

REPORT DOCUMENTATION PAGE

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14. ABSTRACT 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 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) and advanced threat agents. 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.						
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U.S. ARMY TEST AND EVALUATION COMMAND
TEST OPERATIONS PROCEDURE

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

7 January 2021

CHEMICAL DECONTAMINANT TESTING

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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) and advanced threat agents.

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. 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**}.

** Superscript numbers correspond to Appendix C, References.

c. Biological decontaminant test methods (included in previous versions of this TOP) have been removed and placed in TOP 08-2-065².

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. FACILITIES AND INSTRUMENTATION.

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 Section 4.

2.1 Facilities.

2.1.1 Chemical Test Facility.

<u>Item</u>	<u>Requirement</u>
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. Facility must be equipped and certified for work with chemical agents. The chemical agent facility, instruments, and personnel assignments must meet all requirements of Army Regulation (AR) 50-6 ³ and AR 190-59 ⁴ , and the safety requirements of Department of the Army (DA) Pamphlet (PAM) 385-61 ⁵ .
Chemical agent test chamber.	Must be fabricated with appropriate construction materials (e.g., acrylic, stainless steel, glass) 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. Chambers used for this testing should have the capability of environmental controls for temperature and humidity as outlined in the requirements documents.

<u>Item</u>	<u>Requirement</u>
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. Laboratories used for this testing should have the capability of environmental controls for temperature and humidity as outlined in the requirement documents.
Chemical agent test fixture.	Test fixture must be capable of containing 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, as outlined in the requirements documents. 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 Instrumentation.

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

<u>Parameter</u>	<u>Measuring Device</u>	<u>Permissible Error of Measurement</u>
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).

<u>Parameter</u>	<u>Measuring Device</u>	<u>Permissible Error of Measurement</u>
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).
Residual contaminant in samples from contact samplers, coupons, rinsate, or other samples.	MS, GC or LC, FID, FPD, or equivalents.	± 15 percent of the mass of the contaminant per sample or within ± 25 percent of the device MQL.
Temperature.	Thermocouple, remote temperature device, or equivalent.	± 2 °Celsius (°C).
Relative Humidity (RH).	Hygrometer, humidity meter, or equivalent.	± 5 percent RH. <u>NOTE:</u> This means that for an RH target of 50 percent, 45 to 55 percent would be acceptable.
Absolute Humidity (AH).	Psychrometer, hygrometer, humidity meter, or equivalent.	± 0.1 grams per meter cubed (g/m ³)
Differential pressure for test chambers or fixtures only.	Pressure transducer.	± 0.09 millimeter (mm) mercury, or ± 12.0 pascal (± 0.05 inches water gauge).
Wind speed (outside) or airflow (chamber or fixture).	Hotwire anemometer or equivalent.	± 0.1 meters per second (m/sec).
Visual record (still).	Digital color camera.	Image resolution adequate to document details of testing.
Visual record (in motion).	Digital video camera.	Resolution and frame capture rate adequate to document details of testing.

2.3 Test Controls.

<u>Parameter</u>	<u>Tolerance</u> (unless otherwise specified)
Positive control (contaminated but not decontaminated residual liquid) using coupons.	Concentration (mass/area) \pm 15 percent or at the MQL \pm 25 percent. Contaminant per sample (mass/volume) \pm 15 percent or at the MQL \pm 25 percent.
Cross contamination control (neither contaminated nor decontaminated to determine if the test procedures introduce cross contamination).	Concentration (mass/area) \pm 15 percent or at the MQL \pm 25 percent. Contaminant per sample (mass/volume) \pm 15 percent or at the MQL \pm 25 percent.
Negative control (not contaminated, but decontaminated residual liquid) using coupons.	Concentration (mass/area) \pm 15 percent or at the MQL \pm 25 percent. Contaminant per sample (mass/volume) \pm 15 percent or at the MQL \pm 25 percent.
Dose confirmation samples. May be taken before, during, and after test article contamination.	Contaminant per sample (mass/volume) \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 (mass/volume) \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 (e.g., Test and Evaluation Master Plan (TEMP), 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 and 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, System Evaluation Plan (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 design of experiment, 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 as outlined in the Edgewood Chemical and Biological Center (ECBC) Technical Report 980⁶. The minimum number of replicates recommended 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 must be established and coordinated with the test program community of interest before testing begins.

