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TEST OPERATIONS PROCEDURE

\*Test Operations Procedure (TOP) 8-2-061  
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CHEMICAL AND BIOLOGICAL DECONTAMINANT TESTING

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\* This TOP supersedes TOP 8-2-061, 30 September 1967

## 1. SCOPE

### 1.1 Purpose

a. Decontamination is the process of reducing or eliminating the hazards associated with chemical, biological, or radiological contamination in order to accomplish assigned missions. Means of decontaminating personnel, equipment, or areas include absorption, neutralization, weathering, and physical removal of the contaminant and hazard associated with chemical and biological (CB) agents, including emerging threat agents.

b. This TOP describes the test procedures designed to determine the technical performance of the test item(s). These procedures are relative to the criteria cited in applicable Operational Requirements Documents (ORDs) and other requirements and documentation that pertain to a particular test item. Appendix B lists all of the specific engineering tests and test methods required to characterize a decontaminant.

### 1.2 Application

a. This TOP provides the current standard for the planning and conduct of general performance tests of decontaminants, not inclusive of those systems intended for skin or personnel decontamination. The test procedures described herein should be used as the basis of a detailed or abbreviated test plan (DTP or ATP). Any deviations from this TOP should be accounted for in the DTP or ATP. The procedures may require modification for unique items or materials or to satisfy specific testing requirements as specified in a System Evaluation Plan (SEP) or other documentation. However, alteration of this procedure should be made only after full consideration of the possible effects the changes may have upon the reliability and validity of the data to be obtained. Alterations to this procedure will be coordinated between all concerned organizations in advance.

b. The test director (TD) or other appropriate person will be responsible for all phases of planning, coordinating, and reporting of tests and will follow the guidance in this TOP.

### 1.3 Limitations

a. Although environmental analysis, design analysis, and laboratory testing are valuable tools in the materiel acquisition process, there are inherent limitations in analysis and laboratory testing techniques that must be recognized. The methods of this TOP do not include many of the naturally occurring forcing functions that may affect materiel performance or integrity in service use. Further, analytic and laboratory test methods are limited in their abilities to simulate synergistic or antagonistic stress combinations, dynamic (time sequence) stress applications, aging, and other potentially significant stress combinations present in natural field/fleet service environments. Use caution when defining and extrapolating analyses, test criteria, and results.

b. Many of the test procedures and methods referenced in this TOP contain their own limitations that must be observed when using these procedures and methods.

## 2. FACILITIES AND INSTRUMENTATION

Facilities, instrumentation, and safety procedures used for decontaminating chemical agents, biological agents, and simulants are strictly controlled. Additional discussion and requirements for facilities and instrumentation are included in the test procedures of Paragraph 4.

## 2.1 Facilities

### 2.1.1 Chemical Test Facilities

| <u>Item</u>  | <u>Requirement</u>   |
|--|--|
| Chemical laboratory and chemical agent storage facility. | Constructed to ensure safe and secure storage, handling, analysis, and decontamination of chemical agents used for Research, Development, Test, and Evaluation (RDT&E). The quantities of chemical agents will be stored in accordance with (IAW) chemical surety requirements. The chemical agent laboratory, instruments, and personnel assignments must meet all requirements of References 1 and 2 and the safety requirements of References 3 and 4.  |
| Toxic-agent test facility.                               | Constructed to allow contamination, decontamination, and extended residual hazard sampling of test items deliberately contaminated with chemical agent in a temperature- and humidity-controlled environment. Must be equipped and certified for work with chemical agents. All exhaust air must be filtered; equipment, interior surfaces, tools, and waste must be easily decontaminated. No agent may be released to the environment. The facility must be designed to ensure safe and secure storage, transfer, handling, challenge, and disposal of chemical agents, decontaminating solutions, and solvents. The toxic agent test facility and personnel screening must meet all requirements of References 1 and 2 and the safety requirements of References 3, 4, and 5. |
| Chemical agent test chamber.                             | Fabricated with appropriated construction material (e.g., acrylic, stainless steel, glass, etc.), which is used to contain the chemical agents, coupons, coupon holding fixture, and decontaminant(s). The chamber includes doors with seals for ingress and egress of chemical agents, decontaminants, and applicator(s). The chamber may include glove ports or a half-man module.   |
| Personnel change room and shower facility.               | To allow test participants to shower and change into clean test clothing before and after assay of samples, and to reduce cross-contamination and contamination of facilities and non-test personnel.  |
| Environmental control system.                            | Test areas in laboratories and chambers must be equipped with environmental controls that allow air temperature and total air exchange rates to be controlled and maintained at prescribed levels throughout the testing period.   |

Instrumented test range or appropriate operational test facility. To allow observation and measurement of degradation in performance of mission-essential equipment attributable to contamination/decontamination procedures.

### 2.1.2 Biological Test Facilities

| <u>Item</u>   | <u>Requirement</u>   |
|---|--|
| Biological assay laboratories.  | Required to store, analyze, and prepare test quantities of biological agent and simulant materials, to charge disseminating devices, and to prepare samplers.  |
| Chambers for biological simulant testing.   | Equipped with an air intake and an exhaust system, which exhausts through high efficiency particulate filters (capable of retaining 99.7 percent of particles 0.3 $\mu\text{m}$ or greater in diameter) into an exhaust system. The chamber will have sufficient volume to allow free air circulation around the test sample.  |
| A Class III safety cabinet for housing and containing agent biological origin aerosol generators, aerosol mixers, primary containment chambers, and referee samplers for testing biological detector aerosol sensitivity and operation. | A Class III biological safety cabinet that can serve as the secondary physical containment barrier for the pathogen-testing suite. It can be constructed of stainless steel and equipped with an attached autoclave, glove ports, electrical outlets, lighting, connections for a hydrogen peroxide vapor generator, and a disinfectant-filled dunk tank pass through. The Class III biological should maintain certification at Class III cabinet specifications (holds 2.0 inches of water pressure with no more than 10 percent drop for 30 min). All test air leaving the Class III biological safety cabinet should be double exhaust high-efficiency particulate air (HEPA) filtered and use four HEPA filters before being introduced into the building air-exhaust system. |
| A Class II biological safety cabinets for containing agent of biological origin (ABO) solutions for testing biological detector or identifier sensitivity and operation.  | The Class II biological safety cabinets serve as the primary physical containment barrier for testing with ABO solutions. The cabinets can be constructed of stainless steel and equipped with electrical outlets and lighting. Temperature and relative humidity (RH) sensors are mounted as required. These cabinets should be certified to Class II cabinet specifications (holds 2.0 inches of water pressure with no more than 10 percent drop for 30 min).   |

### 2.2 Instrumentation

These instruments or equivalent will be used. Instrumentation unique to a test will be listed in the appropriate section below or in the DTP or ATP.

### 2.2.1 General Test Instrumentation

| <u>Item</u>  | <u>Permissible Error of Measurement</u>  |
|--|--|
| Air temperature.   | $\pm 1^{\circ}\text{F}$  |
| RH.  | $\pm 5$ percent  |
| Wind speed.  | $\pm 10$ percent   |
| Still color camera.  | Adequate to document typical test procedures, details of contamination techniques, and any discrepancies from planned procedures necessitated by operational conditions. |
| Television camera, motion picture camera, and/or recorder. | Adequate to monitor the test chamber and to document time test events and procedures.  |

### 2.2.2 Chemical Test Instrumentation

| <u>Item</u>   | <u>Permissible Error of Measurement</u>  |
|---|--|
| Chemical agent test chamber, test coupon holding fixture.   | Constructed to allow coupons to be affixed and held secure while being decontaminated (potentially using high pressure spray equipment). The fixture should allow incline angle of the test coupon to be varied and should allow distance from test coupon to decontaminant applicator system nozzle to be varied. |
| Sampling chemical agent vapor off-gassing from contaminated surfaces [bubblers, Miniature Automatic Continuous Air Monitoring System <sup>®</sup> (MINICAMS <sup>®</sup> ), solid sorbent tubes, or equivalent] with sampling efficiency >95 percent. | Flow rate, in L/min, $\pm 5$ percent.  |
| Contamination density and droplet size (Printflex <sup>®</sup> cards, Kromecoat <sup>®</sup> cards, filter papers, or equivalent).  | Contamination density, in $\text{g}/\text{m}^2$ , $\pm 10$ percent; droplet size diameter, in mm, $\pm 10$ percent.  |

