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**14. ABSTRACT**

This test operations procedure (TOP) provides the current standard methods for testing the permeation of liquid and vapor challenge chemical agents and simulants through swatches of materials. Swatches can be taken from clothing or equipment that is new, with or without pretreatment(s), and/or material that was previously subjected to periods of wear under varying conditions of field use, storage, and/or environmental exposure(s). These procedures are designed to be used as part of an overall assessment program evaluating the material performance, manufacturing, and integration with other pieces of the protective ensemble.

**15. SUBJECT TERMS**

convective-flow test; Aerosol Vapor Liquid Assessment Group; AVLAG; dual-flow test; liquid challenge/vapor permeation; L/V; vapor challenge/vapor permeation; V/V; liquid challenge/liquid permeation; L/L; swatch test; pretreatments; static diffusion test; aerosol test; rain cabinet; mandrel

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TEST OPERATIONS PROCEDURE

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# PERMEATION TESTING OF MATERIALS WITH CHEMICAL AGENTS OR SIMULANTS (SWATCH TESTING)

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## 1. SCOPE.

### 1.1 Purpose.

a. This test operations procedure (TOP) provides the current standard methods for testing the permeation of liquid and vapor chemical warfare agent (CWA) and simulant challenges through swatches of materials. Such agents include the blister agent, distilled mustard (HD) and the nerve agents, sarin (GB), soman (GD), and persistent nerve agent (VX). These procedures can also be applied to toxic industrial chemicals (TICs) and other emerging threats. The following standardized methods are described in this document:

(1) Liquid challenge/vapor permeation (L/V): A quantitative test for L/V permeation of a liquid chemical agent challenge through a swatch. This method has three test cell configurations: convective-flow, dual-flow, and static-diffusion. The choice of test cell configuration is dependent on the test objectives and/or the air permeability of the swatch.

(2) Vapor challenge/vapor permeation (V/V): A quantitative test for V/V permeation of vapor chemical agent challenge through a swatch. This is done using a convective- or dual-flow configuration depending on the test objectives.

(3) Liquid challenge/liquid permeation (L/L): Qualitative tests to determine liquid penetration of liquid chemical agent challenge through swatches.

(a) The methods for mandrel, expulsion, and inverted expulsion testing of swatches are meant to give indication of chemical resistance of the tested swatch materials under stress from pressure points, etc.

(b) The rain cabinet test of swatches is a qualitative or quantitative (or both) simulant test for liquid penetration of liquid simulant through swatches in the presence of rain.

(c) The aerosol test of swatches is a qualitative test for aerosol penetration of particulate chemical simulant through swatches.

b. The swatch materials are separated into three general categories: air-permeable, air-semipermeable, and air-impermeable. Each of these material types may also be single-layered or

multilayered composites and inert, sorptive, or reactive. Swatches can be taken from clothing or equipment that is new, pretreated in the laboratory, or previously subjected to periods of wear under varying conditions of field use, storage, and/or environmental exposure, including simulated exposure. Swatches may also be taken from manufacturing sources, such as a bolt of material or a sample from a formulation that may be used for the manufacture of protective clothing or equipment.

c. Application of the procedures in this TOP can provide relative ranking or material screening information about the ability of candidate materials to resist chemical agent permeation. The data may also support models that provide additional analyses; however, the creation or use of such models is beyond the scope of this TOP.

d. Swatch testing is a required part of acquisition testing for the test and evaluation process. In addition, swatch testing can be employed as a quality control (QC) measure for production lot testing or surveillance testing of stored equipment.

1.2 Objectives.

a. This TOP provides the basic information necessary to conduct and report swatch agent resistance testing as well as discussing the required facilities, equipment procedures, test and experimental parameters, and the data to be generated using these test methods.

b. This document is a guide to preparing program-specific test plans or other test conduct planning documents (e.g., test plans, operations plans, letters of instruction, etc.).

(1) Procedures described here may need to be tailored to address the particular objectives and requirements for a specific swatch test.

(2) The planning documents will include a rationale for any modification to a TOP procedure used for a swatch test.

1.3 Limitations.

a. The procedures in this TOP are not sufficient to assess the ability of whole ensembles made from tested materials to protect the wearer. These procedures are designed to be used as part of an overall assessment program, evaluating the material performance, manufacturing, and integration with other pieces of the protective ensemble.

b. Results obtained by using these procedures may be compared with results from materials tested during the same or a previous experiment to statistically compare the agent resistance of the materials. Historic swatch testing parameter information is included wherever applicable for reference and comparison with current and/or future threat test parameters. If comparison with previous data is planned, special caution must be taken to use the same test parameters to characterize and optimize the commonality of the data. In addition, a standard reference material must be used as one of the tested samples in a given swatch test.

c. Results obtained by using these swatch test procedures cannot be correlated with specific medical or toxicological values or whole-system performance values, and therefore cannot express an absolute protection value.

d. The data obtained by these procedures cannot be correlated with specific field conditions.

e. The swatch test cell described in this TOP has a limited ability to test swatches with seams and/or closures. The swatch test cells can only be used to test materials that can be cut into 5-cm diameter swatches with exposed surface areas of 10 $cm^2$. Thicknesses should generally be less than approximately 0.32 cm; however, there is some flexibility for greater thicknesses as long as the cup can be sealed. Materials that are not relatively flat (e.g., those that include seams or have varying thicknesses) may not seal properly into the test cells. Each cell seal must be verified before testing swatches that have seams or closures.

f. This TOP is limited to approved standards and procedures. Developments in practices, equipment, and analysis may necessitate establishing new testing baselines. Additionally, standards of performance must be adjusted as technologies advance. Test procedures and parameters listed in this TOP require updating to accommodate new technologies in test items or in test instrumentation. Any updates should be described in the planning documents.

## 2. FACILITIES AND INSTRUMENTATION.

### 2.1 Facilities.

Facilities, instrumentation, and safety procedures used for swatch testing with chemical agents are strictly controlled. Additional discussion and requirements for facilities and instrumentation/equipment are included in the test procedures (Paragraph 4) and Appendix B.

| Item | Requirement |
|---|---|
| Chemical surety laboratory and chemical agent storage facility | Must be constructed to provide the capabilities and procedures needed for work with chemical agents including: (1) secure storage of agents, (2) general and specialized chemical analysis, (3) emergency response provisions, and (4) hazardous waste storage and disposal. |
| Swatch test fixture and control/data system | Contains and provides the required test conditions for each test cell. Must have the ability to control temperature, relative humidity (RH), and airflow to test cells. Fixture data system must be able to record the test conditions every minute. |

2.2 Instrumentation.

| Parameter | Measuring Device | Permissible Error of Measurement |
|---|---|---|
| Air temperature (5° to 49°C) | Thermocouple or other comparable device. | ±0.2°C |
| RH (20 to 90 percent) | Humidity probe or other comparable device. | ±3 percent |
| Airflow [0.1 to 5 standard L/min (slpm)] | Flowmeter, mass flow controller, or other comparable device. | ±3 percent |
| Differential pressure (ΔP) [24.9 ±3.7 Pascal or 0.19 ±0.03 mm mercury (Hg)] | Pressure gauge or other comparable device (L/V and V/V testing). For aerosol penetration testing only, an inclined manometer that allows the operator to read the ΔP across the swatch. | ±1 percent |
| Chemical vapor challenge concentration (10 to 130 $mg/m^3$)[1**] | Real-time (RT) or near real-time (NRT) vapor monitoring system. | ±10 percent of target concentration |
| Chemical vapor permeation sampling (test specific) | Bubblers, RT/NRT monitoring system, solid sorbent tubes (SSTs), or equivalent. | ±15 percent |
| Liquid chemical challenge ($g/m^2$) | A control coupon, analytical balance, and gas chromatograph (GC). | ±15 percent |
| Chemical analysis of permeation samples (µg) | GC, high-performance liquid chromatograph (HPLC), liquid chromatograph (LC), flame photometric detector (FPD), flame ionization detector (FID), mass spectrometer (MS), spectrophotometer, or equivalent. | ±15 percent of calibration standard |
| Aerosol particle sizes and numbers of the aerosol collected (aerosol test specific) | Particle sizer, high-resolution optical aerosol spectrometer, or an optical particle counter, or equivalents. | ±8 percent of size and number of particles /L |

**Superscript numbers and letters correspond to those in Appendix E.

3. REQUIRED TEST CONDITIONS.

a. Swatch testing requires the handling and use of chemical agents. Such testing is strictly controlled by regulations [e.g., Army Regulation (AR) 385-10[2], and Department of the Army (DA) Pamphlet (PAM) 385-61[3]]. Throughout testing, primary emphasis must be on the safety of the fixture operators and other test personnel. Swatch testing with agent must be performed in an approved surety facility.

b. For tests involving threat agents or simulants, qualified and trained operators and standard equipment will be used. The appropriate laboratory will be scheduled to conduct the test, and laboratory technicians will receive appropriate system-operating training before testing begins.

c. The swatch test fixture will contain the test cells needed for each trial and provide the required test conditions for each cell. The fixture data acquisition system will record the test conditions at least every minute. The required test capabilities are:

(1) Ability to provide an oil-free, conditioned airflow to the test cells at a rate ranging from 0.1 to 5 slpm. The flow reading must be accurate to within ±3 percent.

(2) Control of conditioned air and test cell temperature. Historically, the test temperature has been 32.2°±1.67°C, but temperatures may vary depending on test objectives. It is recommended that the test fixture be able to provide temperature control in the range from 5° to 49°C. The temperature measurement must be accurate to within ±0.2°C.

(3) Control of conditioned air and test cell humidity. Historically, the test humidity has been 80±5 percent RH. It is recommended that the fixture be able to provide humidity control in the range from 20 to 90 percent RH (noncondensing). The RH measurement must be accurate to within ±3 percent.

(4) Maintain the ΔP across the swatch at 24.9±3.7 Pascal (or 0.19±0.03 mm Hg) (for convective-flow tests). The ΔP is controlled by modulating the flow of conditioned air through the swatch to change the pressure. The pressure measurement must be within ±1 percent.

(5) Provide a controlled concentration of agent vapor in the range from 10 to 130 mg/m$^3$ ±10 percent to the swatch challenge airflow[1] (for V/V tests).

d. The swatch test cell will be designed to support the swatch with an airtight seal. The cell will also provide access for airflow through the swatch (if applicable) and will minimize loss of chemical agent vapors. Detailed descriptions of the test cell in use as of the date of this TOP are found in Appendix B.

3.1 Test Planning.

a. Test planning documents (e.g., test plan, etc.) must be developed for each test event to document the test objectives and criteria, experimental design (test matrix), test type and conditions, test item storage and tracking procedures, and data management plan.

b. The capability documents [initial capability document (ICD), capability development document (CDD), or the capability production document (CPD)], the concept of operations (CONOPS), the operational test agency's (OTA's) evaluation plan or framework, and the test and evaluation master plan (TEMP) will be used to determine the overall test structure, data required, test objectives and criteria, and data analysis method to be used.

c. Based on the information collected from the capability document, the OTA's evaluation plan or framework, and the TEMP, and in coordination with the customer, the number of test materials and the number of trials that need to be conducted will be determined.

d. A realistic sample size (based on test cost, data objectives, required statistical confidence, value, and availability) for the test material will be determined through review and coordination with the assigned operational test activity evaluator. In all cases, a design of experiment (DoE) method will be used to optimize test-material use and required-data output.

e. Test Objectives and Criteria. The test objectives and criteria will be used to determine the type of testing to be conducted as well as test cell configuration, conditioning and/or pretreatments of the swatch material, trial length, sampler selection, and sampler changes.

f. Each test plan and other planning documents must be reviewed for technical accuracy and conformance to standing operating procedures (SOPs) applicable to the specific instrumentation used; item, system, or materials under test; and tests being conducted.

3.2 Test Cell Configurations.

The following paragraphs include a description of the various test cell configurations used in swatch testing, as well as a discussion on the airflow permeability requirements for the convective-flow cell configuration. In addition, a description of swatch testing methods conducted without test cells, such as mandrel and expulsion testing, is included. A brief outline of the test methods and their applicability is presented in Table 1. **NOTE**: Test results for materials tested using different methods and/or cell configurations cannot be compared. Results are not equivalent.

3.2.1 V/V Testing.

a. There are two test cell configurations for V/V testing: dual-flow configuration and convective-flow configuration (Figures B.1 and B.2).

(1) V/V dual-flow configuration (Figure B.1) is used to test any material's relative ability to resist permeation by a stream of conditioned (temperature, humidity, flow), agent-laden air that is drawn across the top of the swatch while a stream of conditioned (temperature, humidity, flow), clean air is drawn across the underside of the swatch.

TABLE 1. SWATCH TEST METHODS AND APPLICABILITY.

| Test Method | Use With Material Types | Comments |
|---|---|---|
| Convective-flow testing – liquid challenge/vapor penetration (L/V) | Air-permeable and semi-permeable materials | Tests resistance to convective penetration by agent vapor from liquid or thickened agent contamination. |
| Dual-flow testing – L/V | Air-permeable, semi-permeable, and impermeable materials | Tests resistance to diffusive permeation by agent vapor from liquid or thickened agent contamination. |
| Static diffusion testing – L/V | Air-permeable, semi-permeable, and impermeable materials | Tests resistance to diffusive permeation by agent vapor from liquid or thickened agent contamination. |
| Convective flow testing – vapor challenge/vapor penetration (V/V) | Air-permeable and semipermeable materials | Tests resistance to convective penetration by agent vapor challenges. |
| Dual flow testing – V/V | Air-permeable, semi-permeable, and impermeable materials | Tests resistance to diffusive permeation by agent vapor challenges. |
| Mandrel testing – liquid challenge/liquid penetration (L/L) | Air-permeable, semi-permeable, and impermeable materials | Tests resistance to liquid penetration when liquid or thickened agent is applied to material under stress. |
| Expulsion testing – L/L | Air-permeable, semi-permeable, and impermeable materials | Tests resistance to liquid penetration when pressure is applied to material contaminated with liquid or thickened agent. |
| Inverted expulsion testing – L/L | Air-permeable, semi-permeable, and impermeable materials | Tests resistance to liquid penetration when material is pressed against a surface contaminated with liquid or thickened agent. |
| Rain cabinet testing – L/L | Air-permeable, semi-permeable, and impermeable materials | Tests resistance to liquid penetration when driven by rain. |
| Aerosol testing – L/L | Air-permeable and semipermeable materials | Tests resistance to aerosol penetration by liquid chemical or biological particulates. |

(2) V/V convective-flow configuration (Figure B.2) is used to evaluate the capacity of air-permeable and semipermeable protective materials (materials with an air permeability >20 $cm^3/min/cm^2$ that are usually sorptive or reactive) to remove or intercept chemical agent. In this test, conditioned (temperature, humidity, flow, and concentration), agent-laden air is drawn through a test swatch. There is no separate sampling airflow for this configuration because the air passes though the swatch and to the sampler or detector.

b. Both types of V/V tests are run for a specified trial length to determine agent permeation as a function of time or until the agent vapor detected on the clean side of the swatch reaches a set breakthrough concentration (the breakthrough threshold must be defined in the planning documents). For each type of V/V test cell configuration, the test conditions must be documented in the test plan.

3.2.2 L/V Testing.

a. The L/V test method has three test cell configurations: dual-flow, static-diffusion, and convective-flow configuration.

(1) L/V dual-flow configuration is used to test any material's ability to resist permeation by chemical agent. A stream of laboratory air, conditioned to a specific temperature, humidity and flow rate, is drawn across the top of a swatch challenged with droplets of agent. A clean airflow is drawn across the underside of the swatch for sampling. This method causes the agent challenge to evaporate over time. Historically, the top surface airflow rate has been 0.25 L/min, and the underside airflow rate has been 0.30 L/min.

(2) L/V static-diffusion configuration is used to test any material's ability to resist the permeation of chemical agent drops placed on the surface of the swatch. In contrast to the dual-flow configuration, no air is passed across the top of the swatch, keeping the challenge static and less likely to evaporate. A clean airflow is drawn across the underside of the swatch for sampling. Historically, the sampling airflow has been 0.30 L/min.

(3) L/V convective-flow configuration can be used to test only air-permeable or semipermeable materials (materials with an air permeability of >20 $cm^3/min/cm^2$). Liquid agent droplets are placed on the surface of the swatch, and a clean airflow, conditioned to a specific temperature, humidity and airflow rate, is drawn through the test swatch. The test is performed by controlling the flow rate of the air through the swatch and measuring the $\Delta P$ across the top of the swatch. Historically, the $\Delta P$ across the swatch top is maintained at 24.9±3.7 Pascal (or 0.19±0.03 mm Hg).

b. All three L/V test configurations are run for a specified trial length to determine agent permeation as a function of time, or until the agent vapor detected on the clean side of the swatch reaches a set breakthrough concentration threshold as defined in the planning documents.

3.2.3 Air Permeability Test for Convective-Flow Configuration.

Two air permeability test options are available to determine the appropriate test cell configuration and test method for a particular material: the American Society for Testing and Materials (ASTM) Method D737-04[4], and the fixture airflow test. **NOTE**: The ASTM air permeability test may be omitted if all materials undergo the swatch test fixture airflow test (Paragraph 3.2.3.c).

a. To test if the convective-flow configuration for both V/V and L/V testing is possible, ASTM D737-04[4] may be employed with two modifications. The modifications are:

(1) A ΔP of 24.9±3.7 Pascal (or 0.19±0.03 mm Hg) is required.

