, flammability, and other characteristics must be understood prior to any operations involving those specific chemicals. Additional information may be obtained from the Ammunition Control Coordinator.

4.1. Explosive materials handling.

(1) Handling of explosive materials may require special protective measures to ensure the operators are not unnecessarily subjected to any short term or long term harmful effects. Operators will ensure that the properties of ALL chemicals compounds in the ingredients are fully understood prior to operations.

(2) Explosive hazards could result in mass detonation which could cause body harm or death. Knowledge of ammunition reaction and compatibility can be found in the MSDS or Competent Authority located in the CP trailer.

4.2. Resource Conservation and recovery Act (RCRA) material handling.

Special handling precautions will be required for the handling of RCRA restricted chemicals. RCRA labeled chemicals will be handled in accordance with current MSDS and local and U.S. Army regulations.

4.2.1. Personal Protective Equipment (PPE)

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(1) Mandatory PPE:

a. Eye protection

b. Safety Shoes

c. Ammunition Handlers Coveralls.

(2) Optional additional safety equipment may include, but is not limited to:

a. Gloves appropriate for the materials being handled.

b. Hearing protection.

c. M40A1 Military Protective Mask.

d. Respirators with appropriate filter cartridges as assigned by the ECBC MIH.

4.3. Administrative hazards

(1) The hazards involved with explosive materials are in addition to normal industrial hazards.

(2) Common hand tools present unique hazards to each tool. Basic hand tool operation is mandatory to prevent accidents involving tools such as hammers, screwdrivers, knives, saws, clamps, etc.

5.0 Transportation and Issue Involved in the Movement of Chemical Munitions and Chemically Filled DOT Bottles from Igloo H1102 to the Environmental Enclosure (EE).

5.1. Vehicle and driver preparation.

(1) Inspect motor vehicle against current approved Motor Vehicle Inspection (Transportation of Hazardous Materials Inspection) (DD 626).

(2) Supply two drivers that have a current Commercial Drivers License(CDL) with a hazardous materials endorsement and Department of Transportation(DOT) medical card.

(3) Copy of the Code of Federal Regulation 49 part 397 and Emergency Response Guidebook (ERG).

(4) Means of communication (cell phone).

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(5) DOT placards, fuses, chocks, 2- Inspected 10 lbs-“BC” fire extinguishers.

(6) Route Plan.

(7) DA Form 581, Request for Issue and Turn-in of Ammunition.

(8) DD form 1911, Chain of Custody.

(9) Obtain keys to igloo’s H1102 from ECBC AA&E key custodian.

5.2. Surety Measures

(1) Set up an Emergency Personnel Decontamination Station (EPDS).

(2) Perform first entry monitoring. Reference Site Specific Air Monitoring Plan.

(3) If all clear proceed to next step. If not notify PCD.

5.3. Loading Vehicle.

(1) Notify Security to request access to the storage igloo by deactivating the IDS. This should be completed from the road before the next step.

(2) Back vehicle up to 10’ of the igloo door.

(3) Place vehicle in park, turn engine off, set brakes, chock vehicle.

(4) Open the A&B lock(s) and secure it to the hasp of the igloo. Open the igloo door.

(5) Inspect the area/compartment where the items will be loaded onto the transportation platform for any sharp edges, nails, screws or foreign objects that may penetrate the packaging.

(6) Check DD form 581(Request for Issue and Turn in of Ammunition) for identification of items to be pulled. Annotate the Date, Document number, Action/Purpose, Quantity (Gain/Loss), Balance and Printed Name on the Magazine Data Card (DD 3020-R).

(7) Check Items for compatibility (49CFR174.81).

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(8) Using the proper tools and equipment pull each item to be issued and place them in the munitions crate (MC). Cover the top of the MC with a piece of 6 mil plastic sheet and secure the plastic using duct tape.

Note the MC will have the necessary spill containment needed for transporting the items.

(9) Load the MC on the back of the vehicle and secure using cargo straps and eye hooks provided.

(10) The driver and his assistant will agree to the secure load. The doors of the storage compartment will be shut.

(11) Placards will be placed on all four sides of the vehicle indicating the hazard/s stored inside of the vehicle.

(12) Close the door, remove the locks from the hasp and lock the door.

(13) The driver will pull away from the igloo to the side of the road and call security notifying them that the igloo is secure and to reset the IDS.

5.4. Transporting the Chemical Munitions

(1) The Chemical munitions must be transported via the quickest route and no stops will be permitted unless an emergency occurs.

(2) Speed limit will not exceed 15 miles per hour and vehicle will use the right lane of traffic at all times. Four way flashers and head lamps will be used at all times.

