Hattonal Institute for Occupational Safety and Health

National Institute for Occupational Safety and Health National Personal Protective Technology Laboratory Respirator Branch 626 Cochrans Mill Road

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Pittsburgh, Pennsylvania 15236

NOTE: The Respirator Branch maintains an updated index of current procedures.

DETERMINATION OF CBRN TIGHT FITTING AND NON-TIGHT FITTING, POWERED AIR-PURIFYING RESPIRATOR (PAPR) PERFORMANCE DURING DYNAMIC TESTING AGAINST CHEMICAL AGENT DISTILLED SULFUR MUSTARD (HD) VAPOR AND DISTILLED SULFUR MUSTARD (HD) LIQUID CBRN STANDARD TESTING PROCEDURE (STP)

PURPOSE

- This document establishes the procedures for ensuring the level of respiratory protection provided by Chemical, Biological, Radiological, and Nuclear (CBRN) Protection requirements for Powered Air-Purifying Respirator (PAPR) 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); Federal Register Volume 60, Number 110, June 8, 1995.
- The purpose of this Standard Test Procedure (STP) is to describe the test conditions and 1.2 procedures necessary to test and certify manufacturer submitted CBRN PAPR applications. A CBRN PAPR being a complete tight fitting or non-fitting facepiece or hood, properly outfitted with manufacturer unique components and a properly designed manufacturer specified PAPR blower and canisters which are installed per the manufacturer's installation requirements outlined in current user's instructions. This procedure is used to test CBRN PAPR 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 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-0551 for challenge of 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.

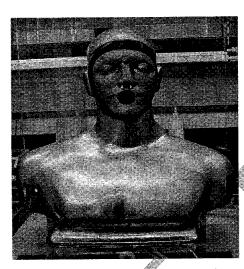
Approvals:	1st Level	2 <u>nd</u> Level	3 <u>rd</u> Level	4 <u>th</u> Level

2. GENERAL:

- 2.1 The STP describes test procedures 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.
- 2.2 Any laboratory using this procedure to supply certification test data to NIOSH/NPPTL is subject to the provisions of the NIOSH Supplier Qualification Program (SQP). This program is based on the tenets of ISO/IEC Procedure 17025, the NIOSH Manual of Analytical Methods (NMAM) and other appropriate NIOSH and NIOSH/NPPTL guidelines. An initial complete quality system audit and follow on audits are requirements of the SQP. Additional details of the SQP and its requirements can be obtained directly from NIOSH/NPPTL.

3. EQUIPMENT. List of necessary test equipment follows:

SMARTMAN, Head/upper torso form or equivalent (see picture 1). Manufactured by ILC Dover, 3.1 Frederica, DE. 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, 114 inch in diameter, leads from the mouth area to the breather pump. Four 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. The four tubes are 1/4" 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.



Picture 1: Head/Upper Torso Manikin, SMARTMAN

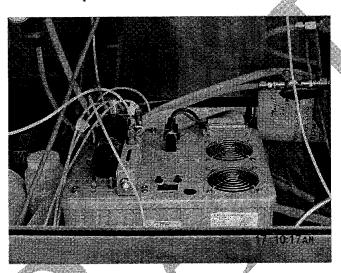
3.2. Leak detector Model TDA-99M or equivalent (see picture 2). Manufactured by Air Techniques, Inc., the TDA-99M is one of the primary tools for accessing aerosol leaks in the mechanical seals of the respirator and proper fit of a respirator to clean SMARTMAN headform under non-toxic oil aerosol conditions. The device generates an Emery 3004 mineral oil aerosol that is used to detect leakage in 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 100mg/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.



Picture 2: Leak detector Model TDA-99M

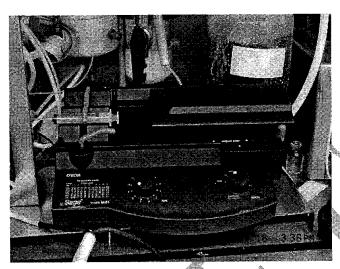
3.3 <u>MINIature Continuous Air Monitoring System, MINICAMS or equivalent (see pic. 3)</u>. The MINICAMS, manufactured by OI Analytical, is a gas chromatograph equipped with a hydrogen flame emission detector and a preconcentrator tube. The preconcentrator tube is a small tube containing an adsorbent material to scrub out agent vapor contained in a sample of air drawn

through it for a set time 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 of time. The Limit of Detection (LOD) is equal to 20% of the 8 hr Time Weighted Average 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. Two MINICAMS are required in order to continuously monitor the interior of the respirator for CBRN respirator certification.



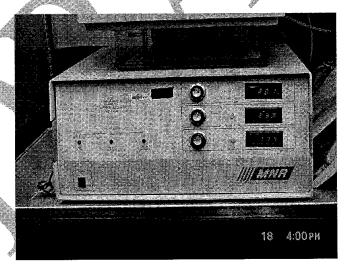
Picture 3: MINIature Continuous Air Monitoring System, (MINICAMS)

3.4 Syringe pump. Sage Model M365 or equivalent (see picture 4). This multirange, 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 ml/hr, Flow Accuracy = +/- 5% Nominal. Technicians must be proficient on the pump's user instructions.



Picture 4: Syringe pump, Sage Model M365

3.5 Flow-Temperature-Humidity Control System or equivalent (see picture 5). This system, referred to as the Miller-Nelson (MN) control system, manufactured by Miller-Nelson Research, Inc., 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 (200-50 L/min.+/- 2%), and relative humidity (20-80 +/- 3%) and temperature (20-30 °C +/- 0.3%).