| <u>Item</u>  | <u>Permissible Error of Measurement</u>  |
|--|--|
| Agent concentration in samples (spectrophotometer, automated or hand-injected gas-liquid chromatograph, or equivalent).  | Agent/sample, in mg, $\pm 8$ percent. (In automated mode. Better precision is achievable at additional cost and time.)           |
| Measuring and counting spot size instrument (Hamamatsu Image Analyzer™, Quantimet™, or equivalent). Hamilton syringe (100 $\mu$ L) fitted with a repeating pipette dispenser system.   | Droplet stain size, in mm, $\pm 10$ percent; droplet stain number by size, $\pm 10$ percent.                                     |
| Chemical contact hazard samplers (silicone rubber samplers or equivalent). The silicone rubber used is 1 mm thick, translucent, unfilled, Poly (dimethylsiloxane) with a durometer reading of 60. The silicone rubber should be rinsed with water and then dried for 24 hr at 85°C (185°F). Circular disks of this material, 3.64 cm in diameter (area of 25 cm <sup>2</sup> ), were used as samplers. | Agent extraction efficiency from sampler, in $\mu$ g/sample, $\pm 10$ percent.   |
| Applying chemical agent to the test sample.  | Contamination density, in g/m <sup>2</sup> , $\pm 10$ percent; droplet size, within range specified for the agent.               |
| Safety monitoring for agent within a test chamber, hood, or toxic facility work area. MINICAMS®, Real-Time Monitor (RTM), Miniature Infrared Analyzer® (MIRAN®), Automatic Continuous Air Monitoring System (ACAMS), Depot Area Air Monitoring System (DAAMS), or their equivalents, may be used in conjunction with a suitable agent containment device or temperature control box.                   | Near real-time. All instruments have differing sensitivities. The available instruments with the best sensitivity shall be used. |
| Rigid containers for holding test samples for agent vapor desorption and sampling. Must be constructed of nonabsorbent material such as stainless steel, glass, etc. Internal dimensions to be measured and reported.  | Interior dimensions $\pm 2$ mm.  |

| <u>Item</u>   | <u>Permissible Error of Measurement</u>   |
|---|---|
| Gas chromatography (GC), GC-mass spectrometry (MS), GC-flame photometric detection (FPD), GC-flame ionization detection (FID), GC-atomic emission detection (AED), or other suitable detectors. | Quality control (QC) or check shot sample, $\pm 15$ percent. Test sample repeat analysis, $\pm 10$ percent. |

Test Assembly Control and Data Acquisition.  $\pm 10$  percent (temperature, RH, and airflow).  
The system will be capable of controlling and/or providing test chamber temperature, RH, and airflow. The system will acquire and record data at a minimum rate as prescribed by test procedures.

### 2.2.3 Biological Test Instrumentation

| <u>Item</u>   | <u>Permissible Error of Measurement</u>   |
|---|---|
| Applying biological agent simulant contamination to the test sample (collision atomizer or equivalent). | Air contamination of $1 \pm 0.5 \times 10^6$ colony forming units (CFU)/L of air. |
| Swab sampling of the test item (calcium alginate swabs, test tubes, and diluents).                      | Swab surface sampling efficiency in CFU/sample, $\pm 10$ percent.                 |
| Assay of biological simulants (microscopes, automatic colony counters, etc.).                           | Number of CFU/sample, $\pm 10$ percent.   |

### 2.3 Data Acquisition and Control

All critical data and information pertaining to each test item shall be acquired, tabulated, analyzed, and displayed by test item control number (TICN), trial by trial control number (TCN), and analytical result by analytical control number (ACN), when applicable. This may include test sample and analytical data chain-of-custody (CoC) information; test sample inspection information; quality assurance (QA) inspection results; target versus actual trial matrices by test type, test conditions by trial by test type; raw analytical results or pass/fail results by test type, QC test results; data analyses models, and/or data analysis results.

## 3. TEST PLANNING

### 3.1 Test Design

Based on the evaluation requirements, a DTP or ATP shall be developed that will include at a minimum a test design, execution matrix, detailed procedures, QA/QC measures, data management, statistical data analysis, and results presentation.



### 3.2 Familiarization

The test planning phase includes identifying potential problem areas by reviewing previous records and the results of similar tests. Relevant Standard Operation Procedures (SOPs) and other procedures to be used shall be reviewed for applicability, completeness, and adequacy. The development of a DTP or ATP requires a review of the applicable SEP and/or other test guidance literature. Familiarity with preceding development and test phases, test criteria, and selection of appropriate samples, methods, sequences, facilities, and test equipment is also recommended. Data from previous similar tests shall be considered in order to avoid duplication of data and to reduce the scope of further testing.

### 3.3 Documentation

The TD shall have all pertinent documentation available. These documents may include Government and manufacturers' publications, requirements documentation, SEP, Safety Assessment Report (SAR), test planning directive, Systems Support Package (SSP) list, Record of Environmental Consideration (REC), TOPs, SOPs, Material Safety Data Sheets (MSDSs), approved DTP, safety release, funding cite, Environmental Assessment (EA), Security Classification Guide (SCG), etc., as required.

### 3.4 Detailed Test Plan or Abbreviated Test Plan

The DTP or ATP document must be prepared, coordinated, and approved before any testing begins. The responsibility for the preparation, coordination, and staffing of this document resides with the assigned TD or project officer, whether Government or industry.

### 3.5 Environmental Documentation

a. An approved EA must be on file covering the storage, use, and disposal of hazardous materials, hazardous waste, and decontaminants used during the execution of the TOP and ATP. The assessment must fully address the potential environmental impact of any specific test being planned. The DTP or ATP must cite the EA and/or a REC, which cites the EA, and the appropriate categorical exclusion. The REC must be approved before testing begins.

b. Compliance with the National Environmental Policy Act (NEPA) requires that an Environmental Impact Assessment for Life Cycle (EIALC) be prepared and that potential environmental impacts be assessed at the earliest practicable stage in the planning process of any new equipment. Testing at Government or contractor facilities must also be assessed for environmental impact. When a proposed action may significantly affect the quality of the environment, is highly environmentally controversial, or is expected to evoke litigation based on environmental issues, a detailed Environmental Impact Statement (EIS) will be prepared and evaluated IAW NEPA processes. Before the test begins, the project officer will ensure that an EIALC, an EIS, or other appropriate documentation has been completed and approved.

### 3.6 Test Readiness Review

If required, programs will undergo a test readiness review (TRR) before testing begins to ensure that the necessary resources are available to effectively and efficiently conduct the test. Representatives from essential support organizations [which may include warfighters, Test Integration Working Group (TIWG), Lead Integrated Product Team (IPT), and contractor(s)] will attend this review and provide input as to their readiness to perform specified requirements. The designated TD will conduct this review and present the status of all critical elements using

red (No Go), yellow (Marginal), and green (Go) designators to determine readiness. The reviewing authority will be in attendance during these proceedings and will decide whether the testing is ready to proceed.

### 3.7 Test Incident Report/Corrective Action Report

Test Incident Reports (TIRs) and Corrective Action Reports (CARs) or equivalent, unless waived by the test sponsor, will be prepared and distributed IAW U.S. Army Developmental Test Command (DTC) Regulation 70-24.

### 3.8 Test Preparation

#### 3.8.1 Security

Security considerations will be adequately determined and provided for, as applicable to each test sample.

#### 3.8.2 Logistical Requirements

Before testing, the TD will ensure that the necessary resources will be available to effectively and efficiently conduct the test.

#### 3.8.3 Test Item Sample Size

The number of test items for each of the tests identified in this TOP may be determined by test item type, test item availability, cost, or other factors. If the sample size is less than optimal, a testing scheme shall be devised to maximize test item utilization and required data output to meet stated objectives and criteria. Sample size will also be determined by the degree of statistical confidence specified in requirements documents. Unless otherwise stated, the sample size is triplicate.

### 3.9 Safety

#### 3.9.1 Safety Statement

The developing agency will ensure that all applicable safety documentation is provided to the test agency 30 days before the start of testing. Documentation includes, but is not limited to, MSDSs, operation manuals, and SARs.

#### 3.9.2 Safety Procedures

a. The primary emphasis in testing must be placed on safety when CB agents are used. CB agents are extremely dangerous and must be handled according to written TOPs. These procedures include, but are not limited to, the testing organization's approved SOPs for agent handling and the procedures specified by the DTP or ATP.

- (1) All SOPs must be reviewed by responsible organizations and approved before testing.
- (2) Procedures that are not included in approved SOPs will not be used.
- (3) A hazard analysis may be required by the testing organization.

(4) All laboratory personnel must thoroughly understand the SOPs that apply to the test and must acknowledge their understanding by signature, if required.

b. DTPs and procedures will ensure performance in the safest manner consistent with fulfillment of the mission.

(1) Plans will include safety procedures, precautions, protective measures, and emergency procedures, as necessary.

(2) Information on the hazards and safety characteristics of the test item(s) as provided by the safety statement and other pertinent information, which may need to be incorporated in DTPs and procedures, will include:

- (a) Evaluation of potential hazards.
- (b) Analysis of risks and limitations.
- (c) Additional precautions, as appropriate.
- (d) Explanation of special test equipment and techniques.

c. DTPs and procedures should include, as appropriate:

(1) Safety procedures, precautions, protective measures, and emergency procedures.

(2) Technical information on the hazards and safety characteristics of the test item, as provided by the safety statement and other pertinent information.

(3) Evaluation of potential hazards, analysis of risks, limitations, and precautions, including special test equipment and techniques.

(4) Special attention should be paid to hazards to operators from exhaust gases.

d. All personnel who participate in the tests defined in this TOP will be:

(1) Thoroughly briefed on the hazards involved.

(2) Briefed on approved procedures and the proper test method to be used.

e. If the test includes the use of SOPs, participants may be required to read and understand applicable sections thereof and sign the supervisor or operator signature sheet.

### 3.10 Quality Assurance

a. All test items, test equipment, and analytical data should be labeled to prevent misidentification during the test process. All test item processing, labeling, and CoC should be verified by an independent QA inspection before beginning the test.

b. Test control samples should be handled as required in the DTP or ATP. The test control samples demonstrate control of the test process across trials and test programs, and verify the lack of positive and negative analytical interferents.

c. If appropriate, and based on program objectives and schedules, an operational readiness inspection (ORI) should be held.

