DETERMINATION OF FULL-FACEPIECE, TIGHT-FITTING, NEGATIVE-PRESSURE, AIR-PURIFYING RESPIRATOR (APR) PERFORMANCE DURING DYNAMIC TESTING AGAINST CHEMICAL AGENT DISTILLED SULFUR MUSTARD (HD) VAPOR AND LIQUID CBRN STANDARD TEST PROCEDURE (STP)

1. PURPOSE

1.1 This document establishes the procedures for ensuring the level of respiratory protection provided by chemical, biological, radiological, and nuclear (CBRN) protection requirements for full facepiece, tight fitting, negative pressure, P100, CBRN Capacity X, air-purifying respirator (APR) submitted for approval, extension of approval, or examined during certification product audits, meet the minimum certification standards set forth in Title 42 CFR, Part 84, Subpart G, Section 84.63(a)(c)(d) and Federal Register, Volume 60, Number 110, June 8, 1995.

1.2 The purpose of this standard test procedure (STP) is to describe the test conditions and procedures necessary to test and certify CBRN APR certification applications. A CBRN APR is a complete tight fitting and full face blank, properly outfitted with manufacturer unique components and a properly designed manufacturer specified APR canister that is installed per the manufacturer’s installation requirements outlined in current user instructions. This procedure is used to test CBRN APR systems against distilled sulfur mustard (HD) vapor and liquid physical states, while the respirator is operated in dynamic mode by means of a breather pump connected to the breathing zone of a manikin headform. Instrumentation is integrated under this static chamber platform for the purpose of generating and controlling challenge concentrations and detecting precise agent permeation and penetration of a tested respirator. This procedure is a separate test under the NIOSH NPPTL Respirator Branch, Certification, Evaluation and Testing Section (CET) heading of CET-APRS-STP-CBRN-0351 for challenge of sulfur mustard (HD) vapor and liquid. This procedure is designed to rigorously test the evaluated respirator as a dynamic breathing system and generate repeatable independent pass or fail results under laboratory conditions.
2. **GENERAL**

2.1 This STP describes a test titled “Determination Of Full Facepiece, Tight Fitting, Negative Pressure, Air-Purifying Respirator (APR) Performance During Dynamic Testing Against Chemical Agent Distilled Sulfur Mustard (HD) Vapor And Distilled Sulfur Mustard (HD) Liquid” in sufficient detail that a team of persons knowledgeable in the appropriate technical field can select equipment with the necessary resolution, conduct the test and determine whether or not the product passes the test.

3. **EQUIPMENT AND MATERIALS**

3.1 **SMARTMAN®, Headform/Upper Torso or Equivalent (Figure 1).** Manufactured by ILC Dover, Frederica, Delaware, The **Simulant Agent Resistant Test Manikin**, a.k.a. SMARTMAN®, is a cast zinc, hollow shell headform representing a static uniform surface that outlines a medium sized human male head, neck, shoulders and upper chest stature. The head features an anatomically correct semi-static surface consisting of dimensional eyes, nose, ears, mouth orifice, forehead and chin. The features are on a movable section of the head to facilitate installing and removing a peripheral front face seal, which is made of silicone rubber and fits into a channel between the face and the permanent part of the head. The seal is inflated to press against the inside of the facepiece seal area to assure against leakage. A face seal adjustment handle allows standard mechanical pressure to be exerted for securing the face seal. The surface of the face of the SMARTMAN® is connected in several places to outside sampling ports by means of stainless steel tubing that is located inside the form and passes out the bottom. The largest tube, 1¼” in diameter, leads from the mouth area to the breather pump. Four

![Figure 1. Headform/upper torso manikin, SMARTMAN®.](image-url)
smaller tubes are present. One tube connects to the center of the left eye; it is blocked off. A tube connects to the lower middle forehead above the bridge of the nose; it is blocked off. There are two metal tubes protruding outward from the oral/nasal region, they are in use. One is used to measure differential pressure by means of a magnehelic gauge, while the second one is used to monitor presence of agent. These four tubes are ¼” in diameter. The SMARTMAN® is mounted and sealed to the floor of an exposure chamber, which is raised by four legs to allow the tubing to exit and connect to the external monitoring devices. A large channel is molded at the bottom of the SMARTMAN® to allow the anchoring of respirator system shrouds as they are intended per manufacturer instructions. Average weight of hollow zinc headform is 85 pounds.

3.2  Leak Detector Model TDA-99M or Equivalent (Figure 2). Manufactured by Air Techniques International in Owings Mills, Maryland, the TDA-99M is one of the primary tools for accessing aerosol leaks in the mechanical seals of the respirator and proper fit of a respirator to a clean SMARTMAN® headform under non-toxic oil aerosol conditions. The device generates a polyalphaolefin (Emery 3004 manufactured by the Henkel Corporation, Emery Group or equivalent) aerosol that is used to detect leakage in the respirator. With the respirator properly installed on the SMARTMAN® and the breather pump operating at manufacturer specifications, the TDA-99M generates a liquid particulate aerosol at a concentration of 100 mg/m³. This aerosol is introduced by the TDA-99M’s pump. The respirator interior is monitored for the presence of aerosol. The leak detector compares the concentration inside with the concentration outside, and calculates a digital percent penetration value.

![Figure 2. Leak detector model TDA-99M.](image)

3.3  MINature Continuous Air Monitoring System, MINICAMS® or Equivalent (Figure 3)). Manufactured by OI Analytical, the MINICAMS® is a gas
chromatograph equipped with a hydrogen flame emission detector and a preconcentrator tube. The preconcentrator tube is a small tube containing an adsorbent material to scrub out agent vapor contained in a sample of air drawn through it for a set period. The tube is then heated to desorb the agent and introduce it into the column and subsequently the detector. By preconcentrating the agent, the detection limit is lowered. The MINICAMS® unique software calculates the amount of agent detected over a specified period. The limit of detection (LOD) is equal to 20% of the 8-hour time weighted average (TWA) for the specific chemical agent being detected. Residual contamination is the amount of challenge agent lingering in the breathing zone of the respirator when a new, clean respirator is mounted on the SMARTMAN®. Residual contamination is quantified at the beginning of each test and the MINICAMS® output must remain stable for a period of 60 minutes prior to the initiation of any test. Minimum of two MINICAMS® are required in order to continuously monitor the interior of the respirator.

Note: Next generation MINICAMS® and support equipment require successful NIOSH validation testing (V-test) documentation before implementation of the next generation equipment used for NIOSH certification/approval testing.

Figure 3. MINIature Continuous Air Monitoring System, (MINICAMS®).

3.4 Syringe Pump, Sage® Model M365 or Equivalent (Figure 4). This multi-range, variable rate infusion pump is used to inject liquid agent at a controlled rate into an air stream to generate a vapor challenge. The challenge concentration can be varied over a wide range to accommodate the requirements. The liquid agent is contained in a syringe connected by a flexible cannula, a small tube for insertion into a cavity or vessel, to the dilution airline. The plunger of the syringe is driven at a controlled rate by the pump to deliver a calculated constant flow of agent. The concentration of agent is adjusted by changing the speed setting of the pump. Rate flow range = 20.0 mL/min to 0.3 L/hr, and flow accuracy of ± 5% nominal. Technicians must be proficient on the pump’s user instructions.
Figure 4. Syringe pump, Sage® Model M365

Note: Next generation syringe pumps or equivalent equipment and support equipment for data logging/automation, require successful NIOSH validation testing (V-test) documentation before implementation of the next generation equipment used for NIOSH certification/approval testing.

3.5 Flow-Temperature-Humidity Control System or Equivalent (Figure 5). Manufactured by Miller-Nelson Research, Inc., this system, referred to as the Miller-Nelson® Control System, is an automated system to control the airflow, temperature, and humidity of an air supply for an operating respirator system. Laboratory specified air and distilled water are supplied to the unit; the three sensors and controlling mechanisms are incorporated electronically, and the unit output is air of the required volume/flow (50–200 L/min ± 2%), and relative humidity (20%–80% ± 3%) and temperature (20°C–30°C ± 0.3%).

Figure 5. Miller-Nelson® flow-temperature-humidity control system.