Picture 5: Flow-Temperature-Humidity Control System

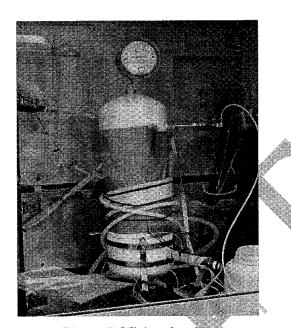
3.6 Exposure Chamber or equivalent (see picture 6). The respirator exposure chamber is constructed of clear, chemical resistant material (Plexiglas ® or Lexan®) or other equivalent material. The floor must be constructed efficiently to support the 85-pound SMARTMAN. The front panel is removable and is held in place with clamps on each edge. The dimensions are approximately 2 ft.

cubed. Four Plexiglas legs 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/Cold Box" is a unit used to perform a fit and leak check on a respirator test unit (no agents used in this chamber) before it is installed in the agent exposure chamber. An "Agent Exposure Chamber/Hot Box" is a chamber used to perform testing of the respirator unit, using live agent.



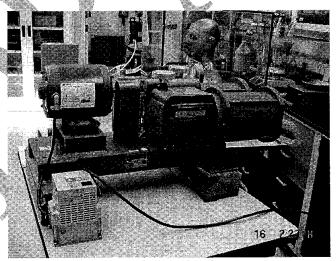
Picture 6: Exposure Chamber

3.7 Agent Mixing Chamber or equivalent (see picture 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 air flow 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 mil spec canister or equivalent).



Picture 7: Mixing chamber

Breather Pump, Model E1R1 or equivalent (see picture 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 pump. The sinusoidal pattern starts at zero flow rate, rises to peak flow of approximately π (3.1416) times the rated test certification flow rate in L/m and drops back to zero. The exhalation stroke of the pump is the same sinusoidal pattern. The volume per breath /tidal is adjustable up to 1.5 liters.



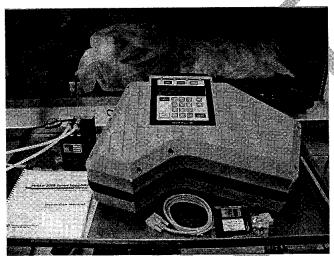
Picture 8: Breather Pump

3.9 <u>Mass Flow Controllers or equivalent</u>. 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 \pm of set point. Photo not provided.

3.10 Ambient Air Analyzer, MIRAN model 1A or 205B, or equivalent (see picture 9 and 10).

Manufactured by Thermo Environmental Instruments Inc. It is an infrared absorption based detector that uses a long path length cell up to 20 meters, into which the air sample is introduced. This analyzer is used to monitor the challenge concentration of the vapor phase of the chemical warfare agents. Model 1A is used for the Sarin (GB) concentration and model 205B or an equivalent is generally used for the HD concentration.



Picture 9: Miran Analyzer, Model 205B

- 3.11 Respirator Systems Required for Testing.
 - 3.11.1 REQUIRED PAPR QUANTITIES: Number of PAPRs required for HD Live Agent Testing (LAT) is three (3) complete respirator systems. Initially, one (1) complete PAPR system which has not been subjected to Durability Conditioning is required for the HD "Qualifier Application". Upon confirmation that the PAPR successfully passed the Qualifier Application HD Test, two (2) additional PAPRs shall be tested for HD "Remainder" LAT after they have been subjected to Durability Conditioning in accordance with CBRN PAPR Statement of Standard.
 - Battery Requirements: In order for the PAPR to be tested for the entire test duration of eight (8) hours, the applicant must supply a battery (for this test only) that is sufficient to operate the PAPR at the rated airflow for nine (9) hours; otherwise, the laboratory manager shall integrate a subsequent power source or an additional battery so that the PAPR operates at the rated flow rate for the entire test duration.
 - 3.11.3 QUALIFIER LAT: HD Live Agent Test canisters, hoods, facepieces, blower units and accessories should be received from the appropriate NIOSH/NPPTL sanctioned source. No alterations to canisters are authorized prior to or during live agent systems testing. No use of duct tape or 100mph tape over inlet and outlet areas is authorized in lieu of no caps

or missing caps. At NIOSH/Tenant Laboratory initial certification qualifier application inventory, canisters received in the Minimum Packaging Configuration are required to be plastic bagged, regardless of type of original packaging, and delivered to LAT lab upon completion of satisfactory certification inventory.

- 3.11.4 REMAINDER LAT: Start of HD Remainder LAT is contingent upon testable PAPR systems completing all required NIOSH/NPPTL CBRN PAPR Durability Conditioning. Durability Conditioned canisters will be attached per manufacturer instructions to available compatible application PAPR assemblies prior to live agent testing by qualified and designated laboratory live agent testing technicians only. Environmentally Durability Conditioned canisters should be received in an air tight durable Ziploc bag and administratively labeled with the date the Durability Conditioning test started, completed, type of damage if any observed, manufacturer and laboratory responsible individual for the Durability Conditioning, phone number and laboratory name. Only the manufacturer's inlet and outlet plugs that come with the canister are to be used to seal up the canister after Durability Conditioning. If no plugs are provided, no other form of sealant is authorized Any canister damage during Durability Conditioning is required to be preserved, not altered and noted on administrative markings for shipment to LAT lab.
- 3.11.5 <u>Annotation:</u> HD Live agent systems test canisters and blower assemblies should be labeled prior to actual Live agent testing with the following administrative information:
 - NIOSH Task Number (TN Number) from NIOSH DEIMS.
 - Model of PAPR from NIOSH DEIMS.
 - 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.
 - Accurate labeling of post tested respirators is mandatory in support of post-test incident investigations.