(3) The driver will stop in a designated area outside the EE and call before entry and proceed when only when the EE supervisor/safety gives the okay that it is safe to do so.

5.5. Unloading Vehicle

(1) Driver will position transport vehicle at least ten feet from the EE door, place the vehicle in park, set the brakes, turn off the vehicle and chock the rear tires.

(2) The transportation container will be opened. The load will be checked for damage due to shifting or bouncing during transportation.

(3) All dunnage will be removed and the items will be off loaded from the transportation vehicle and brought into the EE using a forklift and placed

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inside the EE. All pallets and dunnage will be saved for re-use or destroyed/turned-in IAW federal and local regulations. Good housekeeping will be maintained at all times.

(4) An inventory will be completed against the DD581 and DD 1911 and the items received. The DD581 and DD1911 will be signed by both parties. After all items are destroyed an expenditure sheet will be completed by the ECBC Project Manager.

(5) Within three days a copy of both documents will be sent to the ECBC Property Book Officer.

6.0 Transportation and Turn-in of Chemical Munitions Shipments from EE to Chemical Storage Igloo (H1102)

6.1. Vehicle and driver preparation

(1) Inspect motor vehicle against current approved Motor Vehicle Inspection (Transportation of Hazardous Materials) (DD 626).

(2) Supply two drivers that have a current Commercial Drivers License (CDL) with a hazardous materials endorsement and Department of Transportation (DOT) medical card.

(3) Copy of the Code of Federal Regulation 49 part 397 and Emergency Response Guidebook (ERG).

(4) Means of communication (cell phone).

(5) DOT placards, fuses, chocks, 2- Inspected 10 lbs Class “B” fire extinguishers.

6.2. Loading Vehicle

(1) The driver will position the vehicle at least ten foot from the front of the EE door and place the vehicle in park, turn engine off, set brakes, chock vehicle.

(2) The driver and his assistant will inspect the area/compartment where the items will be loaded onto the transportation platform for any sharp edges, nails, screws or foreign objects that may penetrate the packaging

(3) Check DD form 581(Request for Issue and Turn in of Ammunition) and DD 1911 for identification of items to be turn-in.

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(4) Check Items for compatibility (49CFR174.81).

(5) Using the proper tools and equipment pull each item to be issued and place them in the munitions crate (MC). Cover the top of the MC with a piece of 6 mil plastic sheet and secure the plastic using duct tape. Note the MC will have the necessary spill containment needed for transporting the items.

(6) Load the MC on the back of the transportation vehicle and secure using cargo straps and eye hooks provided.

(7) The driver and his assistant will agree to the secure load

(8) Placards will be placed on all four sides of the vehicle indicating the hazard/s stored inside of the vehicle.

(9) The driver and explosive operator will sign the DD581 and DD 1911 for turn-in.

6.3. Transporting the Chemical Munitions

(1) Ammunition must be transported via the quickest route and no stops will be permitted unless an emergency occurs.

(2) The speed limit will not exceed 15 miles per hour and the vehicle will use the right lane of traffic at all times. Four way flashers and head lamps will be used at all times.

6.4. Unloading Vehicle

(1) Driver's assistant will notify Security to request access to the storage igloo by deactivating the IDS. This should be completed from the road before the next step.

(2) Driver will position the back of the truck approximately 10 feet from the storage igloo door, place the vehicle in park, set the brakes, turn off the vehicle and choke the rear tires.

(3) The igloo will be unlocked by the key holders who were issued the A&B keys from the ammunition key custodian. The lock will be secured to the igloo hasp.

(4) The driver and his assistant will access the transportation storage container. The load will be checked for damage due to shifting or bouncing during transportation.

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(5) All dunnage will be removed and the MC will be placed in the chemical munitions igloo (H1102). All pallets and dunnage will be saved for re-use or destroyed/turned-in IAW federal and local regulations. Good housekeeping will be maintained at all times.

(6) Record the Date, Document number, Action/Purpose, Quantity (Gain/Loss), Balance and Printed Name on the Magazine Data Card (DD 3020-R).

(7) An inventory will be completed against the DD581 and DD1911 for the ammunition received. The DD581 and DD 1911 will be signed by both parties.

(8) The authorized key holders will close the igloo door remove the lock from the hasp and secure the igloo.

(9) The driver will pull the vehicle to the side of the road and call security to reset the IDS.

(10) The driver/assistant will call the ECBC Project Manger/Safety to notify them that the ammunition is secured in the igloo.