(1) The purpose of the ORI is to conduct one or more baseline trials with control samples to demonstrate that the current test methods and procedures yield results that can correlate to historical data.

(2) The ORI results and collected data should be reviewed and compared with the test process and procedures to determine if any changes are needed.

(3) The results of the ORI should be published in a report that details the test procedures used and the results of the data analysis.

e. For chemical analysis of collection solvent from a bubbler or chemical analysis of solid sorbent tubes, conduct the chemical analysis procedure using the appropriate number of standards, blanks, and analytical controls.

f. The sampler selected for use must be well characterized. For example, if a bubbler sampler is used, the collection efficiency at the test temperature and test humidity must be known. If solid sorbent tubes are used, positive steps must be taken to ensure that the capacity of the sorbent is not exceeded and that the absence of residual sorbed chemical has been demonstrated. When solvents are used for collection or extraction, document the stability of the agent in the solvent and, if used as an extractant, the extraction efficiency.

g. The purity of the agent used for each program must be analytically demonstrated at a frequency specified by the testing organization or in the approved DTP or ATP. Procedures used in the testing organization's SOPs and presented in the DTP or ATP will be used. Only chemical agent with purity in excess of 85 percent will be used.

h. If the samples collected during the test must be stored for longer than 1 day before they can be analyzed, several storage controls shall be stored with the test samples. These storage control samples shall then be analyzed with the test samples to show any sample degradation or pickup of interfering chemicals during storage.

i. All aspects of the testing shall be performed with emphasis on acquiring valid, credible, and verifiable results.

#### 4. TEST PROCEDURES

##### 4.1 Introduction

This section describes the decontaminant test procedures. Details of the test procedures are given in Paragraph 4.5.

##### 4.2 Receipt Inspection

The test item will be subjected to the following procedures after its arrival at the test site, with emphasis on the following:

a. Test item inspection:

(1) The test item will be visually inspected and all evidence of damage will be recorded, including:

- (a) Corrosion of hardware.
- (b) Broken connections.
- (c) Cracked or deteriorated hoses, seals, and valve pickings.
- (d) Contamination with foreign material (solid and/or liquid).
- (e) Specific anomalies.

- (f) Evidence of damage, deterioration, or illegible markings.
- (2) The presence of internal damage to the test item will be determined.
- (3) Missing components, instructions, or manuals will be noted.
- b. The length, width, height, and weight of the test item and its packaging will be measured and recorded.
- c. The test item's model, serial number, nomenclature, identifier, manufacturer, lot number and other pertinent information/indicators, if applicable, will be recorded.
- d. The test item will be unpacked and numbered serially for future identification.
- e. The test item power requirements will be determined and recorded.
- f. Photographs will be taken of decontaminant containers, equipment, and any damaged items.
- g. Analytical results or certification of the decontaminant individual constituent component chemical composition will be reviewed/verified.
- h. The MSDS for individual constituent component chemical composition will be reviewed.

#### 4.3 Safety Evaluation

- a. The condition of the test item as received in relation to any potential unsafe aspects during subsequent operation will be noted.
- b. Jagged edges, rust, dents, loose connections, or any other condition or features that may make use of the test item hazardous to personnel will be noted.
- c. If the following tests are conducted by the manufacturer, the test reports are provided with the equipment during shipping. Particular attention will be paid to the results of:
  - (1) The rough handling and surface transport tests.
  - (2) The environmental tests.
- d. The safety aspects, as cited in the safety statement prepared by the developing agency, will be verified.
- e. Data to be included in the safety release recommendation will be collected. The safety release recommendation will be made to DTC in regard to test item's suitability for testing with troops.

#### 4.4 Test Setup/Preparation

##### 4.4.1 Test Item Setup

- a. Unless otherwise specified, the test item will be installed in the test facility in a manner that will simulate service use to the extent practical, with test connections made and instrumentation attached, as necessary.
- b. To test the effectiveness of protective devices, plugs, covers, and inspection plates used during servicing will be in positions appropriate for the test and in their normal (protected or unprotected) mode during operation.
- c. The test items must be protected from unrelated environmental contaminants.

#### 4.4.2 Pretest

a. During the pretest phase, if samplers are to be used they will be installed and operational. Each sampler shall be individually labeled and tested with an appropriate device, located in the sampling area, to ensure that the sampler is operational and will perform as expected. Specific details regarding the type of samplers that will be used and the appropriate methods for verifying that these samplers are operational will be included in the DTP or ATP.

b. If any tests contained in this TOP are conducted at multiple laboratories, the pretest may include an intra-laboratory study to ensure data collected at both sites are compatible.

#### 4.5 Procedures

##### 4.5.1 General

a. This section briefly describes the different types of tests to be discussed in this TOP.

b. Specific tests may be waived or additional tests may be required on a case-by-case basis as agreed upon by the test agency and the test sponsor. Details of test procedures are given in the DTP or ATP, referenced TOPs, or stated procedures.

c. A certification of purity must be supplied when chemical agents are used. Chemical Agent Standard Analytical Reference Material (CASARM) agent will be used to the maximum extent possible. Purity certification should include freezing point depression, nuclear magnetic resonance (NMR), and GC analysis documented for each lot. Chemicals used for preparations, and extractions of decontaminant formulations will be used as received and their purity documented as supplied.

d. Selected simulants may be used for testing purposes when approved by the test agency and test sponsor.

e. Decontaminants will be prepared IAW the manufacturer's instructions and recommendations. Where possible, decontaminants to be tested, as well as any specialized equipment needed for preparation of the candidate formulations, should be acquired from the manufacturer. Quality shall be checked by routine analytical methods (such as pH measurement, titration, etc.). Where applicable, care must be taken so that the pot life (as specified by the manufacturer) of the decontaminant is not exceeded.

f. Data from all tests shall be documented and presented in the final report.

g. Number of replicates conducted shall be sufficient to demonstrate high, statistical confidence in results, or as specifically identified by the test sponsor.

h. The test items must be protected from unrelated environmental contaminants.

##### 4.5.2 Laboratory Test – Contact Hazard Test

a. This test is designed to estimate the contact hazard (or agent available for transfer by contact) after decontamination. Contact hazard is defined as the amount of agent that is present after decontamination that could transfer to skin or other surfaces upon contact. Coupons of equipment materials, component assemblies, or whole test items may be used in this test.

b. The number of replicates conducted must be sufficient to demonstrate high, statistical confidence in results or as specifically identified by the test sponsor.

c. The test may be required to be conducted at low [ $\sim 32^{\circ}\text{C}$  ( $\sim 25^{\circ}\text{F}$ )], ambient [ $21^{\circ}\text{C}$  ( $\sim 70^{\circ}\text{F}$ )], and high [ $\sim 49^{\circ}\text{C}$  ( $\sim 120^{\circ}\text{F}$ )] temperatures, and at low ( $< 20\%$ ) and high ( $> 90\%$ ) RH. Environmental parameters will be clearly defined in the ATP or DTP.

d. Coupons [representing a range of surfaces inherent to the items undergoing decontamination operations (vehicles, vans, weapons, etc.)] will be prepared from materials specified by the test sponsor. An example of a material list is presented in Appendix C.

e. To prevent agent and/or decontaminant spread from the defined testing surface area of the test item/coupon, an appropriate border material will be placed on the periphery of the coupon to prevent agent spread.

f. The coupon will be positioned horizontally, and chemical agent will be applied per the ATP or DTP at a density of  $10\text{ g/m}^2$  ( $1\text{ mg/cm}^2$ ) or as specified by the test sponsor.

g. The equipment or coupons will be covered to prevent/reduce evaporation. The agent will reside for 1 hr (resistance time), or as specified by test sponsor.

h. Decontaminant will be applied in the amount and manner specified by the test sponsor. As a reference, U.S. Army Field Manual (FM) 3-5 (Reference 6) indicates an approximate decontaminant/ agent ratio of 50:1 (weight or volume of decontaminant to mass of agent). Care will be taken to ensure that the entire area is covered by the decontaminant.

i. Decontaminant will reside on the test sample for 15 min, or a time to be specified by the test sponsor or vendor.

j. Unless otherwise specified, the test sample will be rinsed with water and allowed to dry for the amount of time specified in the ATP or DTP. All surfaces of the test sample will be rinsed.

k. A piece of latex dental dam, silicone rubber, or other sorbent material specified by the test sponsor will be applied on top of the test item. A 5.1-cm (2-in) diameter piece of aluminum foil will be placed on top of the sampler. A sponge-like pad will be placed on top of the aluminum foil to increase the surface area contacted on irregular contaminated surfaces.

l. A 1 kg weight will be placed on the sampler for 15 min, or as specified by the test sponsor.

m. The weight and sampler will be removed and the transferred agent will be extracted from the sampler using chloroform or other appropriate extraction solvent.

n. The solvent will be analyzed for agent (GC-FPD is suggested).

o. The following data will be reported:

(1) Total mass extracted from the contact sampler.

(2) Surface area tested, temperature, RH, and agent purity.

(3) TICN, sample item control number (SICN), agent purity, control results, and observations.

#### 4.5.3 Laboratory Test – Off-gassing Test (Vapor Hazard)

##### a. General

(1) This test determines the decontaminant's effectiveness at neutralizing chemical agents as determined by measuring the off-gassing of chemical agents following decontamination.