4. TESTING REQUIREMENTS AND CONDITIONS:

- 4.1. 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.
- 4.2. System Test Conditions:
 - 4.2.1 <u>Breathing Machine</u>:
 - 4.2.1.1 Breathing Machine Requirement (Both Constant Flow and Demand Response)

Airflow = 40 liters per minute Respirations = 36 +/- 2 strokes per minute Tidal Volume = 1.1 liters

4.2.1.2. Breathing Machine Requirement (Demand Response Flow only)

Airflow = 60 liters per minute Respirations = 55 +/- 2 strokes per minute Tidal Volume = 1.1 liters

4.2.2 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, the known volume of the exposure chamber, considering the agent challenge concentration desired, volume of chamber, breather air flow, and dilution air exhausted from the PAPR.

Relative Humidity: $25 \pm 5\%$ RH.

Temperature: $25 \pm 3^{\circ}$ C

4.2.3 <u>SMARTMAN Sampling Point</u>:

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

4.2.4 Mustard (HD) Exposure Test:

4.2.4.1 HD VAPOR: GENERAL.

Vapor Challenge Concentration = 50 mg/m³ + Vapor Challenge Time (disseminator on) = First 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 and PAPR flow rate.

Syringe Injected Volume = as required based upon syringe pump used and PAPR flow rate.

- 4.2.4.2 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 met CASARM agent purity requirements. Proper CASARM storage requirements per local regulation are required to be adhered to.
- 4.2.4.3 Vapor test termination is at the indicated eight (8.0) hours plus required check shots before and after entire test period. In the interest of conserving agent quantities and preserving technician man-hours, a live agent test 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/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.
- 4.2.4.4 Vapor challenge concentration exposure will start immediately after the test chamber has been sealed / closed properly.

4.2.4.5 HD LIQUID: GENERAL.

Drop Size = 20 microliters (μ l).

<u>Liquid Challenge Concentration</u> = Minimum is 0.43 milliliters (32? drops of 20 μl each) and maximum is 0.86 milliliters (43 drops of 20 μl each) depending on type of respirator tested.

<u>Droplet Allocation</u> There shall be 25 drops applied to the facepiece whether it is a face-seal type or a neck-dam type with or without hydration facility. There shall be 7 to 18 applied to components such as the blower, breathing hose(s), multiple canisters, a communications device interface, an electrical power interface and other noted critical interfaces as determined by NIOSH/NPPTL during configuration analysis.

<u>Droplet Schematics</u>: Four (4) NIOSH/NPPTL Schematics of HD Agent Droplet Placement for CBRN PAPR 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/Cheek Style, Monocular, & Drink Component

<u>Appendix C:</u> Chin/Cheek Style, Biocular, Drink Component & Communications Interface

Appendix D: Non-facepiece mounted blower assembly, Biocular, Multiple Canisters, Belt or Strap Mounted Canister Housing, Hose Assembly, Drink Component, Communications Interface, and Housing Power Source Interface with Battery/Power Source installed. If respirator is monocular, substitute monocular droplet sequence for lens area as specified in Appendix B.

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 live agent testing. Facepiece second or third skins or layers must be explicitly specified in the application submission as a required part of the CBRN PAPR configuration prior to live agent testing. 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 droplet applications being on bare facepiece material they are on the shroud covering that depicted area/seam/joint/interface. All other defined criteria apply.

<u>Total Test Time</u>: The total test time of this test is 8 hours. Test start time, Time Zero, begins when the first confirmed vapor is applied to the interior of the agent exposure chamber.

<u>Liquid Time</u>: HD liquid agent is applied to PAPR at hour six (6) mark of the Total Test Time. Total time that CBRN PAPR candidate system is exposed to both HD vapor and HD liquid (Liquid Time) is two (2) hours, the last two hours of the total eight (8) test time period.

- 4.2.4.6 Mustard (HD) agent liquid is required to be Department of Defense, US Army, Chemical Agent Standard Analytical Reference Material (CASARM) grade.

 Agent purity analysis must be NIST traceable, documented and met US Army CASARM agent purity requirements. Proper CASARM storage requirements per local regulation are required to be adhered to.
- 4.2.5 <u>Test Termination Parameters:</u> 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 oversaturation provided a candidate PAPR shows failing criteria prior to the eight (8) hour mark. The termination point should be set at or above the highest calibration point for each one of the MINICAMSs in use.
- 4.3 <u>Safety and Training:</u> Normal laboratory safety practices are required. Laboratory specific regulations such as US Army Regulation 50-6, Chemical Surety apply as required. The practices includes all safety precautions described in the current Occupational Safety & Health Facility Laboratory Safety Manual, the applicable US Army Regulations, the U.S. Army Soldiers and Biological Chemical Command Laboratory Safety Procedures, and any other equivalent manuals/periodicals.
 - 4.3.1. Safety glasses, lab coats, assigned respirator, butyl apron and butyl gloves are required be available/worn as laboratory standard operating procedures apply.
 - 4.3.2. Work and walking surfaces must be maintained free of clutter and non-essential test equipment.
 - 4.3.3. 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.
 - 4.3.4. 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.
 - 4.3.5. The responsible technician must be knowledgeable about the specific CBRN PAPR being tested and should be able to readily identify all CBRN PAPR hardware components, subassemblies and accessories and be able to support accurate trouble shooting of the CBRN PAPR and subassemblies to ensure it is operating properly in accordance with the manufacturer's operating instructions. 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 PAPR material.
- 4.3.6. 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 PAPR materials is the responsibility of the testing laboratory. In accordance with this STP, respirator manufacturers are released of equipment accountability IAW local testing lab test equipment procedures per Department of Defense HAZMAT protocol.
- 4.3.7. 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.
- 4.3.8. 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.