e. Additional consideration must be given in addressing the type of decontamination operations that might be required. Decontamination operations are identified as immediate, operational, thorough, and clearance. Clearance decontamination operations are not addressed in this document. It is possible that immediate, operational, and thorough operations might be performed on the same equipment at different times after a contamination event. The guiding document for decontamination operations is the Army Technical Publication (ATP) 3.11-32⁷, sections D-116 through D-124. The thorough decontamination line or Detailed Equipment Decontamination (DED) line is laid out in Figure D-14 of ATP 3.11-32.

f. The DED calls for an equipment or vehicle pre-wash and a post-decontamination water rinse. The DED also calls for scrubbing (section D-119 of ATP 3.11-32) the vehicle with brushes during application of the decontaminant. Coupon testing laid out in this document should consider these procedures (pre-wash, post-rinse, scrubbing) in the coordination process to address the need, desirability, and execution steps before testing begins.

g. 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 requirements will be followed.

h. Test Incident Report (TIR). Unless waived by the test sponsor, TIRs (or equivalent reports), will be prepared and distributed in accordance with (IAW) U.S. Army Test and Evaluation Command (ATEC) Regulation 73-1⁸ and DA PAM 73-1, Appendix V⁹.

i. 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 310, Global Climatic Data for Developing Military Products¹⁰, for natural/operational environment ranges.

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 (TRR)/Operational Readiness Inspection (ORI).

a. If required, programs will undergo a 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, requirements representatives, Test and Evaluation Integrated Product Team 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 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 pre-operational 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 Quality Assurance (QA)/Quality Control (QC).

a. A chain-of-custody 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) 17025¹¹) 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 of ECBC Technical Report 980⁶.

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.

f. All aspects of the testing will be performed with emphasis on acquiring valid, credible, repeatable, and verifiable data.

3.8 Verification and Validation.

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 the 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 5725¹² 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.

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 plan.

a. 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.

b. Negative control. For negative controls, no contaminant is applied to the coupons, which undergo all other test procedures with the trial coupons, including decontamination. Positive analytical results for contamination on a negative control are an indication of cross contamination most likely showing poor test process control.

4.2 Test Method Outline.

- a. Receipt Inspection (Paragraph 4.5).
- b. Trial Preparation Tasks (Paragraph 4.6).
- c. Chemical Kinetics (Paragraph 4.7).
- d. Agent Decontaminant Reaction Byproducts Test (Paragraph 4.8).
- e. Decontamination Efficacy - Residual Liquid Test Methods (Paragraph 4.9).
- f. Material Compatibility Test (Paragraph 4.10).
- g. Detector Compatibility Test (Paragraph 4.11).
- h. Pot Life Test (Paragraph 4.12).
- i. Shelf Life/Accelerated Aging Test (Paragraph 4.13).

4.3 Hazards.

a. Identified safety hazards are those associated with tests using toxic chemical surety materials, simulants, and hazardous decontaminant chemicals (e.g., chlorine, hydrogen peroxide). Chemical safety guidelines are found in DA PAM 385-61⁵.

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 IAW AR 385-10¹³. 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., guidelines can be found in ISO 17025¹¹). These guidelines can be used for calibrating most chemical analytical equipment (e.g., GCs, LCs) 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). 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 analytical 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 coefficient of determination (R^2) is greater than 0.990 and the calculation of the Relative Percent Deviation (RPD) meets the standards found in ECBC TR-980⁶, then analysis will proceed.
- (4) If the R^2 is less than 0.990, or the RPD for any standard is >15 percent (the lowest concentration standard is >25 percent), 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 0.990, each data point will be evaluated to determine any errors.
 - (b) Method adjustments will be made and the calibration repeated.

(5) If the calibration fails, help will be requested from within the organization or calibration standards will be remade.

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 ± 15 percent of the expected value. If the QC calculated value is within the tolerance range, then the test method will proceed. 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.

g. After any maintenance action to the instrument, two QC samples must have calculated values within the ± 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. TOP 08-2-500A¹⁴ outlines one method of conducting receipt inspection. 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.

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 identification and tracking. The TICN will be marked on all test articles in a location that will not interfere with test procedures. 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. Purity certification will use one of the following methods: freezing point depression, nuclear magnetic resonance, or GC analysis documented for each lot. Advanced threat (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 as-received. 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 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 with a current calibration sticker (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 test items will be stored in a secure, environmentally-controlled location and protected from unrelated environmental contaminants and degradation.