(2) The number of replicates conducted must be sufficient to demonstrate high statistical confidence in results, or as specifically identified by the test sponsor.

(3) The test may be required to be conducted at low [ $\sim 32^{\circ}\text{C}$  ( $\sim 25^{\circ}\text{F}$ )], ambient [ $21^{\circ}\text{C}$  ( $\sim 70^{\circ}\text{F}$ )], and high [ $\sim 49^{\circ}\text{C}$  ( $\sim 120^{\circ}\text{F}$ )] temperatures, and low ( $< 20\%$ ) and high ( $> 90\%$ ) RH.

(4) Coupons [representing a range of surfaces inherent to the items undergoing immediate decontamination operations (vehicles, vans, weapons, etc.)] will be prepared of materials specified by the test sponsor. An example of a material list is presented in Appendix C.

(5) To prevent agent and/or decontaminant spread from the defined testing surface area of the test item/coupon, an appropriate border material will be placed on the periphery of the coupon to prevent agent spread.

(6) The coupon will be positioned horizontally, and chemical agent will be applied per the ATP or DTP at a density of  $10\text{ g/m}^2$  ( $1\text{ mg/cm}^2$ ), or as specified by the test sponsor. The equipment or coupons will be covered to prevent/reduce evaporation. The agent will reside for 1 hr (resistance time; or as specified by test sponsor).

(7) Decontaminant will be applied in the amount and manner specified by the test sponsor. As a reference, FM 3-5 indicates an approximate decontaminant/agent ratio of 50:1 (weight or volume of decontaminant to mass of agent). Care will be taken to ensure that the entire area is covered by the decontaminant.

(8) Decontaminant will reside on the test coupon for 15 min or a time to be specified by the test sponsor.

(9) Unless otherwise specified, the test item will be rinsed with water and allowed to dry for amount of time specified in the ATP or DTP. All surfaces of the test coupon will be rinsed.

(10) The test item/coupon, decontaminant, or both can be analyzed for off-gassing.

b. Test Item/Coupon Off-gassing

(1) Unless otherwise specified, the test coupon surface will be performed as specified by test sponsor.

(2) The test items/coupons will be placed into the testing apparatus specifically designed for such tests. The testing apparatus will consist of a sealed test container/fixture that allows a stream of air to be drawn across the test item/coupon. The off-gas test container/fixture must be made of inert materials, i.e., stainless steel or T6061 aluminum or equivalent.

(3) The flow of air will be specified by the ATP or DTP. The effluent air will pass through a sampler, such as a glass impinger or solid sorbent tubes, containing an inert trapping solvent material, thereby trapping the agent present in the air stream.

(4) At times specified by the test sponsor (e.g., 30 min, 1 hr, and 2 hr) the samplers will be replaced with new samplers. The total test time will be 12 hr and the sampling intervals will be determined by the test sponsor. The sampler media (e.g., agent trapping solvent) will be



analyzed for amount of agent. The 12-hr cumulative mass will be divided by the 12-hr total air volume.

(5) Sampler media will be analyzed by GC-FPD, GC-FID, and/or GC-MS. If near real-time sampling is conducted (e.g., MINICAMS<sup>®</sup>), that data will be reported.

c. Decontaminant Off-gassing

(1) If the decontaminant used is a sorptive powder or some other solid, then the following procedures will be used.

(2) At the end of the specified decontamination time (Paragraph 4.5.4.g), the decontaminant will be tapped and/or scraped into a Petri dish, and the cover placed over the Petri dish as soon as possible to minimize agent vapor loss.

(3) Solid decontaminant will be off-gassed at ambient hood temperature in an off-gassing chamber for 12 hr or other specified time. Samplers (e.g., bubblers containing agent trapping solvent) (nominal flow of 1 L/min) will be removed at specified time intervals. The sampler media will be analyzed for agent presence using GC-FPD, GC-FID, and/or GC-MS.

d. The following data will be reported:

- (1) Sampling time intervals.
- (2) Flow rate of samplers.
- (3) Amount of media in samplers.
- (4) Type of samplers used.
- (5) Analysis technique used.
- (6) Amount of agent remaining in media.
- (7) Temperature, RH, agent purity, and surface area tested.
- (8) TICN, SICN, agent purity, control results, observations.

4.5.4 Laboratory Test – Decontamination Efficacy Test (Chemical)

a. This test is conducted to evaluate the effectiveness of the decontaminant based on percent chemical agent neutralized. This test is performed on coupons because an entire test item cannot be extracted. Therefore, decontamination efficacy testing cannot be performed on a system.

b. The number of replicates conducted must be sufficient to demonstrate high statistical confidence in results, or as specifically identified by the test sponsor. The extraction efficiency subtest must be performed to determine optimum extraction solvent and extraction efficiency.

c. The test may be required to be conducted at low [~32°C (~25°F)], ambient [21°C (~70°F)], and high [~49°C (~120°F)] temperatures, and at low (< 20%) and high (> 90%) RH.

d. Coupons [representing a range of surfaces inherent to the items undergoing immediate decontamination operations (vehicles, vans, weapons, etc.)] will be prepared of materials specified by the test sponsor. An example of a material list is presented in Appendix C.

e. To prevent agent and/or decontaminant spread from the defined testing surface area of the test item/coupon, an appropriate border material will be placed on the periphery of the coupon to prevent agent spread.

f. The coupon will be positioned horizontally and chemical agent will be applied per the ATP or DTP at a density of  $10 \text{ g/m}^2$  ( $1 \text{ mg/cm}^2$ ) or as specified by the test sponsor. The equipment or coupons will be covered to prevent/reduce evaporation. The agent will reside for 1 hr (resistance time), or as specified by test sponsor.

g. Decontaminant will be applied in the amount and manner specified by the test sponsor. As a reference, FM 3-5 indicates an approximate decontaminant/agent ratio of 50:1 (weight or volume of decontaminant to mass of agent). Care will be taken to ensure that the entire area is covered by the decontaminant.

h. Decontaminant will reside on the test coupon for 15 min, or a time to be specified by the test sponsor.

i. Unless otherwise specified, the test item will be rinsed with water and allowed to dry for amount of time specified in the ATP or DTP. All surfaces of the test coupon will be rinsed.

j. Samples of the rinse water will be collected and analyzed for agent.

k. Each coupon will be checked for visible damage or degradation to the surface. Any damage will be noted in the test logbook.

l. Residual agent from the coupon will be extracted using chloroform or other suitable extraction solvent. Extraction times may need to be determined depending on agent, surface, and solvent. Each coupon will be placed in its own sealed sample jar with extraction solvent and gently swirled. The coupons will be allowed to extract for 6 hr, or other specified time.

m. The solvent will be analyzed for agent (GC-FPD, GC-FID, and/or GC-MS).

n. The following data will be reported:

- (1) Temperature of test apparatus.
- (2) RH of test apparatus.
- (3) Decontamination procedures used.
- (4) Mass (mg) of chemical agent deposited on coupons and purity.
- (5) Mass (mg) and concentration ( $\text{mg/m}^3$ ) of chemical agent extracted from samples.
- (6) Volume of the rinse water and the concentration of agent in the rinse water.
- (7) TICN, SICN, agent purity, control results, and observations.

#### 4.5.5 Laboratory Test – Decontamination Efficacy Test (Biological)

##### 4.5.5.1 General

a. This test is conducted to evaluate the effectiveness of a decontaminant based on percent biological agent destroyed.

b. Contaminants will be specified by the test sponsor, but may include spores, vegetative organisms, and viruses [e.g., *Bacillus subtilis* var. *niger* (BG), *Erwinia herbicola* (EH), Male-specific bacteriophage type 2 (MS2)].

c. All procedures will be performed with appropriate personal protective equipment and sterile laboratory techniques. Additionally, sterile materials (including pipette tips, agar plates, and disposable spreaders) will be employed to ensure the integrity of the solutions being tested and to prevent contamination.

#### 4.5.5.2 Effect of Microbial Concentration on Decontaminant Performance

##### a. Spores

(1) An initial concentration of 10 mg of viable spores/10 mL of buffer [e.g., phosphate buffered saline (PBS)], corresponding to  $1 \times 10^8$  viable spores/mL [Industry and U.S. Army Dugway Proving Ground (DPG) Life Sciences standard], will be prepared by vortexing. A series of different concentrations ( $10^8$ ,  $10^6$ , and  $10^4$  spores/mL) will be obtained by serial dilution, or as specified by test sponsor. **NOTE:** If the desired final concentration is  $10^8$  spores/mL, then 1 mg spores must be weighed into each tube (without first suspending in buffer).

(2) The recommended concentration for each decontaminant solution will be added to each dilution tube. The samples containing decontaminants will be incubated at room temperature [25°C (77°F)] for 1 hr with stirring. Decontamination will then be removed by washing the spores a minimum of three times and resuspending in fresh buffer.

(3) Serial dilutions will be performed and samples will be plated and incubated at the appropriate temperature for the appropriate time period. Concentrations of viable spores will be measured by viable count. Controls will consist of spore suspensions treated in an identical manner to the experimental tubes but supplemented with buffer (PBS) as opposed to a candidate decontaminant.

##### b. Vegetative Bacterial Cells

(1) A 10 mL stock solution of  $1 \times 10^8$  CFU/mL in PBS will be prepared. Additional concentrations of  $10^6$  and  $10^4$  CFU/mL will be prepared from this stock solution by serial dilution.

