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This test operations procedure (TOP) describes the test procedures used to characterize and determine the technical performance of a decontaminant. Decontamination is the process of reducing or eliminating the hazards associated with chemical, biological, or radiological contamination in order to accomplish assigned missions. This TOP addresses test methods for decontaminants of chemical contaminants only. Means of decontaminating personnel, equipment, or areas include neutralization, weathering, and physical removal of the chemical contaminant. Chemical contaminants may include chemical warfare agents (CWAs), advanced threat agents, toxic industrial chemicals (TICs) and toxic industrial materials (TIMs). Many of the test methods in this TOP are conducted with only the decontaminant being tested. Some test methods for efficacy require the use of CWAs and decontaminants.

#### 15. SUBJECT TERMS

decontamination; chemical warfare agent; CWA; decontaminant

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## U.S. ARMY TEST AND EVALUATION COMMAND TEST OPERATIONS PROCEDURE

\*Test Operations Procedure (TOP) 08-2-061A DTIC AD No.

20 October 2014

#### CHEMICAL DECONTAMINANT TESTING

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\*This TOP supersedes TOP 08-2-061 Chemical and Biological Decontaminant Testing, dated 19 November 2002.

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#### 1. SCOPE.

#### 1.1 Purpose.

- a. This test operations procedure (TOP) describes the test procedures used to characterize and determine the technical performance of a decontaminant.
- b. Decontamination is the process of reducing or eliminating the hazards associated with chemical, biological, or radiological contamination in order to accomplish assigned missions. This TOP addresses test methods for assessing chemical decontaminant efficacy and the material effects of decontaminants on fielded military equipment (material effects are defined as hardness). Mechanisms of decontaminating equipment or areas include neutralization, weathering, and physical removal of the chemical contaminant. Chemical contaminants may include chemical warfare agents (CWAs), advanced threat agents, toxic industrial chemicals (TICs) and toxic industrial materials (TIMs).
- c. Many of the test methods in this TOP use only the chemical decontaminant. Some test methods in this TOP require the use of CWAs and decontaminants (i.e., methods for testing efficacy and reaction kinetics).

#### 1.2 Application.

This TOP provides the current standard for the planning and conduct of general performance tests of chemical decontaminants. The test procedures described herein will be used as the basis of a test plan. The procedures may require modification for unique items or materials or to satisfy specific testing requirements as specified in the Capability Development Document (CDD). However, alteration of procedures in this TOP will be made only after full consideration of any possible effects the alterations may have upon the reliability and validity of the data to be obtained. Such alterations will be coordinated among all concerned organizations in advance of any testing. Any deviations from this TOP will be documented in the test plan and the test report.

#### 1.3 Limitations.

a. This TOP does not include procedures intended for skin or personnel decontamination testing.

- b. Residual vapor test methods (included in previous versions of this TOP) have been removed and placed in TOP  $08-2-060^{1*}$ .
- c. Biological decontaminant test methods (included in previous versions of this TOP) have been removed and placed in TOP 08-2-065<sup>2</sup>.
- d. This TOP is not intended to develop procedures for decontaminating military equipment.
- e. This TOP does not include procedures for testing decontaminant applicators. **NOTE**: When a decontaminant is part of a decontamination system, the integrated applicator may be required or used in applying the decontaminant to test articles.

#### 2. <u>FACILITIES AND INSTRUMENTATION</u>.

Facilities and instrumentation used for testing CWA decontaminants 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 Facility.

Item	Requirement

Chemical agent facility. Must be designed and constructed to ensure safe and

secure storage, handling, analysis, and decontamination of chemical agents used for

Research, Development, Test, and Evaluation (RDT&E). Facility must be equipped and certified for work with chemical agents. The chemical agent

laboratory, instruments, and personnel assignments must meet all requirements of Army Regulation (AR) 50-6<sup>3</sup> and AR 190-59<sup>4</sup> and the safety requirements of Department of the Army (DA)

Pamphlet (PAM) 385-61<sup>5</sup> and Army Material Command Regulation (AMCR) 385-10<sup>6</sup>.

Chemical agent test chamber.

Must be fabricated with appropriate construction materials (e.g., acrylic, stainless steel, glass, etc.) for containing the chemical agents, coupons, coupon holding fixture, and decontaminant(s). The chamber must include doors with seals for ingress and egress

of chemical agents, decontaminants, and

applicator(s). The chamber may include glove ports.

<sup>\*</sup>Superscript numbers correspond to Appendix C, References.

<u>Item</u>	Requirement
Chemical agent test laboratory.	Equipment, interior surfaces, tools, and waste must be easily decontaminable. All exhaust air from testing must be filtered and monitored to prevent any agent release to the environment. The facility design should ensure safe transfer, handling, challenge, and disposal of chemical agents, decontaminating solutions, and solvents.
Chemical agent test fixture.	Test fixture must be fabricated to contain the contaminants, test articles, and decontaminants. The fixture must be constructed to allow contamination, decontamination, and handling of test articles deliberately contaminated with chemical agent in a temperature- and humidity-controlled environment. The test fixture must include controlled doors with seals for safe ingress and egress of all test materials and equipment. The fixture will include appropriate glove ports for conducting test operations.
Outdoor decontamination facility.	Must be designed for containment of test effluents and provide power and water for multiple system inlet needs. The facility must have sufficient environmental permits for use of multiple simulants and decontaminants.

### 2.2 <u>Instrumentation</u>.

The following instruments or their equivalents will be used. Instrumentation unique to a test will be listed in the test plan.

<u>Parameter</u>	Measuring Device	Permissible Error of Measurement
Contamination density (dose confirmation sample).	Mass spectrometer (MS), gas chromatograph (GC) or liquid chromatograph (LC), flame ionization detector (FID), flame photometric detector (FPD), or equivalents. Gravimetric techniques using a balance.	±15 percent of the mass of the contaminant per sample or within ±25 percent of the device minimum quantification limit (MQL).
Contamination droplet size.	Calibrated repetitive pipette, syringe, or computerized dispensing system.	±10 percent of the droplet size (within the range specified for the contaminant and applicator).

<u>Parameter</u>	Measuring Device		Permissible Error of Measurement
Residual contaminant in samples from contact samplers, coupons, rinsate, or other samples.	equivalents.		±15 percent of the mass of the contaminant per sample or within ±25 percent of the device MQL.
Temperature.	Thermocouple, remote temperature device, thermometer, or equivalent.		±2°C.
Relative humidity (RH).	Hygrometer, equivalent.	humidity meter, or	±5 percent RH.  NOTE: This means that for an RH of 50 percent, 45 to 55 percent would be acceptable.
Absolute humidity (AH).	•	er, hygrometer, eter, or equivalent.	$\pm 0.1 \text{ g/m}^3$
Differential pressure $(\Delta P)$ for test chambers or fixtures only.	Pressure transducer.		±0.09 mm mercury (Hg), or ±12.5 Pascal [(±0.05 inches water gauge (iwg)].
Wind speed (outside) or airflow (chamber or fixture).	Hotwire anemometer or equivalent.		±0.1 m/sec.
Visual record (still).	Digital color	camera.	Image resolution and frame capture rate adequate to document details of testing.
Visual record (in motion).	Digital video	o camera.	Resolution adequate to document details of testing.
2.3 <u>Test Controls</u> .			
Parameter		(unle	<u>Tolerance</u> ss otherwise specified)
Positive control (contaminated but not decontaminated residual liquid) using coupons.		Concentration, in mass/area, $\pm 15$ percent, or at the MQL $\pm 25$ percent. Contaminant per sample, in mass/volume, $\pm 15$ percent, or at the MQL $\pm 25$ percent.	
Negative control (not contaminated, but decontaminated residual liquid) using coupons.		Concentration, in mass/area, $\pm 15$ percent, or at the MQL $\pm 25$ percent. Contaminant per sample, in mass/volume, $\pm 15$ percent, or at the MQL $\pm 25$ percent.	

