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April 6, 1994

LA-SUB--95-146

University of California
Los Alamos National Laboratory
Attention: Dr. William Earl
P.O. Box 1663, Mail Stop G740
Los Alamos, New Mexico 87545

ACCOUNT # 9-XZ2-9020F-1

Dear Dr. Earl:

RE: FINAL REPORT FOR ANALYTICAL SUPPORT OF D + D OPERATIONS

Battelle is pleased to enclose a copy of the Final Report of **Chemical Surety Materiel Decontamination and Decommissioning of Los Alamos National Laboratory Chemical Surety Laboratory Area TA-3, Building SM-29, Room 4009** for work performed under the above mentioned account number.

If you have any questions or comments regarding this report, please do not hesitate to contact me at (614) 424-7956.

Sincerely,

A handwritten signature in black ink, appearing to read "Timothy E. Moore", with a horizontal line extending to the right.

Timothy E. Moore
Program Manager
Hazardous Materials Research Facility

TEM:fc
Enclosure

MASTER

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REPORT

FINAL REPORT

Chemical Surety Materiel

Decontamination and

Decommissioning of Los

Alamos National Laboratory

Chemical Surety Materiel

Laboratory Area TA-3,

Building SM-29, Room 4009

To

University of California

Los Alamos National Laboratory

April 1994

FINAL REPORT

Subcontract Number 9-XZ2-9020F-1

for

**CHEMICAL SURETY MATERIEL DECONTAMINATION AND DECOMMISSIONING OF
LOS ALAMOS NATIONAL LABORATORY CHEMICAL SURETY MATERIEL LABORATORY
AREA TA-3, BUILDING SM-29, ROOM 4009**

to

**UNIVERSITY OF CALIFORNIA
LOS ALAMOS NATIONAL LABORATORY**

APRIL 1994

by

T.E.MOORE and J.M.SMITH

**BATTELLE
Chemical/Biological Defense Department
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1.0 Introduction

The work described in this document was authorized by Los Alamos National Laboratory under subcontract number 9-XZ2-9020F-1.

1.1 Background

From 1982 through 1987, Los Alamos National Laboratory (LANL) performed surety laboratory operations for the U.S. Army Medical Research and Development Command (MRDC). Room 4009 in building SM-29, TA-3, was used as the laboratory for work with the following chemical surety materiel (CSM) agents: sarin (GB), soman (GD), lewisite (L), and distilled mustard (HD) radio-labelled with H^3 or C^{14} . The work was confined to three CSM-certified fume hoods, located in room 4009 (see diagram in Appendix C). The laboratory ceased all active operations during the late 1986 and early 1987 period. From 1987 until 1993 the laboratory was secured and the ventilation system continued to operate. During late 1992, the decision was made to utilize this laboratory space for other operations, thus a decision was made to dismantle and reconfigure this room. LANL sub-contracted Battelle Memorial Institute (BMI) to draw upon the CSM experience of the technical staff from the Hazardous Materials Research Facility (HMRF) to assist in developing a decontamination and decommissioning plan. BMI was subcontracted to devise a CSM safety training course, and a sampling and air monitoring plan for CSM materiel to ensure personnel safety during all disassembly operations. LANL subcontracted Johnson Controls personnel to perform all disassembly operations.

Beginning in early 1993 BMI personnel from the HMRF visited the laboratory to develop both the safety plan and the sample and air monitoring plan. Execution of that plan began in September 1993 and was completed in January 1994.

1.2 Objective

The objective of this work was three fold: (1) To provide near-real-time (NRT) monitoring operations to detect distilled mustard (HD) agent in support of the disassembly crews to alert them to any potential exposure to HD, (2) sample all laboratory residues for the presence of agent off-gassing above the XXX level prior to release for final disposal and (3) develop a CSM safety training course.

The original plan specified for the air monitoring and sampling operations to be conducted with a MINICAMS air monitoring system. The MINICAMS system is a short column (3 meter) gas chromatograph (GC) equipped with a flame photometric detector (FPD) with a sulfur window for the detection of HD. The system automatically draws a representative air sample every 3 to 5 minutes through a heated sampling line. This system was selected for its ease-of-use, accuracy and availability at the time of the operation. The plan also specified that agent calibration standards for the MINICAMS would be obtained by LANL from the Edgewood Research, Development, and Engineering Center (ERDEC) prior to the start of operations. Due to the fact that LANL was not a currently certified surety laboratory, the ERDEC surety office declined to release agent calibration standards to LANL for use on this program. When ERDEC declined to release agent calibration standards, Battelle and LANL technical staff jointly decided to utilize a modified two step sampling plan: (1) Conduct NRT monitoring operations with a MINICAMS calibrated with commercially available "half mustard" solution (2-chloroethyl ethyl sulfide) which is a close analog for HD, for personnel safety and (2) perform waste residue Proof of Decontamination (POD) sampling with Tenax-TA sorbent tubes shipped to the HMRF for analysis.

Prior to approval of this modification three (3) points of concern had to be addressed. (1) the possibility of radioactive materials leaving LANL control, (2) the possibility of agent offgassing during shipment, and (3) chain-of-custody for each sample so that any contamination could be traced back to its origin. These issues were resolved jointly by BMI and LANL technical staff with the changes reflected in the sampling plan.

1.3 Sampling Plan

To reduce any possibility of unintentional release of radioactive materials trapped within the Tenax-TA media, LANL technical staff devised a method of flushing the interior of the sampling tubes and screening the rinsate for radioactive isotopes. This screening operation was performed on one of the three triplicate samples collected at each specified sampling point within the laboratory. Additionally, LANL technical personnel performed a detailed screen of the laboratory for the presence of radioactive particles utilizing swipe tests prior to BMI personnel arriving on site. This preliminary screen indicated no radioactive contamination above action levels present in the laboratory.

To address the issue of offgassing of agent during shipment, the tubes were individually packed in polyethylene bags to prevent any cumulative offgas hazard occurring during the shipment. Additionally, the tubes were capped at each end with plastic caps and the shipping containers were sealed with chain-of-custody seals. (Note: Tenax-TA media must be heated to above 200°C to facilitate agent offgassing, therefore, both LANL and HMRF personnel felt confident that the potential for offgassing conditions occurring during shipment were remote). Lastly, HMRF analytical personnel were directed to conduct all package opening and inventory operations in a manner consistent with HMRF dilute solution (XDS) guidelines and SOPs.

To ensure proper chain-of-custody (COC) of the samples, HMRF QA personnel modified existing Department of Energy (DOE) specifications in the documentation and record keeping associated with each sample set. The guidelines for all QA/QC and COC measures are contained in the document management plan in Appendix E.

2.0 Program Plan

The support provided by Battelle during this program consisted of the following: (1) on-site safety training for personnel unfamiliar with CSM, conducted by Battelle safety personnel, (2) on-site baseline air sampling with both MINICAMS and Tenax-TA tubes, (3) on-site air monitoring during the disassembly phase and (4) off-site analysis of Tenax-TA tubes at the HMRF.

2.1 Training

Battelle safety personnel conducted re-fresher CSM safety training for all involved HMRF technical staff prior to the commencement of on-site sampling operations. Additionally, Battelle safety personnel conducted on-site CSM safety training for all personnel not familiar with CSM and who would be involved in the disassembly operations. This CSM training addressed the physical indications and symptoms of agent exposure, to include immediate self aid/first aid, as well as, self and buddy decontamination procedures. Battelle safety personnel required all personnel to participate in hands-on practice with the Mark I nerve agent antidote kit. Also, material safety data sheets (MSDS) for each agent were reviewed by all participants and copies were kept on-site.

In addition to the CSM specific training, all personnel involved in the operation were required to attend a forty hour hazardous waste operations and emergency response (HAZWOPER) course, as per the Code of Federal Regulations (CFR), Section 1910.120. The forty hour HAZWOPER training was conducted by an outside training firm. Documentation of successful HAZWOPER training was provided to LANL. A mandatory 3-day on-site training period was conducted by LANL technical staff to ensure that all site-specific safety issues were addressed and noted by the visiting BMI technical staff. Additionally, all BMI personnel participated in a LANL sponsored radiological safety and awareness training course prior to entering the laboratory.

2.2 On-Site Baseline Air Sampling

Prior to the start of operations, LANL and BMI technical staff conducted a historical assessment of the types and quantities of agents previously used and stored in the laboratory. This

assessment involved a review of the historical testing records to determine which agents would most likely be encountered during the disassembly operations. This assessment aided in the determination for potential of personnel exposure to CSM materials. This assessment considered the physical properties of the agents (e.g. volatility persistancy) and the locations within the laboratory where they were used. The results of the assessment led the technical staff to conclude that the agent contamination, if present, would be limited to the fume hoods and the ventilation ductwork of the laboratory. The assessment also concluded that the CSM agents which might be encountered were: HD, L and G-series nerve agents. Of the three agents, HD was determined to be the most likely to be encountered. BMI personnel performed all baseline monitoring operations for the HD and G-series nerve agents. The monitoring operations utilized both MINICAMS and Tenax-TA sampling tubes. LANL technical personnel performed the sampling and analysis for Lewisite.

The sampling and analysis for Lewisite were performed as per the Battelle furnished analytical standard operation procedure (SOP) for L collection and analysis (see Appendix F).

In order to successfully calibrate the MINICAMS a close analog of HD was needed. This analog would need to provide a sulfur component in order to mimic HD for detection purposes. (NOTE: the MINICAMS screens for the presence of sulfur in the HD detection mode). Both LANL and BMI technical staff concluded that the commercially available "half-mustard" (2-chloroethyl ethyl sulfide) would be suitable for the purposes of calibration of the MINICAMS.

The MINICAMS is a near real time (NRT) monitoring platform based upon gas chromatography (GC) technology. It is, in effect, a short column (3 meter) GC. The unit is designed to sample and analyze an air sample every 3 to 5 minutes. To monitor for the presence of HD, the unit is equipped with a sulfur window detector. In essence, the unit searches each sample for the presence of a sulfur "peak". Although the system is subject to "false positive" readings, it is a proven safety device which is utilized by Army Explosive Ordnance Destruction (EOD), and Technical Escort Units (TEU), as well as, by most CSM contractor operated laboratories throughout the United States.