7.0 EMERGENCY PROCEDURES

In the event of an upset condition or emergency follow the procedures as identified within the PCAPP EDS Destruction Plan, Annex L Contingency Plan.

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APPENDIX A

Pre-Operational Checklist

DATE:_____

HAZARDOUS OPERATION

** ASSURE BUDDY IS PRESENT IN THE ROOM PRIOR TO THE START OF OPERATIONS **

1. PERSONNEL	YES	NO	N/A
a. Medically Cleared to enter area…….....	_____	_____	_____
b. CPRP/BPRP cleared IAW AR 50-6/50-1....	_____	_____	_____
c. Briefing/Orientation			
1. Chemical Agent Safety Briefing…….....	_____	_____	_____
2. Briefing by Supervisor…....…….....	_____	_____	_____
d. Knowledgeable/Aware of:			
1. Emergency procedures…..…….....	_____	_____	_____
2. Agent sign/symptoms…..…….....	_____	_____	_____
3. SOP contents…… …….....	_____	_____	_____
e. Provided with: (specify what type of each and use requirements)			
1. Protective mask/respirator…….....	_____	_____	_____
2. Gloves, boots….....…….....	_____	_____	_____
3. Eye/Ear protection…..…….....	_____	_____	_____

2. DOCUMENTATION:

a. Current SOP available and signed	_____	_____	_____
b. Entrance(s) posted….....…….....	_____	_____	_____
c. Emergency procedures posted/available...	_____	_____	_____

3. DETECTION EQUIPMENT/MONITORING:

a. First entry monitoring completed…….....	_____	_____	_____
b. Detection equipment available…….....	_____	_____	_____

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4. FIRST AID/EMERGENCY EQUIPMENT:

a. Eyewash/Shower facilities operable and inspected……...…. _____ _____ _____
b. Medical support verified…… …….. _____ _____ _____
c. First Aid kit available and contents current _____ _____ _____
d. Emergency contact numbers posted…….. _____ _____ _____

5. GENERAL:

a. Equipment:
   1. No Spark producing material..……..... _____ _____ _____

Primary Operator: _____

Secondary Operator: _____

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APPENDIX B

Hazard Analysis

of

Accountability and Transportation of Chemical Munitions and DOT Bottles in Support of the Pueblo Agent-Destruction Pilot Plant Explosives Destruction System Project at Pueblo Chemical Depot

This Hazard Analysis was developed by the following:

Names, Titles

Jarad Tucker, Safety Specialist

Frank Evans, Chief Field Technology Branch

The pre-operational survey was performed on: TBD

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CATEGORY	WHAT IF	CONSEQUENCE	CONTROLS	FINAL RAC	COMMENTS
Slip/Trip/Fall	Walking on uneven surfaces	Musculoskeletal injury form Slip/Trip/Fall	ENG: ADM: Keep area clear of debris. PPE: Sturdy footwear	IVD LOW	
Explosives	Enter area with explosive material	- Potential exposure to explosive material - unintentional detonation of explosive material	ADM: Personnel will not handle explosives. - Personnel will not be permitted to carry flame producing material.	IE MED	
Hot and Cold Conditions	Excessive heat or cold conditions are present	Personnel potentially affected by heat or cold stress	ADM: Personnel will limit time in extreme temperature conditions. - Personnel will follow established guidelines for heat and cold stress PPE: Insulated clothing during cold temperatures.	IVD LOW	
Cuts /Abrasions	Handling sharp materials	Personnel cut hands	ENG/ADM: Sharp edges will be reported	IVD LOW	

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CATEGORY	WHAT IF	CONSEQUENCE	CONTROLS	FINAL RAC	COMMENTS
			for reductions. PPE: Gloves will be worn when handling potentially sharp items.		
Chemical Agent	Personnel enter storage area with Chemical Agent	Potentially exposed to chemical agent	ENG: ventilation/filtration controls running ADM: Monitoring will be operational. Clearance required before proceeding, two person rule while agent is present. PPE: Mask slung, boots/gloves	IIE LOW	
Material Handling	Operator drops Munition Crate	Injury to personnel – mechanical impact.	ADM: Utilize MHE as appropriate. PPE: Wear PPE as described.	IIID LOW	
Material Handling	Operator drops Munition Crate	Injury to personnel – chemical exposure	ADM: Utilize MHE as appropriate.	II D LOW	