#### Tolerance

#### (unless otherwise specified)

Dose confirmation samples. May be taken before, during, and after test article contamination.

Parameter

Contaminant per sample, in mass/area,  $\pm 15$  percent, or at the MQL  $\pm 25$  percent.

Process quality samples for GC, LC, or equivalent. These may be samples of a known mass or periodic calibration standards.

Contaminant per sample, in mass,  $\pm 15$  percent, or at the MQL  $\pm 25$  percent.

#### 3. REQUIRED TEST CONDITIONS.

#### 3.1 Familiarization.

The test planning phase includes identifying potential problem areas by reviewing previous records and the results of similar tests. Review and consider data from previous similar tests to avoid duplication of testing. This review may possibly reduce the scope of the current test effort. Relevant laboratory and method-specific standing operating procedures (SOPs) and other procedures should be reviewed for applicability, completeness, and adequacy.

#### 3.2 Test Planning.

- a. Based on the testing requirements in the test planning documentation (Test and Evaluation Master Plan [TEMP], SEP, etc.), a test plan will be developed that will include, at a minimum, a test design, test execution matrix, detailed procedures, quality assurance/quality control (QA/QC) measures, data management, statistical data analysis, and results presentation.
- b. The test plan must be prepared, coordinated with all relevant stakeholders, and approved by the test site before any testing begins. The test procedures described herein must be used as the basis for the test plan; however, the procedures may require modification for unique items or materials to satisfy specific testing requirements in a TEMP, SEP, or other program-specific documentation. Deviations from these procedures will be coordinated among all concerned organizations in advance of any testing, giving consideration to the possible effects the changes may have upon the validity and adequacy of the data. Any deviations from this TOP and the rationale for the deviation will be described in the test plan.
- c. Test Design. The sample size of test articles for test methods identified in this TOP may be determined based on DOE design, confidence required by the customer, test article size, availability, cost, or other factors. The recommended number of replicates is a minimum of five coupons per test condition<sup>7</sup> or the number from the design of experiment (DOE) whichever is greater. The minimum number of replicates for the dose confirmation samples per contamination set will be five. If the sample size is less than recommended, a test execution matrix will be devised to maximize the ability to meet stated objectives and criteria. Statistical confidence limits will be calculated and reported.

- d. When using a chemical agent simulant in the conduct of this TOP, the selected properties of the simulant will be verified as being as closely related to those of the contaminant as possible. Because simulants do not have all of the same physical and chemical properties as the agent, simulant data alone are not sufficient to determine decontaminant performance. An agent-simulant relationship (ASR) must be established and coordinated with the test program community of interest before testing begins.
- e. Security. Security considerations will be adequately determined and provided for in the planning of each test program. The security classification guide (SCG) and the installation operations security (OPSEC) requirements will be followed.
- f. Test Incident Report (TIR). Unless waived by the test sponsor, TIRs (or equivalent reports), will be prepared and distributed in accordance with (IAW) United States (U.S.) Army Test and Evaluation Command (ATEC) Regulation 73-1<sup>8</sup> and DA PAM 73-1, Appendix V<sup>9</sup>.

#### 3.3 Documentation.

The test officer (TO) or principal investigator will have all pertinent documentation available for test planning. These documents may include government and manufacturers' publications, requirements documentation, test planning directive, TOPs, SOPs, safety data sheets (SDSs), approved test plan, SCG, etc., as applicable/required.

#### 3.4 Environmental Documentation.

The test plan must cite the approved environmental documentation for each test program.

#### 3.5 Test Readiness Review/Operational Readiness Inspection.

- a. 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 organizations involved in the test program [which may include Warfighters, program office representatives, Test and Evaluation Integrated Product Team (T&E IPT) representatives, operational test agency (OTA) representatives, and contractor(s)] will participate in this review and provide input to the proposed testing. The designated TO or TO's delegate will conduct this review and present the status of all critical elements.
- b. An operational readiness inspection (ORI) may be required by the performing organization's internal procedures to ensure readiness to begin testing.

#### 3.6 Safety.

- a. It is the responsibility of the user of the TOP to establish appropriate health and safety practices for the execution of procedures in this TOP and handling of generated wastes.
  - b. The primary emphasis in testing using toxic contaminants must be placed on safety.
- c. A composite risk management or hazard analysis may be required by the testing organization.

- d. A preoperational safety survey/inspection may be required before testing can begin.
- e. The SDS(s) for the decontaminant(s) and contaminant(s) will be reviewed and maintained in the laboratory or chamber during testing.

#### 3.7 QA/QC.

- a. A chain-of-custody (CoC) process will be established before testing by labeling all test articles and all test samples to allow tracking of the data flow from test initiation to final data and to prevent misidentification during the test process.
- b. The test control samples will be used to demonstrate control of the test process across trials and throughout the analytical process.
- c. The chemical analysis procedures will be conducted using best laboratory practices [e.g., practices in International Organization for Standardization (ISO) 17025<sup>10</sup>] for standards, blanks, and analytical controls.
- d. The samplers selected for use must be well-characterized. For example, the collection efficiency at the test temperature and test humidity must be known. When using solid sorbent tubes (SSTs), collection efficiency at the expected range of temperatures and humidities must be determined and it must be verified that the capacity of the sorbent is not exceeded by the breakthrough vapor, and the SSTs are clean before their next use. When using solvents for collection or extraction, the stability of the agent in the solvent will be documented. If the solvent is used as an extractant, the extraction efficiency will be documented. Methods for determining collection efficiency, sorbent breakthrough and solvent efficiency are found in Procedure 6-B<sup>11</sup>.
- e. Chemical agent with greater than 90 percent purity is acceptable for use. The purity of the agent must be analytically demonstrated at a frequency determined by the testing organization or based on experience with the agent used. Purity analysis must have been conducted within 12 months of the test [except for persistent nerve agent (VX), which must be purity-analyzed within 6 months].
- f. Samples collected during the test that cannot be analyzed immediately (i.e., within 1 calendar day) will be stored with storage control samples. These storage control samples will be created and placed into storage along with the test samples waiting analysis. The storage control samples will be analyzed along with the test samples to determine whether any test sample degradation occurred during storage.
- g. All aspects of the testing will be performed with emphasis on acquiring valid, credible, repeatable, and verifiable data.

#### 3.8 <u>Verification and Validation (V&V)</u>.

The test procedures used must be verified and validated. Modifications to the procedures or test equipment (fixtures, instrumentation, etc.) must be reviewed for impact on the data collected during testing. Modifications that have a significant impact on the data will need to be verified

and/or validated (e.g., changing the brand of mass flow controller may have no impact on data collected, but changing the humidification system will require verification testing at least). Fixture modifications must be documented in the configuration control plan. Procedure modifications must be documented as soon as possible in SOPs or other controlling documents. ISO 5725<sup>12</sup> provides methods for verification and validation of test methods. Modifications may be necessary because of technical upgrades at the test site, test system requirements, or requirements of the test sponsor.