Due to the fact that actual CSM agent was not available for calibration, a determination was made that a second form of sample collection and analysis would be needed for final POD analysis of all laboratory residues for final POD. LANL and Battelle technical personnel selected TENAX-TA solid sorbent media as an appropriate sampling mechanism for final POD analysis. Tenax-TA media has a high affinity for volatile organic compounds (VOC's) and has been used extensively by Battelle to sample air streams for the presence of agents within the HMRF. The tubes were purchased

through ALLTECH Industries of Chicago, Illinois. The tubes consisted of a 15 cm stainless steel body with a glass lining. The outside diameter was 0.25 inches and the inside diameter was 0.4 millimeters. A total of 100 mg of 60/80 mesh Tenax-TA media was loaded into each tube. The ends of the tube were sealed with glass wool. The tubes were then baked overnight (12 hours) at 250°C to drive off any impurities which might create an interference during collection and analysis. Battelle received individual lots of 20 to 50 tubes at the HMRF. HMRF analytical staff randomly selected 5 percent of each lot for QC confirmation analysis. Each selected tube was analyzed utilizing the same handling and control practices to be employed with the actual sample tubes. All tubes analyzed during the length of this program indicated zero contamination or interferences. Following the QC confirmation the remaining tubes were repackaged and shipped to LANL for use during the program. A total of 300 tubes were procured from ALLTECH, with a total of 147 being consumed during the sampling phase.

Prior to the start of disassembly operations, Battelle technical personnel conducted limited, sampling of the ambient air inside and around the three laboratory fume hoods. The results of this monitoring with the MINICAMS instruments and the TENAX -TA sampling tubes did not indicate the presence of any HD or G- series agent contamination. Lewisite sampling was also negative. A negative agent reading is one which is below the U.S. Surgeon General's allowable exposure level (see Table 2.1).

Listed below is a table of the allowable exposure limits for each of the suspected agents:

Table 2.1 Allowable Exposure Levels

Agent	AEL (mg/m ³)
Sarin (GB)	0.0001
Soman (GD)	0.00003
Distilled Mustard (HD)	0.003
Lewisite (L)	0.000

2.3 Near Real Time Agent Monitoring During Disassembly Operations

During the disassembly phase of operations a MINICAMS unit was used to provide near real time (NRT) air monitoring for HD. The unit was calibrated with a solution of "half mustard" suspended in hexane. The unit was calibrated to alarm at a level of one time weighted average (TWA) for HD (see Table 2.1). The calibration was performed in the manner prescribed in HMRF SOP-IV-054-01 (see Appendix D).

2.4 TENAX - TA Sample Collection

As mentioned above the TENAX -TA solid sorbent collection tubes were selected to provide a second and more reliable form of sample analysis to augment the on-site MINICAMS unit analysis. The tubes were installed at each sampling point in triplicate and exposed to an air stream for a period of sixty minutes with an air flow rate of 0.5 liters/minute. The flow rate was controlled and verified by flow meters. All samples were collected in triplicate with one tube used to screen for the presence of radioactive isotope contamination via the LANL screening method. Following the radioactive screening process the remaining tubes were shipped to Battelle's Hazardous Materials Research Facility (HMRF) located in West Jefferson, Ohio by Federal Express. Proper QA/QC and strict chain-of-custody procedures were developed and implemented to ensure proper sample identification and to assure the correct match between the sampling point and the Tenax tube. The methods employed were derived from existing DOE protocols (see Appendix E). The GC analysis was performed in accordance with HMRF SOP IV-013-01 (see Appendix B). The first tube of each pair was analyzed for HD contamination. The second tube was analyzed for the presence of G-series nerve agent contamination. In the event that the first tube indicated a possible source of HD contamination, the second tube would also be analyzed for HD contamination. Both LANL and HMRF technical staff concluded that the G-series nerve agents were so volatile that there was a very remote possibility that any sample would indicate the presence of G-series agent contamination. In the event that only one tube indicated possible HD contamination, the data would be deemed inconclusive and that sampling point would be resampled.

2.5 Lewisite Collection and Analysis

As stated previously, all Lewisite (L) sample collection and analysis was performed by LANL personnel utilizing the HMRF SOP (see Appendix F). The analysis for L is based upon atomic absorption analysis (AA) for elemental arsenic. The Battelle protocol directs that a bubbler containing 10 mls of 10 percent NaOH - H₂O solution sample an air stream of 0.5 liters/minute for 60 minutes. L sampling and analysis was conducted concurrently with other sampling operations.

According to LANL technical personnel, the L samples were all negative (below AEL for L, see Table 2.1).

2.6 On-Site Disassembly Operations

During on site disassembly operations the decision to modify the original sampling point matrix was made by LANL technical personnel based upon the rate of disassembly operations and the ease of access to the monitoring and sample collection points. Disassembly operations required approximately ten days to complete and involved moving operations from the basement of building SM-29 through the first floor and concluded on the second floor at the laboratory filter train. There were no positive MINICAMS readings noted during this period. The MINICAMS was utilized as a first warning device to alert workers to the potential for agent or agent residue exposure. During the entire disassembly operations, all LANL, Battelle, and other involved personnel were required to wear Level B protective ensemble. (Level B consisted of supplied air respirator, Tyvek protective suit with hood, Tyvek Boot/shoe covers and Latex surgical gloves.) This protective level was directed to be worn in the unlikely event that agent was detected. The laboratory ductwork and other residues were consolidated and placed into a double-polyethylene bags for a period of twelve hours prior to being sampled with the Tenax-TA tubes.

3.0 Results

3.1 MINICAMS Data for HD

The MINICAMS data generated on-site were reviewed jointly by LANL and BMI personnel daily. The MINICAMS data were copied onto 3.5" floppy disks and retained by the LANL COTR. A brief roll-up of MINICAMS data points can be found in Table 3.1.

3.2 GC Data for TENAX -TA Samples

The HMRP GC data roll-up for the Tenax-TA tubes can be found in Appendix A. All samples were negative for detectable limits of agents with the exception of sample numbers: LA33022T, LA35024TU, LA3802T, LA39028T, LA40029T, LA42031T, LA47036T, LAa48037T, LA50039T, LA53042T, LA55044T, LA57046T which indicated HD contamination.

The samples which indicated HD contamination were believed to be a product of some form of interferant. Both LANL and BMI technical staff developed a plan to isolate the source of interference and conclusively demonstrate that the affected sample points were free of HD contamination above 1 TWA.

3.3 TENAX Inferent Contamination and Isolation

As stated in Section 3.2, both LANL and BMI technical staff believed the high HD levels to be the product of some form of sample contamination. In order to isolate the source of contamination the LANL and BMI staff who participated in the Tenax-TA sampling operations held a meeting to determine if the sample points in question were handled in a manner different from the other points. It was quickly discovered that the affected points had been sampled by staff members wearing a substitute version of Latex gloves. The packaging of the originally used gloves indicated that they were "sulfur free" while the substitute gloves packaging had no such indication. A sampling plan was

TABLE 3.1 SUMMARY OF TENAX SAMPLES

Sample Tracking Number	Sampling Location	HD Analysis Result (TWA)	GD Analysis Result (TWA)
LA 07002T	Storage Hood Exhaust	< .5	< .5
LA 06003T	East Hood Floor	< .5	< .5
LA 05004T	East Hood Exhaust	< .5	< .5
LA 12005T	West Hood Floor	< .5	< .5
LA 11006T	West Hood Exhaust	< .5	< .5
LA 19007T	Air Space in Room 4009	< .5	< .5
LA 19008T	Air Space in Room 4009 - Not shipped per W. Earl	< .5	< .5
LA 20009T	Storage Hood P.O.D.	< .5	< .5
LA 21010T	East Hood P.O.D.	< .5	< .5
LA 22011T	West Hood P.O.D.	< .5	< .5
LA 23012T	East Hood Base P.O.D.	< .5	< .5
LA 24013T	10 Foot Piece of Main Duct P.O.D.	< .5	< .5
LA 25014T	10 Foot Piece of Main Duct P.O.D.	< .5	< .5
LA 26015T	Duct Tee (Main) P.O.D.	< .5	< .5
LA 27016T	Storage Hood Duct P.O.D. (1st Piece)	< .5	< .5
LA 28017T	Storage Hood Duct P.O.D. (2nd Piece)	< .5	< .5
LA 29018T	East Hood Duct P.O.D.	< .5	< .5
LA 30019T	Upstream Plenum Environmental Sample	< .5	< .5
LA 31020T	Midstream Plenum Environmental Sample	< .5	< .5
LA 32021T	Downstream Plenum Environmental Sample	< .5	< .5
LA 33022T	8-Foot Piece of Wing 4 Main Duct P.O.D.	1.2 ^a	< .5
LA 34023T	2nd 8 Foot Piece of Wing 4 Main Duct P.O.D.	< .5	< .5

a - Sample result was originally above 0.5 TWA. However, an interferent was discovered associated with the sulphur powder on the poly vinyl gloves and subsequent analyses of duplicate samples minus this interferent yielded results below 0.5 TWA.

TABLE 3.1 SUMMARY OF TENAX SAMPLES (CONTINUED)

Sample Tracking Number	Sampling Location	HD Analysis Result (TWA)	GD Analysis Result (TWA)
LA 35024TU	Q.C Unknown Sample (Poly Vinyl Glove)	2.0 ^a	< .5
LA 36025TU	Q.C Unknown Sample	< .5	< .5
LA 37026TU	Q.C Unknown Sample	< .5	< .5
LA 38027T	P.O.D. of Floor Duct From Mens Room in Wing 4	1.8 ^a	< .5
LA 39028T	P.O.D. of Connecting Ductwork to Men's Room in Wing 4	0.9 ^a	< .5
LA 40029T	3 Foot Piece of Ductwork (Elbowed) From Men's Room - P.O.D.	1.0 ^a	< .5
LA 41030T	P.O.D. of 4 Foot Elbow Duct From Men's Room in Wing 4	< .5	< .5
LA 42031T	P.O.D. of 3 Foot Elbow Duct From Hood in Room 4009	1.3 ^a	< .5
LA 43032T	P.O.D. of 3 Foot Elbow Duct From Hood in Room 4009	< .5	< .5
LA 44033T	P.O.D. of 3 Foot Piece of Main Duct	< .5	< .5
LA 45034T	P.O.D. of Transition Duct to Plenum Tee	< .5	< .5
LA 46035T	P.O.D. of Transition Duct to Men's Room in Wing 4	< .5	< .5
LA 47036T	P.O.D. of Piece of Main Duct Work	0.75 ^a	< .5
LA 48037T	P.O.D. of Piece of Main Duct Work	1.0 ^a	< .5
LA 49038T	P.O.D. of Piece of Off-Chute Ductwork	< .5	< .5
LA 50039T	P.O.D. of Piece of Off-Chute Duct Work	1.3 ^a	< .5
LA 51040T	P.O.D. of Piece of Off-Chute Duct Work	< .5	< .5
LA 52041T	P.O.D. of Piece of Off-Chute Duct Work	< .5	< .5

^a - Sample result was originally above 0.5 TWA. However, an interferent was discovered associated with the sulphur powder on the poly vinyl gloves and subsequent analyses of duplicate samples minus this interferent yielded results below 0.5 TWA.