(2) The swatch material must be conditioned at the swatch test conditions (temperature, humidity, and airflow rate) required in the planning documents.

b. Materials with a measured permeating airflow rate >20 $cm^3/min/cm^2$ at a ΔP of 24.9±3.7 Pascal (or 0.19±0.03 mm Hg) are appropriate for testing in the convective flow configuration. If the measured airflow through the material is <20 $cm^3/min/cm^2$, then the convective configuration should not be used.

c. To determine the test method to be used for materials with permeating airflow rates below 20 $cm^3/min/cm^2$, a fixture airflow test will be conducted on replicate swatches which will be cut and treated (as appropriate) and then placed on the swatch test fixture. These swatches will not be contaminated and will be tested for the duration specified in the planning documents using the convective flow test method. The ΔP data will be analyzed to determine if the test parameters for each replicate swatch were in control. Materials that maintained ΔP control may be tested using the convective flow configuration. Materials without ΔP control must be tested using the dual-flow or static-flow configurations.

3.2.4 L/L Testing.

a. Mandrel Testing.

(1) Application. This procedure is used to test air-permeable, semipermeable, and impermeable materials for liquid agent penetration by applying a droplet of liquid or thickened agent to a swatch of material that is under tension. A piece of M8 sampling material is placed beneath each swatch to determine penetration. The method simulates the chemical exposure of the material in an item being worn that is stressed by the knees, elbows, etc. The test is qualitative (pass/fail) based on a color change in the M8 sampling paper.

(2) Equipment. The equipment used for this test includes a mandrel (41-mm interior-diameter glass tubing), 234-g weights, and M8 chemical agent detector paper. A diagram of the mandrel L/L test assembly is in Figure B.3.

b. Expulsion Testing.

(1) Application. This procedure is used to test air-permeable, semipermeable, and impermeable materials for liquid agent penetration by placing droplets of liquid or thickened agent on a swatch of material and applying pressure on top of the droplets. A piece of M8 sampling paper is placed beneath the swatch. This method simulates a situation in which worn, externally contaminated items are pressed against a surface. This test is qualitative (pass/fail), based on a color change in the M8 sampling paper.

(2) Equipment. The equipment used for this test includes a glass plate, a weight which provides 70.2 $g/cm^2$ of pressure to the contaminated area, and M8 chemical agent detector paper. A diagram of the expulsion L/L test assembly is in Figure B.4.

c. Inverted Expulsion Testing.

(1) Application. This test is similar to the expulsion test (Paragraph 3.2.4.b); however, the liquid or thickened agent droplets are placed on a flat glass surface, and the material swatch is then pressed against the spread-out droplets. This method simulates a situation in which worn material is pressed against an agent-contaminated surface.

(2) Equipment. The equipment used for this test includes a glass plate, a weight which provides 70.2 $g/cm^2$ of pressure to the contaminated area, and M8 chemical agent detector paper. A diagram of the inverted expulsion L/L test assembly is in Figure B.5.

d. Rain Cabinet Testing.

(1) This test is used to determine the effects of rain on agent-resistant materials and can be either qualitative or quantitative. The test swatches are mounted over blotting paper in cups that are challenged with triethyl phosphate (TEP) simulant and then subjected to rain for 30 minutes. The blotting paper can be visually inspected for wetness (yielding a qualitative result) and/or chemically analyzed for simulant penetration (yielding a quantitative result). This procedure can be used with other simulants and/or chemical agents.

(2) A circle of standard blotting paper [made by American Association of Textile Chemists and Colorists (AATCC), Research Triangle Park, North Carolina] is placed in the opening of each stainless steel swatch holder. This is covered with a 7- × 7-mm swatch of the fabric system to be tested. Material is secured on the holder with rubber bands. The surfaces of the material swatches are then spotted with 4 to 5 drops (10 μl) of TEP using a template. Swatches are then placed in the calibrated 1-in/hr rainfall area of the raincourt, the rainfall turned on, and the time noted.

(3) After 15 minutes, the rain is stopped temporarily, and the swatches are checked to see if the blotting paper under the swatch is wet. Swatches with totally wet blotters are removed and the time noted. Rainfall is restarted and swatches are subsequently checked every 30 minutes. Each time a blotter is found to be wet, the swatch is removed and the length of time in rainfall is noted. The test is terminated after 8 hours if no blotters are found to be wet.

(4) Using a Buchner funnel and vacuum pump, wet blotters are extracted with small amounts of distilled water. Any liquid in the swatch holder is added to the extract for analysis. Extracts are analyzed for TEP content using GC analysis. Results are compared with those of standard fabric swatches run at the same time and are analyzed using techniques similar to those used in L/V tests.

e. Aerosol Penetration Testing.

(1) Application.

(a) Aerosol penetration refers to the passing of airborne aerosol through the swatch. This test evaluates the performance of air-permeable or semipermeable materials with measured permeabilities of at least 20 $cm^3/min/cm^2$ to resist penetration by aerosols.

(b) Aerosol penetration of protective clothing is primarily dependent on the physical properties rather than the chemical properties of the aerosol particles and the clothing. Therefore, aerosol testing can be reliably performed with nontoxic chemical and biological simulants that mimic the physical attributes of chemical/biological agents.

(c) The measurements required in this testing are the sizes and counts of the aerosol particles beneath the swatches (downstream) and in the challenge air upstream of the swatches, detected in 1-minute intervals. The sizes and counts of the aerosol particles passing through the swatch are compared with the sizes and counts of the aerosol particles in the challenge airstream to calculate a penetration ratio (or protection factor).

(2) Equipment. The tests should be performed with an aerosol testing apparatus (illustrated in Figure B.6, Appendix B), or a similar apparatus. A 12.7-cm diameter fabric swatch should be mounted in a holder (similar to that shown in Figure B.7) which is placed in the test apparatus. The poly-dispersed challenge aerosol particles should be generated from a nontoxic, low-volatility liquid (e.g., oleic acid). Aerosol particle sizes and numbers are measured with an Aerodynamic Particle Sizer®* (APS™, TSI Incorporated, Shoreview, Minnesota), or equivalent, upstream and downstream of the fabric swatch.

3.3 Experimental Design.

3.3.1 Trial Matrix.

The trial matrix will be designed to balance the test materials among trials and fixtures as much as possible. The test material, positive control, and negative control swatch positions will be randomized within each trial. Randomization will be based on the swatch identification control number (SICN) assigned during the receipt inspection (Paragraph 4.4).

3.3.2 Sample Size.

The sample size will vary in accordance with (IAW) each test program requirements and type of agent challenge. If not specified in an OTA evaluation plan or framework, TEMP, or equivalent document, the sample size for each test/test condition will be determined based on the following factors:

a. The test hypothesis.

b. Selected statistical analysis technique.

c. Confidence or significance level, including alpha and beta.

d. Expected data variability (based on previous test data).

e. A quantifiable difference that is meaningful, e.g., the amount of permeation needed to determine that one material is better than another.

*The use of brand names does not constitute endorsement by the Army or any other agency of the Federal Government, nor does it imply that the brand-named item is best suited for the intended application.

3.3.3 Swatch Pretreatments.

Swatch pretreatments may be used to test how the agent resistance of a material will change when exposed to various contaminants. The planning documents must state what, if any pretreatments will be used. Common pretreatments are: petroleum, oil, and lubricants (POLs), water (fresh and simulated sea water), decontaminants, firefighting foam, insect repellant [N,N-diethyl-meta-toluamide (DEET)], body fluids (simulated sweat, urine, blood, and feces) and other compounds to which personnel may be exposed during a mission. Appendix C contains more information on pretreatments, the constituents for simulated fluids, instructions for application, and time guidelines.

3.4 Environmental Documentation.

Testing will comply with all local, state, and federal regulations. Appropriate documentation will be prepared and submitted and approval received before testing begins.

3.5 Safety.

Applicable safety and surety regulations will be reviewed to ensure that all test procedures are in compliance.

a. Chemical agents are extremely toxic and exposure can be fatal; therefore, during testing with agents, the primary emphasis must be placed on safety.

b. Tests using chemical agents will be conducted IAW DA PAM 385-61[3]and AR 50-6[5], approved agent handling SOPs, and procedures specified in the planning documents.

c. A composite risk management plan and hazard analysis[3] must be written and approved before the test plan is written.

d. All test participants must thoroughly understand the test plan and applicable SOPs, and must acknowledge their understanding and training by signature.

3.6 Quality Assurance (QA) and QC.

3.6.1 QA Planning.

a. A QA plan must be prepared for each test program to ensure that all controllable variables are controlled and that appropriate records are kept throughout the duration of testing. Variables that cannot be controlled must be identified in the test plan. Test variables include but are not limited to: purity and stability of the challenge agents or simulants, purity and stability of any decontaminants, calibration and maintenance of instrumentation and disseminators, accuracy and precision of the laboratory analysis, and quality and uniformity of all swatch samples.

b. The condition of the test item from which swatches are cut at the time of testing is an important test variable. The test items must be inspected IAW TOP 8-2-500[6]. Pretrial inspection of the swatches that is performed as part of a subtest completed before swatch testing is required. Inspection data and certificates of compliance or similar documentation must be reviewed to en-

sure that exterior material surfaces, material finishes, and packaging, meet specifications. Except for situations such as pretreating swatches at the testing facility, the materials should be tested in as-received condition, matching, as closely as possible, the condition in which they would be issued to warfighters in the theater of operations. Swatch testing may be required periodically throughout the equipment life cycle if normal wear or storage is shown to be a major factor in survivability.

c. Test Conduct. Testing must always be conducted IAW approved test documentation, such as technical manuals, field manuals, equipment operating instructions, SOPs, the approved test planning directive, OTA evaluation plan or framework, TEMP, and the test plan. Any deviations from the test documentation will be included in the test plan and/or report and approved by the appropriate authority.

3.6.2 QC Swatches.

Control swatches demonstrate control of all parameters affecting test results across trials and test programs, and verify that the analysis was not affected by the presence of positive and negative interferents. Each trial will have two sets of control swatches, positive and negative. Positive control swatches will be contaminated with the challenge chemical, and negative control swatches will not be contaminated.

a. At least one positive control is required per trial. The positive control must have a well-defined permeation profile. The limits for the positive control will be based on historical data or system verification and validation data. Historically, 6× laundered Joint Service Lightweight Integrated Suit Technology (JSLIST) material has been used as the positive control swatch material. JSLIST material is available from several sources. For dual-flow tests, it is recommended (although not required) that butyl rubber should be used with HD and neoprene should be used with GD to get repeatable and consistent results. The butyl rubber has been obtained from Enterprise Rubber, Inc., Akron, Ohio, and the neoprene obtained from AAA-ACME Rubber Company, Tempe, Arizona. Butyl rubber and neoprene obtained from other manufacturers are acceptable if equivalent specifications are met. The specifications for the butyl and neoprene are in Appendix B.

b. The negative control swatch should be butyl rubber or neoprene (the same as the positive control material), with the option of an uncontaminated test swatch.

**NOTE**: Although unlikely, it is possible for some test items or coatings to cause a false positive. What is more likely is cross contamination due to fixture materials leaching agent or inadequate decontamination. These factors tend to be masked when using a negative control swatch that is made to trap agent. Using an empty cup or a standard negative control material in addition to a test swatch negative control is a viable option to establish the presence or absence of any residual contaminant.

3.6.3 Data Management Plan.

The data management plan will ensure that all data collected will be reviewed and receive a QC check by qualified individuals. This plan will provide for the timely and accurate processing of test data for submittal to the customer.

a. Data Control.

(1) All data will be handled using chain of custody (CoC) procedures.

(2) All test, analytical, and environmental QC data will be audited to ensure the results are within the parameters detailed in the organization's SOPs and approved planning documents.

b. QC Review.

(1) All test and data management procedures and QC data must be verified throughout testing as specified in this TOP or test planning documents.

(2) QC data will be recorded and reported as required by the specific test program.

(3) All aspects of testing will be performed with an emphasis on acquiring quality results that are credible and verifiable.

4. TEST PROCEDURES.

4.1 Test Method Outline.

a. Receipt inspection will be conducted on the test item(s) to document as-tested material conditions (Paragraph 4.4).

b. The agents/simulants will be prepared for application (Paragraph 4.5).

c. Swatch pretreatment, if required, will be conducted (Paragraph 4.6).

d. The sampling/analytical system to be used will be selected and prepared (Paragraph 4.7).

e. The L/V or V/V test cells will be assembled and sealed with the swatches inside (Paragraph 4.8).

f. A fixture check will be performed. Laboratory hood operation will be verified and environmental conditions for the test stabilized (Paragraph 4.9).

g. The L/V or V/V test cells will be installed, and the swatches will be environmentally preconditioned (i.e., conditioned with the correct test temperature and RH) within the fixture, if required (Paragraph 4.10).

h. Agent/simulant challenges will be applied to the swatches and the swatch trial will begin (Paragraph 4.11).

i. The fixture conditions and sampling system will be monitored during trial execution (Paragraph 4.12).

j. The trial will end and the procedures will be followed to collect and analyze all remaining sampling system data (Paragraph 4.13).

k. The L/V or V/V test cells will be dismantled and cleaned and the swatches will be disposed of (Paragraph 4.14).

l. Test data will be reviewed for consistency and acceptability during trial execution or as soon as possible after test (Paragraph 4.15).

4.2 Significance and Use.

a. Application of the procedures in this TOP can provide relative ranking or material screening information about the ability of candidate materials to resist chemical agent permeation. The data may also support models that provide additional analyses; however, the creation of such models is beyond the scope of this TOP.

b. The procedures in this TOP are not sufficient to assess the ability of ensembles made from any test material to protect the wearer. These procedures are designed to be used as part of an overall assessment program evaluating the material performance, manufacturing, and system integration with other pieces of the protective ensemble.

c. Results obtained by using these procedures may be compared with results from the same or a previous experiment to determine any statistical difference in the agent resistance of the materials. If comparison with previous data is planned, special caution must be taken to use the same test parameters to characterize and optimize the commonality of data. In addition, a standard reference material must be used.

d. These procedures are not designed to yield results for correlation to specific medical or toxicological values or to protective system performance. Therefore, results obtained from following these procedures cannot be used to express an absolute protection value.

e. The data obtained by following these procedures cannot be correlated to specific field conditions.

4.3 Calibration and Standardization.

a. Most chemical analytical equipment (e.g., GCs, LCs, etc.) will be calibrated by following the general chemical analytical calibration guidelines in SOP WDC-ANA-004[7].

b. Standards and QC samples will be prepared using the procedures described in SOP DP-0000-M-073[8]. QC samples will be prepared separately from standards[8].

4.4 Receipt Inspection.

a. All test items will undergo a receipt inspection as part of the QA/QC process (Paragraph 3.6.1.b). Test item and/or swatch control and accountability or CoC procedures must be implemented immediately upon receiving the test items/swatches.

b. Both sides of each test item, piece of material, or swatch will be inspected for rips, tears, stains, pinholes, and other damage using visual inspection or light transmission methods. Be aware that pinhole and other damage to impermeable materials may not be observed during the cup pressure test (Paragraph 4.8.b); therefore thorough receipt inspection is critical. Any abnormal characteristics of the swatches will be noted and the observations recorded in a receipt logbook. Surfaces will be inspected for foreign materials not normally present on the item (dust, mud, grease, or markings). Foreign materials may be removed by brushing or vacuuming. The removal of foreign materials will minimize the bias that could cause analytical instruments to overestimate or underestimate the true agent permeability of the material being tested. Where feasible, a photographic record (with metric scale) will be made of all damaged test items/swatches.

c. As part of the receipt inspection and test item tracking process, each test item will be given a test item control number (TICN). Each swatch will be given a SICN based on the TICN. These numbers will be used to uniquely identify each test item by test program and to track the item throughout the test process.

d. The unique SICN will identify each swatch by stratum (location on the original test item from which the swatch was cut), test program, material type, challenge agent, and trial sequence number. The CoC form will be initiated after the swatch is cut from the test item.

e. Swatches and test items will be prepared and stored until needed for trials in a clean, environmentally-controlled storage location. The storage location will have limited or restricted access to minimize the risk of contamination. Limiting access to the storage location is particularly important for materials that are sorptive or reactive.

f. Once individual received or cut swatches are sorted for testing, each will be placed in a sealable plastic bag that has been labeled with the TICN or SICN.

g. Swatches comprising multilayered or composite materials will be kept with the layers together and in the correct order and orientation as the material would be worn. A small, clear plastic bag is recommended for storing multilayered or composite-material swatches.

h. The CoC document for each item/swatch must be updated at the time custody is transferred for further processing. The CoC document must contain at a minimum:

(1) The TICN/SICN for the item.

(2) The date and time the item/swatch was received.

(3) The signature of the individual relinquishing custody.

(4) The signature of the individual receiving custody.

(5) A brief description of the operation conducted with the item/swatch.

(6) The current location of the item/swatch.

4.5 Agents/Simulants.

a. Agent Purity. The purity of the chemical agents used must be known and recorded as test data. Single agents used for swatch testing must be at least 95 percent pure (SOP WDC-ANA-031[9]). If a weapons-grade agent mixture is used, the purity of the defining agent must be measured and recorded as test data. If simulant testing is necessary, a simulant-to-agent correlation must be documented.

b. The agents traditionally used for swatch testing are HD, GB, GD, thickened GD (TGD), and VX.

c. Other potential challenge contaminants [e.g., TICs or other emerging threats] may be used as specified in the TEMP, SEC, and/or test plan.

4.6 Swatch Pretreatment.

If swatch pretreatments are used, the pretreatment process should be conducted before the swatches are assembled into the test cells. See Appendix C for pretreatment procedures. **NOTE**: If swatches are pretreated, no swatch preconditioning will be conducted.