#### 4. TEST PROCEDURES.

#### 4.1 General.

- a. Two types of test controls, the positive control and the negative control, will need to be used during trials. The number of controls used will be outlined in the test planning documentation.
- (1) Positive control. Positive controls use the same type of coupon challenged with the same contaminant and density as the trial coupons. The same test procedures are used; however, positive control coupons do not undergo decontamination. **NOTE**: Test personnel should not assume that positive controls will always have measurable contaminant. For example, it is possible that contaminant may be lost as a result of weathering or other evaporation, even though no decontaminant is applied.
- (2) Negative control. For negative controls no contaminant is applied to the coupons which undergo all other test procedures with the trial coupons. Positive analytical results for contamination on a negative control are an indication of cross contamination most likely showing poor test process control.

#### 4.2 <u>Test Method Outline</u>.

- a. Receipt inspection (Paragraph 4.5).
- b. Trial Preparation Tasks (Paragraph 4.6).
- c. Kinetics (Paragraph 4.7).
- d. Byproducts (Paragraph 4.8).
- e. Coupon Efficacy (Paragraph 4.9).
- f. Material Compatibility (Paragraph 4.10).
- g. Detector Compatibility (Paragraph 4.11).
- h. Pot Life (Paragraph 4.12).
- i. Shelf Life/Accelerated Aging (Paragraph 4.13).

#### 4.3 Hazards.

- a. Identified safety hazards are those associated with testing using toxic chemical surety materials, simulants, and hazardous decontaminant chemicals (e.g., chlorine, hydrogen peroxide, etc.). Chemical safety guidelines are found in DA PAM 385-61<sup>5</sup>.
- b. A test plan must be developed with a safety section (which may include a composite risk management) identifying and addressing all safety concerns for each test conducted using these methods IAW AR 385-10<sup>13</sup>. The safety section of the test plan will be coordinated with the test site's safety office.

#### 4.4 Calibrations and Standards.

- a. General chemical analytical calibration guidelines are found in best laboratory practices (e.g., practices in ISO 17025<sup>10</sup>). These guidelines can be used for calibrating most chemical analytical equipment (e.g., GCs, LCs, etc.) and must be used whenever possible. For each test, a sample sequence will be created that includes the following:
  - (1) A solvent blank to evaluate method interferences.
- (2) At least five calibration standards (ranked low to high or high to low). Preparation of standards must follow test site operating procedures.
  - (3) A second solvent blank to evaluate carryover.
- (4) At least one QC sample per detector to validate the calibration curve, including control samples.
  - (5) A third solvent blank.
  - b. The same method will be used to analyze all samples.
- c. Using the instrument software (where available), the calibration curve will be built from lowest to highest standard concentration.
  - d. Plot information will be evaluated as follows:
    - (1) Curve fit type (linear, quadratic, etc.) will be selected.
    - (2) Point weighting (equal, inverse, etc.) will be selected.
- (3) If the correlation value (R<sup>2</sup>) is greater than 0.995 and the calculation of the relative percent deviation (RPD) meets the standards<sup>7</sup>, then analysis will proceed.
- (4) If the R2 is less than 0.995, or the RPD for any standard is >15%, then one data point with the largest RPD can be removed and the calibration curve recalculated. This is optional.

- (a) If the  $R^2$  is still less than 0.995, each data point will be evaluated to determine any errors.
  - (b) Method adjustments will be made and the calibration repeated.
  - (5) If correlation fails, help will be requested from within the organization.
- e. If all criteria are met, the QC sample will be loaded and processed in comparison with the calibration curve. The GC response will be used to calculate a concentration value for the QC sample.
- f. The calculated value for the QC sample must be within  $\pm 15$  percent of the expected value.
- g. If the QC calculated value is within the tolerance range, then the test method will proceed.
- h. If the QC calculated value is outside of the tolerance range, then a second QC sample will be processed.
- (1) If the second QC calculated value is within the tolerance range, then the test method will proceed.
- (2) If the second QC calculated value is outside of the tolerance range, then corrective actions and recalibration will be performed to the instrument.
- i. After any maintenance action to the instrument, two QC samples must have calculated values within the  $\pm 15$  percent tolerance range or corrective actions and recalibration must be performed.

#### 4.5 Receipt Inspection.

- a. The test articles (which may include coupons, panels, or small items of equipment) will be subjected to a visual receipt inspection after arrival at the test site. Evidence of damage or irregularities in the test articles will be recorded in the laboratory record keeping system and will be documented by still photographs. Damage and irregularities to be considered will include, but are not limited to, the following (if applicable):
  - (1) Corrosion.
  - (2) Broken connections.
  - (3) Cracked or deteriorated surfaces.
  - (4) Contamination with foreign material.
  - (5) Discoloration.
  - (6) Evidence of deterioration or illegible markings.

- (7) Incorrect number of items.
- (8) Missing components, instructions, or manuals.
- (9) Determine if the test article can be operated; and/or is operational.
- b. Each test article's model, serial number, nomenclature, identifier, manufacturer, lot number, and other pertinent information/indicators, if applicable, will be recorded in the laboratory recordkeeping system. Assignment of a test item control number (TICN) to the test article is mandatory for future identification and tracking. The TICN will be marked on small items of equipment in a location that will not interfere with test procedures. Coupons/panels will be labeled on the reverse side with their TICN. The TICN and other pertinent information about the test article will be linked in the laboratory recordkeeping system.
- c. If any items are determined to be not fit for testing, they will be rejected and replaced with items that are in suitable condition for testing.

#### 4.6 Test Preparation Tasks.

- a. Test personnel will ensure that all necessary equipment, materials, reagents, analytical capabilities, and necessary certified/qualified personnel are available for the test.
- b. Any data analysis calculations required to ensure the necessary data are collected will be identified.
- c. A certification of purity must be supplied when chemical contaminants are used. Chemical Agent Standard Analytical Reference Material (CASARM) agent will be used unless weapons-grade agent is required by the test program. A new purity certificate will be required if the CWA vial was opened up to 12 months before testing begins [except for persistent nerve agent (VX), which must be purity-analyzed within 6 months]. Purity certification will use one of the following methods: freezing point depression, nuclear magnetic resonance (NMR), or GC analysis documented for each lot. AT or other chemical contaminants must also have a purity certificate documented in the test report.
- d. Chemicals used for preparation of decontaminant formulations will be used asreceived. Purity will be established based on supplied purity documents. Chemicals used as solvents will be purchased in the highest purity available from the manufacturer or distributor. Simulants will be purchased in the highest purity available from the manufacturer or distributor.
- e. Decontaminants will be prepared IAW the manufacturer's instructions. Quality checks will be performed as necessary by routine analytical methods [such as pH (hydrogen ion concentration) measurement, titration, etc.]. The pot life specified by the manufacturer will not be exceeded. This may require frequent preparation of the decontaminant during trial conduct.
- f. Test fixtures will be powered on and allowed to equilibrate at the specified test conditions. Test personnel will confirm that all equipment is operational (e.g., calibrated IAW required intervals) before the start of the test.

- g. Test personnel will complete the test setup, labeling of vials, trays, jars, etc. and other associated pretest tasks.
- h. Coupons/panels may require cleaning before testing to remove cutting oils or other preparation contaminants. TICN-labeled coupons/panels or small equipment articles will be stored in a secure, environmentally-controlled location. The test articles will be protected from unrelated environmental contaminants and degradation.
  - i. Test personnel will ensure that all calibrated instrumentation has a current calibration.

#### 4.7 Chemical Kinetics.

#### a. General.

- (1) This test determines the time it takes for a liquid 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°C. The stirrer will be started and the contents allowed to thermally equilibrate.
- c. The neutralizing reaction will be initiated by adding 1.00 mL of agent to the decontaminant. The time (t) will be noted (t = 0).
- d. The stirring rate will be adjusted as necessary to ensure complete mixing and homogeneous humidity.
- e. At measured intervals starting at t=2 min\*, a 50- $\mu$ L sample will be collected for GC-atomic emission detection (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 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 freebase form needed for complete extraction into the chloroform.
- (3) When other agents are used in testing, the methodology used to select a quenching solution will be included in the test plan and details of the quenching solution will be included in the test report.