TABLE 3.1 SUMMARY OF TENAX SAMPLES (CONTINUED)

Sample Tracking Number	Sampling Location	HD Analysis Result (TWA)	GD Analysis Result (TWA)
LA 53042T	P.O.D. of Piece of Off-Chute Duct Work	0.75 ^a	< .5
LA 54043	P.O.D. of Plenum Charcoal Filter	< .5	< .5
LA 55044T	P.O.D. of Plenum Charcoal Filter	1.4 ^a	< .5
LA 56045T	P.O.D. of Plenum Charcoal Filter	< .5	< .5
LA 57046T	P.O.D. of Plenum Charcoal Filter	1.2 ^a	< .5
LA 58047T	P.O.D. of Plenum Charcoal Filter	< .5	< .5
LA 59048T	P.O.D. of Plenum Charcoal Filter	< .5	< .5

^a - Sample result was originally above 0.5 TWA. However, an interferent was discovered associated with the sulphur powder on the poly vinyl gloves and subsequent analyses of duplicate samples minus this interferent yielded results below 0.5 TWA.

TABLE 3.2 SUMMARY OF MINICAMS DATA

Sample Tracking Number	Sampling Location	HD Analysis Result (TWA)	GD Analysis Result (TWA)
MC001-MC004	13:55 p.m. - 14:08 p.m. on 9/30/93 - 5 foot sections of off-chute duct from flammable cabinet removed	< .5	N/A
MC005-MC013	14:11 p.m. - 14:35 p.m. on 9/30/93 - 5 foot sections, including risor and crosspiece, over east hood removed	< .5	N/A
MC014	09:47 a.m. - 10:02 a.m. on 10/4/93 - 3 foot elbow duct leading into west hood duct removed	< .5	N/A
MC015	10:03 a.m. - 10:18 a.m. - West hood removed from wall	< .5	N/A
MC016	10:20 a.m. on 10/4/93 - Off-shoot from west hood removed	< .5	N/A
MC017	13:30 p.m. - 13:55 p.m. on 10/4/93 - Removal of east hood and east hood elbow duct (3-foot section) from wall	< .5	N/A
MC018	09:05 a.m. - 09:35 a.m. on 10/5/93 - Removal of main duct work leading from west hood	< .5	N/A
MC019	9:40 - 09:50 a.m. on 10/5/93 - Removal of a 2 foot piece of duct work east of 1st disconnect on main duct	< .5	N/A
MC020	13:30 p.m. - 13:45 p.m. on 10/5/93 - Removal of 1st 10 foot section of main duct work going west to east of lab (Room 4009)	< .5	N/A
MC021	13:50 - 14:30 p.m. on 10/5/93 - Removal of 2nd 10 foot section of duct work (main) going west to east	< .5	N/A
MC022	08:50 a.m. - 10:05 a.m. on 10/6/93 - Removal of cage around storage hood and storage hood and moving the hood out from the east wall	< .5	N/A
MC023	13:15 - 13:55 p.m. on 10/6/93 - Removal of east hood duct work	< .5	N/A

TABLE 3.2 SUMMARY OF MINICAMS DATA (CONTINUED)

Sample Tracking Number	Sampling Location	HD Analysis Result (TWA)	GD Analysis Result (TWA)
MC024	13:58 p.m. + 14:28 p.m. - 14:33 p.m. on 10/6/93 - Removal of last 2 foot section of main duct	< .5	N/A
MC025	13:58 p.m. - 14:06 p.m. on 10/6/93 - Removal of last duct off-chute	< .5	N/A
MC026	14:15 p.m. - 14:23 p.m. on 10/6/93 - Cutting and removing duct work that was connected to the storage hood	< .5	N/A
MC026 (Cont)	08:40 - 08:45 on 10/7/93 - Finished removing duct work that was connected to storage hood	< .5	N/A
MC027	08:50 a.m. - 09:40 a.m. on 10/7/93 - Cutting and removing main duct phlange	< .5	N/A
MC028	14:10 p.m. - 15:00 p.m. on 10/18/93 - Removal of 1st 4 foot piece of duct work connecting room 4009 main duct work and duct work from men's room in Wing 4 (starting at men's foom floor)	< .5	N/A
MC029	08:15 a.m. - 08:30 a.m. on 10/19/93 - Removal of second 4 foot section of duct work in men's room in wing 4	< .5	N/A
MC030	08:45 a.m. - 08:55 a.m. on 10/19/93 - Removal of 4 foot duct elbow in men's room in Wing 4	< .5	N/A
MC031	09:20 - 09:30 - Removal of 8 foot section of duct work in men's room in Wing 4	< .5	N/A
MC032	13:00 p.m. - 13:15 p.m. on 10/19/93 - Removal of next 8 foot duct piece in men's room in Wing 4	< .5	N/A
MC033	13:15 p.m. - 13:25 p.m. on 10/19/93 - Removal of next 4 foot elbow duct in men's room in Wing 4	< .5	N/A

TABLE 3.2 SUMMARY OF MINICAMS DATA (CONTINUED)

Sample Tracking Number	Sampling Location	HD Analysis Result (TWA)	GD Analysis Result (TWA)
MC034	13:25 p.m. - 13:40 p.m. on 10/19/93 - Removal of next 8 foot duct piece in Wing 4	< .5	N/A
MCO35	14:10 p.m. - 14:15 p.m. - Last 8 foot section of duct work removed. All duct work removed, at this point, to plenum.	< .5	N/A

devised to re-sample the affected points, both versions of latex gloves, a sample of the tape used to secure the sampling tubes in a effort to conclusively prove the sampling points to be free of HD contamination, while identifying the source of contamination. Additionally, open Tenax-TA tubes were inserted into the shipping package to determine if the shipping conditions may have provided the source of contamination.

3.4 Tenax-TA Tube Re-Sampling Results

The re-sampled points were clearly shown to be free of HD contamination above 1 TWA, while the substitute latex gloves were shown to be the source of sulfur contamination. Appendix G contains copies of the chromatograms indicating the source of contamination.

4.0 Conclusions

The air sampling support provided by BMI clearly indicated a lack of HD or G-series nerve agent contamination above 1 TWA, respectively, in the former CSM laboratory and associated ductwork located in room 4009 of building SM-29. The BMI staff also demonstrated that Tenax-TA tubes are a viable and cost effective alternative to MINICAMS sampling. However, it was discovered that standard GC analysis can be disrupted by an interferant which, heretofore, had not been encountered (i.e., sulfur cured latex gloves). As a result of this discovery the HMRF analytical staff have adopted the use of non-powdered (non-sulfur) latex gloves for use within the HMRF and at other off-site air monitoring operations.

5.0 References

1. SOP HMRF-I-028-01, Standard Operating Procedure for Sampling and Analysis of Air for Traces of Lewisite (L), 1993.
2. SOP HMRF-IV-054-01, Standard Operating Procedure for Environmental Monitoring Using MINICAMS, November 1993.
3. Program Plan, Chemical Surety Material Decontamination and Decommissioning of Los Alamos National Laboratory Chemical Surety Materiel Laboratory, Area TA-3, Building SM-29, Room 4009, September 1993.
4. SOP HMRF-IV-013-01, Standard Operating Procedure for Analysis of Solutions Containing GA, GB, GD, GF, HD, VX and L by Gas Chromatography, January 1994.

APPENDIX A
GC SAMPLE DATA

LOS ALAMOS G2364-0002

LAED0928 INST #6

HD	TFR	QDL= 50.0ng			
FILE	NAME	AREA	SQRT HD	ug/mL	
NA248	TENAX SPIKE HD 150ng	1460.127	38.212	148.14	150
NA249	TENAX SPIKE HD 100ng	712.736	26.697	103.72	100
NA250	TENAX SPIKE HD 50ng	150.980	12.287	48.14	50
NA251	TENAX SPIKE HEXANE	0.000	0.000	0.00	
NA252	LA07002T-A	0.000	0.000	0.00	
NA253	LA06003T-A	0.000	0.000	0.00	
NA254	LA05004T-A	0.000	0.000	0.00	
NA255	LA12005T-A	0.000	0.000	0.00	
NA256	LA11006T-A	0.000	0.000	0.00	
NA257	TENAX SPIKE HD 150ng	1517.541	38.956	151.01	
NA258	TENAX SPIKE HD 100ng	887.874	29.797	115.68	
NA259	TENAX SPIKE HD 50ng	248.940	15.778	61.60	

Regression Output:

Constant	-0.19218
Std Err of Y Est	1.181973
R Squared	0.99586
No. of Observations	3
Degrees of Freedom	1

X Coefficient(s)	0.259242
Std Err of Coef.	0.016716

LOS ALAMOS G2364-0002

LAGD0928 INST #6

GD TFR QOL= 0.5ng

FILE	NAME	AREA	ug/mL	
NA241	LA05004T-B	0.000	0.00	
NA239	LA06003T-B	0.000	0.00	
NA238	LA07002T-B	0.000	0.00	
NA243	LA11006T-B	0.000	0.00	
NA242	LA12005T-B	0.000	0.00	
NA240	TENAX SPIKE HEXANE	0.000	0.00	
NA235	TENAX SPIKE GD 1.0ng	2.679	1.09	1
NA236	TENAX SPIKE GD 1.5ng	3.621	1.45	1.5
NA237	TENAX SPIKE GD 0.5ng	1.012	0.45	0.5
NA244	TENAX SPIKE GD 1.5ng	0.402		
NA245	TENAX SPIKE GD 1.0ng	0.330		

Regression Output:

Constant	-0.17167
Std Err of Y Est	0.29598
R Squared	0.974906
No. of Observations	3

Degrees of Freedom	1
--------------------	---

X Coefficient(s)	2.609
Std Err of Coef.	0.418579

LOS ALAMOS	G2364-0002	LAGD1012	INST #6	
GD	TFR		QDL= 0.50ng	
FILE	NAME	GD AREA	ug/mL	
NA271	LA20009T-B	0.000	0.00	
NA276	LA21010T-B	0.000	0.00	
NA277	LA22011T-B	0.000	0.00	
NA278	LA23012T-B	0.000	0.00	
NA279	LA24013T-B	0.000	0.00	
NA275	TENAX SPIKE HEXANE	0.000	0.00	
NA272	TENAX SPIKE GD 1.5ng	27.769	1.58	1.5
NA273	TENAX SPIKE GD 1.0ng	13.339	0.84	1
NA274	TENAX SPIKE GD 0.5ng	8.093	0.58	0.5
NA280	TENAX SPIKE GD 1.5ng	27.923	1.59	

Regression Output:

Constant	-3.27566667
Std Err of Y Est	3.7493523
R Squared	0.932294785
No. of Observations	3
Degrees of Freedom	1
X Coefficient(s)	19.676
Std Err of Coef.	5.30238487

LOS ALAMOS	G2364-0002	LAGD1013	INST #6
GD	TFR		QDL= 0.50ng
FILE	NAME	GD AREA	ug/mL
NA285	LA25014T-B	0.000	0.00
NA286	LA26015T-B	0.000	0.00
NA287	LA27016T-B	0.000	0.00
NA288	LA28017T-B	0.000	0.00
NA289	LA29018T-B	0.000	0.00
NA284	TENAX SPIKE HEXANE	0.000	0.00