4.7 Sampling/Analytical System.

Exact preparation will depend on the sampling/analytical system used for each specific test and is subject to local SOPs. However, it should be noted that:

a. Bubblers should be filled with the solvent specified in the program planning document using a calibrated dispenser and the top and side covered and sealed with Parafilm™ (Pechiney Plastic Packaging Company, Chicago, Illinois)[a], or an equivalent material, until ready for use. Bubbler collection efficiency at the temperature, flowrate, and humidity of the test environment must be demonstrated as well as the analyte stability (Appendix B).

b. SSTs must be clean of interferents. The capacity of the SST sorbent must not be exceeded, and SSTs must be free of any residual adsorbed chemical before they may be reused (Appendix B).

c. The chemical analysis will be conducted using the appropriate number of standards, blanks, and analytical controls IAW WDC-ANA-030[10].

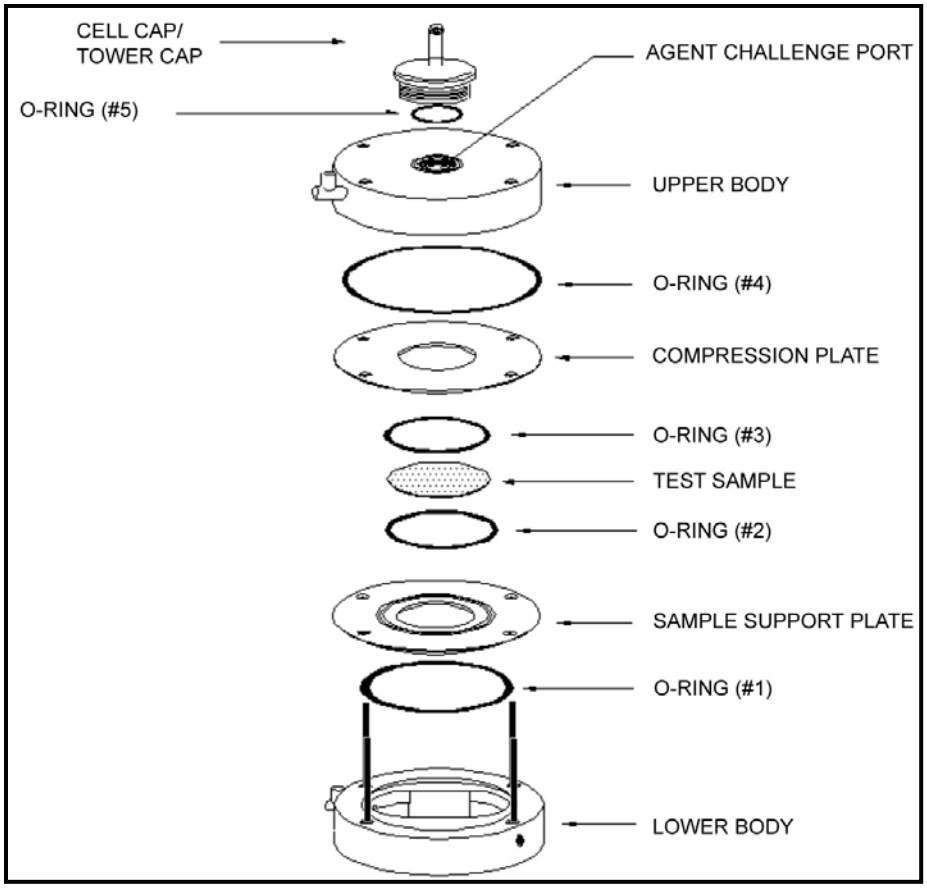
[a]The use of brand names does not constitute endorsement by the Army or any other agency of the Federal Government, nor does it imply that the brand-named item is best suited for the intended application.

d. RT and NRT monitors must be calibrated before use. In addition, a calibration or QC check should be done before and after testing to verify that the system remains in calibration for the test duration.

4.8 Test Cell Assembly and Sealing (L/V or V/V Testing).

a. Test Cell Assembly. The test cell should be assembled IAW the procedures in the following paragraphs. An expanded schematic diagram is provided in Figure 1. **NOTE**: Nitrile O-rings, part numbers 2-227N70, 2-232N70, and 2-224N70 (HydraPak Seals, Inc., Salt Lake City, Utah), or equivalent O-rings should be used.

(1) The trial matrix (Paragraph 3.3.1) should be used to determine which swatch goes in each test cell, including placement of control swatches.




**NOTE**: The cell cap is replaced with the tower cap for the convective flow method (Figure B.2).

Figure 1. Swatch permeation test cell assembly.

(2) The test cell must be configured for the appropriate test method. See Figures B.1 and B.2 for the cell configurations for the different methods.

(3) An O-ring will be placed on the lower body of the test cell. This will be O-ring 1.

(4) The sample support plate will be placed on O-ring 1, and a second O-ring (O-ring 2) will be placed in the groove on the sample support plate.

(5) The swatch will be removed from the storage container and placed in the depression in the sample support plate. O-ring 3 will be placed over the swatch.

(6) O-ring 4 will be placed in the upper body of the test cell, and the compression plate will be positioned over O-ring 4.

(7) The upper body will be inverted and aligned with the lower body.

(8) Using the four cell lugs, the cell halves (upper and lower bodies) will be clamped together to seal the cell.

(9) O-ring 5 will be inserted into the groove around the agent challenge port in the upper body of the test cell, and the cell cap or tower will be screwed into place.

b. L/V and V/V Test Cell Seal Check. Only cells that have passed a pressure test (seal check) will proceed to chemical agent testing. Cell seal checking can be performed only before the agent challenge is disseminated because of the safety risks of handling exposed materials and because materials may become altered or damaged by the chemical agent. The following procedures will be used to verify each test cell seal:

(1) All ports of the test cell will be sealed except for the outlet port.

(2) For convective-flow tests, the cell will be depressurized to 996.4 Pascal or 7.5 mm Hg, allowed to equilibrate to 747.5 Pascal (5.6 mm Hg), and sealed. The pressure will be monitored for 2 minutes. A ≥249.1-Pascal (≥1.9-mm-Hg) drop in pressure will be considered a failure to seal and will require the cell to be reassembled. **NOTE**: The seal check pressures are different than the operating pressure, which is 24.9±3.7 Pascal (or 0.19± 0.03 mm Hg).

(3) For static- and dual-flow methods, the cell will be depressurized to 747.5 Pascal or 5.6 mm Hg. The operator may notice a pressure "bounce" on the manometer. This is caused by the elasticity of some materials that are tested using these methods. When the pressure reaches 747.5 Pascal (5.6 mm Hg), the seal check time will be started. Because of the impermeable nature of the swatch materials tested using these methods, this seal check procedure must be performed for the upper portion of the test cell as well to ensure all ports are reassembled and sealed. A ≥249.1-Pascal (≥1.9-mm-Hg) drop in pressure will be considered a failure to seal and will require the cell to be reassembled.

4.9 Fixture Check/Test Setup.

a. Laboratory hood operation will be verified. The test assemblies or fixtures will be set up in chemical fume hoods.

b. The fixture RH system will be verified as functioning and delivering the correct RH, as specified in the test plan.

c. The temperature will be within permissible error margins, as specified in the test plan.

d. If critical orifices are used, the airflow will be verified as being within 10 percent of the target airflow, as specified in the test plan.

e. The stabilization of the environmental conditions for the test will be verified.

4.9.1 L/L Tests.

An environmental chamber will be used if the temperature and RH of the laboratory room air do not meet the test requirements. The environmental chamber will be installed in the chemical fume hood, and the chamber will be set for the test temperature and RH specified in the planning documents.

4.9.2 L/V and V/V Tests.

a. Optionally, an environmental chamber may be used if the temperature and RH of the laboratory room air do not meet test requirements. The environmental chamber will be installed in the chemical fume hood, and the chamber will be set for the test temperature and RH specified in the planning documents.

b. The cell holder(s) and the manifolds will be installed in the chemical fume hood or environmental chamber and prepared to receive the loaded test cells.

c. The MINICAMS® (a miniature, automatic, continuous air-monitoring system), or equivalent RT/NRT instrument, will be installed.

d. If bubblers are used, they will be filled with the proper collection solvent using a calibrated repipetter or equivalent device. The collection solvent will incorporate an internal standard so adjustments can be made for solvent evaporation/water condensation during sampling.

e. If SSTs are used, they may be purchased prefilled with the appropriate sorbent or prepared for reuse. If being reused, they will be cleaned before refilling by heating and purging. The absence of any residual chemical will be verified by GC analysis.

4.10 L/V and V/V Test Cell Installation and Swatch Preconditioning.

a. Each test cell will be placed in the cell support rack located in the test fixture.

b. For convective-flow mode, the upper pressure monitoring line will be connected to the cell tower, and the lower pressure monitoring line will be connected to the pressure monitoring port.

c. The sampler sidearm will be inserted into the bottom effluent port. For dual-flow test cells, the top effluent port will be connected to the dual-flow vacuum line.

d. The supply air manifold will be connected to the air inlet port(s) located on the test cell. There is one air inlet port on the convective- and static-flow test cells and two air inlet ports on the dual-flow test cell.

e. The sampler sidearm will be connected to the mass flow controller using flexible tubing.

f. Test environmental conditions will be established. Usually the test temperature will be 32.2°±1.67°C and the test RH will be 80±5 percent, but this can vary depending upon test requirements. The airflow or ΔP (depending on the test cell configuration) will be maintained as specified in Table 2 for 30 minutes. **NOTE**: Pretreated swatches will not be preconditioned.

g. For liquid agent challenges, the test cells will be removed from the test assembly immediately before the agent is applied. For vapor challenges, the cells will remain in the test fixture.

TABLE 2. TEST PARAMETERS FOR LIQUID CHALLENGE/VAPOR PENETRATION (L/V) SWATCH TESTING.

| Parameter[a] | | Standard[b] | Rationale |
|---|---|---|---|
| Convective-flow rate | | Flow required to achieve a differential pressure (ΔP) of 24.9±3.7 Pascal (or 0.19± 0.03 mm Hg) across the material | Standard procedure. |
| Dual-flow rate | Top | 0.25 L/min | Standard procedure. |
| | Bottom | 0.30 L/min | |
| Static-diffusion rate | Top | 0 L/min | Standard procedure. |
| | Bottom | 0.3 L/min | |
| Surface density | | As required (historic: 10 g/m$^2$) | Standard procedure. |
| Number and volume of drops (to achieve historically required 10 g/m$^2$ challenge density) | HD | Eight 1-µL drops | Number and volume needed to achieve desired agent challenge density. |
| | VX, GD | Ten 1-µL drops | |
| | TGD | Two 5-µL drops | |
| Temperature | | 32.2°±1.67°C (90°±2°F) | Standardize across test methods. |
| Relative humidity (RH) | | 80±5 percent | Standardize across test methods. |
| Agent purity | | > 95 percent | Minimized interference from impurities. |
| Sampling intervals | NRT samplers | 3 to 30 min per data point | Standard procedure. |
| | Cumulative samplers (bubblers and SSTs) | 1 to 16 hr per data point. | |
| Length of test | Convective-flow | 8 to 24 hr | Specified in the test plan. |
| | Dual-flow | 8 to 24 hr | |
| Positive control material (PCM) and negative control material (NCM) | | 6×-laundered JAM for PCM for convective flow. Neoprene (for GD) or butyl rubber (for HD) for PCM dual/static flow. NCM should be same as PCM, but not contaminated. | Standard procedure. |
| Areas sampled | | Bottom (or top and bottom) of each test swatch depending on test requirement. | Standard procedure. |

[a]HD – distilled mustard; VX – persistent nerve agent; GD – soman; TGD – thickened GD; NRT – near real-time; SST – solid sorbent tube.

[b]ΔP – differential pressure; Hg – mercury; JAM – Joint Services Lightweight Integrated Suit Technology (JSLIST)-approved material.

4.11 Agent Challenge and Swatch Trial Initiation.

4.11.1 Vapor Challenges for V/V Testing.

a. The vapor challenge concentration will be verified by an appropriate RT/NRT monitor. The effluent from the vapor generator will be routed to the waste air until the challenge concentration for the trial has stabilized.

b. V/V Trial Initiation. When conditioning of the swatches is complete (if required) and the vapor challenge has been verified to be at the correct concentration, the vapor challenge will be routed to the test cells and the swatch trial will commence. When the trial starts, the control system will be monitored to ensure that the airflow and other conditions remain constant for each test cell, as detailed in the planning documents. The start and stop times will be recorded.

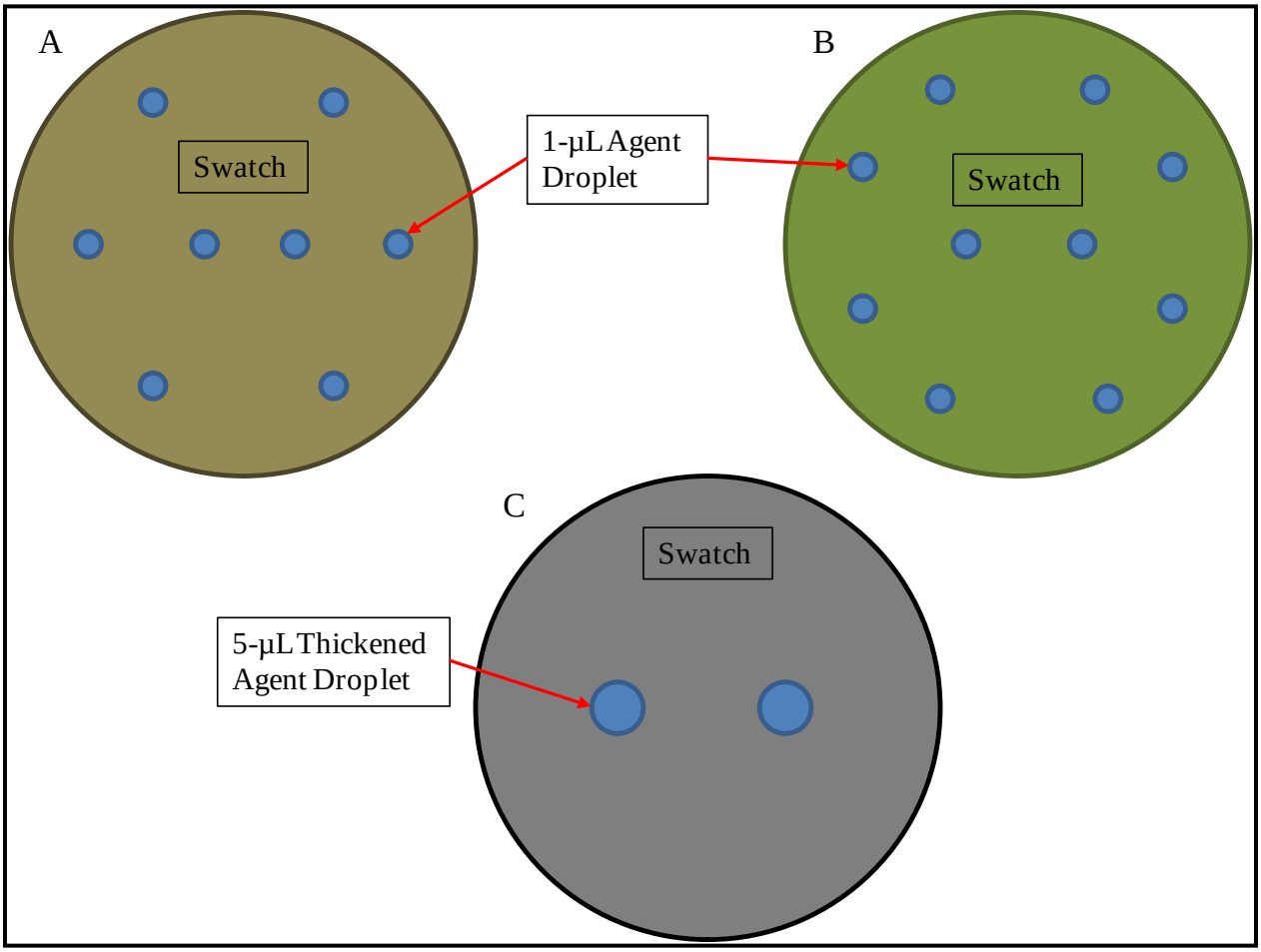
4.11.2 Liquid Challenges for L/V and L/L Testing.

a. Historically, a 10-µg challenge volume has been applied to a swatch with a 10-$cm^2$ exposed area to achieve a 10-$g/m^2$ contamination density. Future programs may require different contamination densities for testing. Contamination densities and spiking patterns must be provided in the test plan. The following examples for neat and thickened forms of agent reflect the historical challenge volumes and drop placement:

(1) Neat Agent. For HD, the recommended droplet pattern consists of six 1-µL droplets evenly spaced around a 2.54-cm diameter circle on the upper swatch surface with two additional 1-µL droplets placed 0.81 cm apart in the center of the swatch (Figure 2, Example A). For nerve agents, the recommended pattern consists of eight 1-µL droplets evenly spaced around a 2.54-cm diameter circle on the upper swatch surface and two 1-µL droplets placed 0.81 cm apart in the center of the swatch (Figure 2, Example B). This will provide the historically required 10-µg challenge on a swatch that has a 10-$cm^2$ exposed area.

(2) Thickened Agent. For thickened nerve agent, two 5-µL droplets should be placed 0.81 cm apart, equidistant from the center of the upper swatch surface (Figure 2, Example C). This will provide the historically required 10-µg challenge on a swatch that has a 10-$cm^2$ exposed area.

(3) If refrigerated agent will be used, the agent will be brought to room temperature so that it will have the proper viscosity.




**NOTE**: Top view schematic of swatches. Examples of the historically required 10-µg challenge on a swatch that has a 10-cm$^2$ exposed area: Swatch A shows placement for distilled mustard (HD); Swatch B shows placement for nerve agents; Swatch C shows placement for thickened nerve agents.