<sup>\*</sup>The standard measurement intervals will be 2 min for the first 10 trial min, and then every 5 min thereafter until a total of 1 hr has elapsed after the agent addition.

- f. Using a micropipette, 1.0 mL of the chloroform layer will be transferred to an autosampler vial.
  - g. The sample will be analyzed with GC-AED or GC-MS.
  - h. The following data will be recorded:
    - (1) GC results, including amount of agent and reaction products.
- (2) Amount of agent remaining at each sample time, and the time required for the agent to become undetectable (if it becomes undetectable).
- (3) Observations made during the neutralization reaction. Observations will include visual inspection for HD droplets in the decontaminant. <u>NOTE</u>: HD is insoluble in water, a component of many decontaminants.
  - (4) pH level.
  - (5) Mass and purity of agent applied.

#### 4.8 Agent-Decontaminant Reaction Byproduct Test.

a. CWA byproducts may be produced through aging or decontamination, and may be as toxic as the original agent. For the purposes of this TOP, only byproducts produced through the decontamination process are being considered. Identification of toxic byproducts is extremely important when considering the hazard presented to unprotected personnel. Although not inclusive, the list in Table 1 shows some common CWA byproducts with Chemical Abstracts Service (CAS®) numbers for reference.

TABLE1. SOME COMMON CHEMICAL WARFARE AGENT (CWA) BYPRODUCTS

Agent <sup>a</sup>	Breakdown Mechanism	Byproduct	Chemical Abstracts Service (CAS®) Number
		Pinacolyl methylphosphonate	616-52-4
GD	Hydrolysis	3, 3-Dimethyl-2-butanol	464-07-3
		Methylphosphonic acid (MPA)	993-13-5
		Mustard sulfoxide	5819-08-9
Oxidation	Mustard sulfone	471-03-4	
HD	and/or elimination	Divinyl sulfone	77-77-0
нр		Divinyl sulfoxide	1115-15-7
	Hydrolygig	Thiodiglycol	111-48-8
	Hydrolysis	2-Chloro-2-hydroxyethyl sulfide	693-30-1

TABLE 1. CONTINUED

Agent <sup>a</sup>	Breakdown Mechanism	Byproduct	Chemical Abstracts Service (CAS®) Number
		Ethylmethylphosphonic acid (EMPA)	1832-53-7
		MPA	993-13-5
	VX Hydrolysis	2-Diisopropylaminoethanethiol	5842-07-9
VX		Methylphosphonothioic acid o-ethyl ester (EMPTA)	18005-40-8
		Hydrogen s-[2-(diisopropylamino)ethyl] methylphosphonothiolate (EA2192)	73207-98-4

<sup>&</sup>lt;sup>a</sup>GD – soman; HD – distilled mustard; VX – persistent nerve agent.

- b. This test procedure is used for initial screening of developmental decontaminants to determine if there is any value in proceeding with development.
  - c. General procedures for byproduct testing are:
- (1) Decontaminant and agents will be mixed in a reaction vessel at a ratio of 50 parts decontaminant to 1 part agent. The ratio may be changed in coordination with the test sponsor and evaluators based on manufacturer's recommendations.
- (2) At time intervals specified in the test planning documents, an aliquot of the mixture will be removed and placed in a secondary container. An appropriate organic solvent will be added to extract organic compounds based on the agent being tested. An aliquot of this mixture will be moved immediately and placed in a GC vial.
- (3) The GC will scan for all organic compound peaks. Each peak found will be analyzed by MS to identify each compound.
- (4) For LC/MS analysis during the aqueous phase: The LC will scan for all peaks. Each peak found will be analyzed on MS to identify organic compounds.

#### 4.9 Decontamination Efficacy – Residual Liquid Test Methods.

a. Quantification of the liquid on or absorbed into the surface of an item does not represent the full measure of residual agent, but rather the measure of contamination that could be bioavailable by touch (human interface) or contact transfer. **NOTE**: As of the date of this TOP, it is not possible to correlate the measured residual liquid with the toxicological effect on an exposed individual.

b. The test surfaces may be coupons\*, component assemblies, or whole test items, but a flat surface is necessary.

#### 4.9.1 Residual Liquid Contact Test Method.

a. This test method is designed to measure the liquid contaminant present on a material surface after the decontamination process that could pose a hazard to Warfighters through transfer to skin. A contact sampler is used as a surrogate for human skin. The contact sampler is used to collect the residual agent from the test article surface. A contact test event is called a touch. A touch is characterized by the contact area, contact pressure, contact duration, and skin condition (wet versus dry). The contact sampler is extracted, and residual liquid collected during the touch is quantified through analysis.

**NOTE**: The contact sampler usually consists of latex dental sampler (Reference 7 Source Document) cut in a 5.1 cm diameter swatch, an aluminum foil 5.1 cm diameter circle, and a 1 kg weight that is 5.1 cm in diameter.

- b. When coupons are used, they will be prepared from materials specified by the test sponsor. An example of a material list is presented in Appendix A. The default coupon size is 5.1 cm in diameter. If coupons of other sizes will be used during testing, a rationale must be included in the test planning documents. Ensure that when using other coupon sizes that the latex dental sampler swatch and the aluminum foil are of the same size as the coupon. It is very important to ensure that the weight is the same size as all of the other contact sample materials and maintains the appropriate pressure as described in reference 7.
- c. To prevent agent and/or decontaminant spread from the surface area of a test coupon, an appropriate border material will be placed on the periphery of the article. The use of a border material may be essential to prevent the agent and/or decontaminant from causing edge effects on the test coupons, especially if the coupons comprise painted surfaces.
- d. Test articles may be required to be tested at varied environmental conditions (temperature and humidity) and will require preconditioning. Fixture control will be more accurately obtained by using AH. However, RH and AH should always be reported.
- e. The use of AH (shown in the equation as *H*) provides a measure of the concentration of water in air. The AH is calculated from the temperature and RH by using Equation 1 and the reference values in Table 2.

#### Equation 1

$$H(T,RH) = \begin{cases} \frac{RH \cdot 13.2238 \cdot \exp\left(\frac{17.27 \cdot T}{T + 237.3}\right)}{T + 273.16} & T > 0 \text{ °C} \\ \frac{RH \cdot 13.2238 \cdot \exp\left(\frac{21.875 \cdot T}{T + 265.5}\right)}{T + 273.16} & T < 0 \text{ °C} \end{cases}$$

<sup>\*</sup>Coupons may represent the range of surfaces inherent to the items undergoing decontamination operations (e.g., vehicles, vans, weapons, etc.) or equipment materials.

Where:

H = absolute humidity (g water/m<sup>3</sup>)

RH = relative humidity (%)

 $T = \text{temperature } (^{\circ}\text{C})$ 

**NOTE**: RH of 10% should entered as 10.0 for this equation.

TABLE 2. REFERENCE VALUES TO VERIFY THE CALCULATION IN EQUATION 1 IS PERFORMED CORRECTLY.