NA281	TENAX SPIKE GD 1.5ng	26.131	1.53	1.5
NA282	TENAX SPIKE GD 1.0ng	15.527	0.95	1
NA283	TENAX SPIKE GD 0.5ng	7.873	0.53	0.5
NA290	TENAX SPIKE GD 1.0ng	15.558	0.95	

Regression Output:

Constant	-1.74766667
Std Err of Y Est	1.204332457
R Squared	0.991373125
No. of Observations	3
Degrees of Freedom	1

X Coefficient(s)	18.258
Std Err of Coef.	1.703183294

LOS ALAMOS	G2364-0002	LAHD1015	INST #6	
HD	TFR		QDL= 150.0ng	
FILE	NAME	HD AREA	SQRT HD	ug/mL
NA291	LA20009T-A	0.000	0.000	0.000
NA296	LA21010T-A	0.000	0.000	0.000
NA297	LA22011T-A	0.000	0.000	0.000
NA298	LA23012T-A	0.000	0.000	0.000
NA299	LA24013T-A	0.000	0.000	0.000
NA301	LA25014T-A	0.000	0.000	0.000

NA302	LA26015T-A	0.000	0.000	0.000
NA303	LA27016T-A	0.000	0.000	0.000
NA304	LA28017T-A	0.000	0.000	0.000
NA305	LA29018T-A	0.000	0.000	0.000
NA295	TENAX SPIKE HEXANE	0.000	0.000	0.000
NA292	TENAX SPIKE HD 100ng	864.676	29.41	105.89
NA293	TENAX SPIKE HD 150ng	1552.379	39.40	147.06
NA294	TENAX SPIKE HD 50ng	228.635	15.12	47.06
NA300	TENAX SPIKE HD 150ng	1564.798	39.56	147.70
NA306	TENAX SPIKE HD 100ng	1162.569	34.10	125.21
NA307	TENAX SPIKE HD 50ng	342.322	18.50	60.98

Regression Output:

Constant	3.6958720776
Std Err of Y Est	1.7513137224
R Squared	0.9897013467
No. of Observations	3
Degrees of Freedom	1

X Coefficient(s)	0.24279559914
Std Err of Coef.	0.02476731618

LOS ALAMOS	G2364-0002	LAGH1021	INST #6	
GD	TFR		QDL= 0.5ng-GD	
FILE	NAME	AREA	ng	
NA336	LA30019T-B	0.000	0.00	
NA337	LA31020T-B	0.000	0.00	
NA338	LA32021T-B	0.000	0.00	
NA335	TENAX SPIKE HEXANE	0.000	0.00	
				CA
NA334	TENAX SPIKE GD 0.5ng	5.228	0.43	0.5
NA333	TENAX SPIKE GD 1.0ng	10.673	1.13	1
NA332	TENAX SPIKE GD 1.5ng	13.057	1.43	1.5
NA339	TENAX SPIKE GD 1.0ng	13.242	1.46	

Regression Output:

Constant	1.823667
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Std Err of Y Est	1.249648
R Squared	0.951515
No. of Observations	3
Degrees of Freedom	1
X Coefficient(s)	7.829
Std Err of Coef.	1.7672692

LOS ALAMOS	G2364-0002	LAGH1021	INST #6	
HD	TFR		QDL= 50.0n-HD	
FILE	NAME	HD AREA	SQRT HD	ng
NA344	LA30019T-A	0.000	0.000	0.00
NA345	LA31020T-A	0.000	0.000	0.00
NA346	LA32021T-A	14.174	3.765	18.33
NA343	TENAX SPIKE HEXANE	0.000	0.000	0.00
NA340	TENAX SPIKE HD 150ng	1576.110	39.700	149.31
NA341	TENAX SPIKE HD 100ng	704.765	26.547	101.37
NA342	TENAX SPIKE HD 50ng	150.453	12.266	49.31
NA347	TENAX SPIKE HD 100ng	1298.465	36.034	135.95

Regression Output:

Constant	-1.26313
Std Err of Y Est	0.460766
R Squared	0.999436
No. of Observations	3
Degrees of Freedom	1
X Coefficient(s)	0.2743432
Std Err of Coef.	0.0065162

LOS ALAMOS	G2364-0002	LAGD1109	INST #6	
GD	TFR		QDL= 0.5ng	
FILE	NAME	AREA	ng	
NA351	TENAX SPIKE HEXANE	0.000	0.000	
NA352	LA33022T-B	0.275	0.042	
NA353	LA34023T-B	0.000	0.000	
NA354	LA38027T-B	0.202	0.039	
NA355	LA39028T-B	0.000	0.000	
NA356	LA40029T-B	0.000	0.000	
				CAL
NA348	TENAX SPIKE GD 1.5ng	32.615	1.499	1.50
NA349	TENAX SPIKE GD 1.0ng	21.599	1.003	1.00
NA350	TENAX SPIKE GD 0.5ng	10.408	0.499	0.50
NA357	TENAX SPIKE GD 1.5ng	34.873	1.600	

Regression Output:

Constant	-0.66633
Std Err of Y Est	0.071443
R Squared	0.999979
No. of Observations	3
Degrees of Freedom	1

X Coefficient(s)	22.207
Std Err of Coef.	0.101036

LOS ALAMOS	G2364-0002	LAGD1110	INST #6
GD	IFR		QDL= 0.5ng
FILE	NAME	AREA	ng
NA361	TENAX SPIKE HEXANE	0.000	0.00
NA362	LA41030T-B	0.000	0.00

NA363	LA42031T-B	0.000	0.00
NA364	LA43032T-B	0.000	0.00
NA365	LA44033T-B	0.000	0.00
NA366	LA45034T-A	0.000	0.00
NA368	LA46035T-B	0.000	0.00
NA369	LA47036T-B	0.000	0.00
NA358	TENAX SPIKE GD 1.5ng	34.611	1.54 1.50
NA359	TENAX SPIKE GD 1.0ng	19.566	0.92 1.00
NA360	TENAX SPIKE GD 0.5ng	10.048	0.54 0.50
NA367	TENAX SPIKE GD 1.5ng	36.543	1.62
NA370	TENAX SPIKE GD 1.0ng	22.850	1.06

Regression Output:

Constant	-3.15467
Std Err of Y Est	2.256388
R Squared	0.983403
No. of Observations	3
Degrees of Freedom	1

X Coefficient(s)	24.563
Std Err of Coef.	3.1910149

LOS ALAMOS G2364-0002

LAGD1111 INST #6

GD TFR QDL= 0.5ng

FILE	NAME	AREA	ng
NA374	TENAX SPIKE HEXANE	0.000	0.00
NA375	LA48037T-B	0.000	0.00
NA376	LA49038T-B	0.000	0.00
NA377	LA50039T-B	0.000	0.00
NA378	LA51040T-B	0.000	0.00
NA379	LA52041T-B	0.000	0.00
NA380	LA53042T-B	0.000	0.00
NA382	LA54043T-B	0.000	0.00
NA383	LA55044T-B	0.000	0.00
NA384	LA56045T-B	0.000	0.00
NA385	LA57046T-B	0.000	0.00
NA386	LA58047T-B	0.000	0.00
NA387	LA59048T-B	0.000	0.00

NA371	TENAX SPIKE GD 1.5ng	33.183	1.51	1.5
NA372	TENAX SPIKE GD 1.0ng	20.886	0.98	1
NA373	TENAX SPIKE GD 0.5ng	10.081	0.51	0.5

NA381	TENAX SPIKE GD 1.5ng	35.667	1.62
NA388	TENAX SPIKE GD 1.0ng	22.594	1.05
NA391	TENAX SPIKE GD 0.5ng	10.638	0.53

Regression Output:

Constant	-1.71867
Std Err of Y Est	0.609106
R Squared	0.998612
No. of Observations	3
Degrees of Freedom	1

X Coefficient(s)	23.102
Std Err of Coef.	0.861407

LOS ALAMOS	G2364-0002	LAH1117A	INST #6	
HD	TFR		QDL= 50.0ng-HD	
FILE	NAME	AREA	SQRT AREA	ng
NA404	TENAX SPIKE HEXANE	0.000	0.000	0.00
NA405	LA38027T-A	2109.276	45.927	181.10

NA406	LA39028T-A	629.952	25.099	99.56	
NA407	LA40029T-A	687.015	26.211	103.92	
NA401	TENAX SPIKE HD 100ng	636.580	25.231	100.08	100
NA402	TENAX SPIKE HD 50ng	153.287	12.381	49.77	50
NA403	TENAX SPIKE HD 25ng	37.131	6.094	25.15	25
NA408	TENAX SPIKE HD 100ng	788.969	28.089	111.27	

Regression Output:

Constant	-0.33129
Std Err of Y Est	0.073454
R Squared	0.999972
No. of Observations	3
Degrees of Freedom	1
X Coefficient(s)	0.255421966
Std Err of Coef.	0.001360111

LOS ALAMOS	G2364-0002	LAHD1117	INST #6	
HD	TFR		QDL= 50.0ng-HD	
FILE	NAME	AREA	SQRT AREA	ng
NA395	TENAX SPIKE HEXANE	0.000	0.000	0.00
NA396	LA33022T-A	1365.477	36.952	122.07
NA397	LA34023T-A	265.341	16.289	36.63
NA392	TENAX SPIKE HD 150ng	1770.808	42.081	143.28
NA393	TENAX SPIKE HD 100ng	1215.482	34.864	113.44
NA394	TENAX SPIKE HD 50ng	320.342	17.898	43.28
NA398	TENAX SPIKE HD 150ng	1847.167	42.979	146.99
NA399	TENAX SPIKE HD 100ng	1237.371	35.176	114.73
NA400	TENAX SPIKE HD 50ng	352.594	18.777	46.92

Regression Output:

Constant	7.431409
Std Err of Y Est	3.979791
R Squared	0.948616
No. of Observations	3
Degrees of Freedom	1