4.4. EQUIPMENT PRE-TEST CONDITIONS: MINICAMS

- 4.4.1 <u>Background Reading:</u> A Laboratory MINICAMS, as opposed to a Field MINICAMS, is used as the detector for agent penetration/permeation into the CBRN PAPR. It consists of a monitor, PC computer, Linear Mass Flowmeter and optional printer or recorder. Before the PAPR 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 may involve the use of a clean M40 mil spec respirator mounted on the SMARTMAN head form. 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 live agent test (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.
- 4.4.2. Unit of Measure. A small volume of air is drawn through a pre-concentrator tube containing an adsorbent material: HD sample volume is per 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.
- 4.4.3 <u>Sample Cycle.</u> 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 PAPR HD analysis requires a total 6-minute cycle (3 minute sample followed by 3 minute purge per detector). The two MINICAMS are required to be synchronized so that one minicam sampling begins when the other minicam sampling ends. Raw data results confirm this requirement.

- 4.4.4. 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.
- 4.4.5. <u>Standardization.</u> 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 uL) of a known standard solution of the agent onto the pre-concentrator tube during the INJECT segment of the test cycle IAW MINICAM calibration SOP. The standard solutions of agent are made in isopropanol, spectrophotometric grade. At least three injections of each quantity of agent should be injected per LAT sequence.
- 4.4.6 Pretest activities for the MINICAMS are as follows:
 Set or verify operational parameters for appropriate agent
 Perform standardization
 Record ASCII file name on Data Sheet
 Standby for start of testing

4.5. EQUIPMENT PRE-TEST CONDITIONS: SMARTMAN HD Vapor Generation.

- 4.5.1. <u>Vapor Concentration</u>. 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 filtered and discharged into the chamber from the filtered and exhaled air of the PAPR.
 - 4.5.1. 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 PAPR 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.
 - 4.5.1.2. Concentration Verification. (Both Constant Flow and Demand Response)
 HD Setup (Mass Balance Analysis Concept or Demonstrated Equivalent)
 Chamber size = 8 ft³
 Target Vapor Challenge Concentration = 50 mg/m³ + 10%.
 Ramp Up Time = 1-2 minutes (time from initial start of injection of HD air mixture to 50 mg/m³, the lowest acceptable challenge concentration)
 Dilution Air from Breather Pump = 40 l/m
 Miller-Nelson Challenge Mixing Air = 50 l/m
 Syringe Rate for Challenge Injection = [Flow Rate of PAPR Dependent]

Total HD expected in 30 minutes = 0.25 ml vapor plus 0.43 to 0.86 ml of liquid drops.

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.

4.6. EQUIPMENT PRE-TEST CONDITIONS: Miller-Nelson (MN) Controller.

- 4.6.1. 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, temperature value and relative humidity values are logged in the technician's lab logbook prior to commencing each actual live agent test.
- 4.6.2. 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.

4.7. EOUIPMENT PRE-TEST CONDITIONS: Syringe Pump.

- 4.7.1. 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.
- 4.7.2. 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.
- 4.7.3. 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.

4.8. EQUIPMENT PRE-TEST CONDITIONS: TDA-99M Aerosol Leak Detector

- 4.8.1. The TDA-99M leak detector is used to detect oil particulate aerosol leaks into the CBRN PAPR 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 PAPR shall be activated in the operational mode (blower operating). Ensure that procedures for the PAPR follow the manufacturer's installation procedures for gasket seal and facepiece donning and interface. Ensure the seal, threaded interface, inlet and outlet areas and canister housing are not changed in any manner prior to or during the live agent test. However, if the canister or canister interface thread is deformed from Durability Conditioning (If applicable), 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 / available for follow up incident review if necessary.
- 4.8.2. Turn on the power and let the leak detector equilibrate, according to the manufacturer's instructions. The PAPR should be placed into operation mode (blower operating). 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.
- 4.8.3. 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 which indicates no leakage, continue with the next item in the LAT procedure; however, if 0.0010% cannot be obtained and there are TDA-99 reading fluctuations due to residual particles from the APER manufacturing process, the laboratory manager shall make a determination whether a true leak exists or residual particles are being detected. If there is evidence of leakage, attempt to find and eliminate the leak. If leakage or contamination is detected, it is at the laboratory manager's discretion whether or not to continue with the test. Annotate the lab book with description and decision.

9. EOUIPMENT PRE-TEST CONDITIONS: Quality Control Measures.

- 4.9.1. SMARTMAN Leak Test Without Respirator Mounted: 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 the recommended static pressure value. 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 head-forms when they are in continuous daily use.
- 4.9.2. <u>Standardization of Instrumentation:</u> 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 ${\bf r}^2 = 0.99$ for HD. Agent solutions must be stored at recommended surety storage temperatures.

- 4.9.3. <u>Calibration of Flows</u>: 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 Air Flow Controller, all electronic flow meters used for the MINICAMS pre-concentrator tube and the flow meters from the breather pump and the syringe pump agent injector.
- 4.9.4 Aerosol Leak Testing: PAPR leak testing using the TDA-99M Aerosol Tester is performed on the facepiece after installation on the SMARTMAN and while the breather pump and PAPA are 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. Values of 0.0009 to 0.0001% penetration of particulate are acceptable.
- 4.9.5 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 oral-nasal 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 PAPR end of test eight (8) hour mark.

5. PROCEDURE (HD Liquid/Vapor Challenge (0551):

5.2 HD LAT (0551)

5.2.1 Assemble PAPR per manufacturer's instructions. Non-Durability Conditioned PAPRs will be tested in the Qualifier Application. For the Remainder Application, Durability Conditioned units per CBRN PAPR Statement of Standard shall be tested. After Durability Conditioning the items, conditioned canisters that were not stored in the

PAPR's Minimum Packaging Configuration (Inside PAPR carrier or case) shall be placed on the PAPR and tested. If the PAPR uses disposable batteries, install new batteries on the PAPR before testing. If the PAPR uses rechargeable batteries, fully recharge the batteries and install them on the PAPR.

NOTE: The applicant must provide a battery(ies) (for this test only) that is sufficient to operate the PAPR at the rated airflow for nine (9) hours; otherwise, the laboratory manager shall integrate a subsequent power source or an additional battery so that the PAPR operates at the rated airflow rate for the entire test duration.