3.6 Exposure Chamber or Equivalent (Figure 6). The respirator exposure chamber is constructed of clear, chemical resistant material (Plexiglas® or Lexan®) or other
equivalent material. The floor must be constructed efficiently to support the 85-pound SMARTMAN®. The front panel is removable and is held in place with clamps on each edge. The dimensions are approximately 2 ft². Four legs made out of the same chemically resistant material are attached to the bottom to hold the chamber above the floor of the hood and allow room for laboratory tubing and the face adjustment handle. A M12A1 military specified air-purifying canister is installed on top of the chamber to filter the air that passes out of the chamber. There are ports in the sides to accommodate tubing for challenge concentration and clean purge air mixtures. An electric fan is installed near the top front to achieve a mixed challenge concentration. A clean exposure chamber is a unit used to perform a fit and leak check on a respirator test unit (no agents are used in this chamber) before it is installed in the agent exposure chamber. An agent exposure chamber is a chamber used to perform testing of the respirator unit, using live agent.

3.7 Agent Mixing Chamber or Equivalent (Figure 7). This chamber is fabricated of PVC pipe, with caps on both ends and three baffles fixed inside to ensure mixing of chemical warfare agent vapor and air. A pressure gauge, mounted on the mixing chamber, indicates internal mixture pressure and serves as a safety pressure indicator. Maximum pressure is indicated per laboratory standard operating procedures. A heating blanket is wrapped around the chamber to facilitate chemical vaporization. This is the primary mixing area that allows the chemical agent syringe pump flow and the regulated airflow from the Miller Nelson controller to mix and generate a specified concentration of chemical warfare agent. When the mixture is not being passed into the exposure chamber for a test, it is passed through a scrubber filter (an M18 military specification canister or equivalent).

Figure 6. Exposure chamber.
3.8. Breather Pump, Model E1R1® or Equivalent (Figure 8). Manufactured by Jaeco Fluid Systems, Inc., it is a breather pump used to replicate breathing. It is a double pump, operated by a single electric motor. The pump design is a modified variable speed motor generating varied strokes per minute. Planetary gears and a Scotch Yoke, producing a sinusoidal breathing pattern, control the pump. The sinusoidal pattern starts at zero flow rate, rises to peak flow of approximately $\pi$ (3.1416) times the rated test certification flow rate in L/min and drops back to zero. The exhalation stroke of the pump is the same sinusoidal pattern. The volume per breath or tidal volume is adjustable up to 1.5 liters.
3.9 Mass Flow Controllers or Equivalent. Manufactured by Tylan Electronic or Brooks Instruments. Mass flow controllers are used to control the flow of sample to the MINICAMS® and the flow of laboratory air to flush out the exposure chamber when the agent challenge is removed. The mass flow controllers are sized to meet the flow requirements. Flows are controlled to ± 2% of set point.

3.10 Ambient Air Analyzer, MIRAN® Model 205B and Model 1A, or Equivalents (Figures 9 and 10). Manufactured by Thermo Environmental Instruments, Inc. in Franklin, Massachusetts, these are infrared absorption based detectors that uses a long path length cell up to 20 meters, into which the air sample is introduced. The Model 250B is used to monitor the challenge concentration of the vapor and liquid phases of HD. The Model 1A is reserved for analyzing the Sarin (GB) concentration.

NO PHOTO CURRENTLY AVAILABLE ON MODEL 205B

Figure 9. MIRAN® analyzer, model 205B.

NO PHOTO CURRENTLY AVAILABLE ON MODEL 1A

Figure 10. MIRAN® analyzer, model 1A.

3.11 Respirator Systems Required for Testing:

3.11.1 Required Quantities: Quantity of respirators for HD Live Agent Testing (LAT) is per NIOSH/NPPTL CBRN APR Statement of Standard, Table 7, Test Sequence and Quantity Matrix. Overall HD LAT quantity is three complete respirator systems. An additional three respirator systems are reserved for the Sarin (GB) LAT. One respirator system is tested as a qualifier in this STP and is commonly known as the Qualifier Live Agent Test (QLAT). Remaining two respirator systems are tested in sequence and are commonly known as the Remainder Live Agent Tests (RLAT). In the case of canisters, 125 canisters only, go through hot, cold, humidity, vibration and 117 of the 125 are manually dropped as a final conditioning action before being tested in one of five types of service life tests. LAT penetration and permeation testing is one of those five types of service life tests for CBRN APR protocol. Initially, one respirator is required in a HD “Qualifier Application” “as is”. Overall, of the two total “as is” qualifier respirators, one is live agent tested against HD and the other is reserved for live agent testing against GB. Then, four environmentally conditioned canisters come out of environmental testing and of those four canisters, two go to HD RLAT and two go to GB RLAT utilizing environmentally conditioned like item respirators. In other words, two CBRN APR “as is” candidate respirators are live agent tested first before or in parallel to any subsequent CBRN APR statement of standard tests commence. The two
Qualifier respirators must each pass their respective LAT under an initial Qualifier Application to proceed on in the remaining test protocol. Upon confirmation that the Qualifier Application is a success and completion of environmental conditioning, remainder LAT is completed by randomly selecting Task Number respirator systems for follow on remainder LAT, GB and HD, in accordance with *CBRN APR Statement Of Standard*, Paragraphs 4.7 and 4.9. Respirators designated as spare queue conditioned respirators are stored and available for use by the testing laboratory in situations where ongoing test processes are compromised by installation power failures, inaccurate test processing or other mid-test termination events identified by the test laboratory manager. In most cases, spare queue respirators are not to be used for additional certification testing because the applicant or manufacturer requests it. NIOSH should be informed of all testing of spare queue inventory conducted by the testing laboratory.

3.11.2 Qualifier LAT: HD LAT canisters and face blanks should be received from the appropriate NIOSH/NPPTL sanctioned source in “as tested or as is” condition. No alterations to the environmentally tested canisters are authorized prior or during live agent systems testing. At the NIOSH tenant laboratory initial certification qualifier application inventory, canisters received “as is” in the ready-to-use/minimum use configuration packaging are required to be unopened, regardless of type of original packaging, and delivered to LAT lab upon completion of satisfactory certification inventory.

3.11.3 Remainder LAT: Commencement of HD RLAT is contingent upon testable canisters completing all required NIOSH/NPPTL CBRN APR environmental factors testing and processing prior to any live agent testing. Environmentally pre-conditioned canisters are attached, per manufacturer instructions, to available compatible application faceblanks prior to live agent testing by qualified and designated laboratory live agent testing technicians only. Environmentally pre-conditioned canisters should be received in minimum packaging configuration and administratively labeled with the start and end dates of environmental testing, the type of damage, if any, observed, the manufacturer, the name of the individual responsible for the environmental testing including a contact phone number and the laboratory’s name. Only the manufacturer’s inlet and outlet plugs that come with the canister are to be used to seal up the canister after environmental testing. If no plugs are provided, no other form of sealant is authorized. Start of live agent testing must commence within 12 hours of receiving the environmentally conditioned canisters and facepieces. Any canister damage during environmental
testing/processing is required to be preserved, not altered and noted on administrative markings for shipment to LAT lab.

3.11.4 HD live agent systems test canisters and respirators should be labeled prior to actual live agent testing with the following administrative information:

— NIOSH task number (TN number)
— Model of APR
— Other routine information that will allow the lab to accurately track receipt, time in test, test results, noted observations, required retest, status of testing, disposal and trend analysis is required to be managed and available for review upon request by current representative of NIOSH/NPPTL.

NOTE: Accurate labeling of post-tested respirators is mandatory in support of post-test incident investigations.

4. TESTING REQUIREMENTS AND CONDITIONS

4.1. Any laboratory using this procedure to supply certification test data to NIOSH will be subject to the provisions of the NIOSH Supplier Qualifications Program (SQP). This program is based on the tenets of ISO/IEC 17025, the NIOSH Manual of Analytical Methods and other NIOSH guidelines. An initial complete quality system audit and follow on audits are requirements of the program. Additional details of the program and its requirements can be obtained directly from the Institute.

4.2. Precision and accuracy (P&A) must be determined for each instrument in accordance with laboratory procedures and NIOSH/NPPTL guidance. Sound practice requires, under NIOSH Manual of Analytical Methods, demonstrating a tolerance range of expected data performance of a plus or minus 25% of a 95% confidence interval of the stated standard requirement. NIOSH/NPPTL P&A tolerance can be higher but not lower.

4.3. Prior to beginning any testing, all measuring equipment to be used must have been calibrated in accordance with the manufacturer's calibration procedure and schedule. At a minimum, all measuring equipment utilized for this testing must have been calibrated within the preceding 12 months using a method traceable to the National Institute of Standards and Technology (NIST).