Relative Humidity (%)	10	50	90
Temperature (°C)	Absolute Humidity (g H <sub>2</sub> O/m <sup>3</sup> air)		
-20	0.088	0.440	0.791
0	0.484	2.421	4.357
20	1.727	8.634	15.541
40	5.099	25.495	45.891

- f. Based on the test planning documentation environmental condition requirements, the test articles will be environmentally conditioned in a conditioning chamber or the test fixture. **NOTE**: It is extremely important in test planning to understand that high temperature and high RH extremes (e.g., 49°C and 90 percent RH) are not always real-world conditions. Test personnel should reference Military Handbook (MIL-HDBK) 310, Global Climatic Data for Developing Military Products<sup>14</sup>, for natural/operational environment ranges.
- g. Chemical agent will be applied to the test article (coupons will be positioned horizontally) per the test plan at a default density of  $10 \text{ g/m}^2$  ( $1 \text{ mg/cm}^2$ ). The application of the agent may require multiple drops of varied sizes up to a single large drop as defined in the test planning documentation.
- h. During testing, the test article should remain uncovered when replicating field conditions. An alternative test method<sup>7</sup> describes covering the test article to minimize headspace and reduce evaporation. The contaminated test article will be allowed to weather/age for 1 hr unless otherwise specified in the test planning documentation. The test article should be maintained at the environmental conditions that are required for contaminant application.
- i. Pretest mixing or preparation of the decontaminant, if necessary, will be performed before the decontamination portion of this test method is conducted. Based on the pot life of the decontaminant, this may require frequent decontaminant preparation throughout testing.
- j. Decontamination of the test article will be conducted. In early screening testing, the decontaminant will be applied in the amount and manner specified by the decontaminant manufacturer. In later developmental testing, the decontaminant may be required to be applied using available standard field methods. Care will be taken to ensure that the entire test surface area is covered by the decontaminant (minimizing decontaminant on the edges of coupons). When using FM 3-11.5<sup>15</sup> methods for decontamination, agitation (e.g., brushing/scrubbing) of

the decontaminant during application is required. It is recommended that a standard hard bristle toothbrush be used when testing coupons and scrubbing is required. When standard field methods are not available, the manufacturer's instructions will be followed.

**NOTE**: As a reference, U.S. Army Field Manual (FM) 3-11.5<sup>15</sup> indicates that an approximate decontaminant/agent ratio is 50:1 (mass of decontaminant to mass of agent).

- k. Decontaminant will reside on the test article for a default time of 30 min, or a time specified by the manufacturer or in test planning documentation.
- 1. The test article will be rinsed with water and allowed to dry until no visible liquid is present unless otherwise specified in test planning documentation. All surfaces of the test sample will be rinsed. Samples of the rinsate may be required for analysis and mass balance calculations. This water rinse represents the FM 3-11.5<sup>15</sup> Detailed Equipment Decontamination post decontamination rinse to remove the decontaminant from the surface of the equipment. Immediate and operational decontamination procedures do not require this water rinse. The second rationale for the water rinse is to remove decontaminant prior to conduct the contact sampling or coupon extraction as the decontaminant may interfere with the analytical instrumentation for sample analysis.
- m. A 5.1-cm diameter piece of latex dental sampler will be placed on top of an identified sample location on the test article (this may be the entire surface of a coupon). A 5.1-cm diameter piece of aluminum foil will be placed on top of the sampler. NOTE: Silicone rubber or another sorbent material may be used instead of latex dental sampler material, but the substitution must be documented in the test report with the rationale and extraction efficiency study results.
- n. A 1-kg weight will be placed on the sampler for 15 min, or as specified by the test planning documentation. This represents one touch. Additional touches of 15 min each may be performed up to a total of 1 hr (or four touches).
- o. The weight will be removed and the sampler and associated foil backer will be placed in a sample jar with extraction solvent. The jar will be agitated periodically during the 1-hr minimum extraction time. **NOTE**: For residual agent absorbed into the surface of coupons, see Paragraph 4.9.2.
- p. The solvent will be analyzed for agent and breakdown products as required. There are no requirements established for the breakdown products. Identification of the presence of the products and/or quantification of the amount of breakdown products will be addressed in the test plan and documented in the test report.
  - q. The following data will be reported:
    - (1) Residual agent results in mass per sampler.
    - (2) Positive and negative control results in mass.

- (3) Dose confirmation results in mass.
- (4) Rinsate results, as needed.
- (5) Temperature.
- (6) RH.
- (7) AH in  $g/m^3$
- (8) Agent purity.
- (9) Extraction solvent used and purity.
- (10) Number and size of drops applied.
- (11) Weathering time.
- (12) Decontaminant preparation method, preparation time, and elapsed time between preparation and application.
  - (13) Decontaminant application method and agitation method, if used.
  - (14) Decontaminant residence time.
  - (15) Rinsate used, method of application, volume, and rinse time.
  - (16) Agent contact time.
  - (17) For test articles, TICN and sample location(s).
  - (18) For coupons, sample item control number (SICN).
  - (19) Visual observations of test article surface and condition.

#### 4.9.2 Absorbed Residual Liquid Test Method.

- a. To determine the level of residual liquid contaminant adsorbed into the surface of the coupon, the coupon will be placed in a sample jar with appropriate extraction solvent. This will be done at the same time that the contact sampler and foil backing are placed into a separate sample jar with extraction solvent (Paragraph 4.9.1.o).
- b. The sample jar holding the coupon will be agitated periodically during the 1-hour minimum extraction time.
  - c. The solvent will be analyzed for agent.
  - d. The following data, in addition to those listed in Paragraph 4.9.1.q, will be reported:

- (1) Test sample GC results from the liquid used to extract the contaminant from the coupon.
  - (2) For each coupon, the SICN.
  - (3) Visual observations of coupon surface and condition before extraction.

#### 4.10 Material Compatibility Tests.

- a. General. Materials to be tested will be specified in the test planning documents.
- b. pH Test. The pH of the decontamination solutions will be measured. The pH meter will be calibrated according to instrument specification.
  - c. Corrosivity Test.
- (1) The objective of this test is to measure any corrosive effects of the decontaminant on several types of metal. This test will be performed IAW the American Society for Testing and Materials (ASTM) (ASTM International, West Conshohocken, Pennsylvania) Standard G31-72(2004)<sup>16</sup>.
- (2) Strip coupons (measuring  $35 \times 50 \times 0.05$  mm) of various materials as listed in the test plan, will be degreased by washing with soapy water followed by thorough rinsing in water and then acetone.
  - (3) The coupons will be weighed.
- (4) 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 for 24 hr.
- (5) After 24 hr the coupons will be removed from the decontamination solution, rinsed with water, and allowed to air dry.
- (6) 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.
  - (7) The coupons will be weighed again.
  - d. Sorption Test (for Nonelastomer or Nonthermoplastic Materials).
- (1) The objective of this test is to determine if the material will adsorb or desorb decontaminants and/or contaminants based on weight change. For continuous-fiber reinforced polymer matrix composite materials, the procedures in ASTM Standard D4762-11a<sup>17</sup> also apply.
- (2) All coupons will be cleaned by washing with soapy water followed by a thorough clear water rinse. After being rinsed, the coupons will be allowed to dry thoroughly.