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CATEGORY	WHAT IF	CONSEQUENCE	CONTROLS	FINAL RAC	COMMENTS
			Training on proper emergency procedures. PPE: Wear PPE as described.		
Material Handling	Operator drops Munition Crate.	Spill	ADM: Utilize MHE as appropriate. Have spill kit readily available.	IVD LOW	
Material Handling	Operator drops Munition Crate	Injury to personnel - Unintentional detonation	ENG: Munition Crate designed specifically for transporting material ADM: Utilize MHE as appropriate. Training on proper emergency procedures.	IE MED	
Struck By/Human Error	Vehicle Accident	Injury to personnel.	ENG: Load properly secured prior to movement.	IIIE LOW	

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CATEGORY	WHAT IF	CONSEQUENCE	CONTROLS	FINAL RAC	COMMENTS
			ADM: Licensed drivers. Placard vehicles. Limited driving speed. PPE: Seatbelts to be worn.		
Struck By/Human Error	Vehicle Accident	Spill	ENG: Load properly secured prior to movement. ADM: Licensed drivers. Placard vehicles. Limited driving speed. Spill kit readily available. Speed limit not to exceed 15 mph Shortest safe route utilized	IIIE LOW	
Weather and Environment	Severe weather occurs during transfer	Injury to personnel.	ENG: Load properly secured prior to ADM: Operation unable to start or discontinued immediately if notified of	IIE LOW	

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CATEGORY	WHAT IF	CONSEQUENCE	CONTROLS	FINAL RAC	COMMENTS
			severe weather.		
Weather and Environment	Severe weather occurs during transfer	Spill	ADM: Operation unable to start or discontinued immediately if notified of severe weather.	IIE LOW	

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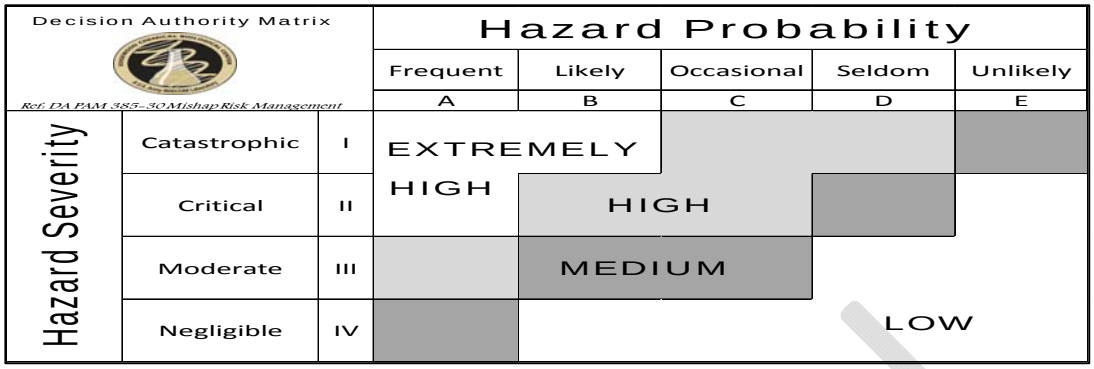

Hazard Probability – *per exposure interval*		
Probability	**Symbol**	**Definition**
Frequent	A	Occurs very often; known to happen regularly.
Likely	B	Occurs several times; a common occurrence.
Occasional	C	Occurs sporadically, but is not uncommon.
Seldom	D	Remotely possible; could occur at some time.
Unlikely	E	Can assume will not occur but not impossible.

Hazard Severity – *per given hazard scenario or mishap*			
Severity	**Symbol**	**Definition**	**ECBC Examples**
Catastrophic	I	Death or permanent total disability (accident risk). Loss of major or mission-critical system or equipment. Major property (facility) damage. Unacceptable collateral damage. Loss of ability to accomplish the mission or mission failure. Irreversible environmental damage. Quantity: Loss > $1M	- Lethal Dose (chem/bio) - Electrocution - Asphyxiation
Critical	II	Permanent partial disability, temporary total disability exceeding 3 months time (accident risk). Extensive (major) damage to equipment or systems. Significant damage to property. Significant collateral damage. Significantly (severely) degraded mission capability or unit readiness. Reversible environmental damage. Quantity: Loss > $200K but <1M	- Exposure to < lethal dose of chem / bio. - Amputation - Hearing Loss
Marginal	III	Minor damage to equipment or systems, or property. Lost days due to injury or illness not exceeding 3 months (accident risk). Degraded mission capability or unit readiness. - Mitigable environmental damage. Quantity: Loss > $20K but < $200K	- Broken bones / sprain - Severe Laceration - Severe strains
Negligible	IV	First aid or minor medical treatment (accident risk). Slight equipment or system damage, but fully functional and serviceable. Little or no property damage. Little or no adverse impact on mission capability. Quantity: Loss < $20K	- Minor cuts - Bruises