- 5.2.2 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 six (6) hour mark, prior to vapor testing re-commencing.
- 5.2.3 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 / seated correctly on the headform. Install blower batteries and turn on the PAPR blower: Refer to section 3.11.2. for battery life requirements. 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 emplaced and the communications device is turned on. Monitor the communications device for malfunctions during the course of the test and visually inspect it and other accessories at the termination of the test for visible cracks and/or breaches of the airflow boundary, a.k.a. compromises of form, fit or function.
- 5.2.4 Turn on the breathing pump (See Requirements in Paragraph 4.2.1. for the prescribed breathing rates). Use the integrated SMARTMAN magnehelic pressure gauge as required as a qualitative indicator of PAPR performance and can serve as a tool for observation purposes.
- Using the TDA 99M, leak test the PAPR in the operational mode (blower operating). Connect the detector inlet to a sample line from the SMARTMAN, allow the PAPR to breath, 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 technician 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 thirty (30) minutes of continuous TDA-99M operations below 0.0010% penetration. If penetration exceeds 0.0010%, the TDA-99M alarm sounds, and/or the digital readout shows higher values, stop the 30 minute test, reanalyze the system and restart a new 30 minute test period. The PAPR must pass a continuous 30 minute test at ≤ 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. Other methods such as allowing the respirator to purge itself of internal offgassing particulates are acceptable provided the purge time is not excessively long. Repeated tests using the TDA-99M on the same tested respirator are not recommended and identified respirators are subject to being replaced to all test progress. 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 and a confirmation analysis by a third party laboratory must confirm the manufacturer's MSDS and the particulates must be determined to be non-toxic in the acute and chronic human toxicology perspective before a NIOSH letter of approval is issued for a passing LAT respirator. If the applicant requests to continue despite the high TDA 99M readings, the testing laboratory manager has the option to continue the test or terminate the test based upon the inability of the respirator to pass the TDA 99M indicator screening test.

- 5.2.6 Before the PAPR facepiece or hood 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/deconned 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. The use of the M40 may or may not be equivalent to the actual use of a tested respirator on the headform, therefore in test situations were background is not stable use all means necessary to gain a steady state background lower than the lowest calibration curve point for both MINICAMS before starting LAT.
- 5.2.7 Remove PAPR from clean exposure chamber and install on SMARTMAN in the HD agent exposure chamber.
- 5.2.8 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 hotbox 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 PAPR facepiece to the SMARTMAN system. Remove the clean airline from the canister system, if it was used to isolate leaks.
- 5.2.9 Turn on the Miller Nelson Flow temperature and humidity controller.
- 5.2.10 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 warfare agent test. Confirm that background level is less than the lowest point on the MINICAM 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 PAPR facepiece or hood, rinse the headform with IPA, redon the facepiece or hood and restart the procedure for characterization as necessary. If the PAPR is removed from the head form, restart the test procedure from paragraph 5.2.3.

- 5.2.11 Set up standard operational mode of test equipment. Ensure all test equipment is within calibration.
- 5.2.12. Set Miller-Nelson for airflow into exposure chamber set to deliver required rates. See paragraph 4.2.2.
- 5.2.13. Ensure MINICAMS are calibrated, check shot is complete and acceptable and ready for the operating mode. Annotate the check shot times and concentrations.
- 5.2.14. 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. Exposure chamber ambient atmosphere detector should be on during baseline determination.
- 5.2.15. Load liquid agent in syringe and set syringe pump to correct flow rate to achieve the required agent challenge concentration. See paragraph 4.7.

5.2.16. HD Vapor Application:

There are two different breathing rate requirements during the first 30 minute vapor challenge phase. The breathing rate shall depend on whether the PAPR being tested is a Constant Flow or a Demand Response type. If the PAPR is a Constant Flow Type, the Breathing machine rate will remain at 40 lpm (36 +/- 2 strokes per minute) during the first 30 minute vapor challenge phase and through out the remainder of the 8 hours test.. If the PAPR is a Demand Response, the breathing rate shall be set at 40 lpm (36 +/- 2 strokes per minute) for the first 15 minutes of the vapor challenge then be increased to 60 lpm (55 +/- 2 strokes per minute) between the 15 minute to 30 minute mark of the vapor challenge. After the 30 minute vapor challenge, the breathing rate for the Demand Response type shall be set back to 40 lpm for the remainder of the test.

5.2.16.1 Vapor Challenge (Constant Flow)

- 5.2.16.1.1. Time zero + One second or start of the agent test is when the first confirmed vapor state of HD is applied to the interior of the 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.
- 5.2:16.1.2. Ensure the breathing pump is set at 40 lpm for the entire duration of the test. Start of the 30 minute vapor exposure is when HD vapor is injected into the HD exposure chamber and initially detected. 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 six (6) hour mark.
- 5.2.16.1.3. Introduce HD vapor agent challenge to the agent exposure chamber by first starting the syringe pumping and turning 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. Using the appropriate Miran detector, monitor the concentration build up in the exposure chamber generated by the mixing chamber. The total flow to the mixing chamber from Miller Nelson is dependent on

the airflow rate of the PAPR, which includes enough excess to make up for the clean air exhausted from the PAPR into the exposure chamber and maintain the constant challenge as required. The syringe flow rate is set to introduce the quantity of agent necessary to generate the challenge concentration required. Record when the MINICAMSs begin monitoring the interior of the facepiece

5.2.16.1.4. 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. Ensure syringe flow is off and the mixing chamber airflow line is bypassed so Miller-Nelson uncontaminated air is flowing to the agent exposure chamber, simulating weathering.