(2) The manufacturer's recommended concentration of each decontaminant will be added to its corresponding dilution tube. The samples containing decontaminants will be incubated at room temperature [25°C (77°F)] for 1 hr. Decontaminant solutions will then be neutralized (sodium thiosulfate for oxidizing agents; concentration to be determined) and removed by washing the vegetative bacteria a minimum of three times and resuspending in fresh buffer.

(3) Serial dilutions will be performed and samples will be plated and incubated at the appropriate temperature for the appropriate time period. Concentrations of viable vegetative bacteria will be measured by viable count. Controls will consist of viable vegetative bacterial suspensions treated in an identical manner to the tubes containing decontaminants, but supplemented with PBS rather than decontaminant.

c. Viruses

(1) Virus (phage) will be suspended at a concentration of  $10^{10}$  plaque forming units (PFU)/mL. The sample will be serially diluted from  $10^{10}$  PFU/mL to  $10^6$  PFU/mL.

(2) The recommended concentration of each decontaminant will be added to its corresponding dilution tube. These tubes containing phage, decontaminant, and PBS will be incubated at room temperature [ $25^{\circ}\text{C}$  ( $77^{\circ}\text{F}$ )] for 1 hr. Following incubation, the phage will be diluted in fresh PBS 100 fold. **NOTE:** Diluting here is necessary; the test may not be as accurate for the first 100 PFU.

(3) Concentrations of viable phage will be measured by the standard plaque forming assay. Controls will consist of phage suspensions treated in an identical manner to the tubes containing decontaminant but supplemented with PBS rather than decontaminant.

4.5.5.3 Contact Time

a. Bacteria or phage will be suspended at a concentration of  $10^6$  CFU/mL or  $10^8$  PFU/mL, respectively.

b. Decontaminants will be added to these simulant suspensions at recommended concentrations.

c. Contaminant/decontaminant mixtures will be assayed as discussed in Section 4.5.5.2 at 15 min, 30 min, 1 hr, 8 hr, 16 hr, and 24 hr to determine the optimum contact time required for maximum effectiveness of the decontaminant.

d. The following data will be reported:

(1) Three logarithmic reduction or greater.

(2) The reduction between 2 and 3 logarithms.

(3) The reduction of less than 2 logarithms.

(4) Other logarithms reduction as required. The data should be normalized so as to compare day to day results, and the standard deviation should be reported.

4.5.6 Laboratory Test – Chemical Kinetics/Reaction Product Test

a. General

(1) This test determines the time it takes for a decontaminant to neutralize chemical agent in a reaction vessel.

(2) Quantities of decontaminant and chemical agent may be varied as directed by the test sponsor based on recommendations from the decontaminant manufacturer.

b. Freshly-prepared decontaminant (50 mL) will be placed into a stirred, jacketed reaction vessel maintained at  $25^{\circ}\text{C}$  ( $77^{\circ}\text{F}$ ). The stirrer will be started and the contents allowed to thermally equilibrate.

c. The reaction will be initiated by adding 1.00 mL of agent and the time will be noted ( $t=0$ ).

d. The stirring rate will be adjusted as necessary to ensure complete mixing and homogenous humidity.

e. At measured intervals (standard will be every 2 min for first 10 min, and every 5 min thereafter until a total of 1 hr elapsed time after agent addition) starting at  $t=2$  min, a 50  $\mu\text{L}$  sample will be collected for GC-AED or GC-MS analysis. The sample will be added to vials containing the quench solution and 2.00 mL of chloroform. This mixture will be vigorously agitated using a vortex mixer, and then the phases will be allowed to separate.

(1) For soman (GD) and distilled mustard (HD), the quench solution is 0.2 M sodium sulfite in water.

(2) For persistent nerve agent (VX), the quench solution is 0.2 M sodium sulfite and 0.2 M sodium carbonate. The sodium sulfite is present to destroy any residual oxidant while the sodium carbonate is present to make certain that the amine group on the VX is entirely in the free base form needed for complete extraction into the chloroform.

f. Using a micropipette, 1.0 mL of the chloroform layer will be transferred to an auto sampler vial.

g. The sample will be analyzed with GC-AED or GC-MS.

h. The following data will be reported:

(1) GC results, including amount of agent and reaction products

(2) Amount of agent remaining at each sample time, and the time required for agent to become undetectable (if it becomes undetectable).

(3) Observations during reaction. Observations should include visual inspection for HD droplets in the decontaminant.

(4) pH.

(5) Mass of agent applied and agent purity.

(6) TICN, SICN, and control results.

#### 4.5.7 Laboratory Test – Material Compatibility Test with Decontaminant

##### 4.5.7.1 General

Materials will be specified by the test sponsor in the ATP or DTP. Suggested materials are listed in Appendix C.

##### 4.5.7.2 pH Test

The pH of the decontamination solutions and a 10 percent aqueous solution for non-aqueous decontamination solutions will be measured. The pH meter will be calibrated according to instrument specification.

##### 4.5.7.3 Corrosivity

a. The objective of this test is to measure any corrosive effects of the decontamination solution on several types of metal. This test will be performed IAW References 7 and 8.

b. Strip coupons [35 x 50 x 0.05 mm (~1.4 x ~2 x ~0.002 in)] of various materials as listed in the DTP or ATP will be degreased by washing with soap followed by thorough rinsing in water and then acetone.

c. The surface area of the coupons will be calculated and the coupons weighed.

d. The coupons will be immersed in a wide mouth jar containing the decontamination solution. These jars, with suitable closures to prevent evaporation, will be conditioned at 30°C (86°F) for 24 hr.

e. After 24 hr the coupons will be removed from the decontamination solution, rinsed with water, and allowed to air dry.

f. The location of corrosion deposits, variations in types of deposits, or variations in corrosion products will be recorded. Any loose corrosion products will be removed from the coupons with a soft brush.

g. The coupons will be weighed.

#### 4.5.7.4 Sorption (Elastomer)/Hardness (Elastomer)

a. The objective of this test is to measure the weight and Shore A hardness changes of an elastomeric material after exposure to the decontamination solution.

b. The sorption test will be performed IAW References 8 and 9.

c. The Shore A hardness test will be performed IAW Reference 10.

d. Coupons will be cut in the form of bars 25 x 50 mm (1 x 2 in) by the thickness of the material.

e. The initial indentation hardness of each type of elastomer will be determined using a Shore Durometer. Each measurement consists of the average of measurements at each of three or five different points distributed over the specimen. These specimens will not be used for further testing.

f. The coupons will be weighed and immersed in closed jars of the decontamination solution and conditioned for 24 hr at 30°C (86°F). After this period, the samples will be removed, rinsed with water, wiped dry and allowed to air dry, weighed, and the final indentation hardness measured.

#### 4.5.7.5 Sorption (Thermoplastic)/Haze and Transmittance (Thermoplastic)

a. The objective of this test is to measure the weight and haze/transmittance changes of a thermoplastic after exposure to the decontamination solution. Haze is the percentage of transmitted light that, in passing through the sample, deviates (by forward scattering) from the incident beam by more than 2.5 degrees. Transmittance is defined as the ratio of transmitted to incident light.

b. The sorption test will be performed IAW References 8 and 11. The haze and transmittance test will be performed IAW Reference 12.

c. The original weight and haze/transmittance will be measured.

- d. The test item will be exposed to the decontamination solutions via filter-paper circles placed on the thermoplastic and then saturated with the decontamination solution.
- e. The thermoplastic will be covered with disposable plastic beakers during the test to minimize evaporation of the decontamination solution.
- f. The filter-paper circles will be resaturated with decontamination solution at regular intervals (approximately every 3 hr or as defined by the test sponsor).
- g. After 48 hr (or time specified by the test sponsor) of exposure to decontamination solution at ambient [ $\sim 25^{\circ}\text{C}$  ( $\sim 77^{\circ}\text{F}$ )] laboratory conditions, the thermoplastic will be removed, rinsed with water, wiped dry and allowed to air dry, and weighed.
- h. The haze and transmittance of the exposed areas of the thermoplastic will be measured.

#### 4.5.7.6 Hardness (Coating)

- a. The objective of this test is to measure the film hardness of a military coating (applied on a coupon) before and after exposure to a decontamination solution. This test will be performed IAW References 8 and 13.
- b. Coupons will be prepared and placed face up in Petri dishes and covered with the decontamination solution. Glass covers will be placed on the Petri dishes to prevent evaporation.
- c. The coupons will be conditioned at ambient [ $\sim 25^{\circ}\text{C}$  ( $\sim 77^{\circ}\text{F}$ )] laboratory conditions for 24 hr.
- d. The coupons will be removed, washed with water, wiped dry, and allowed to air dry.
- e. Film hardness rating will be determined on the exposed and unexposed coupons.