X Coefficient(s)	0.241828691
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Std Err of Coef.	0.056282742
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LOS ALAMOS	G2364-0002	LAHD1118	INST #6		
HD	tfr/bjh	CONC.	QDL= 25.0ng-HD		
FILE	NAME	AREA	SQRT AREA	ng	
NA412	TENAX SPIKE HEXANE	0.000	0.000		
NA435	LA35024T-Y	2555.627	50.553	181.91	
NA436	LA36025T-Y	0.000	0.000		
NA437	LA37026T-Y	0.000	0.000		
NA413	LA41030T-A	0.000	0.000		
NA414	LA42031T-A	1563.911	39.546	142.30	
NA415	LA43032T-A	0.000	0.000		
NA416	LA44033T-A	0.000	0.000		
NA417	LA46035T-A	0.000	0.000		
NA419	LA47036T-A	280.439	16.746	60.26	
NA420	LA48037T-A	841.138	29.002	104.36	
NA421	LA49038T-A	0.000	0.000		
NA422	LA50039T-A	1503.883	38.780	139.55	
NA423	LA51040T-A	0.000	0.000		
NA425	LA52041T-A	0.000	0.000		
NA426	LA53042T-A	445.021	21.096	75.91	
NA427	LA54043T-A	0.000	0.000		
NA428	LA55044T-A	1446.235	38.029	136.85	
NA430	LA56045T-A	0.000	0.000		
NA431	LA57046T-A	1194.457	34.561	124.36	
NA432	LA58047T-A	0.000	0.000		
NA433	LA59048T-A	0.000	0.000		
NA411	TENAX SPIKE HD 25ng	34.805	5.900	22.11	
NA429	TENAX SPIKE HD 25ng	40.592	6.371	23.77	
NA410	TENAX SPIKE HD 50ng	225.654	15.022	54.34	
NA424	TENAX SPIKE HD 50ng	185.704	13.627	49.41	
NA409	TENAX SPIKE HD 100ng	758.051	27.533	98.55	
NA418	TENAX SPIKE HD 100ng	710.939	26.663	95.48	
NA434	TENAX SPIKE HD 100ng	769.001	27.731	99.25	

Regression Output:

Constant	-0.31546
Std Err of Y Est	0.826302
R Squared	0.993999
No. of Observations	7
Degrees of Freedom	5
X Coefficient(s)	0.27790025
Std Err of Coef.	0.009656968

LOS ALAMOS G2364-0002

LAC1222

INST #6

HD	BH	AREA	SQRT AREA	ng
FILE	NAME			
NA451	BKGRND 1	0.000	0.000	0.00
NA449	BLANK	0.000	0.000	0.00
NA448	LA33022T2	0.000	0.000	0.00
NA452	LA38027T2	0.000	0.000	0.00
NA454	LAA2031T2	0.000	0.000	0.00
NA455	LA48037T2	0.000	0.000	0.00
NA456	LA50039T2	0.000	0.000	0.00
NA457	LA55044T2	0.000	0.000	0.00
NA459	LA57046T2	0.000	0.000	0.00
NA444	NA444 HEXANE SPIKE	0.000	0.000	0.00
NA462	SUL GL HMRF	0.000	0.000	0.00
NA463	SUL GL LANL	0.000	0.000	0.00
NA450	TRIP BLANK	0.000	0.000	0.00
NA461	YELL GL 2	0.000	0.000	0.00

NA445	STD 25NG HD	23.308	4.828	26.61	25
NA446	STD 50NG HD	47.735	6.909	47.59	50
NA447	STD 100NG HD	148.502	12.186	100.80	100

NA453	STD 100NG HD	36.668	6.055
NA458	STD 50NG HD	4.180	2.045

Regression Output:

Constant	2.189292137
Std Err of Y Est	0.2979042068
R Squared	0.9969157879
No. of Observations	3
Degrees of Freedom	1

X Coefficient(s)	0.09917229
Std Err of Coef.	0.00551611

APPENDIX B

**STANDARD OPERATING PROCEDURE
FOR ANALYSIS OF SOLUTIONS CONTAINING
GA, GB, GD, GF, HD, VX AND L BY GAS CHROMATOGRAPHY**

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Manual Number:

Battelle SOP Number: HMRF-IV-013-01
Page 1 of 9

Key words: gas chromatography
flame photometric
flame ionization

Title:

STANDARD OPERATING PROCEDURE
FOR ANALYSIS OF SOLUTIONS CONTAINING
GA, GB, GD, GF, HD, VX AND L BY GAS CHROMATOGRAPHY

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Manual Number:

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PRE-OPERATIONAL CHECKLIST

SOP HMRF-IV-013.00

1. Assemble necessary equipment as specified on page 3 so all items are readily available.
2. Ensure that staff conducting the operations have read the SOP, signed page 1A attesting to the fact that they have read and understood the operation and are completely trained in the operation.
3. Ensure that all staff conducting the operation understand the hazards involved with handling XDS, decontaminants and other hazards listed in the SOP.
4. Assure that SARMS have not expired.
5. Assure that hoods used in the operation have been quarterly certified.
6. Assure that local exhaust is in place and if a split stream is used, that it is properly vented.

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Battelle SOP Number: HMRF-IV-013-01

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I. Scope

This Standard Operating Procedure (SOP) describes the procedures for analysis of primarily organic solutions of chemical surety material (CSM) including GB, GD, GA, HD, and VX by capillary column gas chromatography (GC). Aqueous solutions of all these material but HD, which is not stable in aqueous solutions, can be analyzed, but require special handling. Work will be performed in accordance with Bailment agreement DAAA15-89H-0001.

II. Purpose

The purpose of this document is to provide the HMRF staff with a definitive procedure to ensure uniform operation of the gas chromatographic equipment for the purpose of performing XDS analyses. Work will be performed in the analytical lab in accordance with SOP HMRF III-001.

III. References

Standard Operating Procedure for General Provisions for Exempt Dilute Solutions (XDS) in the Hazardous Materials Research Facility (SOP HMRF-III-001).

IV. Definitions

1. See SOP HMRF-II-001, Section IV.

V. Procedure

A. Hazards

Operators will be thoroughly familiar with the hazards associated with the following:

Agents: GB, GD, GA, VX, GF, L, HD

Decontaminants: 10% Sodium Hydroxide (NaOH) for agents
10% HTH for VX, 5% bleach for HD,
10% Alcoholic Caustic for Lewisite

Solvents: Hexane, Diethylphthalate (DEP), Ethylene glycol diacetate

NOTE: The hazards associated with the agents, decontaminants, and solvents listed above are described in the Material Safety Data Sheets as found in the Safety Program Plan and in the binders located beside each SOP Manual in each laboratory.

B. Equipment

In addition to the items specified in SOP HMRF-III-001 the following will be required:

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Battelle SOP Number: HMRF-IV-013-01

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GC with : autosampler
split/splitless injector
flame photometric or flame ionization detector
output device
all associated gases and utilities.

Hamilton syringe or equivalent
Pasteur pipets
Volumetric flasks
Appropriate solvent

C. General Procedure

XDS samples are injected into the GC where the various XDS are separated from each other and from the solvent. The basis for GC separation is the distribution of a sample between two phases. One phase is a stationary phase with a large surface area and the other, the mobile phase, is a carrier gas.

Components to be separated are carried through the column by the carrier gas. The sample mixture is partitioned between the carrier gas and the stationary phase which selectively retards the sample components until they form separate bands in the carrier gas. These component bands elute from the column in the carrier gas stream, are detected and quantitated by the detector, and recorded as a function of time by the output device, which may be a strip chart recorder, an integrator or a laboratory data system. Detectors addressed in this SOP include the flame photometric detector (FPD), flame ionization detector (FID), electron captive detector (ECD) and mass spectrometry (MS).

D. Specific Procedures

1. Interferences

Each sample matrix must be evaluated for possible coextracting and/or coeluting interferences, or sample components which interfere with the proper operation of the detector. Samples should be collected and analyzed under the test conditions but without agent and the chromatograms should be compared to those from samples exposed to or spiked with the analyte of interest.

2. Precision

The relative standard deviation (RSD) for multiple injections of standards greater than 1 $\mu\text{g/mL}$ should be less than 10 percent. Standards less than 1 $\mu\text{g/mL}$ may have a RSD greater than 10 percent. Low levels of HD and VX (0.1 $\mu\text{g/mL}$) have shown 20 to 30 percent RSD.

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The relative standard deviations provided in Table 1 were obtained injecting three sample per day for three days at the concentrations noted. The detector used was the FPD. The FID detector can be expected to give similar results.

TABLE 1. RELATIVE STANDARD DEVIATIONS

$\mu\text{g/mL}$	GB RSD	GD RSD	VX RSD	HD RSD
0.1	0.094	0.111	0.323	0.223*
1	0.019	0.010	0.119	0.059
10	0.028	0.024	0.029	0.046
50	0.032	0.040	0.030	0.079
100	0.011	0.022	0.041	0.053
200	0.032	0.024	0.032	0.053

NOTE: Information not available for GA. Will be inserted as a change memo at a later date.

* 0.5 $\mu\text{g/mL}$

In a similar manner, the detection limit and range information is presented in Table 2. The represented instrument range is achieved using both split- and splitless-injection modes. The represented instrument range cannot be achieved with either mode alone.

TABLE 2. DETECTION LIMIT AND RANGE

CSM	FP DETECTOR	FI DETECTOR
GB	0.05 - 200 $\mu\text{g/mL}$	5 - 500 $\mu\text{g/mL}$
GD	0.05 - 200 $\mu\text{g/mL}$	5 - 500 $\mu\text{g/mL}$
HD	0.05 - 200 $\mu\text{g/mL}$	5 - 500 $\mu\text{g/mL}$
VX	0.05 - 200 $\mu\text{g/mL}$	25 - 500 $\mu\text{g/mL}$

NOTE: Information not available for GA. Will be inserted as a change memo at a later date.

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E. Stock and Calibration Standards

SARM's in hexane are stored at - 70°C. The SARM solutions will be decontaminated and resupplied approximately every six months. All calibration standards are prepared from SARMs whenever possible.

A minimum of three calibration standards will be used to cover the expected concentration range of the samples. All analytical standard solutions should be placed in individual ALS vials, stored in the freezer, and only removed just prior to use. Analytical standards must be prepared fresh each month. Solutions removed from the freezer must be used within 48 hr and then decontaminated.

1. Calibration Standards

The specific procedures stated in HMRP III-001 Section V.F will be followed when making dilutions of XDS.

The calibration standards are prepared by diluting a SARM, for example a 1 mg/mL SARM, with the same solvent as used in the samples to be analyzed. Typical standards are prepared as follows:

1 μ g/mL std - 1 μ L of 1 mg/mL stock/mL of standard prepared
5 μ g/mL std - 5 μ L of 1 mg/mL stock/mL of standard prepared
10 μ g/mL std - 10 μ L of 1 mg/mL stock/mL of standard prepared
15 μ g/mL std - 15 μ L of 1 mg/mL stock/mL of standard prepared
25 μ g/mL std - 25 μ L of 1 mg/mL stock/mL of standard prepared

For standards of less than 1 μ g/mL, dilutions are made from a 10 μ g/mL standard as follows:

0.05 μ g/mL std - 5 μ L of 10 μ g/mL stock/mL standard prepared
0.10 μ g/mL std - 10 μ L of 10 μ g/mL stock/mL standard prepared
0.50 μ g/mL std - 50 μ L of 10 μ g/mL stock/mL standard prepared

F. Sample Preparation

Samples will be prepared as described in the project-specific SOP. The location of sample preparations will also be specified in the project-specific SOP. A minimum of 500 μ L of sample is required for analysis by GC. Experimental quality control samples and matrix spike, matrix spike duplicates, and method blanks should be provided as appropriate.