5.2.16.2 Vapor Challenge (Demand Response Flow)

- 5.2.16.2.1. Time zero + One second or start of the agent test is when the first confirmed vapor state of HD is applied to the interior of the 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.
- 5.2.16.2.2. Ensure the breathing pump is set at 40 lpm (36 +/- 2 strokes per minute) between time zero (0) to the 15 minute mark. Start of the 30-minute vapor exposure is 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 six (6) hour mark.
- 5.2.16.2.3. Introduce HD vapor agent challenge to the agent exposure chamber by first starting the syringe pumping and turning 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. Using the appropriate Miran detector, monitor the concentration build up in the exposure chamber generated by the mixing chamber. The total flow to the mixing chamber from Miller Nelson is dependent on the airflow rate of the PAPR, which includes enough excess to make up for the clean air exhausted from the PAPR into the exposure chamber and maintain the constant challenge as required. The syringe flow rate is set to introduce the quantity of agent necessary to generate the challenge concentration required. Record when the MINICAMSs begin monitoring the interior of the mask
- 5.2.16.2.4. After the PAPR has been exposed to 15 minutes of vapor exposure, increase the breathing pump to 60 lpm (55 +/- 2 strokes per minute) until the 30 minute mark of the vapor challenge phase. After the 30-minute vapor challenge, the breathing rate for the Demand Response PAPR shall be set back to 40 lpm for the remainder of the test.
- 5.2.16.2.5. The vapor syringe pump should run for the prescribed challenge period of 30 minutes, but no more than 31 minutes. After the PAPR has been exposed to 15 minutes of vapor exposure, the syringe rate shall be increased between the 15 minute to 30 minute mark of the vapor challenge test in order to maintain the required HD

concentration in the exposure chamber resulting from the increasing the breathing pump rate to 60 lpm. 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. Ensure syringe flow is off and the mixing chamber airflow line is bypassed so Miller-Nelson uncontaminated air is flowing to the agent exposure chamber, simulating weathering.

5.2.17. Liquid Application of HD droplets:

- 5.2.17.1 At the six (6) hour mark, pre-defined liquid droplets are applied to the PAPR. Liquid drops are delivered to the surface of the PAPR to test permeation of material surfaces. A minimum of 32 and maximum of 43 droplets (size of 20 micro liters each) are specified. Placement of the droplets, via syringe on to the system is specified in (Appendices A, B or C.). The droplets will be placed on the equipment per the numerical sequence and applicable note/nomenclature inserts on the enclosed appendices. Photographs of this manual droplet application must be maintained in the Task Folder. The exposure chamber is closed back up as fast as possible and remaining test cycle time and detection operation proceeds up to the eight (8) hour mark.
- 5.2.17.2 The droplet placement sequence will be starting at the top of the faceblank, working left to right, to the bottom of the face blank following the number sequence marked on Appendix A, B, C or D. 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. Droplet application should be expeditious, safe and done in a timely manner. Annotate the Time Zero + One Second and the HD liquid application time period and liquid exposure time. Visual observation of the tested PAPR for the last two hours of testing is highly recommended.
- 5.2.17.3 Actual guidelines for identification of test configuration for exposure to GB/HD are stated in the current version of the CBRN PAPR Statement of Standard. Two primary concepts are stated. The first is on unique parts, components and/or materials, that form an air pressure boundary between the breathing gas/air and the ambient environment, need to be tested. The second is unique parts, components and/or materials that have a direct effect on the ability of the respirator to provide respiratory protection, such as a facepeice head harness, exhalation valves, inhalation valves, head harness mounts, faceblank seals, canister interfaces, canister housings, eyelens interfaces with locking rings, side voicemeters and/or plugged areas, need to be LAT/tested. The test configuration should conform to one of the enclosed HD droplet schematic appendices. Slight variation is allowed provided it is clearly identified. If the test configuration does not conform, notify NIOSH/NPPTL, provide a digital of the respirator if required to NIOSH and await a new NIOSH droplet application schematic prior to LAT.
- 5.2.17.4 Once the last droplet is applied, conduct a visual check of the breathing PAPR to ensure it is operating properly, 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 PAPR 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/fail criteria and terminate in accordance with paragraph 4.2.5.

- 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.
- 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 (0.030mg/m3) to document the PAPR'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. The pass/fail criteria on Ct are determined after eight (8) hour completion and raw data tabulation. To terminate the test, turn off breather pump. Perform a check shot of agent to ensure 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 injecting the line at the bottom of the exposure chamber. The MINICAMS response should be that indicated on the standard curve for the amount of 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 according to laboratory SOP. Wipe down the interior of the agent exposure chamber and the SMARTMAN using approved cleaning 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 PAPR test.
 - 1. Conduct M40 preparation and background contamination assessment.
 - 2. Install PAPR in clean exposure chamber.
 - 3. Close clean exposure chamber, ensure PAPR blower is operating.
 - 4. Challenge clean PAPR with TDA-99M for 30 minutes
 - Verify that clean PAPR has passed TDA-99M, retest/refit, go to next step or if failure, substitute failed PAPR with a new untested PAPR. Ensure an environmentally pre-tested canister or a qualifier canister is used and properly labeled for LAT.
 - 6. Install PAPR in agent exposure chamber.
 - 7. Photograph PAPR.
 - 8. Close agent exposure chamber, ensure PAPR blower is operating and start breathing pump.
 - 9. Challenge PAPR with TDA-99M for 30 minutes in the hot box.
 - 10. Start MINICAMS.
 - 11. Monitor background inside the mask and do chamber concentration/check shot.
 - 12. Conduct characterization for 60 min.
 - 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 six (6) hour mark apply droplets per required appendix.
 - 27. Monitor PAPR 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 (8.0) 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/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.