4.4. System Test Conditions:

4.4.1. Breathing Machine:
— Airflow = 40 liters per minute (Lpm)
— Respirations = 36 ± 2 strokes per minute
— Tidal Volume = 1.1 liters

4.4.2. Miller-Nelson® Airflow Settings into Exposure Chamber:

Airflow Rate: The actual value here is experiment/method dependent. The value eventually used must be documented. Determine an appropriate airflow rate based upon obtaining a stable concentration and upon the volume of the exposure chamber, considering the agent challenge concentration desired, volume of chamber, breather airflow, and dilution air exhaled from the APR.

Relative Humidity: 50% ± 5% RH.
Temperature: 25°C ± 3°C

4.4.3. SMARTMAN® Sampling Point:

Breathing zone sampling point is a single SMARTMAN® nasal port that allows sequential dual MINICAMS® detections.

4.4.4. Mustard (HD) Exposure Test:

4.4.4.1. HD vapor: General.
— Vapor Challenge Concentration = 50 mg/m³ ± 10%.
— Vapor Challenge Time (disseminator on) = 30 minutes, but no longer than 31 minutes.
— Monitor & Hold Time = 7.5 hours.
— Total Test Time = 8.0 hours.
— Syringe Injection Rate = as required based upon syringe pump used.
— Syringe Injected Volume = as required based upon syringe pump used.

4.4.4.2. Mustard (HD) agent vapor generated is required to be Chemical Agent Standard Analytical Reference Material (CASARM) grade. Agent purity analysis must be NIST traceable, documented and meet CASARM agent purity requirements. Proper CASARM storage requirements per local regulation are required to be adhered to.
4.4.4.3. Vapor test termination is at the indicated 8 hours plus required check shots before and after entire test period. In the interest of conserving agent quantities and preserving technician personnel, a LAT is terminated if at any point in the entire 8-hour period, the respirator detection methods for the breathing zone can show repeatable system failure within the pass or fail protocol described in this current STP. This failure is required to be witnessed by a minimum of two qualified laboratory personnel and noted in the laboratory journal for follow on documentation.

4.4.4.4. Vapor challenge concentration exposure will start immediately after the test chamber has been closed and sealed properly.

4.4.4.5. **HD liquid: General.**

*Drop size:* 20 microliters (µL).

*Liquid challenge concentration:* Minimum is 0.43 mL (thirty-two 20 µL drops) and maximum is 0.86 mL (forty-three 20 µL drops) depending on type of respirator tested.

*Droplet allocation:* Thirty-two drops are used for chin and cheek style respirators with or without hydration facility and one single connected canister. Forty-three drops are used for a non-facepiece mounted APR, which consists of 32 droplets on the faceblank area and 11 droplets on components such as breathing hose(s), multiple canisters, communications device interface, electrical power interface and other noted critical interfaces as determined by NIOSH/NPPTL during configuration analysis.

*Droplet schematics:* Three NIOSH/NPPTL schematics of HD agent droplet placement for CBRN APR application (Facepiece mounted and non-facepiece mounted) are enclosed.

The listing is as follows:

Appendix A: Chin style, biocular, no drink component.

Appendix B: Chin and cheek style, monocular, and drink Component.

Appendix C: Chin and cheek style, biocular, drink component and communications interface.
External shroud or hood: Use of an external shroud or hood attached to the facepiece does not represent the worst case configuration and will therefore be removed, if possible, prior to LAT. Facepiece second or third skins layers must be explicitly specified in the application submission as a required part of the CBRN APR configuration prior to LAT. If they are not, the worst case that will be tested is with the second skin removed. If a hood or shroud is permanently attached to the facepiece, the respirator requires contamination droplets to be placed on those areas of the shroud covering the areas of the depicted facepiece diagram. Droplet application on a shroud stresses the same facepiece areas identified for the non-shrouded facepiece but instead of applications being on bare facepiece material they are on the shroud covering that depicted area/seam/joint/interface. All other defined criteria apply.

Vapor time: The application of vapor HD droplet starts the official total test duration time from when the first droplet is applied and MINICAMs are actively sampling at this time. HD vapor challenge time is a different subset time management of the total test time of 8 hours and could be managed independent of the total test time to allow accuracy of HD vapor and liquid exposure.

Liquid time: HD liquid agent is applied to respirator at the 6th hour during the test procedure. Total time that CBRN APR candidate system is exposed to both HD vapor and HD liquid (Liquid Time) is 2 hours, the last 2 hours of the total 8-hour test time period.

4.4.4.6. Mustard (HD) agent liquid is required to be Department of Defense, US Army CASARM grade. Agent purity analysis must be NIST traceable, documented and meet US Army CASARM agent purity requirements. Proper CASARM storage requirements per local regulation are required to be adhered to.

4.4.5. Test Termination Parameters: The termination of the HD liquid application test is in accordance with the total liquid/vapor test duration time of 8 hours plus the before and after test check shots. The test will be terminated to protect the detectors from over-saturation provided a candidate APR shows failing criteria prior to the 8-hour mark. The termination point should be set at or above the highest calibration point for each one of the MINICAMS® in use.
4.5. Safety and Training. Normal laboratory safety practices are required. Laboratory specific regulations such as US Army Regulation 50-6, Chemical Surety apply as required. The practices include all safety precautions described in the current Centers for Disease Control and Prevention (CDC) General Laboratory Health and Safety Manual, the applicable US Army Regulations, the U.S. Army Soldiers and Biological Chemical Command Laboratory Safety Procedures, and any other equivalent manuals and periodicals.

4.5.1. Safety glasses, lab coats, assigned respirator, butyl apron and butyl gloves are required be available, worn and replaced as laboratory standard operating procedures apply.

4.5.2. Work and walking surfaces must be maintained free of clutter and non-essential test equipment.

4.5.3. When handling any glass laboratory equipment, lab technicians and personnel must wear approved gloves, which are rated appropriate in accordance with current safety and hygiene plans.

4.5.4. Laboratory personnel are required to be trained on this STP and documentation is required to be available for review of said training. Personnel are required to be trained and qualified per local requirements in all applicable standard operating procedures (SOP) appropriate for the test.

4.5.5. The responsible technician must be knowledgeable about the specific CBRN APR being tested and should be able to readily identify all CBRN APR hardware components, subassemblies and accessories and be able to support accurate trouble shooting of the CBRN APR and subassemblies to ensure it is operating in a correct negative pressure mode. The responsible technician must be able to properly align and fit the respirator facepiece on the SMARTMAN® headform without malformation or destruction of any CBRN APR material.

4.5.6. All LAT equipment passing or failing these NIOSH test protocols will be treated as hazardous materials in accordance with local laboratory procedures and methods. Disposal of such contaminated CBRN APR materials is the responsibility of the testing laboratory. In accordance with this STP, respirator manufacturers are released of equipment accountability once equipment is formally tested in the appropriate NIOSH recognized test laboratory. Manufacturer warning and caution statements for the respirators are subject to legal interpretation and most likely will not apply once the respirator has been contaminated by toxic chemical warfare agents and processed thru the decontamination procedure.
4.5.7. Surety lab procedures outlined in applicable SOPs are required to be on hand. Annual training classes focus on the familiarization of required occupational safety and health subjects in accordance with specified surety lab procedures. Refer to appropriate material safety data sheets, manufacturer’s instructions and available current health and safety manuals, or other appropriate documentation for the proper protection and care in handling, storing, and disposing of the contaminated respirators, canisters, subassemblies and chemicals used in this procedure.

4.6. Equipment Pre-Test Conditions: MINICAMS®

4.6.1. Background Reading: A laboratory MINICAMS®, as opposed to a field MINICAMS®, is used as the detector for agent permeation and penetration of the CBRN APR. It consists of a monitor, PC computer, linear mass flow meter and optional printer or recorder. Before the APR is placed on the exposure SMARTMAN®, the MINICAMS® are required to show steady state background readings lower than the lowest point on the current MINICAMS® calibration curves. This is accomplished per local laboratory procedures but a clean M40 mil spec respirator mounted on the SMARTMAN® head form may be used. The SMARTMAN® is then allowed to breathe for the required time (8, 12, 48, 72 hours etc.) until a steady state background is achieved that is lower than the lowest current calibration curve point indicated on the respective MINICAMS® performance results. The LAT for certification is not authorized to start without the background readings of the assigned SMARTMAN® system (tested respirator and headform) being within the limits specified above.

4.6.2. Unit of Measure. A small volume of air is drawn through a pre-concentrator tube containing an adsorbent material: HD sample volume is per the determined value. Agent in the sample is adsorbed on the material. Later in the cycle, the tube is heated to desorb the agent, which then flows through a gas chromatograph column to a flame emission detector. Because the total agent in the sample is detected at one time instead of continuously, the detection limit is much lower. The total quantity of agent detected is calculated back to the sample volume and is expressed as ng/L.