Figure 2. Examples of agent droplet location on swatches.

b. The following equation (Equation 1) will be used to calculate the number of droplets required to achieve the desired contamination density:

*Equation 1*

$$N = \frac{10DA}{\rho VC}$$

Where:

$N$ = number of droplets
$D$ = contamination density (g/m$^2$)
$A$ = area to be contaminated (cm$^2$)
$\rho$ = agent density (g/mL)
$V$ = volume of agent per droplet (µL)
$C$ = purity correction factor

c. Agent Application QC. A QC check will be conducted to verify the challenge level delivered by the spiking method. Verification will be performed gravimetrically as well as with a GC or wet chemistry procedures.

(1) An inert swatch [a Teflon® disc (DuPont™, I.E. du Pont de Nemours and Company, Wilmington, Delaware) or equivalent] will be weighed on an analytical scale. The inert swatch will be spiked and reweighed, and the mass of the actual agent spike will be recorded. Results should show that the application method delivered a mass of agent within ±15 percent of the target mass of 0.01 g (procedures for addressing anomalous results should be included in the test plan). The contaminated swatch will be placed in a jar containing a known volume of solvent for later analysis. This procedure is to be repeated after half of the test swatches have been challenged and again after the last test swatch has been challenged.

(2) The contaminated inert swatches in the jars of solvent will be submitted to the analytical lab. The jars will be agitated for at least 1 hour to ensure that the agent is completely extracted from the swatch. After agitation, the solvent will be transferred to a vial. Analysis will be performed using a GC or wet chemistry procedures. Results should show that the application system delivered a concentration of agent within ±15 percent of the target concentration for each swatch (procedures for addressing anomalous results should be included in the test plan).

d. Agent Application Procedure. The cell caps (Figure 1) will be removed (where applicable), and each swatch will be challenged with agent. After contamination with the challenge, each swatch will be visually inspected, and the following data items will be recorded to verify that the agent was correctly applied.

(1) Quantity of drops and pattern of application.

(2) Swatch surface appearance (wavy, concave, convex, etc.).

(3) Appearance of the drops on the swatch (beaded, absorbed, spread, runoff, wicking, etc.). It is recommended that a digital picture be taken before and after each test to indicate the interaction of the agent drop(s) with the swatch.

e. L/V Trial Initiation. After each swatch cell is challenged, the cell will be reinstalled in the test fixture and the fixture will be allowed to return to operating temperature. After environmental parameters are reestablished, the trial will be initiated by turning on the airflow/pressure control at the cells. When the trial starts, the control system will be monitored to ensure that the airflow and other conditions remain constant for each test cell, as detailed in the planning documents. Critical time points must be collected for all samples:

(1) When each swatch is contaminated (spike time).

(2) When the airflow/pressure is turned on.

(3) When the airflow/pressure is turned off.

(4) The spike-to-trial-start time, which is the time that elapses from the spiking of the first swatch until the trial begins, will be recorded. Every effort will be made to minimize the spike-to-trial-start time and to maintain consistent times between trials and test programs because variations in these times can cause variability in permeation data. Starting cells individually or in small groups can assist in minimizing spike to trial start time.

(5) The start and stop times for each cell will be recorded.

4.11.3 L/L Testing.

Several different test fixtures and procedures are used for L/L swatch testing. All require the use of M8 chemical agent detector paper to ascertain liquid penetration. This detector paper has limited sensitivity for detection of liquid agents and provides only a pass/fail result. Sorptive samplers, such as silicone rubber, may be used in place of the M8 chemical agent detector paper to provide a quantitative estimate of the amount of penetration occurring over the period of the test. In general, each test is conducted for a total of no longer than 1 hour; however, the time could be longer or shorter as designated in the test plan.

a. Mandrel Test.

(1) A 5- × 15-cm material swatch will be placed evenly over a piece of M8 chemical agent detector paper on a 41-mm diameter round glass mandrel so that the outer surface of the swatch faces upwards. The swatch will be stressed by attaching one 234-g weight to each end of the swatch strip.

(2) Six liquid agent drops (5 mg each of neat agent or 8 mg each of thickened agent) will be applied to the surface of the swatch. The detector paper will be examined periodically during the 1 hour after agent application for evidence of liquid penetration. If visible evidence of liquid agent penetration can be seen before 1 hour has elapsed, the time at which the agent penetration became visible will be recorded, and the test will be complete. The test will end after 1 hour if no penetration is observed before that time.

(3) The mandrel test setup diagram is shown in Figure B.3.

b. Expulsion Test.

(1) A 5.1-cm diameter circular (or a 5.1- × 5.1-cm square) material swatch will be placed over a piece of M8 chemical agent detector paper. The swatch and paper will be placed on a flat glass plate with the outer surface of the swatch facing upwards. Then, a single agent drop (5 mg of neat agent or 8 mg of thickened agent) will be applied to the center of the swatch. After a period of 15 seconds, the M8 chemical agent detector paper will be examined for a sign of penetration. If no penetration is visible, a pressure of 70.2 g/cm$^2$ will be applied to the swatch for up to 1 hour. The M8 chemical agent detector paper will be examined periodically for evidence of liquid penetration. The time at which any agent penetration is first visible will be recorded, and the test will be complete. The test will end after 1 hour if no penetration is observed before that time.

(2) The expulsion test setup diagram is shown in Figure B.4.

c. Inverted Expulsion Test.

(1) A single drop of liquid agent (5 mg of neat agent or 8 mg of thickened agent) will be placed on a flat glass plate and allowed to spread for 1 minute.

(2) The test swatch will be placed so that the swatch outer surface faces downwards on top of the drop. The swatch will be covered with a piece of M8 chemical agent detector paper, and a pressure of 70.2 g/cm$^2$ will be applied for 1 hour. The M8 chemical agent detector paper will be inspected periodically for evidence of liquid penetration. The time at which any agent penetration is first visible will be recorded, and the test will be complete. The test will end after 1 hour if no penetration is observed before that time.

(3) The inverted expulsion test setup diagram is shown in Figure B.5.

4.11.4 Aerosol Penetration Testing.

a. Items of instrumentation and equipment specific to aerosol penetration testing are listed in Paragraph 2.2 and Appendix B.

b. Test Procedures Overview.

(1) There are two different aerosol test methods related to swatch testing. These methods are based on testing conducted by the Research Triangle Institute (RTI) International (Research Triangle Park, Durham, North Carolina) and the ASTM International (West Conshohocken, Pennsylvania). The ASTM method uses an optical particle counter to determine the number of mono-dispersed aerosol particles of known diameter. This method does not determine particle size but only counts the number of aerosol particles upstream and downstream of the test swatch. On the other hand, the RTI method uses a single poly-dispersed aerosol with both the particle sizing and counting performed by an optical aerosol spectrometer.

(2) The use of mono-dispersed challenge aerosols is the more precise approach; however, because the test method must be repeated for each particle size of interest, it requires considerably more time. For this reason, the use of the poly-dispersed aerosol approach is more common and cost effective in comparison to the mono-dispersed aerosol approach.

c. Single Poly-Dispersed Aerosol Challenge Method. Tests should be performed with the apparatus illustrated in Figure B.6, or a similar apparatus. A 12.7-cm diameter material swatch should be mounted in a holder similar to that shown in Figure B.7. The holder is placed in the test apparatus.

(1) The swatch may be a single layer of fabric or several layers of a protective overgarment, assembled in the proper sequence.

(2) The challenge aerosol particles should be generated from a nontoxic, low volatility liquid (e.g., oleic acid). A syringe pump will be used to meter the oleic acid at a rate of 0.3 mL/min into a collision type nebulizer.

(3) Air pressure to the nebulizer will be set at ~20 mm Hg (~2758 Pascal) for a flow rate of 300 mL/min. This pressure is well below the nebulizer's normal operating pressure but should be used to keep the resultant aerosol concentration below the saturation limit of the APS™.

(4) After exiting the nebulizer, 24.3 L/min of additional air is added through a porous-tube diluter to achieve the desired 5-cm/sec face velocity through the fabric. The aerosol is passed through a charge neutralizer before passing through the test swatch. Aerosol concentrations upstream and downstream of the fabric are measured with an APS™. The APS™ measures particle concentrations in several sizing channels between 0.4 and 24 μm. The sampling rate of the APS™ is about 5.0 L/min. The APS™ must be calibrated and checked before testing begins.

(5) Before challenging a swatch with an aerosol, the background aerosol particle sizes and numbers from the fabric swatch must be determined in three consecutive 1-minute measurements.

(6) Aerosol particle sizes and number measurements will consist of three upstream measurements, followed by six downstream measurements, followed by another three upstream sampling sequence. Measurements will begin by taking three consecutive upstream 1-minute sample readings. Then the particle sampler's sampling line will be switched to the downstream sample line. Six consecutive 1-minute downstream sample readings will be obtained after 2 minutes. The 2-minute period between the downstream and upstream sample readings will be provided to allow the particle sampler's sample line and optical chamber the chance to "flush out" the old sample and take the new one. The particle sampler will then be switched back to the upstream sample line, and after waiting 2 minutes, three consecutive 1-minute sample readings will be obtained.

(7) The pressure drop will be measured across the fabric with an inclined manometer. The flow rate will be measured through the test swatch with a laminar flow element.

(8) The instruments will be calibrated for measuring the flow rate, temperature, RH, and ΔP across the fabric before testing begins.

(9) The aerosol will be dried or diluted with make-up airflow before injection or dispersion of the aerosol, if necessary. The RH should be held to no more than ±5 percent excursions during a given test.

d. Data required for aerosol testing are significantly different from the other swatch testing methods and are listed separately (Paragraph 5.2)

4.12 Fixture and Sampling System Monitoring During L/V and V/V Trial Execution.

a. Fixture Monitoring. The environmental parameters and flow rates of the fixture will be monitored to ensure they stay within the specified tolerance ranges for the duration of the trial. Environmental parameters will be recorded at least every minute.

b. Sampling System Monitoring.

(1) SSTs. At the end of each sampling interval, one sampler at a time will be changed by first disconnecting the vacuum line and disconnecting the test cell connection. The used sampler will be replaced with a fresh one by first connecting the fresh sampler to the test cell and then to the vacuum line.

(2) RT/NRT Systems. At the beginning, middle, and end of each trial, the calibration of the sampling system will be checked to ensure that accurate data are being generated for the duration of the trial.

4.13 L/V and V/V Trial End and Sampling System Collection Procedures.

a. Trial End. Trial end will be declared when the trial time has elapsed, a breakthrough event has occurred, or the test plan-determined breakthrough concentration has been achieved. The trial will be terminated along with data collection.

b. Sampling System Collection Procedures.

(1) Quality system procedures must be followed IAW local laboratory SOPs. This includes QC checks for analysis, duplicate samples, agent application, etc.

(2) Bubblers. As soon as possible following each bubbler change interval and at the end of the trial, two aliquots of the collection solvent from each bubbler will be transferred to prelabeled sample vials. Using a new pipette for each bubbler, one operator will transfer an aliquot of solvent from the bubbler to each duplicate vial. The vials will be capped, and a CoC form will be used to transfer the vials to the analytical laboratory for analysis. Samples and bubblers will be stored at temperatures between 0° and 4°C when not in use. Bubblers will be cleaned IAW local SOPs provided the method does not expose the bubblers to decontaminants or interferents, such as phosphate-based detergents.

(3) SSTs. Samples will be analyzed as soon as possible via thermal desorption or solvent extraction and analysis. SSTs will be stored at temperatures between 0° and 4°C when not in use. Extraction procedures for SSTs will be conducted IAW local SOPs or program-specific planning documents. Any SSTs reused will be thermally desorbed and verified clean of analyte after analysis or solvent extraction.

(4) RT/NRT Systems. A final calibration check of the sampling system will be conducted to ensure that accurate data are generated for the duration of the trial.

4.14 L/V and V/V Test Cell Cleanup and Swatch Disposal.

a. The fixture will be powered off and the test cells will be removed from the test assembly after the completion of each trial. Each cell will be disassembled, one at a time.

b. Each test swatch will be removed and inspected for obvious abnormalities. It is recommended that a digital picture be taken after each test to indicate the interaction of the agent drop(s) with the swatch. Results of the inspection will be recorded by SICN in a laboratory notebook.

c. The test swatch and O-rings will be placed in a decontamination solution for 24 hours before disposal.

d. Each component of the test cell will be thoroughly rinsed with acetone to remove any residual chemicals. All remnants of the test cell labels will be removed. The cells will be air-dried for 24 hours before being reused.

4.15 Initial Data Review.

The senior test person will review all of the data obtained for consistency and acceptability during trial execution or as soon as possible after the test. Specifically, the following items will be reviewed:

a. The agent challenge QC check (Paragraph 4.11.2.c) will be reviewed to ensure the target challenge concentration was met, if applicable.

b. The analytical results for each sampling period will be reviewed to determine whether the results obtained on replicate test swatches are reasonable and consistent.

c. The analytical results for positive control swatches will be reviewed to determine if the swatches remained within control limits.

d. The negative control swatches will be reviewed to determine if breakthrough occurred in their test cells. The purpose of negative controls is also to determine if there is an issue with the test processes. As an example, samples could come back showing contamination if there is cross-contamination somewhere in the system.

e. Analytical QC and calibration data will be reviewed to determine if the values are within limits specified in the planning documents and SOPs[7,10].

## 5. DATA REQUIRED.

### 5.1 General Data Required.

The following data are required for each trial, as applicable to the method chosen:

a. TICN/SICN of each test swatch.

b. Air-permeability data for each sample by test item and TICN/SICN (if applicable).

c. Test item and swatch inspection data by TICN/SICN.

d. Trial identification.

e. Fixture identification and test laboratory.

f. Swatch pretreatments, if any.

g. Swatch stratum.

h. Swatch challenge (agent or other challenge).

i. Test cell configuration (if applicable) and location in the fixture.

j. Agent application method and QC validation.

k. Results of the negative and positive controls.

l. Mass of agent (μg) collected during each sampling interval and equally weighted arithmetic average mass flux ($\mu g/min \cdot cm^2$) for each SICN.

m. Calibration and QC sample results from all chemical analyses.

n. Agent purity analysis results.

o. Sampler collection efficiency data by agent and test conditions.

p. For convective tests, the ΔP for each test cell throughout the preconditioning and trial execution recorded in Pascal or mm Hg as 1-minute averages.

q. Volumetric sampling airflow rates (L/min) for each test cell during preconditioning and trial execution as 1-minute averages.

r. Temperature and RH for all supply air manifolds and environmental control fixtures during preconditioning and trial execution. These values will be recorded as 1-minute averages.

s. (L/V testing only) For each test cell and swatch, the spike to start and the start and stop times.

t. (V/V testing) Start and stop times for the trial.

u. (L/L testing only) For each test swatch, whether or not the response of the M8 chemical agent detector paper was positive. If positive, record the number of drops of visible penetration on the M8 paper and the time the penetration was first observed. The detector paper should be photographed to provide a reference to return to if/when questions arise about the results.

v. Operator-annotated comments for any observed anomalies during execution of each trial, including observations of each swatch recorded by SICN.

5.2 Data Required for Aerosol Testing of Swatches.

a. The results of the particle size and number measurements will be transferred electronically to the laboratory computer by individual SICN after each trial is completed. The laboratory computer will then relate each analytical result back to its corresponding trial and test item by TICN. The 1-minute averages of the aerosol challenge and penetration particle sizes and number measurements will be calculated by the laboratory computer, and the aerosol penetration value for each swatch will be calculated by SICN and tabulated. Also, the APS™ calibration and QC data will be reported for each chemical analysis. The following data will be reported in the smallest increments that the instrumentation/procedure is designed to achieve in consideration of the expected accuracy of the instrument and the minimum quantification limit (MQL), or in the case of analog instrumentation, the smallest increment that can be easily visually discerned:

(1) The background particle sizes and number measurements for each swatch.

(2) The 1-minute average upstream aerosol particle sizes and number measurements.

(3) The 1-minute average downstream aerosol particle sizes and number measurements.

(4) The purity and identity of the simulant used.

(5) The starting time and length of each sampling period (seconds).

(6) The $\Delta P$ across the swatch.

(7) The flow rate through the test swatch (L/min).

(8) The temperature (°C).

(9) The RH (percent).

6. PRESENTATION OF DATA.

a. Data for individual swatches will be reported in a tabular format and grouped by challenge agent, material type, and material condition (e.g., new versus worn, worn 14 days and laundered versus worn 14 days without laundering).

b. The tables will include the cumulative mass flux, mass flux for each sampling period, pretreatment, test location, fixture number and cell location, test method, test date, SICN, and the average temperature, humidity, and airflow, with the addition of $\Delta P$ if the convective-flow method was used.

c. Each data set will be examined for outliers, but all data will be reported together with the rationale for eliminating any points from subsequent statistical analyses.

d. The test plan must include information on how to handle sampling periods with results below the experimentally derived method quantification limit (MQL) for the individual laboratory. The common practice in acquisition testing has been to set below-MQL values to half the MQL. **NOTE**: There cannot be a predetermined or preset program or interlaboratory MQL imposed on the laboratory(s) by requirement documents. MQLs are determined at the laboratory level.

e. Because MQLs are determined at the laboratory level, issues may arise when data from different laboratories are compared. To resolve this issue, it is recommended that a program quantification limit (PQL) be set at the level of the highest laboratory MQL. This PQL value would be used in the same way that the MQL is used at individual laboratories (Paragraph 6.d), but the value would operate on the interlaboratory level.

f. Procedures for statistical analysis of the data will be developed as specified in the planning documents. Analysis procedures must include:

(a) Ability to address the program criteria.

(b) Type of analysis.

(c) Procedures for analysis of data points below detection limits.