#### 4.5.7.7 Convective Flow For Air Permeable Materials

- a. The objective of this live-agent test is to measure the ability of an air-permeable material to resist convective penetration of a chemical agent after the material had been contaminated with a decontamination solution. This test will be performed IAW Reference 14.
- b. The decontamination solution will be checked before the test for interference (GC peak) and reaction of the decontamination solution vapor with agent.
- c. The testing conditions will be  $80\pm 3$  percent RH and  $32.2\pm 1.1^{\circ}\text{C}$  ( $90\pm 2^{\circ}\text{F}$ ).
- d. A  $10\text{ cm}^2$  sample swatch of the material will be contaminated with 2 mL of the decontamination solution and exposed to the decontamination for 1 hr.
- e. The sample swatch will be dried under test conditions for 30 min and then tested using a liquid challenge/vapor penetration (L/V) convective penetration method.
- f. The sample will be contaminated with  $10\text{ g/m}^2$  ( $10 \times 1\ \mu\text{l}$  drops on  $10\text{ cm}^2$  sample swatch) of agent, and then an air-flow (at a pressure of 0.1 inches of water) will be introduced through the material.
- g. Any chemical agent vapor breaking through the two layers of the material will be measured (via bubbler) over a 24-hr period.
- h. Bubbler samples will be taken after 6, 16, and 24 hr, or as specified by the test sponsor.

#### 4.5.7.8 Reported Data

The following data will be reported:

- a. pH results.
- b. Average corrosion rate (mils/year) and visual observations. **NOTE:** A mil is one-thousandth of an inch.
- c. Observations relating to the evolution of gases/corrosion products. If the gases/corrosion products are hazards, the necessary analysis and quantification of the products will be conducted.
- d. Elastomer sorption results: average percent weight change and visual observations.
- e. Elastomer hardness results: average percent change of indentation hardness and visual observations.
- f. Thermoplastic sorption results: percent weight change and visual observations.
- g. Thermoplastic haze and transmittance results: percent change haze and transmittance, and visual observations.
- h. Coating pencil test hardness results: average gouge measurements, average scratch measurements, and visual observations.
- i. Convective flow results: average weight of agent penetration per unit area of material measured over 24 hr.

#### 4.5.8 Laboratory Test – Detector Compatibility Test With Decontaminant

##### a. General

(1) All detectors will be checked for correct functioning before any other testing. Record results of function checks.

(2) Number of replicates conducted shall be sufficient to demonstrate high, statistical confidence in results or as specifically identified by the test sponsor.

(3) Detectors/alarms used shall be as specified by the test sponsor, but are expected to include the following:

- (a) Improved Chemical Agent Monitor (ICAM).
- (b) U.S. Marine Corp (USMC) Chemical Agent Monitor (CAM).
- (c) M43A1.
- (d) M90D1-C.
- (e) M18A2.
- (f) M256A1.
- (g) Shipboard Chemical Agent Monitor – Portable (SCAMP).
- (h) Improved Point Detection System (IPDS).
- (i) AP2C.



- (j) Lightweight Chemical Detector (LCD).
- (k) Joint Chemical Agent Detector (JCAD).
- (l) M8 and M9 detector paper and tape.
- (m) M272 Water Testing Kit
- (n) Chemical Biological Mass Spectrometer (CBMS).
- (o) Biological Aerosol Warning Sensor (BAWS).
- (p) Hand-Held Assay (HHA).
- (q) Automatic Chemical Agent Detector and Alarm (ACADA).

(4) Agent to decontaminant ratio: Optimize decontaminant to agent ratio and verify prior to test.

b. False Positive vs. Neat Decontaminant

(1) A false positive is defined as the detector alarming to the presence of the neat decontaminant with no agent present.

(2) Decontaminant prepared IAW manufacturers or test sponsor's instruction will be placed in a glass dish in a fume hood (face velocity 100 to 150 ft/min which equates to 1 to 2 mph wind speed).

(3) The inlet of the detector (itself or attached with a flexible Teflon<sup>®</sup> tube) will be placed approximately 2.5 cm (1 in) above the specimen surface to collect the sample vapor.

(4) The detector will be allowed to sample until the unit alarms or until the detector sample time is reached, or up to 5 min if there is no sample time. Precautions will be taken to prevent gross contamination of the detectors. Spectra or recordings may be taken when appropriate.

(5) All detectors responses, aforementioned data requirements, and any conditions or observations deemed potentially relevant to the experiments will also be noted in laboratory notebooks and discussed in the report of data.

(6) The appropriate function check will be conducted after this test is complete.

c. False Negative vs. Agent-Decontaminant Mixture

(1) A false negative is defined as the decontaminant masking the agent (not reacted) in such a way that the detector does not alarm to indicate agent present.

(2) Before testing begins, the functionality of the detectors will be verified.

(3) The decontaminant will be placed in a beaker.

(4) A syringe will measure the required amount of agent and transfer it onto the decontaminant in the beaker. The test will be performed with 10 mg each of VX, HD, and GD. Each agent will be tested separately.

(5) The decontaminant will be mixed thoroughly with the agent.

(6) The beaker will be covered with one-half of a Petri dish.

- (7) The agent-decontaminant mixture will weather for 10 min.
- (8) The Petri dish will be removed during sampling and replaced after each sample period.
- (9) Each detector will be placed to sample the air in the headspace of the beaker.
- (10) The detectors will sample the air based on the manufacturers recommended time and at the time intervals specified by the test sponsor.
- (11) The operator will observe for a response (or lack of response) and response time, while a second individual will annotate the observations. The detectors will be allowed to clear from the exposure (clear down) before simulant checks or new agent exposures are conducted.
- (12) The functionality of the detectors will again be verified.

#### 4.5.9 Laboratory Test – Individual Protective Equipment and Collective Protective Equipment Compatibility

##### a. General

(1) This test determines the effects of decontaminant exposure on individual and collective protection systems hardware. This test is designed to measure any degradation caused by the decontaminant on selective aspects of the system's performance from that of the baseline performance established in the Individual Protective Equipment (IPE) and Collective Protective Equipment (CPE) product performance specifications.

(2) The test may be required to be conducted at low [ $\sim 32^{\circ}\text{C}$  ( $\sim 25^{\circ}\text{F}$ )], ambient [ $21^{\circ}\text{C}$  ( $\sim 70^{\circ}\text{F}$ )], and high [ $\sim 49^{\circ}\text{C}$  ( $\sim 120^{\circ}\text{F}$ )] temperatures, and at low ( $< 20\%$ ) and high ( $> 90\%$ ) RH.

b. Using test fixtures and facilities specified by the test sponsor, the test item will be configured for exposure challenge with the test decontaminant.

c. Decontaminant will be applied to the test item in the amount, manner, and duration specified by the test sponsor.

d. The test item will be evaluated periodically (as specified by the test sponsor) during the conduct of the test and on the completion of the test for compliance with the sponsor's selected requirements from the product's performance specifications. Additional test item performance requirements may be evaluated as specified by the test sponsor. Selected test items may be set aside for evaluation of long term exposure effects.

e. Testing of the physical properties of the test item's materials of manufacture may be performed during the course of the testing and/or on completion of testing at the request of the test sponsor.

f. The following data will be reported:

- (1) Test conditions.
- (2) Type, quantity, and concentration of decontaminant applied.
- (3) Mode and location of decontaminant application.
- (4) Duration of decontaminant application.

(5) Observed test item physical effects, such as changes in consistency and color.

(6) Product performance data as required by the performance specification, or as requested by the test sponsor.

(7) Test item materials physical properties.

#### 4.5.10 Laboratory Test – Pot Life

##### a. General

(1) This test determines the useful life characteristics of the decontaminant under normal use conditions. This test is designed to measure any degradation in critical performance parameters from that of the product's baseline performance resulting from changes in the products physical state or chemical composition during a typical mission period. The test duration shall be 12 hr, or as otherwise specified by the test sponsor.

(2) The test shall be conducted at low [ $\sim 32^{\circ}\text{C}$  ( $\sim 25^{\circ}\text{F}$ )], ambient [ $21^{\circ}\text{C}$  ( $\sim 70^{\circ}\text{F}$ )], and high [ $\sim 49^{\circ}\text{C}$  ( $\sim 120^{\circ}\text{F}$ )] temperatures, and at low ( $< 20\%$ ) and high ( $> 90\%$ ) RH, or as otherwise specified by the test sponsor.

##### b. Procedure

(1) Duplicate samples of the decontaminant will be obtained in the original unopened containers. The decontaminant samples will be prepared IAW standard procedures for field use of the product.

(2) One of the decontaminant samples will be evaluated at the specified test conditions for the critical performance characteristics specified by the test sponsor.

(3) Performance evaluations of the decontaminant samples will be repeated at 4 hr, 8 hr, 10 hr, and 12 hr, or as otherwise specified by the test sponsor.

(4) The following data will be reported:

(a) Test conditions.

(b) Type, quantity, and concentration of decontaminant.

(c) Performance characteristic measurements.

(d) Estimated decontaminant pot life.

#### 4.5.11 Laboratory Test – Shelf Life/Accelerated Aging

##### a. General

(1) This test determines the storage/shelf life characteristics of the decontaminant under normal storage conditions by thermally inducing accelerated aging of the product. This test is designed to measure any degradation caused by the accelerated aging on selective aspects of the decontaminant's performance from that of the product's baseline performance established by Government or contractor test data or product specifications.

(2) Before the start of the accelerated aging test, product properties or performance characteristics to be used to evaluate the decontaminant will be identified. A quantitative

measure of the quality of the decontaminant is to be determined based on these characteristics with a nominal value of 100 when the product is initially produced. This quantitative measure will decrease as the product ages. Products with quantitative measures less than a predetermined threshold for the product shall be considered out of compliance.

b. Procedure

(1) Duplicate samples of the decontaminant in the original unopened containers will be obtained. One of the samples will be evaluated at a nominal ambient storage temperature of 25°C (77°F), for the characteristics specified by the test sponsor.