4.7 Chemical Kinetics.

a. General.

(1) This test determines the time it takes for a liquid decontaminant to neutralize a chemical contaminant in a reaction vessel. The purpose of this testing is to provide a screening mechanism when multiple decontaminant candidates are being tested or to determine the neutralization of a chemical agent by a decontaminant in an ideal situation. This test can also be used to determine if effluent from a decontamination operation can be neutralized.

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

(3) When other agents (ATs) and decontaminants are used in testing, the methodology used to test the kinetics and select a quenching solution will be included in the test plan and details will be included in the test report.

b. Historical methods used for CWAs were as follows:

(1) Freshly-prepared decontaminant (50 milliliters (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.

(2) The neutralizing reaction will be initiated by adding 1.00 mL of agent to the decontaminant. The time (t) will be noted (t = 0).

(3) The stirring rate will be adjusted as necessary to ensure complete mixing.

(4) At measured intervals starting at t = 2 minutes***, a 50- μ 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.

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

(b) For the 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 freebase form needed for complete extraction into the chloroform.

c. Using a micropipette, 1.0 mL of the chloroform layer will be transferred to an autosampler vial.

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

e. The following data will be recorded:

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

*** The standard measurement intervals will be 2 minutes for the first 10 minutes of the trial, and then every 5 minutes thereafter until a total of one hour has elapsed after the agent addition.

(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.

NOTE: 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. Agent neutralization 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. Non-Traditional Agent (NTA) byproducts as a result of decontamination processes have not been identified.

TABLE 1. COMMON CHEMICAL WARFARE AGENT (CWA) BYPRODUCTS.

AGENT ^a	BREAKDOWN MECHANISM	BYPRODUCT	CAS NUMBER
GD	Hydrolysis	Pinacolyl methylphosphonate	616-52-4
		3, 3-Dimethyl-2-butanol	464-07-3
		Methylphosphonic acid (MPA)	993-13-5
HD	Oxidation and/or elimination	Mustard sulfoxide	5819-08-9
		Mustard sulfone	471-03-4
		Divinyl sulfone	77-77-0
		Divinyl sulfoxide	1115-15-7
	Hydrolysis	Thiodiglycol	111-48-8
		2-Chloro-2-hydroxyethyl sulfide	693-30-1

TABLE 1. CONTINUED

AGENT ^a	BREAKDOWN MECHANISM	BYPRODUCT	CAS NUMBER
VX	Hydrolysis	Ethylmethylphosphonic acid	1832-53-7
		MPA	993-13-5
		2-Diisopropylamino ethanethiol	5842-07-9
		Methylphosphonothioic acid o-ethyl ester	18005-40-8
		Hydrogen s-[2-(diisopropylamino)ethyl] methylphosphonothiolate	73207-98-4

^aGD – 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 plan, 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 (not all contaminant that has been absorbed can be extracted) 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, panels, component assemblies, or whole test items, but a flat surface is necessary.

NOTE: Coupons may represent the range of surfaces inherent to the items undergoing decontamination operations (e.g., vehicles, vans, weapons.) or equipment materials.

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 standard 5.1 centimeter (cm) diameter contact sampler usually consists of a latex dental sampler, an aluminum foil 5.1 cm diameter circle, and a 1 kilogram (kg) weight that is 5.1 cm in diameter.

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.

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 plan. 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 applied provides the same pressure.

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. Provide rationale in the test plan for the selection and validation of the chosen material.

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 (a calculation using the RH and temperature). However, RH and AH should always be reported.

e. The use of AH (shown in Equation 1 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.

$$H(T, RH) = \begin{cases} \frac{RH \cdot 13.2238 \cdot \exp\left(\frac{17.27 \cdot T}{T + 237.3}\right)}{T + 273.16} & T > 0^\circ\text{C} \\ \frac{RH \cdot 13.2238 \cdot \exp\left(\frac{21.875 \cdot T}{T + 265.5}\right)}{T + 273.16} & T < 0^\circ\text{C} \end{cases} \quad (\text{Equation 1})$$

where:

H = absolute humidity (g water/m³).

RH = relative humidity (%).

T = temperature (°C).