(d) Specifications of statistical values used (e.g., confidence, probability, power, etc.).

g. A summary data table will be assembled for inclusion into the final test report. The summary table should have the following information: swatch material, any pretreatment applied and application location, cumulative sampling periods, sample size, number of swatches for which no permeation was detected, measurement of central tendency (mean, median, geometric mean, etc.) of the mass flux, measure of variability (variance, standard deviation, logarithmic standard error, etc.), upper and lower bounds for a confidence interval on the mean, and whether the swatch material met the test criteria. Examples of data presentation are in Table 3 and Figure 3.

h. Presentation of Aerosol Testing Data. Examine and summarize the swatch data from each trial and present the data in tabular format. These data include:

(1) Particle Count. Particle counts (counts per minute) for each sizing channel for the upstream and downstream measurements.

TABLE 3. EXAMPLE OF DESCRIPTIVE STATISTICS FOR SWATCH 24-HOUR CUMULATIVE CONCENTRATION × TIME (CT).

| Material[a] | Agent[b] | Treat-ment | Nondetects/ Sample Size | Geometric Mean (mg·min/m$^3$) | Logarithmic Standard Error | 95% Confidence Bounds | |
|---|---|---|---|---|---|---|---|
| | | | | | | Lower | Upper |
| A | GD | Wet Sweat | 6/22 | 400 | 0.46 | 152 | 1049 |
| B | GD | Wet Sweat | 7/22 | 449 | 0.52 | 154 | 1313 |

[a]A and B are generic placeholders for two different materials presented for swatch testing.
[b]Soman.

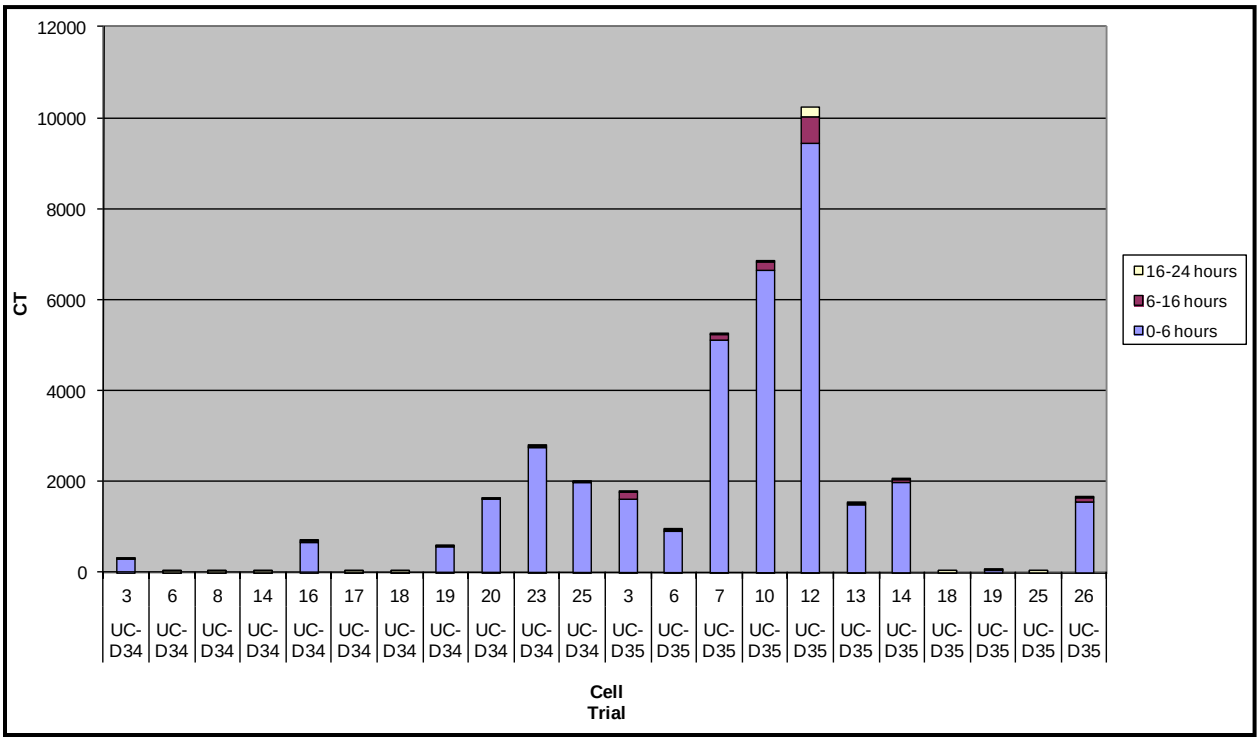



Figure 3. Example of a swatch permeation data plot for cumulative concentration × time (CT).

(2) Aerosol Penetration. The aerosol penetration for a particle size range is calculated using the average of six downstream measurements divided by six upstream measurements for each sized channel (Equation 2).

*Equation 2*

$$A_P = \frac{\overline{D}}{\overline{U}}$$

Where:

$A_P$ = the aerosol penetration.

$\overline{D}$ = the average (mean) of six downstream measurements.

$\overline{U}$ = the average (mean) of six upstream measurements.

(3) Aerosol Retention. The aerosol retention is a measure of the fraction of the aerosol retained by the fabric. The amount of aerosol retained in the fabric is simply Equation 3:

*Equation 3*

$$A_R = 1 - A_P$$

Where:

$A_R$ = the aerosol retention (efficiency).

$A_P$ = the aerosol penetration.

# APPENDIX A. GLOSSARY.

| | |
|---|---|
| Breakthrough | A mass or concentration of a challenge contaminant found on the initially clean side of a contaminated test swatch, indicating that the contaminant has permeated or penetrated the material being tested. Breakthrough threshold levels are defined for each test program and should be specified as needed in the test plan or other test planning documents. |
| Candidate material | A nonstandard material, supplied as a flat piece, a bolt, or a manufactured item, from which swatches may be cut for testing. |
| Cell | A test cup used to hold a test swatch for permeation or penetration testing. Cups have top and bottom sections with the test swatches attached to the lower section. |
| Challenge | The chemical agent or agent simulant (or other chemical compound) that is placed on one side of a test swatch. The challenge can be in liquid or vapor form. The challenge amount is usually stated in planning documents in units of $g/m^2$ for liquid or $mg/m^3$ for vapor. |
| Penetration | The flow of a chemical through closures, pores, seams, pinholes, or imperfections in a protective clothing material on a nonmolecular level[11]. |
| Permeation | The process by which a chemical moves through a protective clothing material on a molecular level. Permeation involves the following: (1) sorption of molecules of the chemical into the contacted surface (usually the outside) of a material, (2) diffusion of the sorbed molecules among the material molecules, and (3) desorption of the molecules from the noncontacted surface (usually the inside) of the material into the collecting medium[11]. |
| Sampler | A device used to collect vaporous or liquid compounds to determine the presence of a challenge chemical. |
| Swatch | A measured section of fabric cut from a bolt of material, a garment, cap, clothing liner, mask, glove, footwear, etc. The swatch should be selected to be representative of the area of the material to be tested. |

## APPENDIX A GLOSSARY.

| | |
|---|---|
| Test control swatches | Both positive and negative test control swatches must be used in each trial. The negative control swatch will be a swatch of a material (defined in the test plan) that is conditioned the same as the test swatches except that agent is not applied to the upper surface. The positive control swatch for evaluating air-permeable and semipermeable materials will be a swatch of a baseline material, such as 6× laundered Joint Services Lightweight Integrated Suit Technology (JSLIST) overgarment material, neoprene, or butyl rubber. |
| Test assembly/ test fixture | An assembly or fixture contained inside an environmental conditioning chamber and consisting of test cells into which swatches can be sealed and tested for permeability/penetrability. The fixture also includes a sampler or samplers attached to the test cells, air delivery manifolds; vacuum manifolds; delivery air-conditioning system; vapor generation and control system; pressure-measuring devices; airflow control and monitoring devices; temperature- and humidity-measuring devices. |
| Volatility | The saturated vapor concentration of a chemical at any given temperature. Listed in Table A.1 are the estimated volatilities at a room temperature of 20°C (68°F) of the chemical agents historically used in permeation or penetration testing. |

Table A.1. ESTIMATED CHEMICAL AGENT VOLATILITIES AT 20°C (68°F).

| Sarin (GB) ($mg/m^3$) | Soman (GD) ($mg/m^3$) | Distilled Mustard (HD) ($mg/m^3$) | Persistent Nerve Agent (VX) ($mg/m^3$) |
|---|---|---|---|
| 13,427 | 2,685 | 590 | 7.3 |

**NOTE**: These numbers represent the theoretical saturation concentrations. In practice, because of humidity and other variable test factors, the actual maximum concentrations are much lower.

# APPENDIX B. LIST OF EQUIPMENT AND ADDITIONAL INFORMATION.

## FIGURE LIST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# APPENDIX B. LIST OF EQUIPMENT AND ADDITIONAL INFORMATION.

1. Test Equipment List.

Thermocouple or other temperature-measuring device.

Humidity probe.

Mass flow controllers or flow meters.

Still color camera.

Flexible tubing.

Nitrile O-rings.

Test cells.

Agent/simulant vapor generator.

M8 chemical detection paper, or equivalent.

Standard laboratory American Association of Textile Chemists and Colorists (AATCC) blotting paper.

Aerosol nebulizer that uses an air jet to nebulize a liquid and is designed for constant output of both particle size and particle concentration in particles/L.

Aerosol charge neutralizer. (Aerosols generated by atomization are very often electrically charged. The charge is undesirable because it increases particle loss to the transporting and sampling systems. The aerosol must be neutralized to ensure proper functioning of the system.)

Laminar flow element (aerosol testing only). This device operates using capillary flow and may be used for measurement of flow as well as calibration of the flow.

Device for uniformly cutting 5-cm diameter swatches. Other swatch sizes may be cut by hand or by using a device to ensure more accuracy and ease in cutting to exact size.

Liquid agent dispensing device for uniform application of liquid agent (≤2 g total force during application), or equivalent.

Gas-tight syringe (50 to 500 µL) and syringe needles for application of thickened agent, or equivalent dissemination applicator.

Bubblers, near real-time (NRT) monitoring system, solid sorbent tubes (SSTs), or equivalent sampler.

# APPENDIX B. LIST OF EQUIPMENT AND ADDITIONAL INFORMATION.

Gas chromatograph (GC), high-performance liquid chromatograph (HPLC), liquid chromatograph (LC), flame photometric detection (FPD), flame ionization detection (FID), mass spectrometer (MS), spectrophotometer, or equivalent.

Positive control material with a well-defined permeation profile. **NOTE**: Control limits for the positive control swatches should be based on historical data or on system verification and validation data. Historically, the material used has been 6× laundered Joint Service Lightweight Integrated Suit Technology (JSLIST) material. JSLIST material is available from several sources. For dual-flow tests, repeatable and consistent results have been obtained by using the following positive control materials:

a. For distilled mustard (HD) trials: 10- × 10-inch butyl rubber sheets (Enterprise Rubber, Inc., Akron, Ohio), part number "Dugway" Butyl Sheet, thickness of 0.015±0.005 inches; durometer hardness of 40A, lot number 427, or equivalent material. **NOTE**: The specifications from this manufacturer were given in English/American Standard measurements, not metric, and there is a risk that if the material or equivalent was ordered using metric conversions, it may not be equivalent.

b. For soman (GD) trials, 1 $ft^2$ neoprene sheets (AAA-ACME Rubber Company, Tempe, Arizona); part number SS-.017X36-34000, thickness of 0.017±0.006 in; durometer hardness of 40A, lot number 11401-001, cure data 1Q05, or equivalent material. **NOTE**: The specifications from this manufacturer were given in English/American Standard measurements, not metric, and there is a risk that if the material or equivalent were ordered using metric conversions that it may not be equivalent.

## 1. TEST EQUIPMENT – ADDITIONAL INFORMATION.

### 1.1. Samplers.

a. Bubbler. A bubbler is a cumulative sampler. A collection solvent having good solubility for the chemical vapor to be sampled is placed in the bubbler. Generally, 10 mL of solvent is used. During operation, air is drawn through the bubbler at a fixed, known flow rate and any chemical vapor in the air dissolves in the collection solvent. Before testing, the collection efficiency of the bubbler and the selected collection solvent for the chemical sampled must be determined at the airflow rate, relative humidity (RH), and temperature to be used for testing. The stability of the solvent must be known so that postcollection loss by decomposition can be avoided by establishing storage conditions and a maximum storage time. At the completion of each prescribed sampling period, each bubbler must be replaced with a fresh bubbler. An aliquot of the collection fluid from the used bubbler should be analyzed to determine the amount of the chemical collected. There is usually a sufficient sample volume to perform a repeat analysis should the initial value obtained appear questionable, need to be verified, or need to be diluted to bring its concentration into the range of the analytical method used; however, the sensitivity of the analysis may be limited by dilution of the analyte. Testers should be aware that water condenses in bubblers. Under certain conditions, the water may react with the dissolved analyte; such reaction may impair quantification of the analyte.

APPENDIX B. LIST OF EQUIPMENT AND ADDITIONAL INFORMATION.

b. SST. SSTs are cumulative samplers. SSTs are either metal (stainless steel is preferred) or glass tubes that contain a small quantity of a solid sorbent material [e.g., Porapak Q® (Bellefonte, Pennsylvania), Tenax® GC or Tenax® TA (Buchem BV, Apeldoorn, The Netherlands), etc.]. This sampler is aspirated for a prescribed period of time in a manner similar to bubblers. Following the prescribed sampling period, the SST should be replaced with a fresh tube and the used one analyzed to determine the amount of the chemical collected. As with the bubbler, the results obtained by the SST are averages over the sampling period, and details of permeation or breakthrough are lost.

(1) The analysis is done conventionally by thermally desorbing the chemical from the tube into a GC set up to measure the chemical. The major advantage of the SST is that it has a higher sensitivity than the bubbler because the entire sample (rather than just a portion) is analyzed at once. This is also a disadvantage because no portion of the collected sample is reserved in case reanalysis is needed. Care must be taken to ensure that the sorptive capacity of the solid sorbent is not exceeded during the sampling period. Frequently, the thermal desorption step is not completed properly, leading to a low estimate of the amount of the chemical collected. If special care is not taken to ensure the tube is completely purged and cleaned before the next use, there may be carryover of challenge chemical to the next use of the tube. As a precaution to prevent carryover, all SSTs should be identified by number, and used tubes should be cleaned by simultaneously heating them and purging them with nitrogen. Each individual tube should be subjected to the GC analysis procedure to demonstrate that it is blank (free of chemical contaminants) before being stored in a sealed, contaminant-free glass jar until the next use.

(2) Alternatively, the solid sorbent can be extracted with an appropriate solvent and the extract analyzed. This reduces the sensitivity but obviates problems arising from one-shot thermal desorption.

c. NRT and real-time (RT) samplers. NRT and RT samplers are automated analytical instruments that sample and analyze the effluent from the swatch sampling ports either continuously (for RT samplers) or at set intervals that are less than 15 minutes apart (for NRT samplers). The advantage of these systems (over bubblers and SSTs) is that permeation curves can be constructed from the data. From these data (permeation curves), other permeation details like breakthrough time and steady-state permeation can be determined which provide additional indicators of material performance. The disadvantage is that calibration checks are required before and after each trial to ensure that the system remains in calibration for the duration of the trial.

1.2. Test Cells.

**NOTE**: Comparison of test results for materials tested using different methods/cell configurations is not appropriate. Results are not equivalent.

a. Dual-/static-flow (Figure B.1) and convective-flow test cells (Figure B.2) are standard for testing materials to determine relative abilities to resist permeation by a chemical agent.

APPENDIX B. LIST OF EQUIPMENT AND ADDITIONAL INFORMATION.

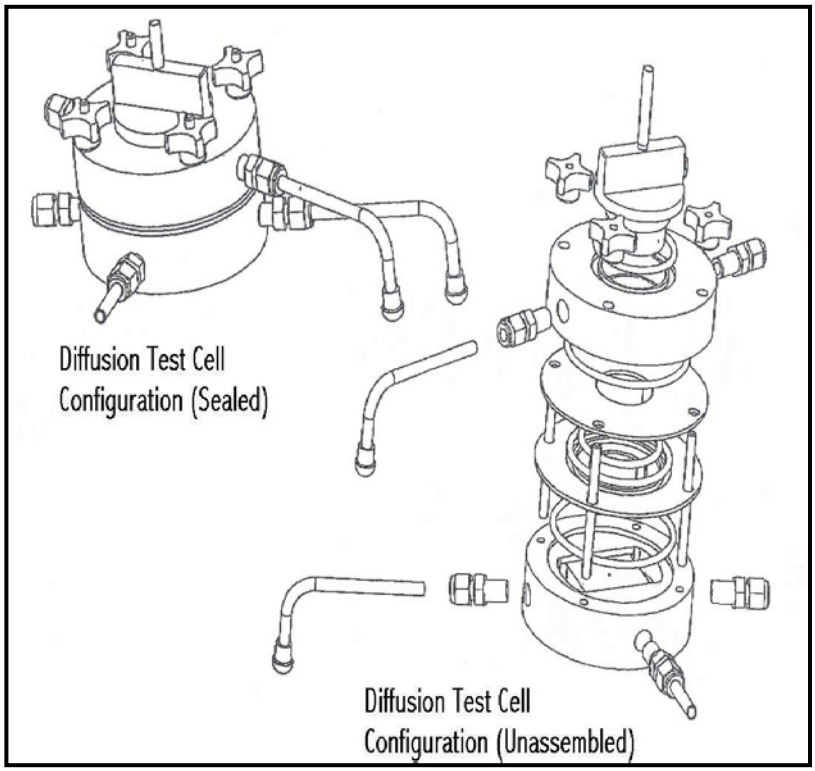



Figure B.1. Sealed and unassembled views of the liquid challenge/vapor penetration (L/V) test cell for the dual-flow and static-flow configuration.

b. The dual-/static-flow cells (Figure B.1) are advantageous because they can be used to test all material types (air-permeable, semipermeable, and nonpermeable) in the same configuration.

c. The convective-flow test cell (Figure B.2) is advantageous for vapor challenge/vapor permeation (V/V) testing because it can be used to measure the capacity of sorptive or reactive materials to remove agents from a stream of air containing agent vapor that is passed through the test swatch.