- (3) All coupons must be the same size for direct comparison. A minimum of five replicates is required. Each coupon will be weighed and assigned a TICN.
- (4) As required by the test objective described in the test plan, contaminant or decontaminant will be applied to the coupon. A residence time based on test plan requirements will be allowed. Isopropyl alcohol will be used to remove contaminants and a water rinse will be used to remove decontaminants. The coupons will be allowed to dry thoroughly.
  - (5) Each coupon will be weighed.
  - e. Sorption (Elastomer) and Hardness (Elastomer) Test.
- (1) 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.
  - (2) The sorption test will be performed IAW ASTM Standard D471-12<sup>18</sup>.
- (3) The Shore A hardness test will be performed IAW ASTM Standard D2240- $05(2010)^{19}$ .
  - (4) Coupons will be cut in the form of bars measuring  $25 \times 50$  mm.
- (5) The initial indentation hardness of each type of elastomer will be determined using a 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.
- (6) The coupons will be weighed and immersed in closed jars of the decontamination solution and conditioned for 24 hr at 30°C. After this period, the samples will be removed, rinsed with water, wiped dry and allowed to air dry, weighed, and their final indentation hardness measured.
- f. Sorption (Thermoplastic), Hardness (Plastic), and Haze and Transmittance (Thermoplastic) Test.
- (1) The objective of this test is to measure the weight, hardness, 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.
  - (2) The sorption test will be performed IAW ASTM Standard D543-06<sup>20</sup>.
  - (3) The hardness test will be performed IAW ASTM Standard D785-08<sup>21</sup>.
- (4) The haze and transmittance test will be performed IAW ASTM Standard D1003-11e1<sup>22</sup> for transparent or translucent materials. The original weight and haze/transmittance will be measured.

- (5) For liquid or foam decontaminant solutions:
- (a) The test article will be exposed to the decontamination solutions via filter-paper circles placed on the thermoplastic and then saturated with the decontamination solution.
- (b) The thermoplastic will be covered with disposable plastic beakers during the test to minimize evaporation of the decontamination solution.
- (c) The filter-paper circles will be resaturated with decontamination solution at regular intervals (approximately every 3 hr or as defined by the test plan).
- (d) After 24 hr (or a different duration specified in the test plan) of exposure to the decontamination solution at ambient laboratory temperatures (~25°C), the thermoplastic will be removed, rinsed with water, wiped dry and allowed to air dry, and weighed.
  - (6) For decontaminant powders:
- (a) The decontaminant will be dusted on the surface of the test article. A towelette will be pressed against the decontaminant and swiped from left to right once and from top to bottom once. Alternatively, the towelette may be swiped in a circular motion twice. The amount of pressure to be applied on the towelette will depend upon the manufacturer's recommendation, and must be described in the test plan.
  - (b) The decontaminant will be removed by light brushing, pressurized air, or rinsing.
  - (7) For decontaminant wipes:
- (a) The decontaminant wipe will be used as described in the CONOPS developed by each using Service with consideration given to the manufacturer's recommended use described in the test plan. The amount of pressure to be applied on the wipe will depend upon the manufacturer's recommendation and must be described in the test plan.
- (b) The decontaminant will be removed from the surface of the test article by rinsing with water or wiping with a dry towelette.
- (8) The final weight and haze/transmittance of the thermoplastic will be measured, as needed.
  - g. Hardness (Coating) Test.
- (1) 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 ASTM Standard F502-08<sup>23</sup> and ASTM Standard D3363-05(2011)e2<sup>24</sup>.
- (2) Coupons will be prepared, placed face up in Petri dishes, and covered with the decontamination solution. Glass covers will be placed on the Petri dishes to prevent evaporation.
- (3) The coupons will be conditioned at ambient laboratory temperatures ( $\sim 25^{\circ}$ C) for 24 hr.

- (4) The coupons will be removed, washed with water, wiped dry, and allowed to air dry.
  - (5) Film hardness rating will be determined for coupons before and after exposure.
  - h. The following data will be reported:
    - (1) pH results.
    - (2) Average corrosion rate (mils/year). **NOTE**: 1 mil = .001 in.
    - (3) Visual observations.
- (4) Observations relating to the evolution of gases/corrosion products. If the gases/corrosion products are hazardous, the necessary analysis and quantification of the products will be conducted.
- (5) Elastomer sorption results: average percent weight change and visual observations.
- (6) Elastomer hardness results: average percent change of indentation hardness and visual observations.
  - (7) Thermoplastic sorption results: percent weight change and visual observations.
- (8) Thermoplastic haze and transmittance results: percent change in haze and transmittance and percent change in visual observations.
- (9) Hardness (coating) test results: average gouge measurements, average scratch measurements, and visual observations.
- (10) Convective flow results: average weight of agent penetration per unit area of material measured over 24 hr.

#### 4.11 Detector Compatibility Test.

#### a. General.

- (1) All detectors will be checked for correct functioning before any other testing. Results of function checks will be recorded as part of the test documentation.
- (2) Fielded detectors/alarms will be selected in coordination with the test sponsor and the operational test agencies and may include (but are not limited to) some or all of the following:
  - (a) Improved Chemical Agent Monitor (ICAM).
  - (b) U.S. Marine Corps (USMC) Chemical Agent Monitor (CAM).
  - (c) M43A1 Chemical Agent Automatic Alarm Detector Unit.

- (d) M90D1-C Chemical Warfare Agent Detector.
- (e) M18A2 Chemical Agent Detector Kit.
- (f) M256A1 Chemical Agent Detector Kit.
- (g) Shipboard Chemical Agent Monitor Portable (SCAMP).
- (h) Improved Point Detection System (IPDS).
- (i) AP2C Vapor and Liquid Agent Detector.
- (j) Lightweight Chemical Detector 3 (LCD 3).
- (k) Joint Chemical Agent Detector (JCAD).
- (1) 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) Handheld Assay (HHA).
- (q) Automatic Chemical Agent Detector and Alarm (ACADA).
- (3) Detector/alarm posttest functionality checks will be conducted and the results recorded.
  - b. False Positive Versus Neat Decontaminant Test.
- (1) A false positive is defined as a detector alarm to neat decontaminant with no agent present.
- (2) Decontaminant prepared IAW manufacturer's or test sponsor's instruction in the test plan will be placed in a glass dish in a fume hood.
- (3) The inlet of the detector [itself or attached with a flexible Teflon<sup>®\*</sup> tube (DuPont<sup>TM</sup>, E.I. du Pont de Nemours and Company, Wilmington, Delaware)] will be placed approximately 2.5 cm 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 sampling time is reached (up to 5 min if there no sampling time is specified by the

<sup>\*</sup>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 it is best suited for its intended application.

manufacturer). Precautions will be taken to prevent gross contamination of the detectors. Spectra or recordings may be taken when appropriate.

- (5) All detectors responses, data requirements, and any conditions or observations deemed potentially relevant to the experiments will be noted in laboratory notebooks and discussed in the report.
  - c. False Negative Versus Agent-Decontaminant Mixture Test.

This test method requires additional development or refinement and will be place back in this document upon completion or at the next review cycle. Conduct of this test method during this interval will be fully described in the detailed test plan for any SUT and coordinated with the test sponsor.

#### 4.12 Pot Life Test.

Pot life is defined as the period of time the decontaminant remains efficacious after mixing or after opening the container. Pot life testing is most often used as an initial screening or a bench test for decontaminant performance.

#### 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, as required by the TEMP, or as otherwise specified by the manufacturer because of product limitations.
- (2) The test will be conducted at low temperatures ( $\sim$ -32°C), ambient temperatures (21°C), and high temperatures ( $\sim$ 49°C); and at low RH (< 20 percent) and high RH (> 90 percent), or as otherwise specified by the test plan.
- (3) This test procedure does not require the use of any challenge material, such as CWAs or nontraditional agents (NTAs), with the decontaminant.

#### b. Procedure.

- (1) Properties or performance characteristics to be used to evaluate continued decontaminant efficacy will be identified in the test plan. A quantitative measure of the identified performance characteristics will be determined. The quantitative measure will be assigned a nominal value of 100 (representing 100 percent) for the level present when the decontaminant is initially produced/opened. Examples of quantitative measures would be:
- (a) The amount of oxidizer or other reactive species (for reactive decontaminant). Testers will determine the initial amount of free chlorine in a chlorinated decontaminant when the product is mixed/opened (this is the 100 percent amount). After an elapsed time, the remaining amount of free chlorine will be determined and compared with the 100 percent

amount. For liquid decontaminants, testers will ensure that the solution is well-mixed before removing an aliquot for analysis.