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Appendix 2-7

SOP#CSC-194

STANDING OPERATING PROCEDURE

Location: Pueblo Chemical Depot

Igloo #: H1103

(check all that apply)
_____Chemical Agent
_____Biological/Toxin
_____ NTA
_____Radioactive Material, Radiation, and/or Lasers
__X__ Industrial Chemicals
_____Pyrotechnics/Explosives
_____ other (specify:)

DIRECTORATE OF PROGRAM INTEGRATION

ECBC

Title: Accountability and Transportation of Hazardous Waste in Support of the Pueblo Agent-Destruction Pilot Plant Explosives Destruction System Project at Pueblo Chemical Depot

SOP#: CSC-194

Submitted by:

_____
Dennis G. Hall, Chief, Applications Integration Branch

Environmental Quality Office: _____
Gerald Starnes

Safety and Health Office: _____
D. Todd Nay

Approved by:

_____
Timothy A. Blades, Deputy Director, CB Operations
Directorate of Program Integration

APPROVAL DATE: _____

Prepared by: RDCB-DPO-A/Dennis G. Hall, (410) 436-2393

Draft Final

Title: Accountability and Transportation of Hazardous Waste in Support of the Pueblo Agent-Destruction Pilot Plant Explosives Destruction System Project at Pueblo Chemical Depot

Operators Statement:

I have read and understand, or have had read to me and understand, the general and specific safety and environmental requirements, personnel and hazardous material limits, work description, and inspection requirements described in this SOP, the Hazard Analysis, and Preparation of Standing Operating Procedures.

Print Name	Signature	Date

Supervisors Statement:

I have read and understand, or have had read to me and understand, the general and specific safety and environmental requirements, personnel and hazardous material limits, work description, and inspection requirements described in this SOP, the Hazard Analysis, and Preparation of Standing Operating Procedures. I have verified that the operators are trained in and understand this SOP, and that the tasks can be executed in a safe and efficient manner.

Supervisor's Name	Signature	Date
Supervisor's Name	Signature	Date

Draft Final

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5.0 TRANSPORTATION AND RECEIPT HAZARDOUS WASTE SHIPMENT FROM THE PUEBLO AGENT-DESTRUCTION PILOT PLANT (PCAPP) EXPLOSIVE DESTRUCTION SYSTEM (EDS) ENVIRONMENTAL ENCLOSURE (EE) TO IGLOO H1103..... 7

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1.0 STATEMENT OF WORK

This Standing Operating Procedure outlines the Edgewood Chemical Biological Centers (ECBC) accountability and transportation of Hazardous Waste in support of the Pueblo Agent-Destruction Pilot Plant Explosives Destruction System (EDS) project at Pueblo Chemical Depot.

2.0 EQUIPMENT, TOOLS, AND SUPPLIES

ITEM:	QUANITY:
Pallet Jack	1 each
Fire symbol	#1, #2, #3, #4 as required
Chemical Symbol	as required
Chocks	2 per carrier
Fire Extinguisher 10 BC	2 per carrier
DOT Placards	4 per carrier
Dunnage	as required
Plastic sheet, 6mil	as required
Tape	as required
Hammer	as required
Bar, wrecking carpenter	as required
Pliers, common	as required
Forklift, 6000 lb or higher	as required
Vehicle, 626 qualified	as required
MSDS, IHC, CA's	as required
Universal Drum, 95 Gallon	as required
Pad, sorbent (15" X 19")	110 each
Sorbent socks (3" X 4')	12 each
Sorbent socks (3" X 12'')	8 each
Sorbent pillows (18" X 18")	8 each
Disposable bags	10 each
Nitrile gloves	as required
Goggles	as required
Emergency handbook	as required
Shovel plastic	as required
Spill Pallet	as required

3.0 SAFETY REQUIREMENTS

General Safety Requirements

(1) Whenever an electrical storm approaches the installation, operational personnel will cease hazardous waste operations and evacuate from magazine locations.

(2) Employees will not tamper with any safety devises or protective equipment.

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(3) Any defect or unusual condition noted that is not covered by this SOP will be reported immediately to the supervisor.

(4) Appropriate fire symbol, Department of Transportation (DOT) symbol or chemical hazard symbol shall be displayed in such a manner as to be easily visible from all roads of approach.

(5) Care will be taken to limit exposure to a minimum number of personnel for a minimum amount of time. This will minimize exposure to the amount of hazardous material consistent with safe and efficient operations.

(6) Any location containing a fire symbol of “1” shall evacuate immediately when threatened by fire or explosion.