(2) Fifteen test samples of the decontaminant will be transferred into clean, non-reactive, thermally stable, hermetically sealable containers; the containers will be tightly closed. The original product containers may be used for this purpose, if deemed suitable. Each test sample container should contain sufficient product to perform all characterization testing prescribed herein.

(3) Five test samples will be placed in each of three test temperature environments selected on the basis of an evaluation of the products physical composition or as specified by the test sponsor. The selected environmental conditions should not, however, result in unrealistic failure modes that could never occur under real-time ambient conditions. Humidity conditions should be constant, at less than 20 percent RH, or as otherwise specified by the test sponsor.

(4) Once a month, over a period of 7 months, a product sample should be drawn from the test containers, and the quantitative measure of the decontaminant should be determined and recorded.

(5) The following data will be reported:

- (a) Test conditions.
- (b) Type, quantity, and concentration of decontaminant. For multi-component decontaminants, the corresponding values for all components should be recorded.
- (c) Performance characteristics used to calculate the products quantitative measure of quality.
- (d) Quantitative measurement values for each sample collected during the test period.
- (e) Estimated product shelf-life based on analysis of test data.

5. PRESENTATION OF DATA

a. The TD (or other responsible party) shall review the data for consistency and acceptability. Specifically, the following will be reviewed:

- (1) The results of each trial to determine if these are reasonable and consistent.
- (2) The precision obtained on the analytical controls.
- (3) The length of time for each trial.

(4) The printouts of the recorded results of the chemical concentrations, air temperatures, and pHs to ensure that the target chemical challenge concentration and air temperature were achieved and maintained during the test period.

(5) The interference control results, in order to determine if false positive values were obtained, and if so, whether the analytical procedure was changed to a method that will discriminate between chemical agent and interferent.

(6) Any notable observations made by the test operators.

b. Decontamination data will be analyzed IAW the experimental design specified in the SEP or the DTP or ATP.

c. Data to be included in the safety release recommendation will be collected. The safety release recommendation will be made to DTC in regard to test item's suitability for testing with troops.

d. The description of the test item, number of items tested, and conditions upon receipt will be presented in tabular form.

e. Photographs, drawings, or other methods that substantiate the conclusions, and recommendations shall be used to substantiate results included in the report.

f. Data from these subtests will be presented in narrative form and will clearly indicate whether an agent has an effect on the test item (its components, materials, chemicals, and/or systems) or vice versa.

g. The results of the operational check tests performed at the conclusion of the various environmental tests will be presented in narrative or other suitable form.

h. Data derived from these subtests will be presented in narrative form supplemented by drawings, photographs, charts, tables, graphs, or any other suitable means of displaying information. The report will clearly conclude whether the test item meets the criteria established in applicable specifications. Recommendations relative to further testing and methods to overcome malfunctions will also be included.

APPENDIX A. ABBREVIATIONS

ABO – agent of biological origin  
ACADA – Automatic Chemical Agent Detector and Alarm  
ACAMS – Automatic Continuous Air Monitoring System  
ACN – analytical control number  
AED – atomic emission detection  
AR – Army Regulation  
ASTM – American Society for Testing and Materials  
ATP – abbreviated test plan  
BAWS – Biological Aerosol Warning Sensor  
BG – *Bacillus subtilis* var. *niger*  
CAM – Chemical Agent Monitor  
CAR – corrective action report  
CARC – Chemical Agent Resistant Coating  
CASARM – Chemical Agent Standard Analytical Reference Materials  
CB – chemical and biological  
CBMS – Chemical Biological Mass Spectrometer  
CFU – colony forming units  
CoC – chain of custody  
CPE – Collective Protective Equipment  
DAAMS – Depot Area Air Monitoring System  
DEM – diethyl malonate  
DOT – Department of Transportation  
DPG – U.S. Army Dugway Proving Ground  
DTP – detailed test plan  
DTC – U.S. Army Developmental Test Command  
EA – environmental assessment  
EH – *Erwinia herbicola*  
EIALC – Environmental Impact Assessment for Life Cycle  
EIS – Environmental Impact Statement  
EPA – Environmental Protection Agency  
FID – flame ionization detection

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FM – Field Manual  
FPD – flame photometric detection  
GC – gas chromatograph  
GD – soman , non-persistent agent  
HD – distilled mustard  
HEPA – high-efficiency particulate air  
HHA – Hand-Held Assay  
IAW – in accordance with  
ICAM – Improved Chemical Agent Monitor  
IPDS – Improved Point Detection System  
IPE – Individual Protective Equipment  
IPT – Integrated Product Team  
IR – infrared  
JCAD – Joint Chemical Agent Detector  
JSFDS – Joint Service Family Decontamination System  
JSLIST – Joint Service Lightweight Integrated Suit Technology  
LCD – Lightweight Chemical Detector  
L/V – liquid challenge/vapor penetration  
MeS – methyl salicylate  
MIL-STD – military standard  
MINICAMS<sup>®</sup> – Miniature Automatic Continuous Air Monitoring System<sup>®</sup>  
MIRAN<sup>®</sup> – Miniature Infrared Analyzer<sup>®</sup>  
MS – mass spectrometry  
MS2 – male-specific bacteriophage type 2  
MSDS – material safety data sheet  
NBC – nuclear, biological, and chemical  
NEPA – National Environmental Policy Act  
NMR – nuclear magnetic resonance  
ORD – Operational Requirements Document  
ORI – operational readiness inspection  
PBS – phosphate buffer saline  
PEG 200 – polyethylene glycol 200

PFU – plaque forming units  
QA – quality assurance  
QC – quality control  
REC – record of environmental consideration  
RDT&E – research, development, test, and evaluation  
RH – relative humidity  
RTM – Real-Time Monitor  
SAR – Safety Assessment Report  
SCAMP – Shipboard Chemical Agent Monitor – Portable  
SCG – Security Classification Guide  
SEP – System Evaluation Plan  
SICN – sample item control number  
SOP – Standing Operation Procedure  
SSP – System Support Package  
TCN – trial by trial control number  
TD – test director  
TEP – triethyl phosphate  
TIC – toxic industrial chemical  
TICN – test item control number  
TIR – test incident report  
TIWG – test integration working group  
TOP – Test Operations Procedure  
TRR – test readiness review  
USMC – U.S. Marine Corp  
VX – methylphosphonothioic acid, persistent nerve agent



APPENDIX B. DECONTAMINATION TESTS

Table B.1. Developmental Tests, Facilities, Instrumentation/Equipment and Accuracy, and Specific Standardized Decontamination Tests Not Otherwise Described in This TOP.

| Developmental Test   |  | Test Method or Procedure        |
|--|--|---------------------------------|
| <b>Effectiveness</b>   |  |                                 |
| Chemical Agents/<br>TICs <sup>a</sup>                        | Contact Hazard                         | Paragraph 4.5.2                 |
|  | Off-gassing                            | Paragraph 4.5.3                 |
|  | Decontamination efficacy (chemical)    | Paragraph 4.5.4                 |
|  | Decontamination efficacy (biological)  | Paragraph 4.5.5                 |
|  | Chemical kinetics/Reaction Product     | Paragraph 4.5.6                 |
| Material compatibility with decontaminant                    |  | Paragraph 4.5.7                 |
| Detector compatibility with decontaminant                    |  | Paragraph 4.5.8                 |
| Individual and Collective Protective Equipment Compatibility |  | Paragraph 4.5.9                 |
| Pot Life   |  | Paragraph 4.5.10                |
| Shelf Life/Accelerated Aging                                 |  | Paragraph 4.5.11                |
| <b>Compatibility</b>   |  |                                 |
| Materials  | Corrosivity                            | ASTM <sup>b</sup> G31-72 (1995) |
|  | Sorption (Elastomers)                  | ASTM D471-98                    |
|  | Hardness (Elastomers)                  | ASTM 2240-97                    |
|  | Sorption (Thermoplastic)               | ASTM D543-95                    |
|  | Haze and Transmittance (Thermoplastic) | ASTM D1003-97                   |
|  | Hardness (Coating)                     | ASTM D3363-92a                  |
|  | Visual Observation                     | ASTM F502-93                    |
|  | Convective Flow                        | TOP <sup>c</sup> 8-2-501        |
| <b>Chemical/Physical Properties</b>                          |  |                                 |
| pH   | pH meter                               |                                 |
| Freeze Point   | ASTM D1016-94                          |                                 |
| Freeze-Thaw Stability  | ASTM D2337-84 (1996) e1                |                                 |
| Flash Point  | ASTM D93                               |                                 |
| Viscosity versus Temperature                                 | ASTM D445                              |                                 |
| Specific Gravity   | ASTM D 891                             |                                 |
| Solubility Tests   | ASTM D2030-97                          |                                 |
| Agent Adsorbent Capacity of the Decontaminant (Adsorbent)    | ASTM F-726-99                          |                                 |

Table B.1. Developmental Tests, Facilities, Instrumentation/Equipment and Accuracy, and Specific Standardized Decontamination Tests Not Otherwise Described in This TOP (Cont'd).