- (b) The amount of sorptive capacity remaining (for sorbent decontaminant). Testers will determine the initial sorptive capacity of a sorbent powder when the package is opened (this is the 100 percent capacity). After an elapsed time, the remaining sorptive capacity will be determined and compared with the 100 percent capacity.
- (2) This quantitative measure value will decrease as the product ages after mixing or opening. When the quantitative value decreases to levels less than a predetermined threshold (e.g., 50 may be a threshold minimum value compared with the initial nominal value of 100) the decontaminant will be considered ineffective. The time it takes for a decontaminant to decrease to this threshold level will be considered the decontaminant's pot life.
- (3) Multiple samples of the decontaminant will be obtained from the original unopened containers. The number of samples will be determined based on a minimum of three replicates for the baseline product (e.g., sorbent mitts) and three replicates for the product to be aged, which may mean three replicates for each sampling period. The decontaminant samples will be prepared IAW the manufacturer's instructions or standard procedures for field use of the decontaminant.
- (4) One of the decontaminant samples will be evaluated for the quantitative measure as a baseline for comparison with the aged product. Both samples (baseline and the product to be aged) will be prepared at the specified test conditions (temperature and humidity) identified in the test plan.
- (5) The quantitative measure will be evaluated from the aged decontaminant samples at the intervals specified in the test plan (e.g., at 4, 8, 10, and 12 hr) until values decrease below the threshold level. The decontaminant's pot life will be estimated from these sampling results.
  - (6) The following data will be reported:
  - (a) Test environmental conditions [i.e., temperature and humidity (RH and AH)].
  - (b) Type, quantity, and concentration (if applicable) of decontaminant.
  - (c) Performance characteristic measurements.
  - (d) Estimated decontaminant pot life.

#### 4.13 Shelf Life/Accelerated Aging Test.

#### 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 in the test plan. A quantitative measure of the quality of the decontaminant (e.g., sorptive capacity) will be determined based on the identified characteristics. The quantitative measure will be assigned a nominal value of 100 when the decontaminant is initially produced or opened. This quantitative measure value will decrease as the product ages. When the quantitative measure value decreases below a predetermined threshold the product will be considered out of compliance.

#### b. Procedure.

- (1) Multiple 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 for the characteristics identified in the test plan.
- (2) 105, five replicates for each condition and sample period (Reference 7), unopened test samples of the decontaminant will be transferred into clean, nonreactive, thermally-stable, hermetically-sealable containers and tightly closed. The original product containers may be used as the outer container, if deemed suitable, in which case no transferring would be necessary. Each test sample container must contain sufficient product to perform all characterization testing prescribed herein.
- (3) 35 of the test samples will be placed in each of three test temperature environments selected on the basis of an evaluation of the product's physical composition or as specified by the test sponsor. Humidity conditions will be constant, at less than 20 percent RH, or as otherwise specified by the test plan.
- (4) Once a month, over a period of 7 months, five product samples will be drawn from the original unopened test containers (packaging), and the decontaminant quantitative measure value will be determined and recorded.
  - (5) The following data will be reported:
  - (a) Test environmental conditions (i.e., temperature and humidity).
- (b) Type, quantity, and concentration (if applicable). For multi-component decontaminants, the corresponding values for all components will be recorded.
- (c) Performance characteristics used to calculate the product's 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. <u>DATA REQUIRED</u>.

Data required are listed under the individual subtests in Paragraph 4.

#### 6. PRESENTATION OF DATA.

- a. Photographs will be presented of any visible effect from contaminant or decontaminant on the test article.
  - b. Kinetic data versus time will be presented in tabular form.
  - c. Decontamination efficacy results will be presented by agent in tabular form.
- d. Decontamination efficacy data will be analyzed IAW the experimental design specified in the test plan.
- e. Environmental data for each trial will be presented graphically [e.g., temperature and/or humidity (RH and AH) versus time].
- f. Material compatibility subtest results will be presented in tabular form with photographs demonstrating significant effects.
  - g. Pot life results will be presented graphically versus time.
  - h. Shelf life results will be presented graphically versus time.
- i. Data collected 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. COUPON MATERIALS.

#### 1. MATERIAL SELECTION.

Coupons will be prepared of materials representing a range of surfaces inherent to the items undergoing decontamination operations (vehicles, weapons, etc.). The selection and prioritization of materials to meet the needs of each program of record will be coordinated between the test sponsor and operational test agencies. There are a wide variety of items comprising hundreds of potential materials that could be encountered during decontamination. A sample listing of highest-priority materials was compiled by material developers to condense the potential materials to a manageable number and facilitate a cost-effective test and evaluation program for a decontaminant. Factors considered are the expected impact of the material on mission/combat readiness, likelihood of the materials to be exposed to contamination, and the cost of the material to replace.

#### 2. SAMPLE LISTING OF HIGH-PRIORITY MATERIALS.

**NOTE**: Coupon materials are not limited to the following.

- (1) Chemical agent-resistant coating (CARC) on steel (tactical vehicles).
- (2) Aircraft topcoat paint on aluminum (aircraft).
- (3) Low-infrared (IR) paints on aluminum or steel (aircraft & ships).
- (4) Ship deck antiskid coating on steel.
- (5) Polyurethane, epoxy, and alkyd paints on metals (commercial vehicles).
- (6) Aluminum alloys, forged and cast (aircraft surfaces & structural members).
- (7) Aluminum, oxidized aluminum (vehicle substrate surface).
- (8) Stainless and high strength steel alloys (aircraft and engine structural members).
- (9) Nickel-based and other superalloys (aircraft and engine structural members).
- (10) Carbon/stainless steels (vehicle, munitions substrate surface).
- (11) Brass/bronze/copper and nickel alloys (munitions substrate surface).
- (12) Composite and laminate materials (aircraft surface and structural members).
- (13) Aircraft composites (aircraft).
- (14) Tire rubber (aircraft, vehicles).
- (15) Polycarbonates/Lexan® (SABIC Innovative Plastics, Pittsfield, Massachusetts) (aircraft canopy/window materials, tactical vehicles).

### APPENDIX A. COUPON MATERIALS.

- (16) Glass (commercial vehicles, tactical vehicles).
- (17) Butyl rubber (mask, gloves/boots).
- (18) Silicon rubber (M40 mask).