(7) Work locations will be maintained in a neat and orderly condition.

(8) All hand tools shall be maintained in a good state of repair.

(9) Personnel in proximity to steel banding operations will wear a face shield or safety eye wear. Operators handling metal banding will also wear protective gloves.

(10) Operators lifting materials will use proper hand tools, assume proper lifting position, and avoid twisting when lifting or carrying.

(11) Each Material Handling Equipment (MHE) and/or vehicle operator will have in his possession a valid operator’s permit for the particular piece of equipment to be operated.

(12) MHE and other lifting devices will have the load rating and the date of next inspection marked on them IAW TB 43-0142, Safety Inspection and Testing of lifting Devices. The load rating will not be exceeded and the equipment will not be used without a current inspection date.

(13) Steel-toed safety shoes will be worn by all personnel engaged in material handling operations.

(14) Protective gloves will be worn when handling wooden boxes or sharp objects.

(15) Hazardous waste containers shall not be handled roughly. Large items, packaged in DOT approved containers designed to permit dragging, rolling or towing, may be so moved when necessary during handling for storage and transportation.

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(16) Any hazardous waste containers determined to be too dangerous to be handled will be reported immediately to supervisory personnel. Operations will be suspended and if warranted, personnel will be evacuated pending further instruction. Doors of operating buildings will not be bolted or locked when operations are being conducted. Transient personnel must comply with the same safety requirements as operating personnel.

(17) Department of Transportation placards will be displayed on vehicles used in transporting hazardous waste intra-depot if required.

(18) Compatibility will be maintained IAW DOT regulations when hauling ammunition.

(19) Igloo movements will be made with appropriate material handling equipment.

(20) All operators shall have a current Commercial Drivers License with a Hazardous Materials Endorsement, DOT Medical Card and a U.S. Government Motor Vehicle Operators Identification Card.

(21) Appropriate housekeeping must be observed at all times.

(22) All dunnage and packing materials must be removed when not in use.

4.0 HAZARDS INVOLVED

All hazard information is held in the MSDS file LOCATED IN THE Command Post (CP). Information such as toxicity, flammability, and other characteristics must be understood prior to any operations involving those specific chemicals. Additional information may be obtained from the Site Supervisor.

4.1. Resource Conservation and recovery Act (RCRA) material handling.

Note: All waste containers will be labeled IAW regulations and PCD's hazardous waste permit.

(1) Handling of hazardous waste may require special protective measures to ensure the operators are not unnecessarily subjected to any short term or long term harmful effects. Operators will ensure that the properties of ALL chemicals compounds in the ingredients are fully understood prior to operations.

(2) Hazardous wastecontainers could result in a release which could cause body harm or an environmental impact. Knowledge of ammunition reaction and compatibility can be found in the MSDS or Competent Authority located in the CP trailer.

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(3) Special handling precautions will be required for the handling of RCRA hazardous waste . RCRA hazardous waste labeled chemicals will be handled in accordance with current MSDS and local and U.S. Army regulations.

4.2. Personal Protective Equipment.

(1) Mandatory Personal Protective Equipment (PPE):

a. Eye protection.

b. Safety Shoes.

c. Ammunition Handlers Coveralls.

(2) Optional additional safety equipment may include, but is not limited to:

a. Gloves appropriate for the materials being handled.

b. Hearing protection.

c. M40A1 Military Protective Mask.

d. Respirators with appropriate filter cartridges as assigned by the ECBC Site Safety and Health Officer.

5.0 TRANSPORTATION AND RECEIPT HAZARDOUS WASTE SHIPMENT FROM THE PUEBLO AGENT-DESTRUCTION PILOT PLANT (PCAPP) EXPLOSIVE DESTRUCTION SYSTEM (EDS) ENVIRONMENTAL ENCLOSURE (EE) TO IGLOO H1103.

5.1. Vehicle and driver preparation.

(1) Inspect motor vehicle against current approved Motor Vehicle Inspection (Transportation of Hazardous Materials Inspection) (DD 626).

(2) Supply two drivers that have a current Commercial Drivers License (CDL) with a hazardous materials endorsement and Department of Transportation(DOT) medical card.

(3) Copy of the Code of Federal Regulation 49 part 397 and Emergency Response Guidebook (ERG).

(4) Means of communication (cell phone).

(5) DOT placards, fuses, chocks, 2 - Inspected 10 lbs-“BC” fire extinguishers.

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(6) Route Plan.

(7) Obtain keys to igloo's H1102 from ECBC AA&E key custodian.