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G. Instrument Setup

Instrument setup is described in an equipment-specific Operator's Manual which is kept on file at the HMRP. Check to see if the local exhaust vent is in place and operating. A typical setup includes the parameters listed below. In addition, the gas flow requirements for each detector are set according to the instrument operator's manual. Under these conditions the approximate retention times for the CSM are indicated in Table 3, while a typical chromatogram is included in Figure 1.

Column:	DB-5, HP-1, SPB-20, SE-52, Carbowax 20M, or other appropriate column
Carrier Gas:	Helium at a velocity of 35 cm/sec
Injector Temp:	225°C
Detector Temp:	225°C
Temp Program:	40°C for 2 min 20°C/min to 250°C 250°C for 10 min

TABLE 3. TYPICAL RETENTION TIMES

CSM	RETENTION TIME
GB	4.2 min
GD	7.7 min
HD	9.2 min
VX	13.1 min
GA	3.8 min

H. Instrument Shut Down

1. For overnight shut down, simply set the oven temperature at 220°C.
2. For long-term shut down, set the oven temperature at 220°C. Turn off the detector power and detector gases. See the Instrument Manual for details.

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I. Quality Control

All gases and solvents will be of the highest purity available. New lots of solvent should be checked to assure that coeluting interferences do not exist in the solvent. The instrument will be calibrated with at least two sets (minimum of three each) of calibration standards that cover the expected range of interest. After every fifth unknown sample a single calibration standard will be run to confirm instrument performance. Analysis of the check sample will be based on the standard curve generated by the sets of calibration standards.

The standard curve should have a value for $r \geq 0.993$ and the signal-to-noise ratio for the lowest standard should be at least 3:1. Values for the check samples should be in the control range described in Section III. If the check samples are not in the control range new standards will be prepared and the samples rerun.

The number of method blanks, matrix spikes, and matrix spike duplicates to be run with the unknown samples will be indicated in the project-specific test plan. The method blanks, matrix spikes, and matrix spike duplicates typically total 10 to 20 percent of the number of unknown samples run, with approximately equal numbers of method blanks, matrix spikes, and matrix spike duplicates.

Samples not in the expected range will be reanalyzed with standards to cover the concentration range of the samples, each batch of unknown samples will be analyzed with an appropriate method blank, method spike, and duplicate samples.

J. Calculations

The standard curve and analysis of samples are calculated by the data system using peak area and a linear regression program. Additional calculations, as required, will be described in the relevant project specific SOPs.

K. Decontamination

Remaining XDS, pipets, flasks, gloves, etc., used in this procedure will be decontaminated and verified to be decontaminated in accordance with SOP III-001 Section V.F.9 and 10, and V.G. and H.

L. Emergency Procedures

Action necessitated by emergencies will be conducted as outlined in SOP III-001, Section V.C. and D (page 13-17).

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M. Special Safeguards

GCs will be provided with local exhaust (such as an elephant trunk) near the point that the column exhausts. Any split stream work will also be vented. Sealed GC vials will be placed and removed from the instrument wearing the minimum XDS clothing while wearing a pair of surgical gloves.

N. First-Aid/Self-Aid Procedures

If physical injuries occur, first aid or self aid will be administered and the nurse located on site will be called. If extensive injury results the ambulance will be summoned by dialing 4-5435 or 4-5438. If the telephone line is inoperable or busy, use the two-way radio located in the secretary's office.

APPENDIX C

LOCATION SKETCH OF ROOM 4009 (not to scale)

Figure C.1 Location Sketch of Room 4009 (not to scale)

APPENDIX D

**STANDARD OPERATING PROCEDURE
FOR ENVIRONMENTAL MONITORING USING MINICAMS**

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Key words: environmental
monitoring
MINICAMS
confidence limit

STANDARD OPERATING PROCEDURE FOR ENVIRONMENTAL MONITORING USING MINICAMS

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PRE-OPERATIONAL CHECKLIST

SOP HMRF-IV-054-01

- ___ 1. Ensure that there is sufficient compressed gases to complete the test.
- ___ 2. Ensure that staff conducting the operations have read the SOP, signed page 1A attesting the fact that they have read and understood the operation, and completed dry runs, and are completely trained in the operation.
- ___ 3. Ensure that all staff conducting the operations understand the hazards involved with handling CSM and decontaminants.
- ___ 4. Check the retention time for the agent of interest.

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I. Scope

This Standard Operating Procedure (SOP) details the techniques to be used for operating the Miniature Chemical Agent Monitoring System (MINICAMS). This operation is performed in support of contracts associated with Bailment Agreement DAAA15-89-H-0001.

II. Purpose

The purpose of this document is to provide the HMRF staff with a definitive procedure to ensure uniform operation of the MINICAMS for the purpose of performing low-level CSM monitoring during CSM operations within the HMRF.

III. References

Standard Operating Procedure for General Provisions for Exempt Dilute Solutions (XDS) in the Hazardous Materials Research Facility (SOP HMRF-III-001).

IV. Definitions

See SOP HMRF-III-001.

V. Procedure

A. Hazards

Operators will be thoroughly familiar with the hazards associated with the following:

Agents: GB, VX, HD, GD, GA

Decontaminants: 10% Sodium Hydroxide (NaOH) for GB, GD, and GA
10% HTH for VX, 5% bleach for HD

Solvents: Hexane, Acetone, Isopropyl alcohol

NOTE: The hazards associated with the agents, decontaminants, and solvents listed above are described in the Material Safety Data Sheets as found in the Safety Program Plan and in the binders located beside each SOP Manual in each laboratory.

B. Health and Safety Procedures

All procedures referred to in SOP HMRF-III-001, Sections V.A. and V.B.1 will be followed.

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C. Equipment

In addition to the items specified in SOP HMRF-III-001 the following will be required:

- MINICAMS
- 10 μ L Syringe
- 25 μ L Syringe.

D. General Procedure

For CANL - 3 CONSECUTIVE 1 TWA INJECTIONS

The MINICAMS will be calibrated daily using calibration standards with known concentrations of 0.5, 1, and 2 times the established time weighted average (TWA) concentration for the target chemical agent.¹ The calibration will be checked using an injection of the 1 TWA calibration standard at the mid point of an 8 hr operation or after 4 hours of operation to verify that shifts in the instrument's response has not occurred. If the results from the calibration check varies by more than 15 percent from the calibration results, calibration will be reestablished.

E. Specific Procedure

1. Work with Drinking Water Standard (DWS) will be carried out wearing the following minimum clothing.
 - a. Blue surgical scrub suit
 - b. Fully fastened lab coat
 - c. Safety shoes
 - d. Protective eye wear
 - e. Supplied-air full-face respirator readily available
 - f. Latex surgical gloves (2 pair)

¹

Airborne exposure limits (AEL)s established by the U.S. Army Surgeon General is the maximum exposure to a chemical vapor based upon an 8-hour time weighted average (TWA) concentration for an unmasked worker: 3.0×10^{-3} for HD mg/m³, 3.0×10^{-5} for GD mg/m³, 1.0×10^{-4} for GB mg/m³, 1.0×10^{-5} for VX mg/m³, and 1.0×10^{-4} for GA mg/m³.

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2. Minimum protective clothing for all XDS operations will be in accordance with SOP HMRF-III-001, Steps V.B.6.b. From the stock solution, prepare the required standards by dilution with hexane either as follows or equivalently.
 - a. 10 $\mu\text{g/mL}$ Analytical Standard - Dilute 10.0 μL of the 1 mg/mL stock standard per mL of desired volume standard needed. Cap, vortex, and label appropriately.
 - b. 0.02 $\mu\text{g/mL}$ Calibration Standard - Dilute 2 μL of the 10 $\mu\text{g/mL}$ analytical standard per mL of desired volume standard needed. Cap, vortex, and label appropriately.

NOTE: The 0.02 $\mu\text{g/mL}$ calibration standard is used for the GB, GD, VX, GA calibration.

- c. 0.20 $\mu\text{g/mL}$ Calibration Standard - Dilute 20 μL of the 10 $\mu\text{g/mL}$ analytical standard per mL of desired volume standard needed. Cap, vortex, and label appropriately.

NOTE: The 0.20 $\mu\text{g/mL}$ calibration standard is used for the HD calibration.

3. Calibrate the MINICAMS each day in accordance with the procedures defined in the operator's manual using injections of standards with known concentrations of 0.5, 1, and 2 times the established TWA for the target agent. The minimum MINICAMS response at 1 TWA will be $> 2.5 \text{ nA}$ and the R^2 value for the regression of the calibration curve must be greater than 0.99. Record the date, time, agent, retention time and initials of the person doing the calibration in the Instrument Log Book. Rinse the syringe with hexane or acetone. The rinsate will be placed in the appropriate decon solution. The syringe will be returned to the analytical laboratory when monitoring is completed.
4. Once calibrated, position the MINICAMS sample probe in the breathing zone of the work area to be monitored. The MINICAMS will now monitor without constant observation, but must be checked several times throughout the monitoring period.
5. The data generated during the monitoring period will be evaluated based on the known retention time of the agent of interest. If the retention time of any air sample varies from the retention time established from the calibration by more than 1.0 seconds or if the peak width is < 1 second or > 3 seconds, it will be documented as a "non-agent" response (e.g., materials other than agent are occasionally observed by the MINICAMS, but can only be identified by the variance in retention time).

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NOTE: To calculate the mean TWA values, sum all the values recorded above the confidence limit which eluted at the correct retention time on the MINICAMS print-out. Divide the sum total by the total number of readings. Record this value in the log book as the Relative TWA Average.

F. Emergency Procedures

Any actions necessitated by emergencies will be conducted as described in SOP HMRF-II-001, Section V.E.19 through V.G.

G. First Aid/Self Aid Procedure

If physical injuries occur, first aid or self aid will be administered and the nurse located on site will be called. If extensive injury results the ambulance will be summoned by dialing 4-5435 or 4-5438. If the telephone line is inoperable or busy, use the two-way radio located in Harry Hawkin's office.

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6. The MINICAMS calibration will be checked at the half-way point of an 8 hr operation. This will be accomplished using a "check sample" of known concentration (e.g., typically the 1 TWA standard). The result with the check sample must be within 15 percent of the expected value. The minimum XDS personal protective clothing will be worn as outlined in V.E.1.

NOTE: Following the calibration, check the MINICAMS printout to ensure it has been properly annotated with "SPIKE" and the spike sample concentration. A service note appears on the mini-net printout. This will prevent uncertainty when reviewing the records as to why an alarm was observed and reported by the MINICAMS during the monitoring operation.