1.3. Test Fixtures.

a. Liquid Challenge/Vapor Permeation (L/V) and V/V. Two manifolds are required for the V/V and L/V test fixtures (assemblies). One manifold supplies either clean, conditioned air to the test cells for the L/V tests, or agent vapor for the V/V tests, and the second manifold provides the vacuum to draw effluent air through the test cells and samplers. Unused positions on each manifold must be blocked.

APPENDIX B. LIST OF EQUIPMENT AND ADDITIONAL INFORMATION.

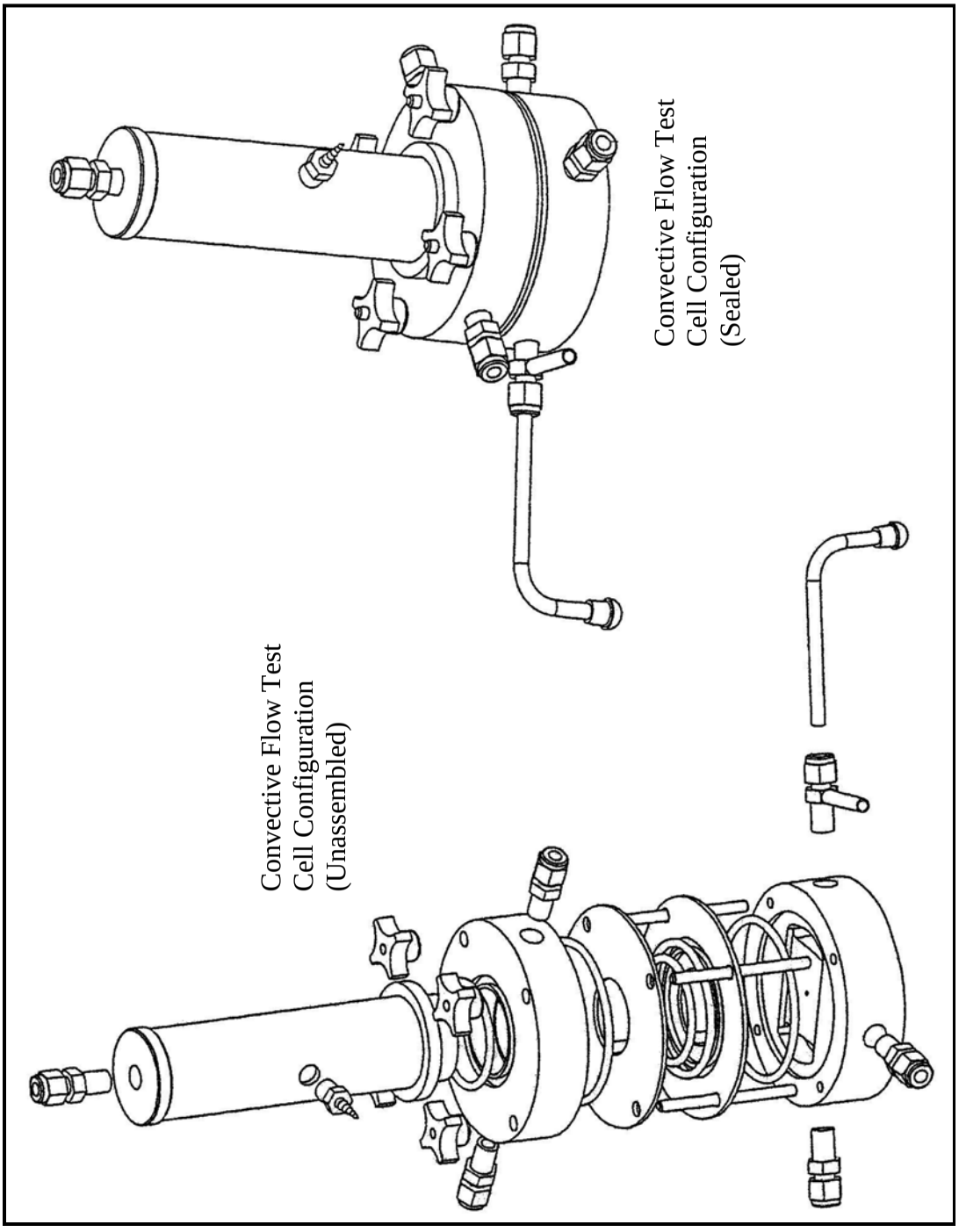



Figure B.2. Unassembled and sealed views of the liquid challenge/vapor penetration (L/V) test cell in the convective-flow configuration [pressure differential ($\Delta P$) >0].

APPENDIX B. LIST OF EQUIPMENT AND ADDITIONAL INFORMATION.

b. Liquid Challenge/Liquid Penetration (L/L).

(1) Mandrel Test. This test requires a length of glass tubing 41 mm in diameter, two 234-g weights, and one piece of M8 chemical agent detector paper per test swatch. The test set-up for this test is shown in Figure B.3.

(2) Expulsion Test. For each test swatch, this test requires a hard (glass) surface, a piece of M8 chemical agent detector paper, and a 454-g stainless steel cylindrical weight with a diameter of 2.87 cm and an end surface area of 6.47 $cm^2$. The test setup for this test is shown in Figure B.4.

(3) Inverted Expulsion Test. For each test swatch, this test requires a hard (glass) surface, a piece of M8 chemical agent detector paper, and a 454-g stainless steel cylindrical weight with a diameter of 2.87 cm and an end surface area of 6.47 $cm^2$. The test setup for this test is shown in Figure B.5.

c. Aerosol Penetration Testing. A schematic diagram for a swatch aerosol penetration testing apparatus is in Figure B.6, and a schematic diagram for the fabric holder is in Figure B.7.

1.4. Flexible Tubing.

a. Connections between cells, samplers, airflow controllers, manifolds, etc., should be made with flexible tubing, when appropriate. All tubing used to conduct air that could contain chemical vapor must have a low tendency to absorb the chemical.

b. Tubing made from the following fluorinated polymers have been found acceptable, although others may be appropriate for use: Teflon® fluorinated ethylene propylene (FEP) (DuPont™, I.E. du Pont de Nemours and Company, Wilmington, Delaware), Teflon® perfluoro-alkoxy (PFA), and Teflon® polytetrafluoroethylene (PTFE).

c. In all cases, only the minimum necessary length of tubing should be used, and tubing length between similar parts should be consistent.

APPENDIX B. LIST OF EQUIPMENT AND ADDITIONAL INFORMATION.

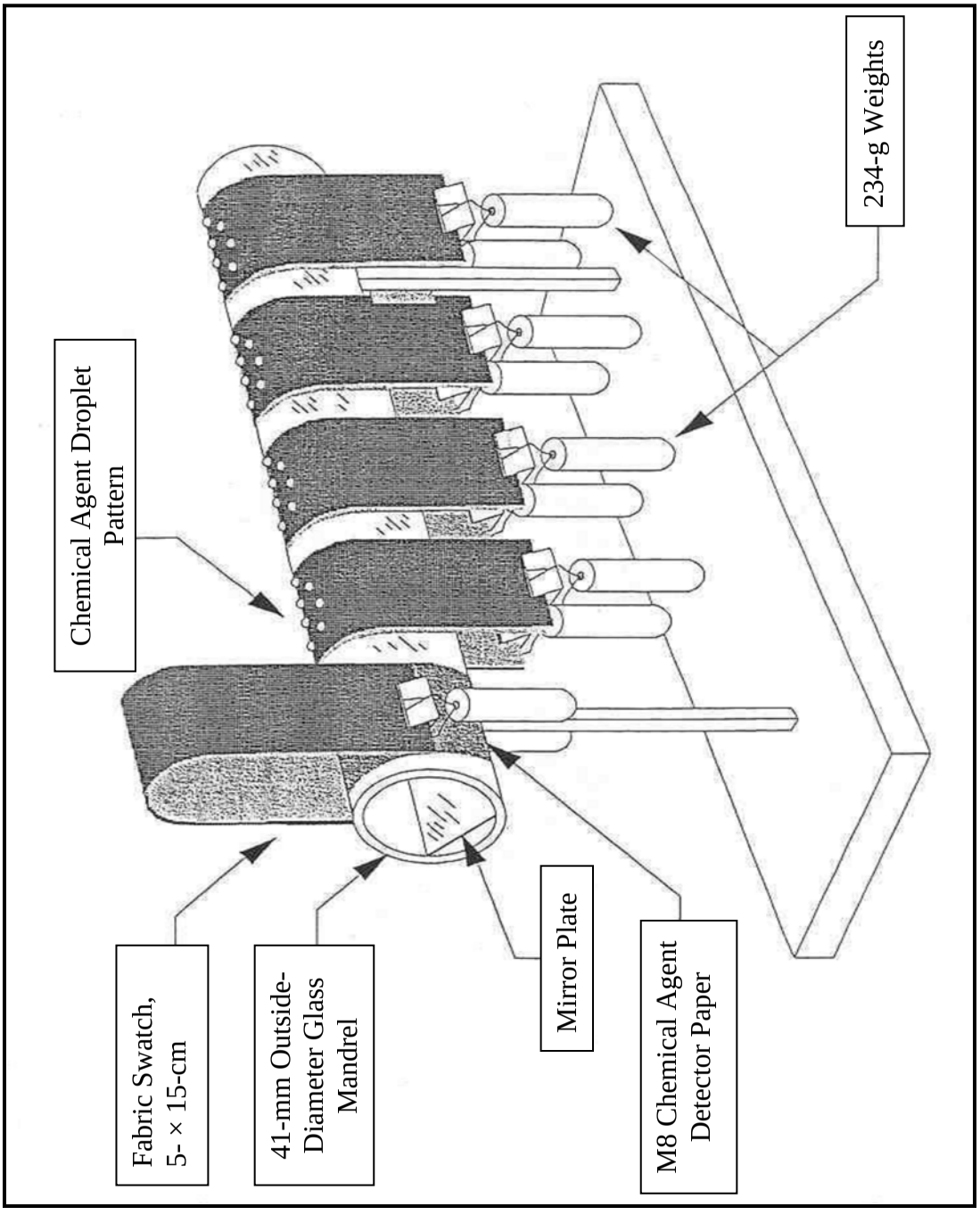



Figure B.3. Drawing of mandrel liquid challenge/liquid penetration (L/L) test assembly.

APPENDIX B. LIST OF EQUIPMENT AND ADDITIONAL INFORMATION.

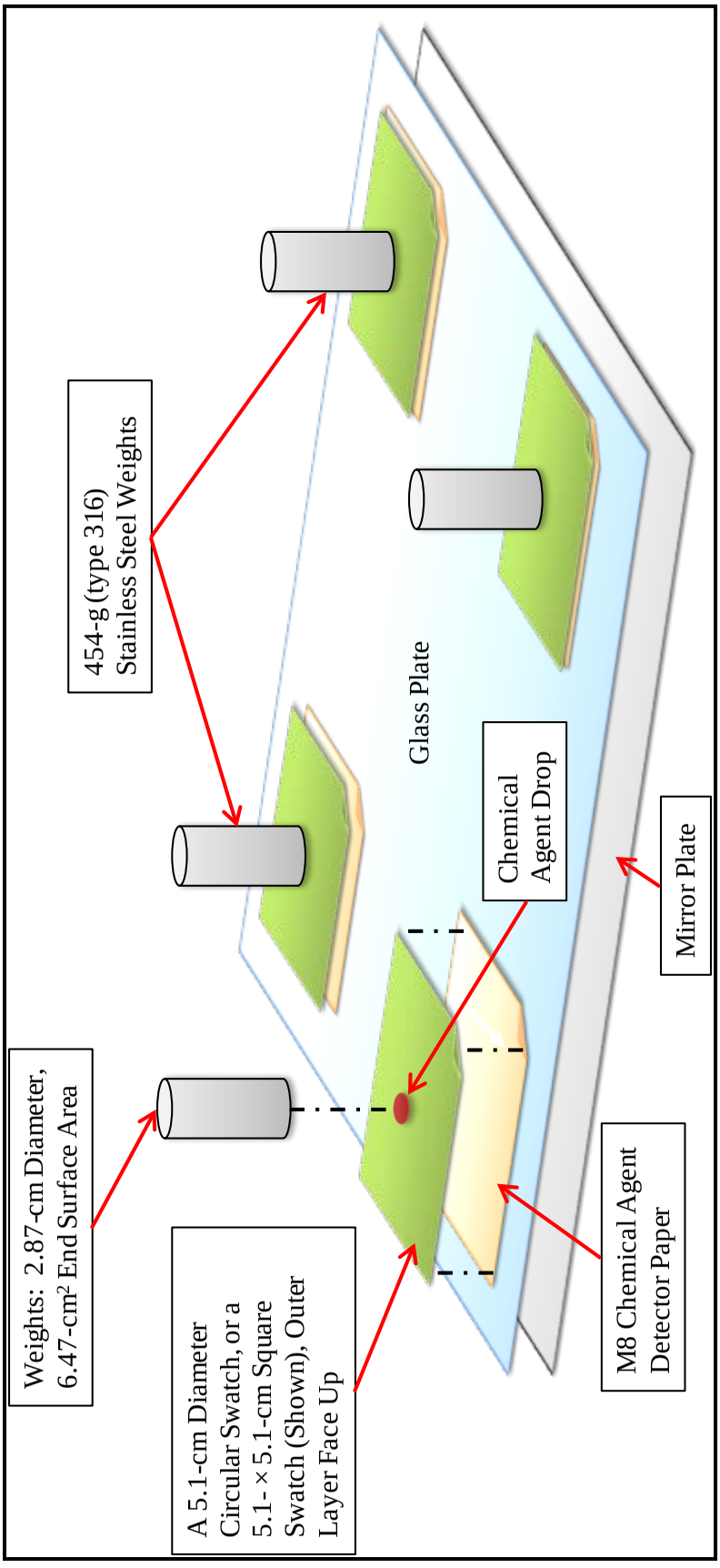



Figure B.4. Diagram of an expulsion liquid challenge/liquid penetration (L/L) test assembly.

APPENDIX B. LIST OF EQUIPMENT AND ADDITIONAL INFORMATION.

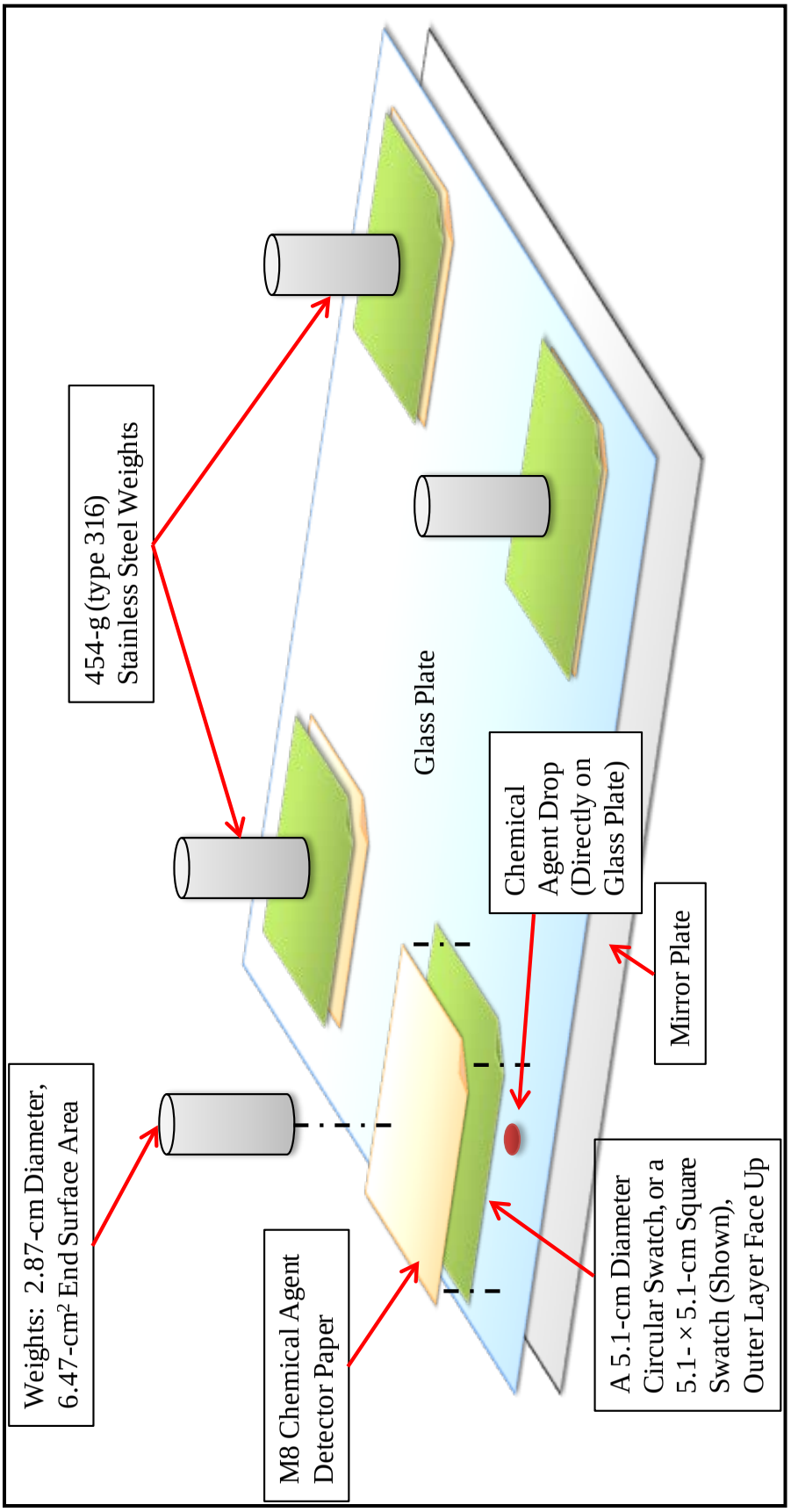



Figure B.5. Diagram of an inverted expulsion liquid challenge/liquid penetration (L/L) test assembly.