5.2. Loading Vehicle.

(1) The driver will position the vehicle at least ten feet from the front of the EE door and place the vehicle in park, turn engine off, set brakes, chock vehicle.

(2) The driver and his assistant will open the rear door of vehicle and inspect inside of the storage compartment for any sharp edges, nails, screws, or foreign objects that may penetrate the packaging.

(3) Check DD form 1911(Material Courier Receipt) for identification of items to be collected.

(4) Check Items for leaks and compatibility (49CFR174.81).

(5) Using the proper tools and equipment load the item on the back of the vehicle and secure using cargo straps and eye hooks provided.

(6) The driver and his assistant will agree to the secure load. The doors of the storage compartment will be shut.

(7) Placards will be placed on all four sides of the vehicle indicating the hazard/s stored inside of the vehicle.

(8) The driver or assistant will sign the DD1911 for change of custody.

5.3. Transporting the Hazardous Waste.

(1) The hazardous waste must be transported via the quickest route and no stops will be permitted unless an emergency occurs.

(2) The speed limit will not exceed 15 miles per hour and the vehicle will use the right lane of traffic at all times. Four way flashers and head lamps will be used at all times.

5.4. Unloading Vehicle.

(1) Drivers assistant will notify JPM-E (P) HW Contractor to request access to igloo H1103. This should be completed from the road before the next step.

(2) Driver will position the back of the truck approximately 10 feet from the igloo door, place the vehicle in park, set the brakes, turn off the vehicle, and choke the rear tires.

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(3) The driver and his assistant will open the door of the storage compartment. The load will be checked for damage due to shifting or bouncing during transportation.

(4) All dunnage will be removed and the waste will be placed in the hazardous waste igloo (H1103). All pallets and dunnage will be saved for re-use or destroyed/turned-in IAW federal and local regulations. Good housekeeping will be maintained at all times.

(5) The hazardous waste will be turned over to the JPM-E (P) HW Contractor.

(6) The JPM-E (P) HW Contractor will sign the DD 1911 for change of custody of the hazardous waste

(7) The driver/assistant will call the ECBC Project Manger/Safety to notify them that the hazardous waste is secured in the igloo.

6.0 EMERGENCY PROCEDURES

In the event of an upset condition or emergency follow the procedures as identified within the PCAPP EDS Destruction Plan, Annex L Contingency Plan.

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APPENDIX A

Pre-operational Checklist

DATE:_____

HAZARDOUS OPERATION

	YES	NO
a. Medically Cleared….....	_____	_____
b. CPRP/ cleared IAW AR 50-6.....	_____	_____
c. Daily Safety Briefing…….....	_____	_____
d. Knowledgeable/Aware of:		
1. Emergency procedures…..…….....	_____	_____
2. Agent sign/symptoms…..…….....	_____	_____
3. SOP contents……..…….....	_____	_____
e. Provided with:		
1. Protective mask/respirator…….....	_____	_____
2. Coat, coveralls, apron…..…….....	_____	_____
3. Gloves, boots….....…….....	_____	_____
4. Eye/Ear protection…..…….....	_____	_____
5. Protective suit/ensemble. . ……. . . .	_____	_____
f. Current SOP available and signed	_____	_____
g. MSDS/SDS/HHDWS for chemicals used…	_____	_____
h. Spill kit available……	_____	_____
i. Eyewash/Shower facilities operable and inspected…….....	_____	_____
j. Additional Equipment needed…….....	_____	_____
k. Medical support verified…… …….	_____	_____
l. First Aid kit available and contents current	_____	_____
m. Emergency contact numbers posted……....	_____	_____
n. Waste packaged/labeled.....…….....	_____	_____

Primary Operator: _____

Secondary Operator: _____

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APPENDIX B

HAZARD ANALYSIS

OF

ACCOUNTABILITY AND TRANSPORTATION OF HAZARDOUS WASTE IN SUPPORT OF THE PUEBLO AGENT-DESTRUCTION PILOT PLANT EXPLOSIVES DESTRUCTION SYSTEM PROJECT AT PUEBLO CHEMICAL DEPOT

This Hazard Analysis was developed by the following:

D. Todd Nay, Safety and Health Office

Dennis Hall, Chief, applications Integration Branch

The pre-operational survey was performed on: TBD

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CATEGORY	WHAT IF	CONSEQUENCE	CONTROLS	FINAL RAC	COMMENTS
Material Handling	Operator drops hazardous waste container.	Injury to personnel – mechanical impact.	ADM: Utilize MHE as appropriate. PPE: Wear PPE as described.	IIID LOW	
Material Handling	Operator drops hazardous waste container.	Injury to personnel – chemical exposure	ADM: Utilize MHE as appropriate. Training on proper emergency procedures. PPE: Wear PPE as described.	IVD LOW	
Material Handling	Operator drops hazardous waste container.	Spill	ADM: Utilize MHE as appropriate. Have spill kit readily available.	IVD LOW	
Material Handling	Hazardous waste container exceeds safe lifting weight.	Injury to personnel.	ADM: Utilize MHE as appropriate.	IIID LOW	
Struck By/Human Error	Vehicle Accident	Injury to personnel.	ENG: Load properly secured prior to movement. ADM: Licensed drivers.	IIIE LOW	

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CATEGORY	WHAT IF	CONSEQUENCE	CONTROLS	FINAL RAC	COMMENTS
			Placard vehicles. Limited driving speed. PPE: Seatbelts to be worn.		
Struck By/Human Error	Vehicle Accident	Spill	ENG: Load properly secured prior to movement. ADM: Licensed drivers. Placard vehicles. Limited driving speed. Spill kit readily available.	IIIE LOW	
Weather and Environment	Severe weather occurs during transfer	Injury to personnel.	ENG: Load properly secured prior to ADM: Operation unable to start or discontinued immediately if notified of severe weather.	IIE LOW	
Weather and Environment	Severe weather occurs during transfer	Spill	ADM: Operation unable to start or discontinued immediately if notified of severe weather.	IIE LOW	

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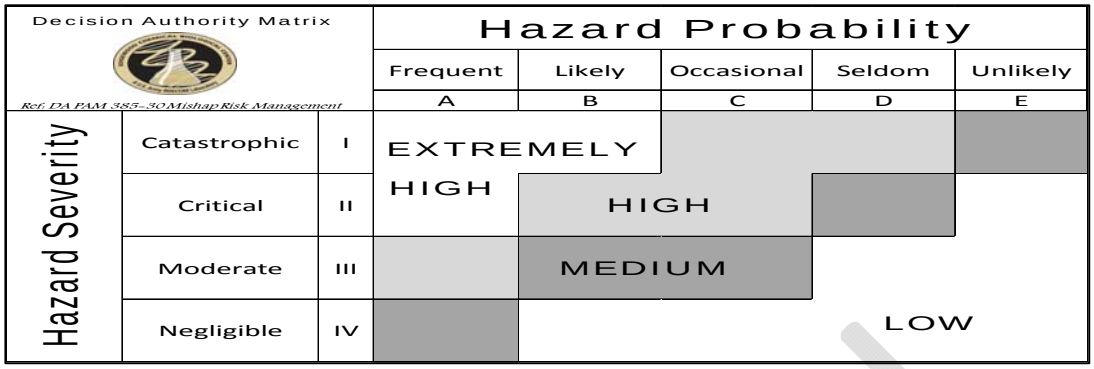

Hazard Probability – *per exposure interval*		
Probability	**Symbol**	**Definition**
Frequent	A	Occurs very often; known to happen regularly.
Likely	B	Occurs several times; a common occurrence.
Occasional	C	Occurs sporadically, but is not uncommon.
Seldom	D	Remotely possible; could occur at some time.
Unlikely	E	Can assume will not occur but not impossible.

Hazard Severity – *per given hazard scenario or mishap*			
Severity	**Symbol**	**Definition**	**ECBC Examples**
Catastrophic	I	Death or permanent total disability (accident risk). Loss of major or mission-critical system or equipment. Major property (facility) damage. Unacceptable collateral damage. Loss of ability to accomplish the mission or mission failure. Irreversible environmental damage. Quantity: Loss > $1M	- Lethal Dose (chem/bio) - Electrocution - Asphyxiation
Critical	II	Permanent partial disability, temporary total disability exceeding 3 months time (accident risk). Extensive (major) damage to equipment or systems. Significant damage to property. Significant collateral damage. Significantly (severely) degraded mission capability or unit readiness. Reversible environmental damage. Quantity: Loss > $200K but <1M	- Exposure to < lethal dose of chem / bio. - Amputation - Hearing Loss
Marginal	III	Minor damage to equipment or systems, or property. Lost days due to injury or illness not exceeding 3 months (accident risk). Degraded mission capability or unit readiness. - Mitigable environmental damage. Quantity: Loss > $20K but < $200K	- Broken bones / sprain - Severe Laceration - Severe strains
Negligible	IV	First aid or minor medical treatment (accident risk). Slight equipment or system damage, but fully functional and serviceable. Little or no property damage. Little or no adverse impact on mission capability. Quantity: Loss < $20K	- Minor cuts - Bruises

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