4.6.3 Sample Cycle. Operation of the MINICAMS® requires the use of compressed house air, hydrogen, and nitrogen, of a high purity. The operating manual recommends operating parameters (temperature, timing, pressures, etc.) and cycle times (3, 5, 10 or 15 min.), depending on the laboratory application. CBRN APR HD analysis requires a 6-minute cycle. The two MINICAMS® are required to be synchronized so that one MINICAMS® sampling begins when the other MINICAMS® sampling ends. Raw data results confirm this requirement.
4.6.4. HD Detection Principle. The MINICAMS® is installed in accordance with the operating manual and local lab SOP. The appropriate optical filter for HD must be installed in front of the photomultiplier tube. In principle, when HD burns in a hydrogen flame, a chemical element (sulfur) is formed that emits radiation at a unique wavelength. The optical filter isolates the radiation and allows it to pass into the photomultiplier tube (PMT), whose output voltage is correlated with the quantity of agent burned in the flame.

4.6.5. Standardization. MINICAMS® are configured in accordance with the operating manual and the specific method for the chemical warfare agent that is being used. In order to quantify the agent in the sample, the MINICAMS® must be standardized. Standardization is accomplished by injecting a small quantity (1.0 or 2.0 µL) of a known standard solution of the agent onto the pre-concentrator tube during the INJECT segment of the test cycle. The standard solutions of agent are made in spectrophotometric grade isopropanol. At least three injections of each quantity of agent should be injected per LAT sequence.

4.6.7. Pretest activities for the MINICAMS® are as follows:
— Start the MINICAMS®
— Set or verify operational parameters for appropriate agent
— Perform standardization
— Record ASCII file name on Data Sheet
— Standby for start of testing

4.7. Equipment Pre-Test Conditions: SMARTMAN® HD Vapor Generation and Liquid Application

4.7.1. Vapor Concentration. The vapor challenge for SMARTMAN® testing is generated by injecting the required quantity of liquid agent into the volume of air that passes through the exposure chamber to give the challenge in mg/m³. This is accomplished by a combination of controlled airflow from the Miller-Nelson air controller and a syringe pump for injecting the agent through a heated “tee” into the air stream. Determine the volume of air needed to pass through the exposure chamber per minute (flow rate) and the quantity of agent necessary to give the specified challenge concentration for this flow rate, taking into account the volume of air discharged into the chamber from the exhalation air of the APR.

Ramp-up Time: Conditions for each individual system will have to be determined for each laboratory setup to achieve the required ramp up time
for CBRN APR exposure concentration. The ability to accurately detect and quantify this agent ramp up time is a requirement for the testing laboratory and ramp up time and agent duration exposure time graphs should be available to confirm agent exposure duration in accordance with the procedures of this STP.

4.7.2. Concentration Verification Summary:

- Chamber size = 8 ft³
- Target Vapor Challenge Concentration = 50.00 mg/m³ ± 10%.
- Ramp-Up Time = 1–2 minutes (Time from initial start of syringe pump to 50 mg/m³, lowest acceptable challenge concentration.)
- Dilution Air from Breather Pump = 40 L/min
- Miller-Nelson® Challenge Mixing Air = 50 L/min
- Example: Syringe Rate for Challenge Injection = 1.0 mL/min

4.7.3. Agent Dispersion. From the pump operating manual, select the syringe size and pump rate that will inject the required amount of agent per unit time. Draw up the total amount of agent needed into the syringe, with a small excess, connect one end of the cannula (the small flexible tube that is inserted into the air duct) to the syringe, and the other end to the heated tee in the air duct. Clamp the barrel of the syringe onto the pump and move the plunger drive until it contacts the end of the plunger. Turn on the power to the pump. The plunger will be activated and agent will be injected into the air stream as a vapor from the heated tee. The mixture passes into the mixing chamber where it is thoroughly mixed, ready to be introduced into the exposure chamber for the test.

4.8. Equipment Pre-Test Conditions: Miller-Nelson® Controller

4.8.1. The Miller-Nelson® unit receives compressed air from the laboratory house air supply system. Operate the Miller-Nelson® according to the manufacturer’s instructions. The sensors for relative humidity and temperature must be calibrated, as well as the flow controller, since it is important that the total flow through the test system be known in order to
supply the requisite amount of agent from the syringe pump. Insure the total flow value is logged in the technician’s lab logbook prior to commencing each actual LAT.

4.8.2. Set the readout panels on the Miller-Nelson® according to paragraph 4.2.2. Ensure the Miller-Nelson® is properly configured for current test procedure and all required airlines are secure to inlet ports of SMARTMAN® and Miller-Nelson® systems. Allow the clean air to flow through the mixing chamber and the M18 filter until it is time to start the test.

4.9. Equipment Pre-Test Conditions: Syringe Pump

4.9.1. A syringe pump is used to inject liquid agent into the dilution air stream at a controlled rate such that the concentration of agent in air is that required for the challenge specified for the test. Manual setting of the syringe pump controls allows the pump rate to be changed by using a turn knob.

4.9.2. Select the size syringe that will hold sufficient agent for the challenge period and the total volume of air required. Fill the syringe to the volume determined and attach the syringe to the fitting on the flexible cannula. The cannula is normally made of plastic with Luer locks on each end. One end of the cannula is attached to the heated tee in the dilution airline. Set the syringe in the holder and clamp it in place. Move the drive block until it is firmly against the end of the plunger.

4.9.3. Set the switch on the pump to the setting required for the size syringe and the injection rate. Turning on the power switch will start the drive block pushing the plunger of the syringe to begin generating the agent challenge concentration. Turning off the power switch will stop the drive block from pushing against the plunger and stop the challenge agent concentration flow at the predetermined time.

4.10. Equipment Pre-Test Conditions: TDA-99M Aerosol Leak Detector

4.10.1. The TDA-99M leak detector is used to detect oil particulate aerosol leaks into the CBRN APR after it has been installed on the SMARTMAN® headform cold box. Ensure the canister and all components are correctly mounted and tightened per the current manufacturer instructions and specifications. The APR is operated under negative pressure using air supplied to the APR from the Miller-Nelson® filtered house air of the laboratory. Ensure that procedures for the APR follow the manufacturer’s installation procedures for gasket seal and facepiece donning and interface. The canister and gasket, if provided, should be the same product that has undergone recent NIOSH/NPPTL environmental factors testing or
in the “as is” ready-to-use condition. Ensure the seal, threaded interface, inlet and outlet areas and canister housing are not changed in any manner prior to or during the LAT. However, if the canister or canister interface thread is deformed from environmental testing, continue with the test by attaching the canister per manufacturer instructions. Do not make any corrections for canister deformities at this time. Ensure the lab book is annotated and digital photos are taken and available for follow up incident review if necessary.

4.10.2. Turn on the power and let the leak detector equilibrate, according to the manufacturer’s instructions. The APR should be in static non-breathing mode. Turn on the breather pump to activate the negative pressure test respirator. Connect the detector inlet to a sample line from the SMARTMAN®. When aerosol is being generated, direct the wand to various portions of the facepiece and all mechanical seals or joints to detect any leak paths. If no localized leaks are found, replace the front panel of the exposure chamber and start the actual TDA-99M test.

4.10.3. Connect the TDA-99M to a port into the exposure chamber and fill the chamber with aerosol. Maintain a constant aerosol concentration inside the exposure chamber for 30 minutes. Check the display on the TDA-99M for detection of aerosol inside the facepiece. When the detector indicates a maximum penetration of less than 0.0010 % for 30 minutes, continue with the next item in the LAT procedure. If there is evidence of leakage, attempt to find and eliminate the leak. If a leak is detected during the process isolate the canister by using a dedicated clean Teflon tube to draw clear air from outside the chamber in accordance with local laboratory SOP. Ensure Teflon tube data and configuration drawing is annotated in lab book.