- Laboratory manager and technician must complete all required Test Data 5.2.23.2 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 PAPR 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 PAPR test data collection. NIOSH DEIMS, current version, will assign TNs, maintain the CBRN PAPR test queue and process all initial, lab and final reviews. NIOSH DEIMS for CBRN PAPR should be reviewed every three working 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 bi-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 = 0.0865 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 para 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 PAPR 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/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); Volume 60, Number 110, June 8, 1995. A Two-fold pass/fail criterion is required for successful passing of HD testing. The criterion is as follows as indicated in final PAPR Statement of Standards dated ****:

Vapor-liquid sequential challenge of PAPR with distilled sulfur mustard (HD)

Agent	Challenge Concentration	Duration of Challenge (min)	Breathing Machine Airflow Rate (L/min)	Maximum Peak Excursion (mg/m³)	Maximum Breakthrough (concentration integrated over minimum test time) (mg- min/m³)	Number of Systems Tested	Minimum Test Time (hours)
HD- Vapor	50 mg/m ^{3*}	30	40 60§§	0.30 [‡]	3.0 %	3	8 ^{††}
HD- Liquid	0.43 to 0.86 ml*,†, **	120	40	0.30 [‡]	3.0 [§]	3	2

- * Vapor challenge concentration will start immediately after the test chamber has been sealed. Minimum Test Time for liquid exposure starts after the first liquid drop is applied.
- † Liquid Volume dependent on accessories used with the respirator. Minimum volume is 0.43 ml based on the respirator only.
- ‡ Three consecutive sequential test data points at or exceeding 0.3 mg/m³ will collectively constitute a failure where each test value is based on a detector sample time of approximately two (2) 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 respirator at hour six (6) of the vapor test cycle.
- †† The test period begins upon initial generation of vapor concentration and ends at eight (8) hours.
- §§ The breathing rate shall be increased to 60 lpm during the 15 minute to 30 minute mark of the vapor challenge phase while testing Demand Response PAPR.
 - 6.1.1 Distilled Sulfur Mustard (HD) Penetration and Permeation Test:

<u>Challenge</u>: $50 \text{ mg/m}^3 + 10 \%$ for 30 minutes (but not more than 31 minutes) proceeded by a maximum of forty three (43) 20 μ l liquid droplet application at the 6 hour mark.

Test Time: 8.0 hours.

Two-Fold Pass/Fail Criteria:

- a) Maximum Agent Breakthrough (Ct) = 3.0 mg-min/m³. Concentration integrated over minimum service life is Ct and it is based on a detection sample time of approximately three (3) minutes for eight (8) hours. The Ct 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 (3) consecutive data points at or exceeding the peak value constitutes a failure where each test value is based on detection sample time of approximately three (3) minutes for eight (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 potential failure based upon assessment of NIOSH CBRN PAPR Guidelines for Identification of Test Configurations for Exposure to GB/HD, current version.

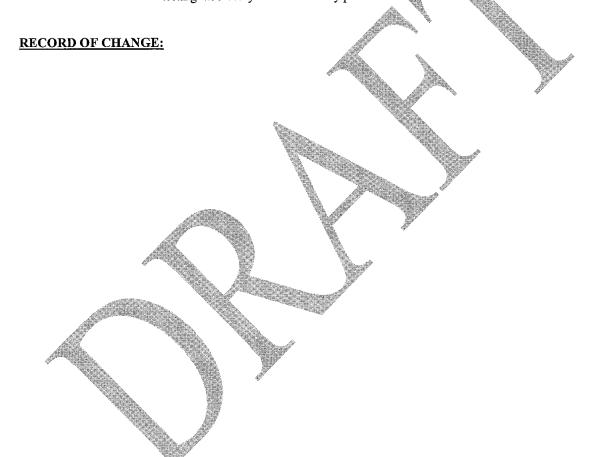
NOTE: Any PAPR test that fails as a result of laboratory test equipment failure or malfunction, laboratory electrical power loss or incorrect technician operating procedures/actions will be considered by NIOSH/NPPTL as a test termination and immediate mandatory retest upon lab supervisor confirmation.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

- 6.1.2 Precision and accuracy must be determined for each instrument in accordance with laboratory procedures and follow on NIOSH/NPPTL guidance to be published. Sound practice under NIOSH Manual of Analytical Methods supports a plus or minus 25% tolerance of a 95% confidence interval. NIOSH/NPPTL CBRN STP P&A tolerance can be higher but not lower.
- 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 Powered Air-Purifying Respirator (PAPR) 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); Federal Register Volume 60, Number 110, June 8, 1995.

7. RECORDS/TEST SHEETS

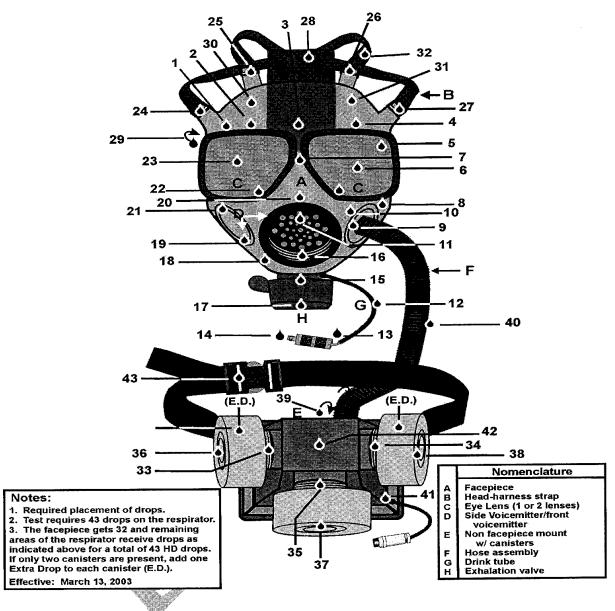
- 7.1. All test data will be recorded in the NIOSH DIEMS and STP recognized formatted CBRN PAPR test data sheets (Appendix D). All applicable data, graphs and photographs taken/made by laboratory technicians, technician supervisors or the equivalent will remain on file at the actual lab where the test was conducted, is required to be retrievable within 24 hour notification and be 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 Certification Evaluation and Testing (CET) Section Chief and await further instructions on prepare/action any uncontaminated hardware for return 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 be 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 D, LAT