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

TEMPERATURE (°C)	ABSOLUTE HUMIDITY (g H ₂ O/m ³ air)		
	RELATIVE HUMIDITY	RELATIVE HUMIDITY	RELATIVE HUMIDITY
	10 percent	50 percent	90 percent
-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 plan environmental condition requirements, the test articles will be environmentally conditioned in a conditioning chamber or the test fixture.

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 g/m² (1 mg/cm²). The application of the agent may require multiple drops of varied sizes up to a single large drop as defined in the test plan. Document (quantitatively or photographically) the appearance of the droplets at the time of application and before applying the decontaminant.

h. During testing, the test article should remain uncovered when representing field conditions. An alternative test method (ECBC TR-980) describes covering the test article to minimize headspace and reduce evaporation. The use of one of the alternatives should be described in the test plan. The contaminated test article will be allowed to weather/age for a time

specified in the test plan. The test article should be maintained at the environmental conditions that are required for contaminant application.

i. As required by the customer and outlined in the test plan, a pre-rinse of the test articles will be performed after the weathering period and before applying the decontaminant.

j. 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.

k. 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 ATP 3-11.32 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: Historically an approximate decontaminant/agent ratio is 50:1 (mass of decontaminant to mass of agent) was used. This ratio can be modified based on manufacturer recommendations.

l. Decontaminant will reside on the test article for a default time of 30 minutes, or a time specified by the manufacturer or in the test plan.

m. The test article will be rinsed with water and allowed to dry until no visible liquid is present unless otherwise specified in the test plan. All surfaces of the test sample will be rinsed. The mechanism for applying the rinse will depend upon the requirements coordinated with the customer. If possible, the rinse may be applied to replicate the M12 or M26 decontamination apparatus.

n. This water rinse represents the ATP 3-11.32 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.

o. Samples of the rinsate may be required for analysis and mass balance calculations. The second rationale for the water rinse is to remove decontaminant before starting the contact sampling or coupon extraction as the decontaminant may interfere with the analytical instrumentation for sample analysis.

p. A 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). An equal sized piece of aluminum foil will be placed on top of the sampler.

q. A 1-kg weight will be placed on the sampler for 15 minutes, or as specified by the test plan. This represents one touch. Additional touches of 15 minutes each may be performed up to

a total of 1 hour (or four touches)⁶. The four touches at 15 minute intervals is the recommended default method unless a different touch schedule is outlined in the TEMP. If there is a different schedule in the TEMP, then the test plan should accommodate that schedule.

r. 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 hour minimum extraction time. If the test item is a coupon, the coupon will also be extracted for residual agent analysis.

NOTE: For residual agent absorbed into the surface of coupons, see Paragraph 4.9.2.

s. 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.

t. 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) 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 after the decontamination process. 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.q).

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.s, will be reported:

(1) Test sample GC results from the liquid used to extract the contaminant from the coupon.

(2) Each coupon's SICN.

(3) Visual observations of coupon surface and condition before extraction.

4.10 Material Compatibility Tests.

a. Materials to be tested will be specified in the test planning documents.

b. pH Test. The pH of the decontamination solutions will be measured.

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) International Standard G31-72(2004)¹⁵.

(2) Strip coupons (measuring 35 × 50 × 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 hours.

(5) After 24 hours 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 materials that are not elastomers or thermoplastics).

(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 International Standard D4762-11a¹⁶ also apply.

(2) All coupons will be cleaned by washing with soapy water followed by a thorough 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 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 International Standard D471-12¹⁷.

(3) The Shore A hardness test will be performed IAW ASTM International Standard D2240-05(2010)¹⁸.

(4) Coupons will be cut in the form of bars measuring 25 × 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 hours 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 International Standard D543-06¹⁹.

(3) The hardness test will be performed IAW ASTM International Standard D785-08²⁰.

(4) The haze and transmittance test will be performed IAW ASTM International Standard D1003-11e1²¹ 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 hours or as defined by the test plan).

(d) After 24 hours (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, 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. The same operator should perform these operations to minimize the variability of the pressure.

(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 concept of operations 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.

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 International Standard F502-08²² and ASTM International Standard D3363-05(2011)e2²³.

(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 (~25 °C) for 24 hours.

(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 inch.

(3) Visual observations.

(4) Observations relating to the evolution of corrosion products. If the corrosion products are expected to be 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.