| Developmental Test  |                                 | Test Method or Procedure         |
|---|---------------------------------|----------------------------------|
| <b>Toxicity</b>   |                                 |                                  |
| Rat Oral LD <sub>50</sub>   |                                 | EPA <sup>d</sup> 870.1100        |
| Rabbit Eye Irritancy  |                                 | EPA 870.2400                     |
| Rabbit Dermal Irritancy   |                                 | EPA 870.2500                     |
| DOT <sup>e</sup> Test Battery                                       | Rat Inhalation                  | 49 CFR <sup>f</sup>              |
|   | Rat Oral                        | 49 CFR                           |
|   | Rabbit Dermal                   | 49 CFR                           |
|   | Rabbit Dermal LD <sub>50</sub>  | EPA 870.1200                     |
| Aquatic Toxicity Testing  | Microtox                        | ASTM D-5660                      |
|   | Daphnia magna Assay (Acute)     | EPA 540/P-91/009                 |
|   | Ceriodaphnia Assay (Acute)      | EPA 600/4-91/002                 |
|   | Fathead Minnow Assay (Acute)    | EPA 540/P-91/009                 |
|   | Sheepshead Minnow Assay (Acute) | EPA 600/4-85/013                 |
|   | Ceriodaphnia Assay (Chronic)    | EPA 600/4-91/002                 |
|   | Fathead Minnow Assay (Chronic)  | EPA 600/4-91/002                 |
|   | Mysid Shrimp Assay (Chronic)    | EPA 600/4-91/003                 |
|   | Fate and Persistence in Water   |                                  |
| Terrestrial Toxicity Testing  | Ames Assay                      | EPA 712-C-96-219                 |
|   | Earthworm Assay                 | EPA 560/76-82-002                |
|   | Pot Worm Test                   | ISO/CD 16387:2001                |
|   | Early Seedling Growth Test      | EPA 712-C-96-347                 |
|   | Foliar Exposure Test            | EPA 712-C-96-347                 |
|   | Fate and Persistence in Soil    |                                  |
| <b>Worldwide Operational Environmental Conditions</b>               |                                 |                                  |
| NBC <sup>g</sup> Survivability - Container                          | Decontaminability               | TOP 8-2-111 or 8-2-510           |
|   | Hardness                        |                                  |
|   | Compatibility                   |                                  |
| <b>Packaging, Handling, Storage, Shipping, and Transportability</b> |                                 |                                  |
| Low Pressure (Altitude)   |                                 | MIL-STD <sup>h</sup> 810F, 500.4 |
| High Temperature  |                                 | MIL-STD 810F, 501.4              |
| Low Temperature   |                                 | MIL-STD 810F, 502.4              |
| Temperature Shock   |                                 | MIL-STD 810F, 503.4              |
| High Humidity   |                                 | MIL-STD 810F, 507.4              |
| Low Humidity  |                                 | MIL-STD 810F, 507.4              |
| Sand and Dust   |                                 | MIL-STD 810F, 510.4              |

Table B.1. Developmental Tests, Facilities, Instrumentation/Equipment and Accuracy, and Specific Standardized Decontamination Tests Not Otherwise Described in This TOP (Cont'd).

| Developmental Test  |  | Test Method or Procedure                       |
|---|--|--|
| Packaging, Handling, Storage, Shipping, and Transportability (Cont'd) |  |  |
| Fungus  |  | MIL-STD 810F, 508.5                            |
| Salt Fog  |  | MIL-STD 810F, 509.4                            |
| Explosive Atmosphere  |  | MIL-STD 810F, 511.4                            |
| Solar Radiation   |  | MIL-STD 810F, 505.4                            |
| Air Transport   | Interface Standard for Transportability Criteria                 | MIL-STD-1366D                                  |
|   | Helicopter and Plane Drops Along with Shipboard Drops from Docks | TOP 4-2-601 and 602                            |
| Airborne Delivery   | Low Velocity Air Drop  | MIL-STD-814                                    |
|   | Interface Standard for Transportability Criteria                 | MIL-STD-1366D                                  |
|   | Helicopter Flight  | MIL-STD-209J and MIL-STD-913                   |
| Highway Transport   | Transportation Vibration System                                  | MIL-STD 810F, 514.5, Procedure I               |
|   | Loose Cargo Bounce System  | MIL-STD 810F, 514.5, Procedure II              |
| Rail Transport: Rail Impact   |  | MIL-STD 810F, 516.5, Para 4.5.8, Procedure VII |
| Ship Transport  | Lifting and Tie-down Provisions                                  | MIL-STD-209J                                   |
|   | Mechanical Vibrations of Shipboard Equipment                     | MIL-STD-1657-1 and 2                           |
|   | Helicopter and Plane Drops Along with Shipboard Drops from Docks | TOP 4-2-601 and 602                            |
| Other Considerations  |  |  |
| Human Factors   |  | MIL-STD 1472F                                  |
| Safety  |  | AR <sup>g</sup> 385-16                         |

<sup>a</sup>Toxic Industrial Chemicals.

<sup>b</sup>American Society for Testing and Materials.

<sup>c</sup>Test operations procedure.

<sup>d</sup>Environmental Protection Agency.

<sup>e</sup>Department of Transportation.

<sup>f</sup>Code of Federal Regulations.

<sup>g</sup>Nuclear, biological, and chemical.

<sup>h</sup>Military standard.

<sup>i</sup>Army Regulation.

APPENDIX C. COUPON MATERIAL AND CONTAMINANTS

A. Coupon Material

Coupons [representing a range of surfaces inherent to the items undergoing immediate decontamination operations (vehicles, vans, weapons, etc.)] will be prepared of materials specified by the test sponsor. There are a wide variety of items comprised of hundreds of potential materials that could be encountered. To reduce this list to a manageable number and thus to facilitate a cost-effective test and evaluation program for a decontaminant, a listing of assumed highest priority materials was developed by materiel developers based on the expected impact on mission/combat readiness, likelihood of being exposed to contamination, and cost to replace. In addition to the aforementioned prioritization that is focused on restoration of combat readiness and mission execution, compatibility with individual protective equipment is also proposed to be included among high priority materials to be evaluated. Consequently, materials from aircraft, tactical and support vehicles, munitions, runways/flight decks, and individual protective equipment were deemed of primary importance and thus included in this listing:

(1) List A

- (a) Chemical agent resistant coating (CARC) (tactical vehicles).
- (b) Aircraft topcoat paint (aircraft).
- (c) Low-infrared (IR) paints (aircraft & ships).
- (d) Ship deck anti-skid.
- (e) Polyurethane, epoxy, and alkyd paints (commercial vehicles).
- (f) Aluminum alloys, forged and cast (aircraft surfaces & structural members).
- (g) Aluminum, oxidized aluminum (vehicle substrate surface).
- (h) Stainless and high strength steel alloys (aircraft and engine structural members).
- (i) Nickel based and other superalloys (aircraft and engine structural members).
- (j) Carbon/stainless steels (vehicle, munitions substrate surface).
- (k) Brass/bronze/copper and nickel alloys (munitions substrate surface).
- (l) Composite and laminate materials (aircraft surface and structural members).
- (m) Aircraft composites (aircraft).
- (n) Tire rubber (aircraft, vehicles).
- (o) Polycarbonates/Lexan<sup>®</sup> (aircraft canopy/window materials, tactical vehicles).
- (p) Glass (commercial vehicles, tactical vehicles).
- (q) Asphalt (runways and parking areas).
- (r) Concrete (runways and parking areas).
- (s) Standard tent, soft shelter material.

(2) List B

- (a) Joint Service Lightweight Integrated Suit Technology (JSLIST).
- (b) Battle dress overgarment.
- (c) Butyl rubber (mask, gloves/boots).
- (d) Silicon rubber (M40 mask).
- (e) Cotton, polyester materials (uniform materials).
- (f) Collective protection, soft shelter material.

(3) It is recommended that the A List materials be evaluated in terms of both decontamination effectiveness (effectiveness of a candidate decontaminant to decontaminate threat agents from a respective material) and materials compatibility (to determine the effects of the decontaminant on the surface being decontaminated).

(4) It is recommended that the B List materials be evaluated in terms of material compatibility and material performance only. Further, it is also recommended that the above list be recognized as a general guide to be used in the development of detailed test plans, and that the above list may be modified as deemed necessary to provide materials that are most generally representative of the categories of equipment mentioned within that list.

**B. Contaminants**

Contaminants will be specified by the test sponsor. Likely contaminants include:

- (1) Chemical agents.
  - (a) G agents.
  - (b) VX agents.
  - (c) H agents.
  - (d) Thickened agents.
- (2) Toxic industrial chemicals (TICs).
  - (a) n-Butyl isocyanate.
  - (b) Parathion.
  - (c) Sulfuric acid.
- (3) Chemical agent simulants
  - (a) Diethyl malonate (DEM).
  - (b) Methyl salicylate (MeS).
  - (c) Polyethylene glycol 200 (PEG 200).
  - (d) Triethyl phosphate (TEP).

APPENDIX D. REFERENCES

Required References

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Forward comments, recommended changes, or any pertinent data that may be of use in improving this publication to U.S. Army Developmental Test Command, (CSTE-DTC-TT-M), 314, Longs Corner Road, Aberdeen Proving Ground, MD 21005-5055. Technical information may be obtained from the preparing activity: West Desert Test Center (CSTE-DTC-DP-WD-C-C) U.S. Army Dugway Proving Ground, Dugway, UT 84022-5000. Additional copies are available from the Defense Technical Information Center, Cameron Station, Alexandria, VA 22304-6145. This document is identified by the accession number (AD No.) printed on the first page.