7. If the result with the check sample is not within 15 percent of the expected value and neat agent manipulations are in progress, (1) the CSM operation will be suspended, (2) the MINICAMS will be recalibrated following the procedure outlined in step V.E.1-3 and (3) when a proper calibration is established and the MINICAMS is properly positioned for monitoring, the CSM operation will be restarted.
8. When monitoring is completed, the environmental log book will be filled out with the data obtained from the analysis period. Note the log book number on the MINICAMS print-out.
9. Note the end time of the monitoring period in the environmental monitoring log book.
10. The data generated during the monitoring period will be evaluated/documented based on the known retention time of the agent of interest and the average of the TWA measurements for that agent.
 - If the retention time of any air sample varies from the retention time established from the calibration by more than 1.0 second or the peak width is < 1 second or > 3 seconds it will be documented as a "non-agent" response (e.g., materials other than agent are occasionally observed by the MINICAMS, but can only be identified by the variance in retention time).
 - The mean TWA values obtained throughout the monitoring period will be recorded based on the operational confidence limits of the MINICAMS. For example, if the confidence limit of the MINICAMS is 0.20 TWA and, the average of the MINICAMS results during the monitoring period is 0.27 TWA, the monitoring results will be recorded as 0.27 TWA. However, TWA values below the confidence limit (e.g., 0.20 TWA) will be recorded as values less than the confidence limit (e.g., < 0.20 TWA).

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Environmental Monitoring					Environmental Monitoring				
Sample	Description	Date	Start Time	End Time	Sampled by	Analysis TC GC HC CSM	Comments	Analysis Date	Analysis Result, $\mu\text{g/L}$ Avg.
-01									-01
-02									-02
-03									-03
-04									-04
-05									-05
-06									-06
-07									-07
-08									-08
-09									-09
-10									-10
-11									-11
-12									-12
-13									-13
-14									-14
-15									-15
-16									-16
-17									-17
-18									-18
-19									-19
-20									-20
-21									-21
-22									-22
-23									-23
-24									-24
-25									-25
-26									-26
-27									-27
-28									-28

FIGURE 1. ENVIRONMENTAL MONITORING LOG

Sample and Document Management Guidelines

The sample and documentation guideline establishes required procedures and documentation which will be incorporated into SOP's or study plans for all sampling and analysis teams.

The need for sample and document management procedures is important for three reasons:

1. establishing the authenticity of the evidence collected,
2. facilitating interpretation of sampling and analysis results, and
3. standardizing data entries for input into the data management system.

Traditional procedures used for collecting, handling, documenting and shipping samples may fail to meet the demands required for hazardous waste sampling situations of this nature.

There is a definite need to view sampling and analysis results in context to address questions concerning data quality and application. Knowledge of the circumstances surrounding collecting, handling, transporting, and analyzing samples will play an important role in determining how the analytical data are used and the kinds of interpretations that may develop.

A definite need for uniformity in data recording is a must. Various groups or organizations will be involved in the Los Alamos sampling task data gathering undertaking. As a result, consistent, standardized recording becomes essential to the development of an effective and efficient data management system.

Documentation of Field Sampling

Record-keeping and documentation of field activities are essential for a thorough investigation. A written record of all field data, observations, and events serves the following:

1. Ensure timely, correct, and complete analysis for all parameters requested
2. Satisfies applicable quality assurance requirements
3. Ensures that all essential information is consistently acquired and preserved for current use and future reference
4. Acts as evidence in court proceedings
5. Establish a chain-of-custody record for samples.

APPENDIX E

SAMPLE DOCUMENT MANAGEMENT GUIDELINES

Project Logbook

The project logbook is used to record general information, events and activities of the sampling staff, as well as any other related information that becomes available to project personnel. The project logbook should contain the names of all individuals participating in the sampling effort and their respective signatures, so that each individual recording information can easily be identified.

The logbook will be bound, and each page numbered. The cover will clearly display the title, "Project Logbook #1", Other information written on the cover will include:

1. Name of the sampling organization, " Battelle"
2. Name of the site. "Los Alamos"
3. Name of the person responsible for the logbook.

A Field Sampling leader or the Supervisor will complete the logbook, and will assure that it is included in the project file after a QA review has been performed.

General Field Logbook

The Field sampling Leader or Supervisor will use this book to record activities of the sampling team as samples are collected and to record quantitative information and measurements for each physical sample collected. If sampling is performed by the Site (Los Alamos personnel), once the sample is received, along with chain of custody information, then information should be recorded concerning the specific sample once Battelle personnel have signed the chain of custody form. Such information should include the name of the person who relinquished the sample, collection point, sampling time, and other information specific to each sample.

The General Field Logbook will clearly display the title, "General Field Logbook" # 1, Other information on the cover will include:

1. Name of sampling organization, "Battelle".
2. Name of site, "Los Alamos".
3. Name of person responsible for the log book.

Each logbook will be bound with consecutive numbered pages, and contain the following information:

1. Table of contents (See attachment 1)
 - a. Sampling location
 - b. Date sample collected
 - c. Sample ID number(s)
 - d. Sample log sheet (page number). Include page number within the field logbook where quantitative information and measurements were recorded for a given location or sample ID number(s).

Maintaining standardized records facilitates the collection of the data required to conduct site activities. Use of standardized forms also ensures that the same type of information will be recorded consistently. These records provide the basis for decisions regarding the existence of environmental problems at the Los Alamos site.

General Procedures

Field logbooks and sample labels will be completed using waterproof ink. If an error is made on any of the field documents, a correction will be made by drawing a single line through the error and entering the correct information. All corrections must be initialed and dated. Should a field document be damaged, lost or destroyed, the serial number and the disposition must be recorded. Field documents that are voided must not be destroyed; they shall be maintained in the study files for accountability.

Sample Identification Codes

Sample identification (ID) numbers will be assigned to each physical sample that is requested. The requirements of the sample ID number are (1) that it be unique and distinguish the sample from other similar evidence and (2) that it be traceable throughout the process.

To meet these two requirements, the sample ID number will contain the following information:

<u>Character(s)</u>	<u>Define</u>
1 and 2	Survey site
3 and 4	Air Sample Position (see Table C 1)
5,6 and 7	Sample Number
8	Sample Media

The purpose of the Survey site character is to reference the site from which the sample is obtained. For our purpose use LA (Los Alamos).

The purpose of the sample number is to reference each physical sample that is collected from a designated location.

The sample container is used to distinguish sample material collected at a designated site, under a designated location, and sample number in a specific container.

In summary, a sample ID number such as LA 01-001 T would indicate the following:

LA	Los Alamos
01	Sample number
001	Sample number
T	Tenax Tube

- k. **Preservatives.** Indicate the type and volume of preservative added and/or conditions under which the sample was kept (e.g. temperature).

Instrument Calibration Logbook

The Instrument Calibration Logbook (see attachment 4) is used (1) to document proper instrument calibration and/or test procedures and (2) to identify field instruments at the Los Alamos Site that are damaged or malfunctioning. If MINICAM is used, calibrations will be recorded in a green, bound notebook.

A separate Instrument Calibration Logbook will be maintained for each instrument used.

At a minimum, the Instrument Calibration Logbook should contain the following:

1. Instrument name, model, and serial number
2. Date and time of calibration and/or test
3. Methods and type of standards used for calibration and/or test
4. Signature of individuals performing the calibration and/or test

The Instrument Calibration Logbook will be completed by the individual performing the calibration. Following a QA check, the log, or copy thereof, will be placed in the study file.

Sample Labels

The Sample Label is used to maintain sample identity. It is recommended that Standard I-CHEM bottle labels be used (See attachment 5) but another label can be substituted as long as the following information is present:

Site name	Record the site (Los Alamos) at which the sample was taken.
Date	Record the date of sample collection.
Time	Record the time at which the sample was taken.
Preservative	Record the type and volume of preservative, if any, which was added.
Analysis	Indicate the type of analysis that will be performed by the analytical laboratory as specified in the sampling plan.

2. Activity log sheet (see attachment 2)

- a. Record the date when the form was initiated.
- b. Record how many pages or log sheets were used to record the activity for a given day.
- c. Team members. Name all the members of the team involved in the activity.
- d. Narrative (include time and location). Create a chronological record of the teams activities throughout the day, include the time and location of each activity. Include descriptions of any general problems encountered and the name of site personnel contacted by the Battelle team for permits, logistics, security, technical, or other information.
- e. Recorded by (signature) Indicate the individual responsible for the entries contained on the form.
- f. QA checked by (signature) Indicate the individual who perform the QA check on the completed form.

3. Sample log sheet (see attachment 3)

The Sample Log Sheet is used to record the following information:

- a. Date of Collection
- b. Sample ID Number. Indicates the first 6 characters of the sample ID number assigned to the physical sample.
- c. Time of collection
- d. Sample location. Indicate the exact location of sampled area (specified by code and/or description) as referenced in the sampling plan.
- e. Sampling point. Indicate the exact point at which the sample was collected as indicated by code, description, and/or coordinates.
- f. Describe the sampling material. What type of material from which the sample came from.
- g. Indicate the type of sample collected.
- h. Recorded by (signature). Indicate the individual who recorded entries contained on the form.
- i. QA Checked by (signature). Indicate the individual who performed the QA check on the completed form.
- j. Analysis. Indicate the analysis that will be performed by the analytical laboratory as specified in the Study Plan.

Airbills

Airbills are used to assist the shipping personnel in complying with shipping requirements set forth by the U.S. Department of Transportation.

Airbills will be completed by the shipping Coordinator. The customer copy will be held in the temporary field office until the shipment is received in the analytical laboratory. The copy is then placed in the project file.

The sample label will be completed by the individual collecting the sample in the bottle. Following a QA check, the label information will be entered into the General Field Logbook. The container holding the sample will be disposed of following analysis.

Chain-of-Custody

Chain-of Custody records (attachment 6) are used to record transfer in the possession and custody of samples. The record attests that, between sample collection and laboratory analysis, the sample was constantly under custody.

Serialized Chain-of-Custody forms will contain the following information:

1. Survey Site Name. "Los Alamos"
2. Samplers (signature). Record the signatures of all individuals who physically collected the samples and initialed the Chain-of Custody Records.
3. Date of Collection. Record the date of sample collection.
4. Sample ID Number. Indicates the ID number(s) of physical samples(s).
5. Analysis(optional). Indicates the analysis that will be performed on respective samples.
6. Remarks. Make comments about the individual sample(s). Example "sample container was broken and disposed of".
7. Relinquished by/received by. Indicates the individual who relinquishes possession and custody of the sample(s) and the individual accepting the possession and custody of the sample(s). Also included are the date and time of the transfer. Note that the first person to relinquish the sample must be the person who collected the sample(s).
8. Package received with Chain-of-Custody seals Intact. Analytical Laboratory Sample Custodian will check to indicate the conditions of the Custody Seals and the integrity of the contents.
9. Sample labels and Chain-of-Custody documentation. The analytical Laboratory Sample Custodian will check to indicate that all the contents of the package are in order.
10. Comments will be made about the samples inventoried on the Chain-of Custody Record (e.g., shipped via Federal Express, Airbill number).