4.11. Equipment Pre-Test Conditions: Quality Control Measures

4.11.1. SMARTMAN® Leak Test: Because the SMARTMAN® is made of cast zinc, it is possible for leak paths, after extensive LAT or incorrect assembly, to form through the metal casting, allowing chemical warfare agent vapor to pass through the headform cavity into the interior of the respirator mounted on the headform. To check for these invisible leak paths, install a clean peripheral seal on the headform and inflate it to maximum pressure of 3 psi. Flood the interior of the headform with a known rated helium concentration and purity. Use the probe of the helium leak detector to check the entire surface and the seal for presence of helium. Any leak found by the helium leak probe procedure must be diagnosed and eliminated, if possible. The leak test is to be performed initially on each new or reconditioned SMARTMAN® and monthly on the SMARTMAN® headforms when they are in continuous daily use.
4.11.2. Standardization of Instrumentation: Standardize the MINICAMS® by using liquid standard solutions of the agents at various concentrations. These solutions are to be made in accordance with US Army, *ECBC CAT IOP #214, Preparing Standard Agent Solutions for Instrumentation*, or equivalent laboratory procedures. A stock solution is the primary solution made by weighing a quantity of agent into a volumetric flask and diluting to volume. This solution may be used for two weeks, unless deterioration is noted before that time. The stock solution is diluted further to make a series of standard solutions that are used to standardize the MINICAMS®. The standard solutions may be used for one week, unless MINICAMS® analyses indicate that the solutions are deteriorating. Class A glassware must be used for all volumetric work. Calibration curves should have a minimum correlation of $r^2 = 0.99$ for HD. Agent solutions must be stored at 4°F or lower.

4.11.3. Calibration of Flows: Since flow rates are used in several aspects of this test, it is necessary to use calibrated flow meters to set the flows used in the instruments. Flow meters are calibrated by the US Army Test Methods & Development Equipment (TM&DE) and Metrology Laboratory, in accordance with ISO 17025 procedures or equivalent and use instruments traceable to NIST. Flow meters to be checked against calibrated meters are the Miller-Nelson® airflow controller, all electronic flow meters used for the MINICAMS® preconcentrator tube and the flow meters from the breather pump and the syringe pump agent injector.

4.11.4. Aerosol Leak Testing: APR leak testing using the TDA-99M aerosol tester is performed on the facepiece after installation on the SMARTMAN® and while the breather pump is operating. Allow the TDA-99M to stabilize in its initial detection procedure. When readings are stable within ± 2 end place digits of 0.0000, the TDA-99M aerosol tester is ready to begin detecting potential leak paths. If there is no leak, the display on the TDA-99M should read 0.0000% penetration.

4.11.5. MINICAMS® Detector Response: Check the response of the MINICAMS® detector, before and after each LAT. This is done by injecting an aliquot of standard solution that contains a known concentration of agent near the mid-range of the standard curve. Inject the aliquot into the end of the heated sample line from the oronasal sampling port; it is necessary to disconnect the line from the bottom of the chamber to do this. This is called a “Check shot”. Repeat it at the end of the test to assure that the detector response has not changed during the test. The response of the detector should fall on the standard curve at the value expected for the amount of agent in the aliquot, or within 10% of that value. Record results of check shot in laboratory notebook. If the check
shot does not reproduce a verifiable result, repeat the check shot. If the second check shot is not within the 10% parameter, the test is invalid. A quantifiable check shot should be made at the maximum, within 3 hours of the start of the actual test and at the APR end of test 8-hour mark.

5. **PROCEDURE**

5.1 **HD LAT (0351)**

5.1.1 Assemble APR per manufacturer’s instructions. Ensure the packaged canister is received as specified in paragraph 3.11 and CBRN APR Statement of Standard, Paragraph 4.10, Table 7, Note 2. The last note here identifies the canisters only being in being in the “minimum manufacturer’s recommended packaging”, which is interpreted as the ready-to-use assembly for the canister. Facepieces will be tested “as is” when submitted in the qualifier application, conditioned in the remainder application and married up with appropriate ready-to-use or conditioned canisters in accordance with CBRN APR Statement of Standard.

5.1.2 Take digital photographs of the assembled unit prior to start of LAT. These photographs are required to be available for NIOSH/NPPTL review and they should accurately and clearly indicate how the actual manual placement of the HD droplets occurred at the 6-hour mark, prior to vapor testing re-commencing.

5.1.3 Mount the respirator on the SMARTMAN® in the clean exposure chamber. The facepiece should be mounted to the SMARTMAN® per the manufacturers’ operating user instructions with special emphasis on head harness fitting and canister tightness. Ensure all parts of the facepiece are mounted and seated correctly on the headform. If a shroud or other accessory is being tested as part of the respirator system, ensure that they are mounted properly and serviceable. If batteries are required for communications devices, ensure they are in place and the communications device is turned on. Monitor the communications device for malfunctions during the course of the test and visually check it and other accessories at the termination of the test for visible cracks or breaches of the airflow boundary i.e., compromises of form, fit or function.

5.1.4 Turn on the breathing pump. Insure that the APR is in the negative pressure mode by conducting negative pressure seal check(s) with protected hand over required surfaces of the tested respirator per on hand current manufacturer operating instructions or use instructions. Use the integrated SMARTMAN® magnehelic pressure gauge as required to support confirmation of negative pressure cycles. Magnehelic pressure gauge should be used only as a qualitative indicator of APR performance
and it can serve as a tool for observation purposes. Do not allow the tested CBRN APR to go into or maintain positive pressure for any reason such as misdirected house air from air flow tubing cracks, kinks or failure.

5.1.5. Using the TDA-99M, leak test the APR in the negative pressure mode. Connect the detector inlet to a sample line from the SMARTMAN®, allow the APR to breathe, and create a stable value on the TDA-99M. When the aerosol is being generated, direct the wand to various portions of the facepiece and all mechanical seals and joints to detect any leaks. The aerosol will be detected inside the facepiece if it finds a leak path. If any leaks are found, they must be corrected by the authorized laboratory technicians or manager only. If a leak is found through the canister, connect a clean airline from outside the exposure chamber. If no localized leaks are found, replace the front panel of the exposure chamber. Connect the TDA-99M to a port into the exposure chamber and fill the chamber with the aerosol challenge. Maintain the aerosol challenge inside the chamber for 30 minutes of continuous TDA-99M operations below 0.0010% penetration. If penetration exceeds 0.0010%, the TDA-99M alarm sounds or the digital readout shows higher values, stop the 30-minute test, re-analyze the system and begin a new 30-minute test period. The APR must pass a continuous 30-minute test at \( \leq 0.0009\% \) penetration parameters in separate clean exposure chamber and an agent exposure chamber to be considered qualified to progress on in the agent testing. If after repeated attempts a successful leakage test cannot be achieved, the laboratory manager may use alternative means to seal the facepiece to the headform such as sealing the facepiece and nosecup to the headform using non-toxic adhesive with the concurrence of the NIOSH/NPPTL. All adhesive designated for CBRN certification use is required to undergo verification testing (V-test) prior to use as a CBRN LAT capable substance successful performance to the given STP using three consecutive V-test trial samples. If the respirator continues to fail the TDA-99M, the manufacturer must supply confirmed MSDS on the type of particulate being picked up by the TDA-99M. In addition, the manufacturer must obtain an analysis by a third party laboratory confirming the manufacturer’s MSDS, the particulates must be determined to be non-toxic in the acute and chronic human toxicology perspective.

5.1.6. Before the APR facepiece can be placed on the SMARTMAN® in the agent exposure chamber, the MINICAMS® must have shown a steady state background lower than the lowest point on the calibration curves. This is accomplished by installing a M40 mil spec respirator on the SMARTMAN®. Ensure a new or sanitized decontaminated M40 respirator is used. The SMARTMAN® should be allowed to breathe until
the steady state background is lower than the lowest calibration curve point indicated on the respective MINICAMS®.

5.1.7. Remove APR from clean exposure chamber and install on SMARTMAN® in the HD agent exposure chamber.

5.1.8. Mount the respirator on the SMARTMAN® in the agent exposure chamber. Conduct a TDA-99M 30-minute test by repeating steps of paragraph 4.8, with the exception that once the agent exposure chamber is sealed and no leaks are confirmed with the TDA-99M, the TDA-99M aerosol challenge is discontinued. Ensure the agent exposure chamber is purged of Emery oil particulates for at least 15 minutes. Insure that the removal of the TDA-99M test hardware does not disturb the established seal of the APR facepiece to the SMARTMAN® system. Remove the clean airline from the canister system, if it was used to isolate leaks.

5.1.9. Turn on the Miller-Nelson® flow, temperature and humidity controller.

5.1.10. MINICAMS® Background Characterization. A background characterization must be run before every agent test. The MINICAMS® should be monitored for a period of 60 minutes prior to the initiation of the chemical agent warfare test. Confirm that background level is less than the lowest point on the MINICAMS® calibration curve. If the background level is not less than required, trouble shoot the SMARTMAN® system, do not start the test, advise laboratory manager and if necessary remove the APR facepiece, decon the headform, re-don the facepiece and restart the procedure for characterization as necessary. If the APR is removed from the head form, restart the test procedure from paragraph 5.1.3.