APPENDIX B. LIST OF EQUIPMENT AND ADDITIONAL INFORMATION.

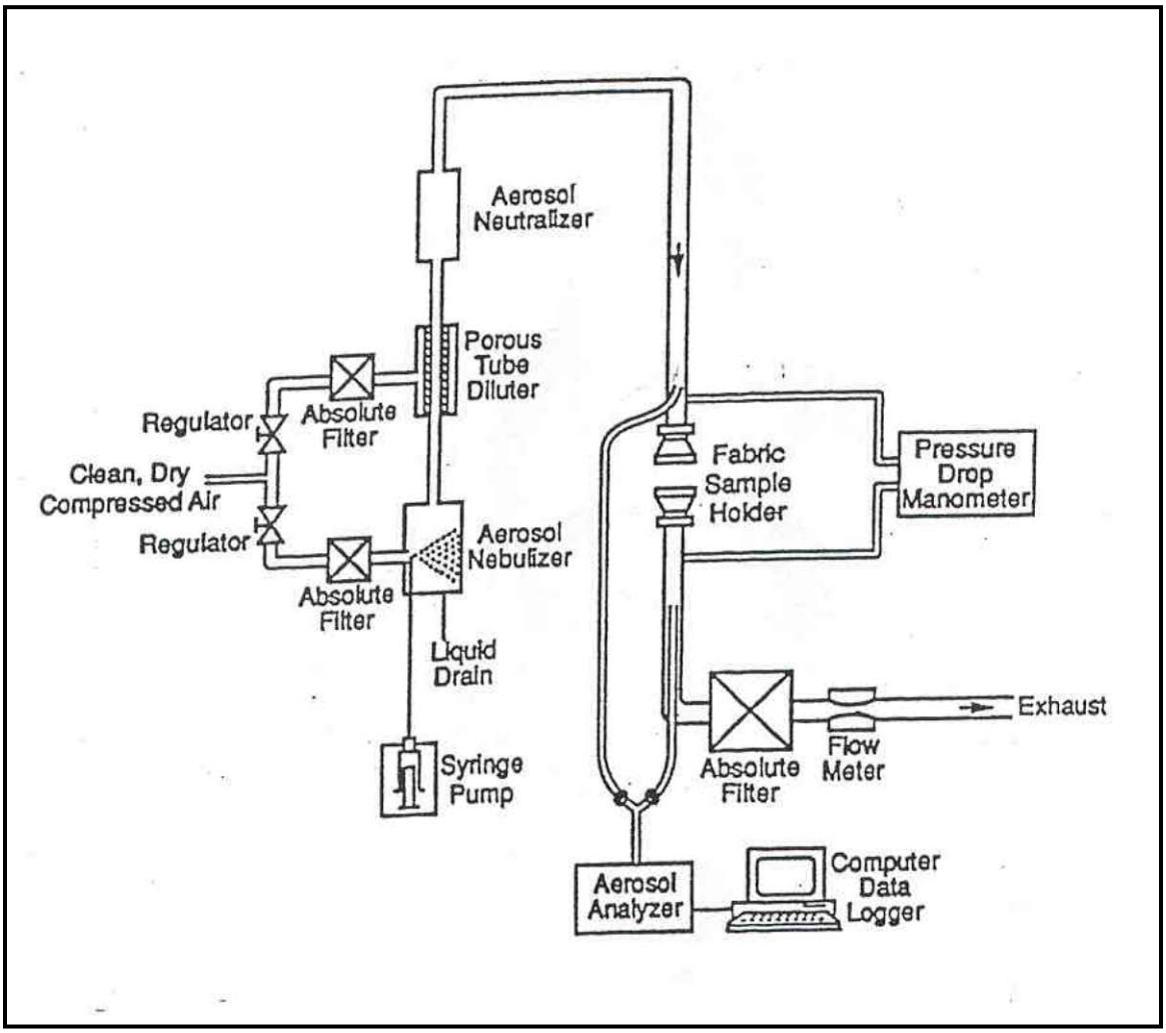
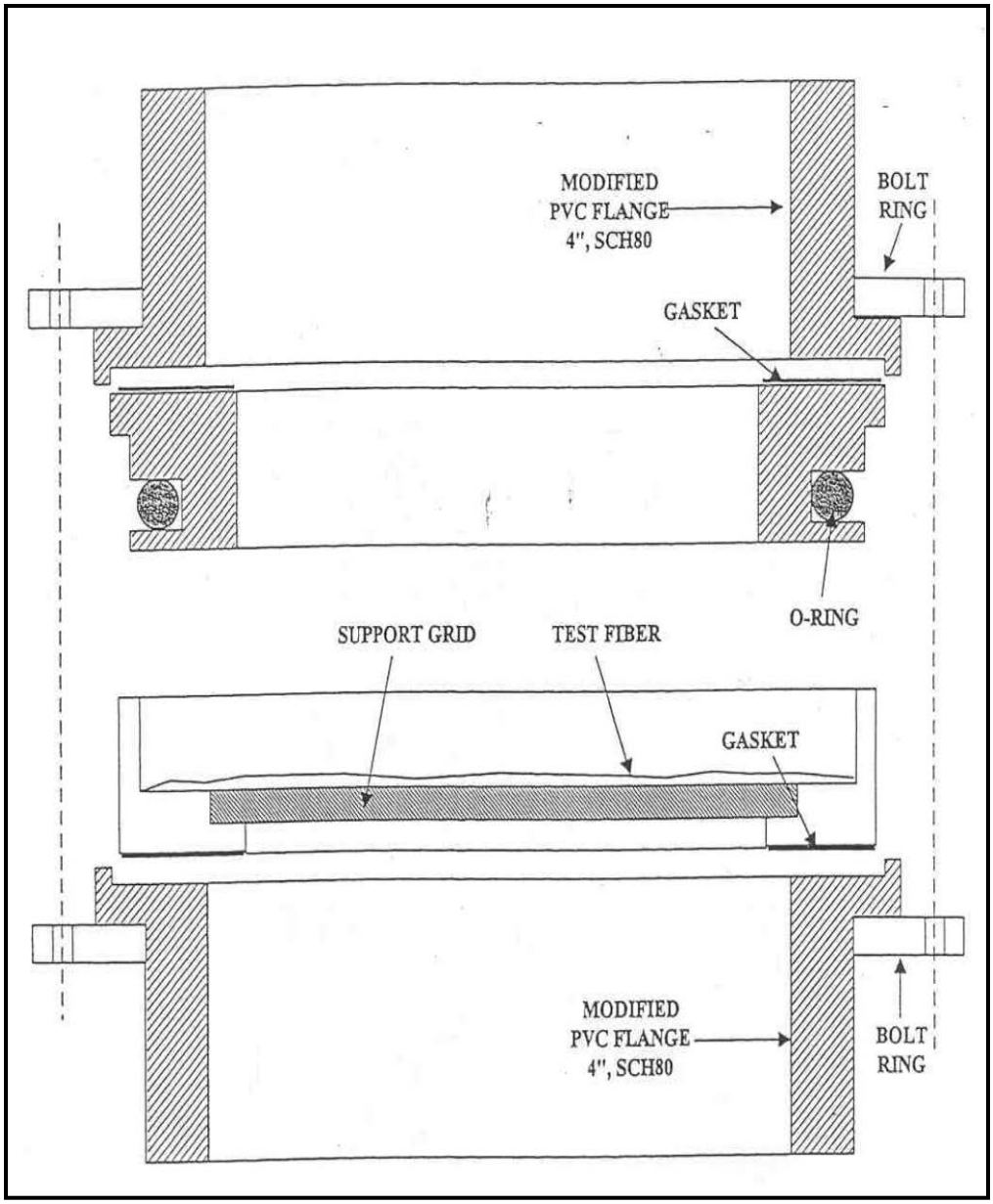



Figure B.6. Schematic diagram of test apparatus for measuring the aerosol penetration of fabric swatches.

APPENDIX B. LIST OF EQUIPMENT AND ADDITIONAL INFORMATION.




**NOTE**: PVC – polyvinyl chloride.

Figure B.7. Schematic diagram of swatch test fabric holder for aerosol testing.

# APPENDIX C. TESTING PRETREATED SWATCHES.

## 1. PURPOSE.

This appendix describes swatch pretreatments and the procedures to be used for testing material swatches that have been pretreated before chemical agent resistance testing in accordance with (IAW) this test operations procedure (TOP). Pretreatments include, but are not limited to, petroleum, oil, and lubricants (POLs), water, body fluids, decontaminants, fire fighting foam, and other compounds to which personnel are exposed during their missions.

## 2. DESCRIPTION OF TREATMENTS.

A range of pretreatments may be used, depending upon the particular test program. This appendix describes the most commonly used pretreatments (based on historical testing). However, other pretreatments or combinations of pretreatments may be used to address program-specific requirements. The compatibility of each pretreatment with the analytical systems and application methods must be demonstrated as part of test preparation. Commonly used pretreatments include:

a. POLs.

(1) Diesel fuel.

(2) Jet propulsion fuel number 8 (JP-8).

(3) Small arms lubricant.

(4) Hydraulic fluid.

**NOTE**: Diesel fuel and JP-8, as well as POLs in general, are not distinct chemical species. Their chemical composition can vary depending on the source of the crude oil from which they have been distilled and the manufacturing process. This can potentially affect agent resistance results.

b. Water and Body Fluids.

(1) Salt water.

(2) Fresh water.

(3) Sweat.

(4) Urine.

(5) Feces.

APPENDIX C. TESTING PRETREATED SWATCHES.

c. Decontaminants.

(1) M258A1 Individual Decontamination Kit.

(2) M291 Individual Decontamination Kit, Skin (SDK).

(3) M295 Individual Equipment Decontamination Kit.

(4) 10 percent high-test hypochlorite (HTH) solution.

(5) 5 percent sodium carbonate solution.

(6) Hot soapy water (HSW): [1 lb Detergent, General Purpose, Liquid, National Stock Number (NSN) 7930-00-282-9699, in 5 gallons water at 38°C (100°F)].

(7) Supertropical bleach (STB).

d. Aqueous fire-fighting foam (AFFF).

e. Insect repellant [DEET (N,N-diethyl-meta-toluamide)].

3. FORMULATIONS FOR SIMULATED FLUIDS.

3.1. Simulated Sea Water.

The following formulation for simulated seawater was calculated from data obtained from Sverdrup, Johnson, and Fleming[12].

| Ingredient | Amount |
|---|---|
| Sodium chloride | 26.83 g |
| Magnesium sulfate | 3.32 g |
| Magnesium chloride | 2.35 g |
| Calcium chloride | 1.11 g |
| Potassium chloride | 0.51 g |
| Potassium carbonate | 0.32 g |
| Strontium bromide | 0.10 g |
| Boric acid (ortho) | 0.02 g |
| Water | to 1 L |

APPENDIX C. TESTING PRETREATED SWATCHES.

3.2. Simulated Sweat.

The simulated sweat will be refrigerated after preparation and then brought to room temperature before pretreating test swatches. The following formulation for simulated sweat was described by Ferrell, Rousseau, and Aneja[13].

| Ingredient | Amount |
|---|---|
| Sodium chloride | 8.0 g |
| Potassium sulfate | 0.5 g |
| Sodium sulfate | 0.1 g |
| Magnesium sulfate | 1.02 g |
| Calcium chloride | 0.04 g |
| Urea | 0.5 g |
| Glucose | 0.15 g |
| Lactic acid | 1.0 g |
| Pyruvic acid | 0.03 g |
| Ammonium hydroxide | Add to bring pH (negative ion activity) to 7.5 |
| Water | to 1 L |

3.3. Simulated Urine.

The simulated urine will be refrigerated after preparation and then brought to room temperature before pretreating test swatches. The following formulation for simulated urine was calculated based on data showing the major constituents present in natural urine obtained from Altman and Dittmer[14]. The proper amount of aqueous ammonium hydroxide ($NH_4OH$) will be added to provide 0.82 g of $NH_4OH$.

## APPENDIX C. TESTING PRETREATED SWATCHES.

| Ingredient | Amount |
|---|---|
| Urea | 17.50 g |
| Creatinine | 1.15 g |
| Uric acid | 0.10 g |
| Glucose | 0.35 g |
| Glutamic acid | 0.20 g |
| Alanine | 0.05 g |
| Asparagine | 0.13 g |
| Aspartic acid | 0.085 g |
| Cystine | 0.065 g |
| Glutamine | 0.16 g |
| Glycine | 0.11 g |
| Histidine | 0.82 g |
| Citric acid | 0.55 g |
| Phenol | 0.16 g |
| Sodium chloride | 7.62 g |
| Potassium sulfate | 1.90 g |
| Calcium sulfate | 0.85g |
| Potassium chloride | 0.79 g |
| Phosphoric acid | 0.84 g |
| Potassium bicarbonate | 0.16 g |
| Ammonium hydroxide | 0.82 g |
| Water | to 1 L |

APPENDIX C. TESTING PRETREATED SWATCHES.

3.4. Simulated Fecal Material.

a. The simulated fecal material will be refrigerated after preparation and then brought to room temperature before pretreating test swatches. The following formulation for simulated fecal material was calculated based on data showing the major constituents present in natural fecal obtained from Altman and Dittmer[14]. The proper amount of aqueous $NH_4OH$ will be added to provide 1.5 g of $NH_4OH$.

b. The additional solids (see following ingredients list) will be some type of pulverized crude fiber, such as dry small red beans (44.4 percent fiber), dry string beans [(haricot) 25.4 percent fiber], dry black turtle beans or dry pinto beans (39 percent fiber), dry lima beans (34.3 percent fiber), dry lentils (28.1 percent fiber), split peas (24.4 percent fiber), wheat bran, oat bran, corn bran, or rice bran, etc. In order to standardize the mixture, the additional solids will be prepared by grinding up dry small red beans, with substitution of another source of fiber only if the beans are unavailable. The total sum of solids will be 410 g.

c. The dry solids will be well ground and mixed in a blender. The fats will then be added and mixed well, followed by the water. The entire mixture will then be thoroughly blended to form a spreadable paste. If the mixture is too stiff, more water will be added. To allow the fiber to soak up water, the mixture will be stored for 1 day before use.

| Ingredient | Amount |
|---|---|
| Additional solids | 100.0 g |
| Electrolytes: | |
| Calcium chloride | 21.0 g |
| Magnesium sulfate | 7.5 g |
| Potassium chloride | 13.0 g |
| Ammonium hydroxide | 1.5 g |
| Fatty acids: | |
| Myristic acid | 1.8 g |
| Linoleic acid | 1.8 g |
| Deoxycholic acid | 21.0 g |
| Stearic acid | 23.0 g |
| Oleic acid | 13.0 g |
| Linolenic acid | 2.7 g |
| Phenol | 1.5 g |

## APPENDIX C. TESTING PRETREATED SWATCHES.

| | |
|---|---|
| Amino acids: | |
| Arginine | 3.8 g |
| Histidine | 1.7 g |
| Isoleucine | 4.3 g |
| Leucine | 5.6 g |
| Lysine | 5.7 g |
| Threonine | 4.0 g |
| Valine | 4.6 g |
| Fats: | |
| Unsalted butter | 42.0 g |
| Safflower oil | 22.0 g |
| Soaps: | |
| Sodium myristate | 1.9 g |
| Sodium palmitate | 39.0 g |
| Sodium stearate | 49.0 g |
| Sodium margarate | 1.3 g |
| Sodium palmitoleate | 1.1 g |
| Sodium oleate | 7.0 g |
| Bile acids: | |
| Deoxycholic acid | 3.6 g |
| Cholic acid | 1.0 g |
| Lithocholic acid | 1.0 g |
| Phospholipids: | |
| Lecithin | 1.0 g |
| Cephalin | 1.0 g |
| Sphingomyelin | 1.0 g |
| Phosphatidic acid | 1.0 g |
| Purine bases: | |
| Hypoxanthine | 0.6 g |
| Adenine | 0.6 g |
| Guanine | 0.6 g |
| Xanthine | 0.6 g |
| Water | 1360 g |

# APPENDIX C. TESTING PRETREATED SWATCHES.

## 4. PRETREATING THE SWATCHES.

a. The test swatch (a circular piece of material comprising all layers of a protective garment in the correct sequence) will be placed in a test fixture so that the swatch is secured to permit application of the pretreatment and to prevent the pretreatment from running off.

b. The amount of pretreatment, location of application, and weathering should be determined based on the guidance listed herein and program-specific requirements, and must be appropriately documented.

### 4.1. Amount of Pretreatment.

Amount of pretreatment is usually 2 mL unless otherwise dictated by the program-specific mission exposure or as described in Field Manual (FM) 3-11.5[15]. This pretreatment amount is based on covering the 10-$cm^2$ surface area of the swatch. Some pretreatments may also require immersion and/or total saturation.