ACADA Automatic Chemical Agent Detector and Alarm

AD No. accession number

AED atomic emission detection

AH absolute humidity

AMCR U.S. Army Materiel Command Regulation

AR Army Regulation

ASR agent-simulant relationship

ASTM American Society for Testing and Materials

ATEC U.S. Army Test and Evaluation Command

BAWS Biological Aerosol Warning Sensor

CAM Chemical Agent Monitor

CARC chemical agent resistant coating

CAS Chemical Abstracts Service

CASARM Chemical Agent Standard Analytical Reference Materials

CBMS Chemical Biological Mass Spectrometer

CoC chain of custody

CWA chemical warfare agent

DA Department of the Army

DOE design of experiment

DTIC Defense Technical Information Center

EA2192 hydrogen s-[2-(diisopropylamino)ethyl] methylphosphonothiolate

EMPA ethylmethylphosphonic acid

EMPTA methylphosphonothioic acid o-ethyl ester

FID flame ionization detection

FM field manual

FPD flame photometric detection

GC gas chromatograph

GD soman

HD distilled mustard

Hg mercury

HHA handheld assay

IAW in accordance with

ICAM Improved Chemical Agent Monitor
IPDS Improved Point Detection System

IPT integrated product team

IR infrared

ISO International Organization for Standardization

iwg inches water gauge

JCAD Joint Chemical Agent Detector

LC liquid chromatography

LCD 3 Lightweight Chemical Detector 3

MIL-HDBK military handbook

MPA methylphosphonic acid

MQL minimum quantification limit

MS mass spectrometry

NMR nuclear magnetic resonance

NTA nontraditional agent

OPSEC operational security

ORI operational readiness inspection

OTA operational test agency

PAM pamphlet

pH hydrogen ion concentration

QA quality assurance QC quality control

R<sup>2</sup> correlation coefficient

RDT&E research, development, test, and evaluation

RH relative humidity

SCAMP Shipboard Chemical Agent Monitor – Portable

SCG Security Classification Guide

SDS safety data sheet

SEP system evaluation plan

SICN sample item control number SOP standing operating procedure

solid sorbent tube

standing operating proce

SST

t

T&E test and evaluation

time

TEMP test and evaluation master plan

TICN test item control number
TIM toxic industrial material

TIR test incident report

TO test officer

TOP Test Operations Procedure

TRR test readiness review

U.S. United States

USMC U.S. Marine Corps

V&V verification and validation

VX methylphosphonothioic acid, persistent nerve agent

 $\Delta P$  differential pressure

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TOP 08-2-061A 20 October 2014

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# TECMIPT Test Operations Procedures (TTOP) 08-2-061A Chemical Decontaminant Testing

Decontamination Capability Area Process Action Team (CAPAT):

William G. Davis, Dugway Proving Ground Deborah L. Beier, Dugway Proving Ground

CAPAT Review & Concurrence: April 2014



DISTRIBUTION A. Approved for public release: distribution unlimited.

#### REFERENCES:

(a) Chemical and Biological Defense Program (CBDP) Test and Evaluation (T&E) Standards Development Plan, dated 19 July 2010.

(b) Memorandum of Understanding (MOU) Among the Department of National Defence of Canada the Secretary of State for Defense of the United Kingdom of Great Britain and Northern Ireland and the Secretary of Defense on Behalf of the Department of Defense of the United State of America concerning the Research, Development and Acquisition of Chemical, Biological and Radiological Defense Materiel, dated June 2000. Amendment One, dated August 2006.

TECMIPT Test Operations Procedure (TOP) 08-2-061A,			
Chemical Deco	ontaminant	Testing Concurrence Sheet	
LT Col Kevin Reilly Marine Corps Operational Test Activity (MCOTEA)	t & Evaluation	Mark F. Thomas Joint Program Executive Office of Chemical Biological Defense (JPEO-CBD) Test & Evaluation	
Signature	Date 5/30/14	Signature Date	
Grant D. Schaber, Civ, DAF Acting Director of Operations Air Force Operational Test and (AFOTEC)	· ·	Laurie K. Richter, Lt Col, USAF Joint Requirements Office for Chemical, Biological, Radiological and Nuclear Defense (JRO-CBRND)	
Grant Sell	14 May 14	Declicites 11Jun 14	
Signature	Date	Šignature Date	
Jeffery Bobrow Operational Test and Evaluatio (OPTEVFOR)	on Force	Deborah Shuping Deputy Under Secretary of the Army – Test and Evaluation (DUSA-TE)	
gh_	2/19/14	Doborah Shuping	
Signature	Date	Signature Date 13 May	
Deirdre Sumpter US Army Test and Evaluation Army Evaluation Command (A	Command/US ATEC/AEC)	Mike Roberts Joint Science and Technology Office (JSTO)	
Deirdee Sunpto	5 May 14	Taichael.rober Digitally signed by michael.roberts@dtra.mil DN: cn-michael.roberts@dtra.mil Date: 2014.04.14 11:45:54-04'00'	
Signature	Date '	Signature Date: 04/14/2014	
Bill Davis		Brent Mantooth	
William Wair		Brent Mantorth 13 May 2019	
Signature	Date	Signature Date 6 level concurrence from their organization. If the	

Note: CAPAT member's signature represents an O6 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.

#### **TECMIPT Chair Endorsement**

AMXAA-CD 24 July 2014

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

SUBJECT: Test and Evaluation Capabilities and Methodologies Integrated Product Team (TECMIPT) Test Operations Procedure (TTOP) 08-2-061A, Chemical Decontaminant Testing

- The Decontamination Capability Area Process Action Team (CAPAT) has completed
  the subject TTOP in accordance with the DUSA-TE Instructions to the TECMIPT, the
  Standards and Development Plan, and the TECMIPT Standard Operating Procedure.
  All signatory members of the CAPAT have provided their concurrence to this TTOP.
  The final TTOP is enclosed.
- 2. Based on the concurrence of the CAPAT, I recommend the CBRND T&E Executive endorse this TTOP as a Department of Defense Test and Evaluation Standard.

Encl

for Water K Gods RONALD O. PRESCOTT TECMIPT Chair

#### Office of the 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

DUSA-TE

JUL 3 0 2014

#### MEMORANDUM FOR DISTRIBUTION

SUBJECT: Endorsement of Test and Evaluation (T&E) Capabilities and Methodologies Integrated Process Team (TECMIPT) Test Operations Procedure (TTOP) 08-2-061A, Chemical Decontaminant Testing

- Reference: Memorandum, DUSA-TE, and 19 July 10, subject: Chemical and Biological Defense Program (CBDP) Test and Evaluation (T&E) Standards Development Plan.
- TTOP 08-2-061A was developed, coordinated, and approved by the members of the Decontaminant Capability Area Process Action Team (CAPAT) in accordance with the reference. The U.S. Army Test and Evaluation Command (ATEC) approved according to their TOP approval process.
- 3. I endorse this TTOP as a DoD T&E Standard for decontamination 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.
- My point of contact for this action is Ms. Deborah Shuping, (703) 545-1119, deborah.f.shuping.civ@mail.mil.

Encl

JAMES C. COOKE CBRN Defense T&E Executive

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#### Office of the Deputy Under Secretary of the Army Endorsement (continued)

#### DUSA-TE

SUBJECT: Endorsement of Test and Evaluation (T&E) Capabilities and Methodologies Integrated Process Team (TECMIPT) Test Operations Procedure (TTOP) 08-2-0661A, Chemical Decontaminant Testing

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CSTE-TM 20 October 2014

#### 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) 08-2-061A, Chemical Decontaminant Testing, Approved for Publication

1. TOP 08-2-061A, Chemical Decontaminant 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 describes the test procedures used to characterize and determine the technical performance of a decontaminant. Decontamination is the process of reducing or eliminating the hazards associated with chemical, biological, or radiological contamination in order to accomplish assigned missions. This TOP addresses test methods for decontaminants of chemical contaminants only. Chemical contaminants may include chemical warfare agents (CWAs), advanced threat agents, toxic industrial chemicals, and toxic industrial materials. Many of the test methods in this TOP are conducted with only the decontaminant being tested. Some test methods for efficacy require the use of CWAs and decontaminants.

- 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.amy.mil/.
- 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.

RAYMOND G. FONTAINE Associate Director, Test Management Directorate (G9)

FOR

MICHAEL J. ZWIEBEL Director, Test Management Directorate (G9) Forward comments, recommended changes, or any pertinent data which may be of use in improving this publication to the following address: 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, (TEDT-DPW), U.S. Army Dugway Proving Ground, Dugway, UT 84022-5000. Additional copies can be requested through the following website: <a href="http://www.atec.army.mil/publications/topsindex.aspx">http://www.atec.army.mil/publications/topsindex.aspx</a>, 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.