The Chain-of-Custody Record will be initialed by the sampler, reviewed and signed during any change in custody, and QA reviewed and signed. A copy of the record will be held in the temporary field file until the original is received at the analytical laboratory. The original record will be shipped with the sample to the laboratory, where it will be QA reviewed and signed by the Sample Custodian. All Chain-of-Custody records and Federal Express Bills will be stapled together and placed in the Study File.

ATTACHMENT 2

LOS ALAMOS

TASK TEAM ACTIVITY LOG SHEET

Date (dd/mm/yy): _ _ / _ _ _ _ / _ _

Page ____ of ____

Task Team Members:

Narrative (include time and location):

RECORDED BY: _____ (Signature) QA CHECK BY: _____ (Signature)

ATTACHMENT 1

GENERAL FIELD LOOKBOOK

Table of Contents

[illegible]

ATTACHMENT 4

INSTRUMENT CALIBRATION AND TEST LOG

Instrument Name and Model: _____

Serial Number: _____

[illegible]

Sample Log Sheet

[illegible]

21

Recorded by _____
QA Check By _____

Form No.

CHAIN OF CUSTODY RECORD

[illegible]

ATTACHMENT 5

Sample Bottle Label

I-Chem Research, Inc. 23727-F Elchler Street Hayward, California 94545 (415) 782-3806		104 Quincey Blvd. New Castle, Delaware 19720 (302) 323-3806	
		(800) 316689			
SITE NAME			DATE		
ANALYSIS			TIME		
			PRESERVATIVE		
SPECIALTY CLEANED CONTAINERS					

Manual Number:

Battelle SOP Number: HMRF-I-028-01

Page 1 of 5

Key words: bubblers
AA
standards

STANDARD OPERATING PROCEDURE FOR SAMPLING AND ANALYSIS OF AIR FOR TRACES OF LEWISITE (L)

Originated by: _____ Date: _____
Research Technician
Hazardous Materials Research Facility

Approved by: _____ Date: _____
Quality Assurance Officer
Chemical/Biological Defense Department

Approved by: _____ Date: _____
Director, Environmental Safety and Health
National Security Division

Approved by: _____ Date: _____
Manager
Hazardous Materials Research Facility

Effective Date: _____

Distribution List:

Quality Assurance
SOP Manuals

Battelle
Chemical/Biological Defense Business
505 King Avenue
Columbus, Ohio 43201

APPENDIX F

STANDARD OPERATING PROCEDURE FOR SAMPLING AND ANALYSIS OF AIR FOR TRACES OF LEWISITE(L)

Manual Number:

Battelle SOP Number: HMRF-I-028-01

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I. Scope

This SOP defines the procedures used to perform environmental (air) sampling of Lewisite(L).

II. Purpose

The purpose of this SOP is to describe the sampling and analysis protocols to be used to sample environmental for Lewisite (L) at levels of $0.003 \mu\text{g}/\text{m}^3$ or greater when Lewisite testing is being performed in the HML or LITF facilities at the HMRF. This SOP is in support of contracts associated with Bailment Agreement DAAA15-89-H-0001.

III. References

Military Specifications MIL-L-10658A, 5 August 1964.

Standard Operating Procedure HMRF-II-001 (Handling CSM).

IV. Definitions

L agent Vesicant CSM, also called Lewisite. Lewisite is an organoarsenical that is extremely corrosive to skin upon contact.

V. Procedures

A. Hazards

Operators will be thoroughly familiar with hazards associated with the following:

Agents	L
Solvent	0.1N NaOH, acetone
Chemical	Arsenic

NOTE: The hazards associated with these materials are described in the Material Safety Data Sheets in the Safety Program Plan located beside each manual in each lab.

B. Equipment

Bubblers
Ice Bath
Flameless Atomic Absorption Unit (AA)
Teflon Tubing

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PREOPERATIONAL CHECKLIST

To ensure a safe operation, the following list of items should be completed prior to any operations with CSM using this SOP.

1. Ensure that staff conducting the CSM operations have read the SOP, signed page 1A attesting to the fact that they have read and understand the operation, and ensure that staff are completely trained in the operation.
2. Ensure that all staff conducting the operation understand the hazards involved with handling CSM and decontaminants.
3. Ensure that the equipment listed in Section B is readily available and in good working order.

Manual Number:

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b. Calibrate the system using the external standard method as follows:

- (1). Prepare calibration standards of As in 0.1N NaOH at levels of approximately 5, 10, 20, 50 and 100 ng/mL.
- (2). Inject 25 μ L of each calibration standard using an autoinjector and tabulate the absorbencies. Use the results to prepare a calibration curve of absorbance versus concentration.
- (3). The working calibration curve must be verified daily by the measurement of two or more calibration standards. If the response for any standard varies from the predicted response by more than ± 10 percent, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve may be prepared.

c. Analyze the samples from the bubblers as follows:

- (1). Inject 25 μ L of the sample using an autoinjector and record the resulting absorbance.
- (2). If the response for the peak exceeds the working range of the calibration curve, dilute the sample and reanalyze.
- (3). Determine the concentration of L in the sample by referring to the calibration curve, correcting for 36 percent arsenic in Lewistite.

d. Record results of analysis

VI. Quality Control

None

VII. Emergency Procedures

Any actions necessitated by emergencies conducted as described in SOP HMRF-II-001, Section V.E.19 through V.G.

VIII. First Aid/Self Aid

If physical injuries occur, first aid or self aid will be administered and the nurse located on site will be called. If extensive injury results, the ambulance will be summoned by dialing 4-5435 or 4-5438. If the telephone is inoperable or busy, the two-way radio located in the Facility Engineers office will be used.

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Manual Number:

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C. General Instructions

Air is drawn through paired bubblers containing 10 mL of 0.1N NaOH at ambient temperature at a flow rate of 2 L/min. At the end of the sampling period, the 0.1N NaOH is analyzed by using a flameless atomic absorption spectroscopic method. This procedure is selective for total arsenic. Any arsenic found in the bubbler solution is presumed to be present as a result of L in the atmosphere. L contains 36 percent (w/w) arsenic, in accordance with the chemical structure and Military Specification MIL-L-10658A, 5 August 1964.

D. Specific Instructions

1. Sampling

- a. Personnel performing the environmental sampling will wear PPE required for XDS operations as described in HMRF-II-001, Section IV.O, Minimum XDS Work Clothing (Middle Level).
- b. Fill the required number of bubblers with 10 mL each of 0.1N NaOH. (2 bubblers per sample site).
- c. Operate the bubblers in an ice bath for the required sampling times at a flow rate of 2.0 L/min.
- d. At the completion of the sampling period, retrieve all bubblers and transport them to the analytical area for analysis.

2. Sample Analysis

- a. Establish the following flameless atomic absorption (AA) spectroscopic operating parameters:

Drying Temperature:	110°C
Drying Time:	50 seconds (04 15 seconds at 200°C)
Charring Temperature:	1000°C
Atomization Temperature:	2400°C
Atomization Time:	10 seconds
Purge Gas and Mode:	Argon, interrupt
Purge Gas Flow Rate:	40 mL/min
Wavelength:	193.7 nm

7:59:40 12/29/1993

NA449
GC2016

Inject time: 10:27:17 12/23/1993
Inetrimgt+ 6 Vial 155

APPENDIX G

7:58:30 12/29/1993

NA444
CC2016

Instrument: E

Inject time: 8:32:22 12/23/1993
Vial 155

TRIP BLANK

7:59:54 12/29/1993

NA450
CC2015

Inject time: 10:49:04 12/23/1993
Inst: mgp + F
Win: 165

7:58:44 12/29/1993

NA445

Scratch method

Inject time: 8:54:58 12/23/1993

Instrument: F

Wial 155

8:00:08 12/29/1993

NA451
GC2016

Instrument: 6

Inject time: 11:12:15 12/23/1993

Vial: 165

STD 100NG HD

7:59:13 12/29/1993

NA447
Scratch method

Inject time: 9:41:00 12/23/1993
Instrument: 6 Vin1.155

7:58:59 12/29/1993

NA446
QC2016

Inject time: 9:17:22 12/23/1993
Instrument: 6 Vial: 155

SUL GL HMR

8:07:05 12/29/1993

NA462
CJ2016

Inject time: 15:38:40 12/23/1993
Instrument: F
Vial: 155

7:59:27 12/29/1993

NA448
GC2016

Instrument: A

Inject time: 10:05:04 12/23/1993
Vial: 165

8:06:50 12/29/1993

NA461
QC2016

Inject time: 15:16:41 12/23/1993
Instrument: 5

SUL GL LAM

8:07:20 12/29/1993

Detector Response mVolts

Elution Time

Minutes

NA463
QC2m16

Inject time: 16:00:56 12/23/1993
Instrument: 165

8:00:49 12/29/1993

NA454
02016

Inject time: 12:43:54 12/23/1993
Instrument: 6 Vol: 155

8:00:21 12/29/1993

NA452
GC2016

Instrument: 6

Inject time: 11:34:27 12/23/1993
Vial: 165

8:01:17 12/29/1993

NA456
002016

Inject time: 13:27:20 12/23/1993
Instrument: 6

LA48037T2

8:01:03 12/29/1993

Detector Response mVolts

0 10 20 30 40

Elution Time

8.7

8.8

8.9

9.0

9.1

9.2

9.3

9.4

9.5

Minutes

NA455

Q7716

Instrument

Inject time: 13:05:29

12/23/1993

11:21:46

8:06:20 12/29/1993

NA459

002015

Inject time: 14:33:32 12/23/1993

Instrument: 6

Vol 135

LA55044T2

8:01:30 12/29/1993

Detector Response mVolts

Elution Time

Minutes

NA457
QC2016

Inject time: 13:49:50 12/23/1993
Instrument: 5 vid 1165

05394W

June 10, 1994

ACCOUNTS PAYABLE

Los Alamos National Laboratory
Accounts Payable MS P240
PO Box 1663
Los Alamos
New Mexico 87545

'94 JUN 14 AM 11 58

Ref 324
NF
6/16
to hyn 6/17

Invoice # CU 128

Your ref: PR # 2798D0014-3W

Our ref: YU94/4/2

SARP for Package 2799E

For the following services under the above subcontract up to June 10, 1994:

Review of actions, establishing work schedule and discussions with the LANL technical officer

Review of 2999 SARP and preparing for meeting with the ANL review team

Compiling report CTRU 94/03 for the DOE Alternative PRA

Project management

36 hrs manager rate @ \$120/hr	=	\$4,320.00
10 hrs professional rate @ \$95/hr	=	\$950.00

Invoice Total

\$5,270.00

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Robert A Vaughan, President

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