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



NIOSH/NPPTL Schematic of HD Agent Droplet Placement for CBRN PAPR Application (Single Visor)



Appendix C
NIOSH/NPPTL Schematic of HD Agent Droplet Placement for CBRN
PAPR Application (Hood Type)



APPENDIX E: CBRN PAPR HD LAT Certification Test Data Summary Sheet;

NIOSH Procedure No. CET-APRS-STP-CBRN -0551

(PAPR) Vapor a A. B. C. D. E. F. G.	ITLE: Determination of Tight Fitting Facepiece and Hood Style Powered Air Performance During Dynamic Testing Against Chemical Agent Distilled Sulnd Distilled Sulfur Mustard (HD) Liquid. Task Number (TN): Manufacturer: PAPR Model #/Type: Test Start Date of Qualifier Application: Test Start Date of Remainder Application: Test End Date of Remainder Application: Primary P/N and Subject for LAT Configuration: Gaining NIOSH CBRN Approval Numbers:	r-Purifying Respirator Ifur Mustard (HD)
3. SUPPO A. B.	Each of three CBRN Tight Fitting Facepiece and Hood Style Powered Air-F (PAPR) shall demonstrate no permeation or penetration of Distilled Sulfur M and Distilled Sulfur Mustard (HD) liquid equal to or greater than the stated excursions and the stated cumulative concentration over time (Ct) including excursion data points for the duration of the eight (8) hour test. HD vapor che will start immediately after the test chamber has been sealed. Minimum Ser exposure starts after the first liquid drop is applied. Liquid volume used is components used with the respirator. Minimum volume of 0.43mL and may 0.86mL is based on the respirator final tested configuration as using one or canisters on a specific respirator assembly. Three consecutive sequential test exceeding 0.3mg/m3 will collectively constitute a failure where each test valuation of the test. Liquid agent is applied to the complete respirator at hour duration of the test. Liquid agent is applied to the complete respirator at hour the test period begins upon initial generation of HD vapor concentration an respirator system is tested in the AS IS/Ready to Use configuration and rem systems are tested in the environmentally pre-conditioned configuration prior to LAT in accordance with User Instructions? RTING REQUIRED DATA: Has Canister expired prior to LAT in accordance with User Instructions? Is Canister bent, cracked or disfigured prior to any portion of LAT? Does Canister to facepiece connection violate Form, Fit or Function?	Mustard (HD) vapor maximum peak all maximum peak nallenge concentration rvice Life for liquid dependent on ximum volume of multiple PAPR t data points at or alue equals to or a 3 minutes. The tabe exceeded for the ar 6 of the test cycle. In the definition of the test cycle and ends at 8 hours. One aining two respirator
D. E. F. G.	Is Facepiece fully serviceable prior to any portion of LAT? Are Sub Assemblies free of visible deformations or aberrations? Are remainder components environmentally conditioned prior to LAT? Does blower operate prior to LAT?	YES or NO YES or NO YES or NO YES or NO
Task Number: Manufacturer:	STP No.: Reference No.:	

Test Title: Determination of Tight Fitting Facepiece and Hood Style Powered Air-Purifying Respirator (PAPR) Performance During Dynamic Testing Against Chemical Agent Distilled Sulfur Mustard (HD) Vapor and Distilled Sulfur Mustard (HD) Liquid.

H. Did each PAPR pass or fail maximum peak excursions?							PASS of FAIL			
I. Did each PAPR pass or fail Ct?									PASS or FAIL	
J. Was all lab test equipment verified calibrated prior to LAT?								PASS or FAIL		
	K.	Were the	e SAF Configuration	Part N	umbers verified	l on the ac	tual testab	le hardwa	are?	
	120		• • • • • • • • • • • • • • • • • • • •				/#P		PASS or FAIL	
<u>QUALI</u>	FIER TE	ST # 1	Total Test Time]	Max Peak Excu	ursions	<u>Ct</u>	<u>RESULT</u>	• -	
(e.g. TN	1-HD 1		480 minutes or less	Í	Quantity failing	g 🐔	Value	Pass/Fail	1)	
REMAI	NDER T	EST # 2	Total Test Time		Max Peak Exci	arsions	<u>Ct</u>	<u>RESULT</u>	.	
(TN – H	HD 2)		Same as above (sab)).	sab		sab	sab		
<u>REMAI</u>	NDER T	<u>EST # 3</u>	Total Test Time	:	Max Peak Exc	ursions	<u>Ct</u>	<u>RESUL7</u>		
(TN – E	HD 3.)		sab		sab		sab	sab		
4.	COMM	ENTS:								
	(e.g. Cl	BRN PAI	PR candidate was test	ed in n	on-environmen	tally cond	itioned /M	[inimum]	Packaging	
	Configu	ration for	r initial Qualifier App	licatio	n. CBRN PAP	R passed i	nitial Qua	lifier App	olication LAT.	
	Howeve	er, when a	a Durability Condition	ned PA	PR was tested	in Remain	der Appli	cation tes	ts, candidate	
	CBRN I	PÁPR fai	led max peak excursi	on crite	eria and exhibit	ed caniste	r thread d	ents and b	ends as a result	of
			e – conditioning prio							
5.		TURES:								
	A. Lab	oratory T	echnician:			Da	ate:			
á	B. Labo	oratory Su	ipervisor:		D	ate:			_	
4	Legible	Signatur	es infer concurrence	with te	st summary fine	dings as in	dicated al	oove.		