5.1.11. Set up standard operational mode of test equipment. Ensure all test equipment is within calibration.

5.1.12. Set Miller-Nelson® for airflow into exposure chamber set to deliver required rates. See paragraph 4.4.2.

5.1.13. Load liquid agent in syringe and set syringe pump to correct flow rate to achieve the required agent challenge concentration. See paragraph 4.7.

5.1.14. Ensure MINICAMS® are calibrated, the check shot is complete and acceptable and ready for the operating mode. Annotate the check shot times and concentrations.

5.1.15. Ensure that the challenge concentration instrument is calibrated and is ready for analysis. Monitor agent exposure chamber during the 60 minutes
background characterization period. Characterization reading should reflect a steady state condition.

5.1.16. HD Vapor Application:

5.1.16.1. Time zero plus 1 second or start of the agent test is when the first confirmed vapor state of HD is applied to the interior of the agent exposure chamber while the MINICAMS® are actively sampling. Vapor is pumped in for 30 minutes, turned off and agent decay cycle is monitored for 5.5 hrs. At the 6-hour mark, pre-defined liquid droplets are applied per the exact enclosed droplet appendix or a NIOSH approved modified droplet appendix. Hotbox is closed back up as fast as possible and remaining test cycle time and detection operation proceeds up to the 8-hour mark. Droplet application should be expeditious, safe and done in a timely manner. Annotate the time zero plus 1 second and the HD liquid application time period and liquid exposure time. Visual observation of the tested APR for the last 2 hours of testing is highly recommended.

5.1.16.2. The 30-minute vapor exposure begins when HD vapor is injected into the HD exposure chamber. Annotate this starting time. Ensure time management is accomplished that allows the efficient tracking of two simultaneous events: one clock for total test time from start of HD exposure for 8 hours and another clock for HD vapor at 30 minutes. If necessary, the vapor clock can convert to the liquid agent clock at the 6-hour mark.

5.1.16.3. Introduce HD vapor agent challenge to the agent exposure chamber. Turn the valve inline from bypass of the mixing chamber to direct flow from the mixing chamber to the internal ambient space of the agent exposure chamber. The total flow to the chamber is approximately 50 L/min from Miller-Nelson®, which includes enough excess to make up for the clean air exhausted from the mask into the chamber and maintain the constant challenge as required. The concentration of challenge agent monitored using the appropriate MIRAN® detector. The syringe flow rate is set to introduce the quantity of agent necessary to generate the challenge concentration required. Record when the MINICAMS® begin monitoring the interior of the mask. It is required to start the MINICAMS® before introducing agent into the chamber so that the first sampling period will coincide with the first agent challenge.
5.1.16.4. The vapor syringe pump should run for the prescribed challenge period of 30 minutes, but no more than 31 minutes. At the end of the challenge period, turn off the syringe pump. Record the total volume used by the syringe pump, the elapsed time and the airflow rate delivered from the Miller-Nelson®. These values will be recorded in the laboratory notebook and test data sheets. Ensure syringe flow is off and the mixing chamber airflow line is bypassed so uncontaminated air is flowing through the Miller-Nelson® to the agent exposure chamber, simulating weathering.

5.1.17. Liquid Application of HD droplets:

5.1.17.1. Liquid drops are delivered to the surface of the APR to test permeation of material surfaces. A minimum of 32 and a maximum of 43 droplets (20 µL each) are specified. Placement of the droplets, via syringe or equivalent glove box methods, on to the system is specified in Appendixes A, B and C of this STP. The droplets will be placed on the equipment per the numerical sequence and note inserts identified in the appendixes. Photographs of this manual droplet application process should be maintained in the task number folder. The liquid application of HD occurs at the 6-hour mark, after the vapor challenge begins and ends. The droplets usually stay until the test cycle ends.

5.1.17.2. CBRN APRs with hose assemblies, chest or back mounted canisters or specific submittal mandatory components require the maximum droplet quantity of 43 drops. HD droplets 1-32 apply to the facepiece and the canister. Droplets 33 to 43 are applied in accordance with the stated appendixes. If no hose assembly or a modified hose assembly is used, droplets 33 to 43 are used on any Y adapters, communications devices, or other respirator mandatory submittal application items in accordance with droplet patterns enclosed and conducive to targeting seams, material interfaces and obvious breathing airflow channel areas.

5.2.17.3 The droplet placement sequence is in accordance with local laboratory policy and is recommended to start at the top of the face blank, working left to right, to the bottom of the face blank and follow the droplet number sequence identified in Appendixes A, B and C of this STP. Prior to actual droplet application, technicians should prepare by marking location of actual droplets on a digital photograph of the equipment being
tested and use this “dotted” schematic during the course of testing. If an external shroud is permanently mounted on the face blank, apply droplets per standard schematic based on respirator configuration but on surfaces of the shroud that mirror the routine face blank areas. If not permanently affixed, remove the shroud prior to test in order to test the “worst case configuration”.

5.2.17.4. Guidelines for identification of test configuration for exposure to GB/HD are stated in the current version of the CBRN APR Statement of Standard. The focus is on obvious pressure boundary concerns and any protective material surface that appears to be a possible concern related to penetration or permeation. The test configuration should conform to one of the enclosed appendixes. If the test configuration does not conform, notify NIOSH/NPPTL as soon as possible, provide a digital of the respirator if required and await a new NIOSH droplet application schematic.

5.2.17.5. Once the last droplet is applied, conduct a visual check of the breathing APR to ensure it is in still in negative pressure mode, no parts or accessories are out of place and that there is no immediate destruction of material due to the recent exposure to HD droplets. Mount the front panel of the agent exposure chamber. If APR starts to disintegrate immediately during or just after droplet application occurs, finish the droplet application process and close up the box immediately. Allow the test to run, monitoring the pass or fail criteria and terminate in accordance with paragraph 4.4.

5.2.18. A detector is used to monitor the challenge concentration in the exposure chamber. This will be recorded on a computer with compatible software capable of accurately depicting the agent concentration versus time.

5.2.19. System Hold or Agent Decay Monitor Time involves the continuance of flushing the agent exposure chamber with the Miller-Nelson® uncontaminated air to allow the simulation of the natural effects of ambient environmental conditions while the persistent vapor effects of HD react. Ensure the breathing machine operates at the same rate for the remaining 7.5 hours.

5.2.20. Test Surveillance: The laboratory technicians should monitor the entire test to make sure all components of the system function, collect data as required and monitor the breakthrough concentration to protect the MINICAMS® against saturation. If the system is failing, the
MINICAMS® must capture three maximum peak excursions to document the APR’s failure before MINICAMS® can be taken off line from detecting.

5.2.21. Procedures for Termination of Test:

5.2.21.1. The test should be terminated when the full time for the test has elapsed or three confirmed quantifiable consecutive maximum peak excursions are verifiable at any time in the test trial cycle. The pass/fail criteria on Ct are determined after 8-hour completion and raw data tabulation. To terminate the test, turn off breather pump. Perform a check shot of agent to assure that the detection system is still operating correctly. Take an aliquot of one of the mid-range standard solutions of agent with a microliter syringe and inject it into the nasal sampling port by disconnecting the line from the bottom of the exposure chamber. The MINICAMS® response should be that indicated on the standard curve for agent contained in the aliquot. The response must be within 10% of the correct value for the final check shot test to be valid.

5.2.21.2. Turn off the MINICAMS® per laboratory SOP. Turn off Miller-Nelson® airflow through the exposure chamber. Remove the test respirator; separate it into components as necessary and double-bag the components in accordance with laboratory SOP. Remove the bagged components to the decontamination hood for temporary storage. If requested, a manufacture representative may review the items at this time to perform reverse engineering analysis. The test respirator will then be decontaminated, monitored and disposed of according to laboratory SOP. Wipe down the interior of the agent exposure chamber and the SMARTMAN® using approved decontaminating solution. Dispose of cleaning materials according to laboratory SOP. Test Service Agreements (TSA) between the manufacturer and the test laboratory on materials swatch testing or entire respirators is authorized off line from the timeline of the conduct of this standardized test procedure. However, in support of the TSA testing concept, the NIOSH/NPPTL CBRN Respirator Research and Development (R&D) Test Program, current letter, provides for prioritized use of dedicated NIOSH test equipment in support of research and development that is separate and distinct from certification.