### 4.2. Location of Pretreatment Application.

a. Pretreatments that normally would contaminate the external layer of the clothing (e.g., POLs, water, and AFFF) will be applied by thoroughly wetting the exterior surface of the swatch with the pretreatment. Pretreatments that normally would contaminate the interior surface of the clothing (e.g., sweat, urine, and feces) will be applied by thoroughly wetting the interior surface of the swatch with the pretreatment. Insect repellant (e.g., DEET) may be applied onto the exterior and/or interior layers of the swatch.

b. If the program-specific requirements include simulating the entire decontamination process, the specified decontaminant(s) may be rinsed with running water after the appropriate decontaminant application time. If the requirement of the program is to determine the effect of the decontamination compound itself, then the rinsing procedure will be omitted.

c. The pretreatment locations of decontaminants on swatch interior or exterior are listed below by decontaminant:

# APPENDIX C. TESTING PRETREATED SWATCHES.

| Decontaminant | Swatch Pretreatment Location |
|---|---|
| M258A1 Individual Decontamination Kit | Exterior |
| M291 Individual SDK | Interior and/or exterior |
| M295 Individual Equipment Decontamination Kit | Exterior |
| 10 percent HTH solution | Exterior |
| 5 percent sodium carbonate solution | Exterior |
| HSW: [1 lb Detergent, General Purpose, Liquid, NSN 7930-00-282-9699, in 5 gallons water at 38°C (100°F)] | Exterior |
| STB | Exterior |

4.3. Pretreatment Weathering.

Swatches with the pretreatments may be tested either wet or dry depending on the program-specific requirements. The pretreatment will remain on the swatches at ambient temperature for 1 hour; then the excess liquid will be drained off and blotted. To be tested wet, the swatch will be tested immediately thereafter. If the pretreatment on the material will be tested in a dry state, then the pretreated swatches will be exposed to air at 21°C (70°F) and held at this temperature for 24 hours.

4.4. Impact of Pretreatment Application.

a. Some pretreatments may affect the air permeability of the material. For convective-flow swatch testing, this may cause the air permeability to drop below the threshold for testing (Paragraph 3.2.3).

b. All materials that will be tested in the convective-flow configuration and tested with pretreatments should be assessed for air permeability after being pretreated. If the pretreatment reduces the air permeability below the threshold, then the material should not be tested in either the treated or untreated state using the convective-flow test cell configuration. A different test cell configuration should be selected (Paragraph 3.2.3).

# APPENDIX D. ABBREVIATIONS.

| | |
|---|---|
| ΔP | differential pressure |
| AATCC | American Association of Textile Chemists and Colorists |
| AD No. | accession number |
| AFFF | aqueous fire-fighting foam |
| APS™ | Aerodynamic Particle Sizer® |
| AR | Army Regulation |
| ASTM | American Society for Testing and Materials |
| ATEC | U.S. Army Test and Evaluation Command |
| ATTN | attention |
| CDD | capability development document |
| CoC | chain of custody |
| CONOPS | concept of operations |
| CPD | capability production document |
| CT | concentration × time |
| CWA | chemical warfare agent |
| DA | Department of the Army |
| DEET | insect repellent; N,N-diethyl-meta toluamide |
| DoE | design of experiment |
| DPG | U.S. Army Dugway Proving Ground |
| DTC | U.S. Army Developmental Test Command |
| FEP | fluorinated ethylene propylene |
| FID | flame ionization detection |
| FM | field manual |
| FPD | flame photometric detection |
| GB | sarin (isopropyl methylphosphonofluoridate) |
| GC | gas chromatograph(y) |
| GD | soman (pinacolyl methylphosphonofluoridate) |
| HD | distilled mustard (bis-(2-chloroethyl) sulfide) |
| Hg | mercury |
| HPLC | high-performance liquid chromatography |
| HSW | hot soapy water |
| HTH | high-test hypochlorite |

## APPENDIX D. ABBREVIATIONS.

| | |
|---|---|
| IAW | in accordance with |
| ICD | initial capabilities document |
| JAM | JSLIST-approved material |
| JP-8 | jet propulsion fuel number 8 |
| JSLIST | Joint Services Lightweight Integrated Suit Technology |
| LC | liquid chromatograph(y) |
| L/L | liquid challenge/liquid permeation |
| L/V | liquid challenge/vapor permeation |
| MINICAMS® | a miniature, automatic, continuous air-monitoring system |
| MQL | method quantification limit |
| MS | mass spectrometer |
| NCM | negative control material |
| $NH_4OH$ | ammonium hydroxide |
| NRT | near real time |
| NSN | National Stock Number |
| OTA | operational test agency |
| PAM | pamphlet |
| PCM | positive control material |
| PFA | perfluoroalkoxy |
| pH | negative ion activity |
| POL | petroleum, oil, and lubricants |
| PQL | program quantification limit |
| PTFE | polytetrafluoroethylene |
| PVC | polyvinyl chloride |
| QA | quality assurance |
| QC | quality control |
| RAR | rapid action revision |
| RH | relative humidity |
| RT | real time |
| RTI | Research Triangle Institute |

# APPENDIX D. ABBREVIATIONS.

| | |
|---|---|
| SDK | M291 Individual Decontamination Kit, Skin |
| SICN | swatch identification control number |
| sLpm | standard L/min |
| SOP | standing operating procedure |
| SST | solid sorbent tube |
| STB | supertropical bleach |
| TEMP | test and evaluation master plan |
| TEP | triethyl phosphate |
| TGD | thickened GD |
| TIC | toxic industrial chemical |
| TICN | test item control number |
| TOP | test operations procedure |
| VDLS | VISION Digital Library System |
| VISION | Versatile Information Systems Integrated ON-line |
| V/V | vapor challenge/vapor permeation |
| VX | persistent nerve agent; O-ethyl-S-(2-diisopropylaminoethyl) methyl phosphonothiolate |

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# APPENDIX E. REFERENCES.

1. Institute for Defense Analysis, Alexandria, Virginia, Chemical Challenge Study, December 2006.

2. Headquarters, Department of the Army (DA), Washington, DC, Army Regulation (AR) 385-10, The Army Safety Program, 27 August 2007 [Rapid Action Revision (RAR) 004, 4 October 2011].

3. Headquarters, Department of the Army (DA), Washington, DC, Pamphlet (PAM) 385-61, Toxic Chemical Agent Safety Standards, 17 December 2008.

4. American Society for Testing and Materials (ASTM) International, West Conshohocken, Pennsylvania, www.astm.org, ASTM Standard D737-04e2, Standard Test Method for Air Permeability of Textile Fabrics, DOI: 10.1520/D0737-04R08E02, Revised 2008.

5. Headquarters, Department of the Army (DA), Washington, DC, Army Regulation (AR) 50-6, Nuclear and Chemical Weapons and Materiel, Chemical Surety, 28 July 2008.

6. U.S. Army Test and Evaluation Command (ATEC), Aberdeen Proving Ground (APG), TOP 08-2-500, Receipt Inspection of Chemical and Biological (CB) Materiel, 1 July 1984.

7. U.S. Army DPG SOP WDC-ANA-004**, Procedures for the Analysis of Liquid Samples by Gas Chromatographic Methods, Revision 7, 25 January 2012.

8. U.S. Army DPG SOP DP-0000-M-073**, Preparation and Verification Procedures for First Dilution, Stock A and Working Solutions, Revision 14, 21 June 2012.

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10. U.S. Army DPG SOP WDC-ANA-030**, Procedure for Gas Chromatography (GC) Analysis of Swatch Bubble Solutions, Revision 7, 23 April 2012.

11. American Society for Testing and Materials (ASTM) International, West Conshohocken, Pennsylvania, www.astm.org, ASTM Standard F739-07, Standard Test Method for Permeation of Liquids and Gases Through Protective Clothing Materials Under Conditions of Continuous Contact, DOI: 10.1520/F0739-07, 2007.

12. Sverdrup, H. U., Johnson, M. W., and Fleming, R. H., The Oceans, Their Physics, Chemistry, and General Biology, Prentice Hall, Inc., New York, New York, 1942.

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## APPENDIX E. REFERENCES.

14. Altman, Philip L. and Dorothy S. Dittmer, Biological Handbook of Metabolism, Vol. III, 2d ed., pp 1489-1512, Federation of American Societies for Experimental Biology, Bethesda, Maryland, 1974.

15. Headquarters, Department of the Army (DA), Washington, DC, Field Manual (FM) 3-11.5, Multiservice Tactics, Techniques, and Procedures for Chemical, Biological, Radiological, and Nuclear Decontamination, 4 April 2006.

** SOPs are included only to serve as examples of the types of procedures used at U.S. Army Dugway Proving Ground (DPG), Utah, and as a reference for other installations. Many SOPs are specific to a particular installation, facility, or instrument, and may not apply to other installations, facilities, or instruments without modifications. It is expected that each installation will have its own equivalent SOPs. These equivalent SOPs must be provided to the test and evaluation community interested in this test method in order to properly understand the data produced, any differences between test method application between installations, and therefore the ability to compare data produced by different installations. If an installation does not have an equivalent SOP already in place, these or other similar procedures could be used as temporary guides until appropriate SOPs are developed. The most current version of these SOPs can be requested through the U.S. Army Test and Evaluation Command (ATEC) or through access to the Versatile Information Systems Integrated ON-line (VISION) Digital Library System (VDLS).

For information only (related publications)

a. Department of Defense (DoD), Washington, DC, Military Standard (MIL-STD)-882E, Standard Practice for System Safety, 11 May 2012.

b. Headquarters, Department of the Army (DA), Washington, DC, Army Regulation (AR) 190-59, Military Police, Chemical Agent Security Program, 11 September 2006.

APPENDIX F. APPROVAL AUTHORITY.

CAPAT Cover Sheet

# *TECMIPT Test Operations Procedures (TTOP) 08-2-501A, Permeation Testing of Materials With Chemical Agents or Simulants (Swatch Testing)*

Individual Protection Capability Area Process Action Team (CAPAT)

Key Contributors: Brent Baxter, Deborah Beier, Bryan Fausett, Aaron Orland, Scot Westwood (Dugway Proving Ground), and Gene Stark

CAPAT Review & Concurrence: April 2013

**Test and Evaluation Capabilities and Methodologies Integrated Process Team (TECMIPT) Participants:**

# APPENDIX F. APPROVAL AUTHORITY.

## CAPAT Signature Sheet

### Test Operations Procedures (TOP) 08-2-501A, Permeation Testing of Materials With Chemical Agents or Simulants (Swatch Testing)

The Individual Protection (IP) Capability Area Process Action Team (CAPAT) of the Test and Evaluation Capabilities and Methodologies Integrated Process Action Team (TECMIPT) has completed review of this document. The CAPAT recommends approval of this document. If a representative non-concurs, a dissenting position paper will be attached.

**Concurrence Sheet for the Test Operations Procedure (TOP) 8-2-501, Permeation Testing of Materials With Chemical Agents or Simulants (Swatch Testing)**

| | |
|---|---|
| Lt Col Kevin Reilly<br>Marine Corps Operational Test & Evaluation Activity (MCOTEA)<br>Signature Date 21 FEB 13 | Steven Tackett<br>US Army Test and Evaluation Command (ATEC)/U.S. Army Evaluation Command (AEC)<br>Signature 1 Mar 2013 Date |
| Nevin K. Elden, Colonel, USAF<br>Director of Operations<br>Air Force Operational Test and Evaluation Center (AFOTEC)<br>Signature 31 Jan 13 Date | Laurie K. Richter, Lt Col, USAF<br>Joint Requirements Office (JRO) for Chemical, Biological, Radiological, and Nuclear Defense<br>Signature 19 Feb 13 Date |
| Jeffery Bobrow<br>Assistant Chief of Staff, Expeditionary Warfare, Commander Operational Test and Evaluation Force (COMOPTEVFOR)<br>Signature 8 Apr 13 Date | Deborah Shuping<br>Office of the CBRN Defense T&E Executive<br>Signature Date 1/22/13 |
| Curt Wilhide<br>Joint Program Executive Office for Chemical and Biological Defense (JPEO CBD)<br>Signature 12 Jun 2013 Date | Michael Roberts<br>Joint Science and Technology Office (JSTO)<br>michael.roberts@dtra.mil Digitally signed by michael.roberts@dtra.mil DN: cn=michael.roberts@dtra.mil Date: 2013.03.26 14:55:11 -04'00'<br>Signature Date 02/07/13 |
| Charlie Walker<br>Individual Protection CAPAT Chair<br>Signature Date 130409 | |

**Note**: CAPAT members' Signature represents an 06 level concurrence from their organization. If the CAPAT representative is not empowered at this level, he/she must coordinate the concurrence/non-concurrence process within his/her organization, and prior to the specified suspense date for the document.

# APPENDIX F. APPROVAL AUTHORITY.

## T&E Capabilities and Methodologies Integrated Process Team (TECMIPT) Chair Endorsement

AMXAA-CD 15 August 2013

MEMORANDUM FOR

Chemical, Biological, Radiological and Nuclear Defense (CBRND) Test and Evaluation (T&E) Executive, Office of the Deputy Under Secretary of the Army, Taylor Building, Suite 8070, 2530 Crystal Drive, Arlington, VA 22202

SUBJECT: Test and Evaluation Capabilities and Methodologies Integrated Process Team (TECMIPT) Test Operations Procedure (TTOP) 8-2-501, Permeation Testing of Materials with Chemical Agents or Simulants (Swatch Testing)

1. The Individual Protection Capability Area Process Action Team (CAPAT) has completed their review of the subject TTOP in accordance with the DUSA-TE Instructions to the TECMIPT, the Standards and Development Plan, and the TECMIPT Standard Operating Procedure (SOP). All signatory members of the CAPAT have provided their concurrence to this TTOP. The CAPAT signature sheets and the ATEC Approval for Publication memorandum are enclosed.

2. Based on the concurrence of the CAPAT, I recommend the CBRND T&E Executive endorse this TTOP as a Department of Defense (DoD) Test and Evaluation (T&E) Standard.

Encl

RONALD O. PRESCOTT
TECMIPT Chair

# APPENDIX F. APPROVAL AUTHORITY.

## Deputy Under Secretary of the Army Endorsement

DEPARTMENT OF THE ARMY
OFFICE OF THE DEPUTY UNDER SECRETARY OF THE ARMY
102 ARMY PENTAGON
WASHINGTON, DC 20310-0102

AUG 2 3 2013

DUSA-TE

MEMORANDUM FOR DISTRIBUTION

SUBJECT: Endorsement of TECMIPT Test Operations Procedure (TTOP) 08-2-501, Permeation Testing of Materials with Chemical Agents or Simulants (Swatch Testing)

1. Reference: Memorandum, DUSA-TE, 19 July 10, subject: Chemical and Biological Defense Program (CBDP) Test and Evaluation (T&E) Standards Development Plan

2. The Individual Protection (IP) Capability Area Process Action Team (CAPAT) developed, coordinated, and approved TTOP 08-2-501 in accordance with the reference. The U.S. Army Test and Evaluation Command (ATEC) approved the TOP in accordance with the TOP approval process.

3. I endorse this TTOP as a DoD T&E Standard for chemical swatch testing and encourage its broad use across all test phases. All T&E Standards are for government associated program access and use. They are stored in Army Knowledge Online (AKO), located at https://www.us.army.mil/suite/files/22142943 and on the National Institute of Standards and Technology (NIST) website at http://gsi.nist.gov/global/index.cfm/L1-4/L2-19/A-664.

4. My point of contact for this action is Ms. Deborah Shuping, (703) 545-1119, deborah.f.shuping.civ@mail.mil.

Encl

JAMES C. COOKE
DOD CBRN T&E Executive

# APPENDIX F. APPROVAL AUTHORITY.

Approval for Publication, Director, Test Management Directorate (G9), ATEC-HQ

CSTE-TM 30 July 2013

MEMORANDUM FOR

Commanders, All Test Centers
Technical Directors, All Test Centers
Directors, U.S. Army Evaluation Center
Commander, U.S. Army Operational Test Command

SUBJECT: Test Operations Procedure (TOP) 08-2-501A, Permeation Testing of Materials with Chemical Agents or Simulants (Swatch Testing), Approved for Publication

1. TOP 08-2-501A, Permeation Testing of Materials with Chemical Agents or Simulants (Swatch Testing), has been reviewed by the U.S. Army Test and Evaluation Command (ATEC) Test Centers, the U.S. Army Operational Test Command, and the U.S. Army Evaluation Center. All comments received during the formal coordination period have been adjudicated by the preparing agency. The scope of the document is as follows:

This TOP provides the current standard methods for testing the permeation of liquid and vapor challenge chemical agents and simulants through swatches of materials. Swatches can be taken from clothing or equipment that is new, with or without pretreatment(s), and/or material that was previously subjected to periods of wear under varying conditions of field use, storage, and/or environmental exposure(s). These procedures are designed to be used as part of an overall assessment program evaluating the material performance, manufacturing, and integration with other pieces of the protective ensemble.

2. This document is approved for publication and has been posted to the Reference Library of the ATEC Vision Digital Library System (VDLS). The VDLS website can be accessed at https://vdls.atc.army.mil/.

3. Comments, suggestions, or questions on this document should be addressed to U.S. Army Test and Evaluation Command (CSTE-TM), 2202 Aberdeen Boulevard-Third Floor, Aberdeen Proving Ground, MD 21005-5001; or e-mailed to usarmy.apg.atec.mbx.atec-standards@mail.mil.

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Forward comments, recommended changes, or any pertinent data, which may be of use in improving this publication to the Range Infrastructure Division (CSTE-TM), US Army Test and Evaluation Command, 2202 Aberdeen Boulevard, Aberdeen Proving Ground, Maryland 21005-5001. Technical information may be obtained from the preparing activity: Director, West Desert Test Center, U.S. Army Dugway Proving Ground, ATTN: TEDT-DPW, Dugway, UT 84022-5000. Additional copies can be requested through the following website: http://itops.dtc.army.mil/RequestForDocuments.aspx, or through the Defense Technical Information Center, 8725 John J. Kingman Rd., STE 0944, Fort Belvoir, VA 22060-6218. This document is identified by the accession number (AD No.) printed on the first page.