5.2.22. Example step sequence of activities for HD liquid and vapor CBRN APR test.
1. Conduct M40 preparation and background contamination assessment.
2. Install APR in clean exposure chamber.
3. Close clean exposure chamber and ensure APR is breathing.
4. Challenge clean APR with TDA-99M for 30 minutes
5. Verify that clean APR has passed TDA-99M, retest/refit, go to next step or if failure, substitute failed APR with a new untested APR. Ensure an environmentally pre-tested canister or a qualifier canister is used and properly labeled for LAT.
6. Install APR in agent exposure chamber.
7. Photograph APR.
8. Close agent exposure chamber, ensure APR is in negative pressure mode and start breathing pump.
9. Challenge APR with TDA-99M for 30 minutes in the agent exposure chamber.
10. Start MINICAMS®.
11. Monitor background inside the mask and do chamber concentration/check shot.
12. Conduct characterization for 60 minutes.
13. Ensure front panel is readily available for replacement on agent exposure chamber.
14. Start makeup air using Miller-Nelson Controller while simultaneously starting ambient challenge concentration detection profile software, if not already started.
15. Start syringe pump.
16. Record start time of MINICAMS®.
17. Initiate challenge agent.
18. Record 30-minute vapor start time.
19. Record challenge agent start time, vapor exposure.
20. Ensure chamber ambient challenge concentration is ramping up as expected.
21. Monitor MINICAMS® for detection, noted penetration peaks and saturation prevention.
22. Stop syringe pump at 30-minute mark, but no later 31 minutes.
23. Record stop time for challenge agent vapor exposure.
24. Record hold time start (7.5 hours).

25. At 5.5-hour mark, conduct final preparations for HD droplet application.

26. At the 6-hour mark, apply droplets per required appendix.

27. Remain with test platform, monitor APR for visible cracks, catastrophic breakage or failure criteria. Monitor clock exposure times, do not exceed 8 hours total and ensure respirator does not convert to positive pressure for any reason. Testing laboratory is responsible for meeting all STP compliance requirements concerning test time; check shots, concentrations and pass/fail criteria assessment and determination.

28. Eight hours, end test.

29. Record end time and local time at 8.0 hours

30. Conduct check shot instrumentation

31. Record check shot results and time of last end of test check shot.

32. Prepare system for decon and removal of tested components.

5.2.23. Data Analysis

5.2.23.1. Lead technician is responsible for accurately maintaining a laboratory notebook and all required records. Hardcopies of data should be annotated when pertinent events occur, such as a catastrophic failure, obvious airflow boundary cracks, accessory cracks/failure, check shots and test start and end times. Lab supervisor must gain NIOSH approval prior to pre-approving any deviation from NIOSH standard test procedure and signoff in the technician’s notebook as to the appropriate NIOSH approved change. Notebooks should be signed and dated. Copies of all hardcopies of data that are generated shall be kept in the assigned task folder with NIOSH task number.

5.2.23.2. Laboratory manager and technician must complete all required test data sheets in accordance with Appendix D. Originals of all test data sheets must be completed and retained in the task file. Test data sheets for CBRN APR candidates are managed by digital exchange of pre-formatted blank file forms easily transferred in standard email. No NIOSH DEIMS electronic forms exist for CBRN APR test data collection. NIOSH DEIMS, current version, will assign TNs, maintain the CBRN APR test queue and process all initial, lab and final reviews. NIOSH DEIMS for CBRN APR should be reviewed, at the
minimum, every three days by the lab technician supervisor, principal investigator or equivalent. While an application is active and has information for update, the transfer of applicable files should be updated daily with all applicable information to allow timely feedback and prevention of miscommunication. Key fob holders of testing lab are required to insure all summary data sheets are reviewed for accuracy prior to final submission to NIOSH/NPPTL in a timely manner.

5.2.23.3. Transfer the permeation data from the MINICAMS® computer into a computer for analysis by Microsoft Excel. This table will contain data from the nasal area and associated time markers. The resulting table will each have four columns: 1) elapsed time 2) volume collected 3) sample collection time and 4) nanograms per sample. Convert the nanograms into a concentration, ng/L, by multiplying nanograms by a factor obtained by dividing the actual sample volume (typically 200 mL but may be considerably less) into 1000 mL/L (Example: 1.73 ng x 1/0.2 L = 8.65 ng/L). Convert nanograms/liter to milligrams/cubic meter by dividing by 1000 (Example: 8.65 ng/L / 1000 = 0.00865 mg/m³). Ct (concentration x time) is calculated by multiplying the collection duration time times the concentration (Example: 0.0865 mg/m³ x 2 min = 0.173 mg·min/m³). The cumulative Ct is calculated by adding the Ct value for each sample time. Using Excel’s chart wizard feature, a plot of concentration vs. time and the Ct vs. time can be generated and printed using all the data in the table.

5.2.23.4. Challenge Concentration Data: Using the computations generated from Paragraph 5.2.23.3 above. Plot the challenge concentration versus time and produce two data graphs that track total detection events covering any maximum peak excursions over 8.0 hours and total concentration over time known, as Cumulative Ct, covering the total 8.0 hours of potential cumulative and instantaneous dosages. If the test is terminated prior to the completion of 8.0 hours due to APR failure, MINICAMS® saturation preventive measures or other situations as outlined, laboratory technician, supervisor or equivalent is responsible for accurately recording all applicable maximum peak excursions and cumulative Ct detected in the tested time.
6. PASS OR FAIL CRITERIA

6.1. The criterion for passing is set within the authority of 42 CFR, Part 84, Subpart G, Section 84.63(a), (c) & (d) and Federal Register, Volume 60, Number 110, June 8, 1995. A two-fold pass or fail criterion is required to be met for successful passing of HD testing. The criterion is:

6.1.1. Distilled Sulfur Mustard (HD) Penetration and Permeation Test:

Challenge: 50 mg/m³ + 10% for 30 minutes (but not more than 31 minutes) proceeded by a maximum of forty-three (43) 20-μL liquid droplet application at the 6-hour mark.

Test Time: 8.0 hours.

Two-fold Pass or Fail Criteria:

a) Maximum Agent Breakthrough ($C_t$) = 3.0 mg·min/m². Concentration integrated over minimum service life is $C_t$ and it is based on a detection sample time of approximately 2 minutes for 8 hours. The $C_t$ data value, including all maximum peak excursion data points, must not be exceeded for the duration of the test.

b) Maximum Peak Excursions = 0.30 mg/m³. Three consecutive data points at or exceeding the peak value constitutes a failure where each test value is based on detection sample time of approximately 2 minutes for 8 hours.

NOTE: Any visible respirator deterioration in assigned material components such as breakage, distortion, hazing of lens, cracking or separation shall also constitute a system warning and qualify as a failure based upon lab manager final determination with consultation from NIOSH/NPPTL.

NOTE: Any APR test that fails as a result of laboratory test equipment failure or malfunction, laboratory electrical power loss or incorrect technician operating procedures/actions will be considered by NIOSH/NPPTL as a test termination and immediate mandatory retest upon lab supervisor confirmation. In this case, the testing laboratory, at no additional cost to NIOSH/NPPTL or the manufacturer concerned, retests the respirator.

6.2. This test establishes the procedures for ensuring the level of respiratory protection provided under special Chemical, Biological, Radiological, and Nuclear (CBRN) requirements for Full Facepiece, Tight Fitting, Negative Pressure, P100, APR submitted for approval, extension of approval, or examined during certification.
product audits, meet the minimum certification standards set forth in 42 CFR, Part 84, Subpart G, Section 84.63(a)(c)(d).

7. RECORDS AND TEST DATA SHEETS

7.1. All test data will be recorded in the NIOSH DIEMS and STP recognized formatted CBRN APR test data sheets (Appendix D). All applicable data, graphs and photographs taken or prepared by laboratory technicians, technician supervisors or their equivalent will remain on file at the actual lab where the test was conducted, and is required to be retrievable within 24-hour notification and maintained in accordance with local administrative SOPs and NIOSH/NPPTL Respirator Branch CET historical filing requirements.

7.2. All videotapes and photographs of the actual test being performed by testing laboratory personnel, or of the test equipment shall be maintained in the task file as part of the permanent record.

7.3. All equipment failing any portion of this test will be handled as follows:

7.3.1. If a failure occurs on a new certification application (qualifier or remainder application), testing laboratory will send a test report to the NIOSH NPPTL Respirator Branch, Equipment Evaluation Team/Certification Evaluation and Testing (CET) “Team Leader” and await further instructions on return of any uncontaminated hardware to the manufacturer.

7.3.2. If the failure occurs on hardware examined under NIOSH directed off-the-shelf audit, the hardware will be examined by a technician and the Laboratory Manager for cause. All equipment failing or passing any portion of this test will have been contaminated with chemical warfare agent. Upon request, the contaminated equipment can be viewed by designated NIOSH personnel in support of incident investigation proceedings. Upon completion of NIOSH proceedings, the equipment is disposed of in accordance with testing laboratory chemical surety practices.
Appendix A, LAT
NIOSH/NPPTL Schematic of HD Agent Droplet Placement for CBRN APR Application (Facepiece Mounted)

Notes:
1. Required placement of drops.
2. Test requires 32 drops on the facepiece.
3. The respirator receives 25 drops on the facepiece and 7 drops on the canister.
4. Pattern starts at #1 drop, 11 o’clock position, goes clockwise and stops at drops 31 and 32.
5. Drops 9 and 21 are on the gasket interfaces between lens and connector.
6. F & G do not exist on this configuration.

Effective: March 13, 2003

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Appendix B, LAT

NIOSH/NPPTL Schematic of HD Agent Droplet Placement for CBRN APR Application (Facepiece Mounted w/ Drink Tube)

Notes:
1. Required placement of drops.
2. Test requires 12 drops on the facepiece.
3. The respirator receives 25 drops on the facepiece and 7 drops on the canister.

Effective date: March 13, 2003

Nomenclature

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<td>B</td>
<td>Head-harness strap</td>
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<tr>
<td>C</td>
<td>Eye Lens (1 or 2 lenses)</td>
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<tr>
<td>D</td>
<td>Side Voicemitter/Front Voicemitter</td>
</tr>
<tr>
<td>E</td>
<td>Canister</td>
</tr>
<tr>
<td>F</td>
<td>Drink tube housing</td>
</tr>
<tr>
<td>G</td>
<td>Drink tube</td>
</tr>
<tr>
<td>H</td>
<td>Exhalation valve</td>
</tr>
</tbody>
</table>
Appendix C, LAT

NIOSH/NPPTL Schematic of HD Agent Droplet Placement for CBRN APR Application (Facepiece Mounted w/ Commo)

Notes:
1. Required placement of drops.
2. Test requires 32 drops on the facepiece.
3. The respirator receives 25 drops on the facepiece and 7 drops on the canister.

Effective: March 13, 2003
Appendix D

CBRN APR HD LAT Certification Test Data Summary Sheet

(Test Data Sheet 1 of 3)

1. TEST TITLE: Determination of Full Facepiece, Tight Fitting, Negative Pressure, P100, Air-Purifying Respirator (APR) Performance During Dynamic Testing Against Chemical Agent Distilled Sulfur Mustard (HD) Vapor and Distilled Sulfur Mustard (HD) Liquid.

A. Task Number (TN): _____________________________________________
B. Manufacturer:   ________________________________________________
C. APR Model #/Type: __________________________________________
D. Test Start Date of Qualifier Application: _____________________________
E. Test Start Date of Remainder Application: ______________________
F. Test End Date of Remainder Application: ______________________
G. Primary P/N and Subject for LAT Configuration:______________________

2. REQUIREMENT:

Three of each CBRN Full Facepiece, Tight Fitting, Negative Pressure, Air-Purifying Respirators (APR) shall demonstrate no permeation or penetration of Distilled Sulfur Mustard (HD) vapor and Distilled Sulfur Mustard (HD) liquid equal to or greater than the stated maximum peak excursions and the stated cumulative concentration over time (Ct) including all maximum peak excursion data points for the duration of the eight (8) hour test. HD vapor challenge concentration will start immediately after the test chamber has been sealed. Minimum Service Life for liquid exposure starts after the first liquid drop is applied. Liquid volume used is dependent on components used with the respirator. Minimum volume of 0.43 mL and maximum volume of 0.86 mL is based on the respirator final tested configuration of one APR canister on a specific respirator assembly. Three consecutive sequential test data points at or exceeding 0.3 mg/m³ will collectively constitute a failure where each test value equals to or exceeds 0.3 mg/m³ and is based on a detector sample time of approximately 3 minutes. The cumulative Ct, including all maximum peak excursion data points, must not be exceeded for the duration of the test. Liquid agent is applied to the complete respirator at hour 6 of the test cycle. The test period begins upon initial generation of HD vapor concentration and ends at 8 hours.

OVERALL RESULT: PASS or FAIL
CBRN APR HD LAT Certification Test Data Summary Sheet

(Test Data Sheet 2 of 3)

3. SUPPORTING REQUIRED DATA:
   A. Has Canister expired prior to LAT in accordance with User Instructions? YES or NO.
   B. Is Canister bent, cracked or disfigured prior to any portion of LAT? YES or NO.
   C. Does Canister to facepiece connection violate Form, Fit or Function? YES or NO.
   D. Is Facepiece fully serviceable prior to any portion of LAT? YES or NO.
   E. Are Sub Assemblies free of visible deformations or aberrations? YES or NO.

HD CBRN APR LAT Certification Summary Continuum:

Task Number: _______________________    STP No.: ___________________
Manufacturer: _______________________   Reference No.: ___________________
Test Title: Determination of Full Facepiece, Tight Fitting, Negative Pressure, Air-Purifying Respirator (APR) Performance During Dynamic Testing Against Chemical Agent Distilled Sulfur Mustard (HD) Vapor and Distilled Sulfur Mustard (HD) Liquid.

   F. Did each APR pass or fail maximum peak excursions? PASS or FAIL
   G. Did each APR pass or fail Ct? PASS or FAIL
   H. Was all lab test equipment verified calibrated prior to LAT? PASS or FAIL

<table>
<thead>
<tr>
<th>QUALIFIER TEST # 1</th>
<th>Total Test Time</th>
<th>Max Peak Excursions</th>
<th>Ct</th>
<th>RESULT</th>
</tr>
</thead>
<tbody>
<tr>
<td>(e.g., TN – HD 1)</td>
<td>480 minutes or less</td>
<td>Quantity failing</td>
<td>Value</td>
<td>Pass or Fail</td>
</tr>
<tr>
<td>REMAINDER TEST # 2</td>
<td>Total Test Time</td>
<td>Max Peak Excursions</td>
<td>Ct</td>
<td>RESULT</td>
</tr>
<tr>
<td>(TN – HD 2)</td>
<td>Same as above (sab)</td>
<td>sab</td>
<td>sab</td>
<td>sab</td>
</tr>
<tr>
<td>REMAINDER TEST # 3</td>
<td>Total Test Time</td>
<td>Max Peak Excursions</td>
<td>Ct</td>
<td>RESULT</td>
</tr>
<tr>
<td>(TN – HD 3.)</td>
<td>sab</td>
<td>sab</td>
<td>sab</td>
<td>sab</td>
</tr>
</tbody>
</table>
CBRN APR HD LAT Certification Test Data Summary Sheet

(Test Data Sheet 3 of 3)

4. COMMENTS:
   (e.g., CBRN APR candidate was tested in minimum packaging configuration for initial Qualifier Application. CBRN APR passed initial Qualifier Application LAT. However, when conditioned configuration was tested in Remainder Application tests, candidate CBRN APR failed max peak excursion criteria and exhibited canister thread dents and bends as a result of environmental conditioning prior to Remainder LAT.)

5. SIGNATURES:
   A. Laboratory Technician: _________________________ Date: _______________
       (Signature)
       __________________________
       (Printed Name)

   B. Laboratory Supervisor: _________________________ Date: _______________
       (Signature)
       __________________________
       (Printed Name)

Legible Signatures infer concurrence with test summary findings as indicated above.
## Revision History

<table>
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<tr>
<th>Revision</th>
<th>Date</th>
<th>Reason for Revision</th>
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<tr>
<td>0</td>
<td>23 December 2003</td>
<td><strong>Interim Guidance Version:</strong> Initial version dated March 18, 2003 was converted from an interim guidance STP to a draft final STP dated December 24, 2003. Integration of actual DIEMS test data sheet was standardized between the STP and the NIOSH administrative project management system (DIEMS). Incorporated new third party laboratory name change: SBCCOM to RDECOM.</td>
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<tr>
<td>0.0</td>
<td>30 September 2004</td>
<td><strong>Final STP Version:</strong> Incorporated next generation equipment guidance on MINICAMS® and syringe pump into STP.</td>
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<tr>
<td>0.1</td>
<td>24 October 2005</td>
<td>Update header and format to reflect lab move from Morgantown, WV No changes to method</td>
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<td></td>
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<td>No changes to method</td>
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