(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 hours.

4.11 Detector Compatibility Test.

a. The basic concept of detector compatibility testing is to answer two questions: do the detectors provide an incorrect response (false positive) when exposed to a decontaminant, and do the detectors provide an incorrect response (false negative) to CWA contamination after exposure or during exposure to a decontaminant.

(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 system evaluators and may include (but are not limited to) some or all of the following:

- (a) Improved Chemical Agent Monitor (ICAM).
- (b) M43A1 Chemical Agent Automatic Alarm Detector Unit.
- (c) M18A2 Chemical Agent Detector Kit.
- (d) M256A1 Chemical Agent Detector Kit.
- (e) Shipboard Chemical Agent Monitor - Portable.
- (f) Improved Point Detection System (IPDS).
- (g) AP4C Vapor and Liquid Agent Detector.
- (h) Lightweight Chemical Detector 3.
- (i) Joint Chemical Agent Detector.
- (j) M8 and M9 detector paper and tape.
- (k) M272 Water Testing Kit.

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- (l) Chemical biological MS.
- (m) Biological aerosol warning sensor.
- (n) Handheld assay.
- (o) Automatic Chemical Agent Detector and Alarm (ACADA).

(3) Detector/alarm post-test functionality checks will be conducted and the results recorded.

b. False Positive Test.

(1) A false positive is defined as a detector alarm to a decontaminant when no agent is present.

(2) The decontaminant must be prepared IAW manufacturer's or test sponsor's instruction as outlined in the test plan. Decontaminant wipes may require a packet to be opened to remove the wipe. The decontaminant will be placed in a container in a fume hood for exposure to the detector being tested.

NOTE: In the test planning process, these procedures will require significant coordination with the test sponsor and the evaluation community to ensure that detectors are used in an operationally correct manner so that valid data will be produced. Some detectors require a vapor source (e.g., ICAM or IPDS) and others require droplet exposure (e.g., M8 or M9 paper).

(3) The inlet of the vapor detector will be placed above or near the decontaminant specimen surface to collect the sample vapor. This step may require special apparatus to be designed and built to achieve this step. Coupons of various substrates may also be used to present liquid decontaminants for exposure.

(4) The detector will be allowed to sample until the unit alarms or until the detector sampling time is reached (up to 5 minutes if no sampling time is specified by the manufacturer or evaluator). Precautions will be taken to prevent gross contamination of the detectors. Spectra or recordings may be taken when appropriate.

(5) All detectors responses 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 Test.

(1) A false negative is defined as a detector failing to alarm to an agent when a decontaminant is present.

(2) Vapor Detectors

(a) The detector must be allowed to come to a ready state in a clean air environment.

- (b) The detector must be confidence checked at the beginning and end of each trial day. Record the confidence check results.
 - (c) The vapor generator must be allowed to bring the agent to a steady state and have a referee confirmation that the challenge level of the vapor is at the desired level.
 - (d) Challenge the detector in the agent laden airstream. If the detector alarms, immediately remove from the agent challenge and place back in the external clean air source for a period of 120 seconds. Record the time to alarm and the clear down time. This step is performed as a positive control of detector performance.
 - (e) If no alarm or the wrong alarm occurs the detector should be allowed to continue sampling for up to 30 minutes, or until a positive response is achieved. The detector should then be moved back to the external clean air for a period of at least 120 seconds. Record the lack of alarm or any incorrect alarms.
 - (f) Apply the decontaminant being tested to a new (never used) agent contaminated coupon. Expose the detector to the coupon for a time described in the test plan (recommend a minimum of 60 seconds). Record any alarms.
 - (g) Repeat the steps in Paragraph 4.11.c(1)(d) and record any lack of alarm or any alarms and subsequent clear down time.
 - (h) Repeat the steps in Paragraphs 4.11.c(1)(a) through 4.11.c(1)(g) for the required number of replicate trials.
- (3) Non-vapor Detectors (usually color change paper tickets):
- (a) Determine detector lot functionality with three replicate exposures to the agent being tested. Record the response/color change.
 - (b) Prepare the decontaminant being tested. Apply the decontaminant to the surface of a new (never used) coupon of the material or substrate being tested.
 - (c) Place a detector ticket on a portion of the coupon with the decontaminant under the ticket (Figure 1).

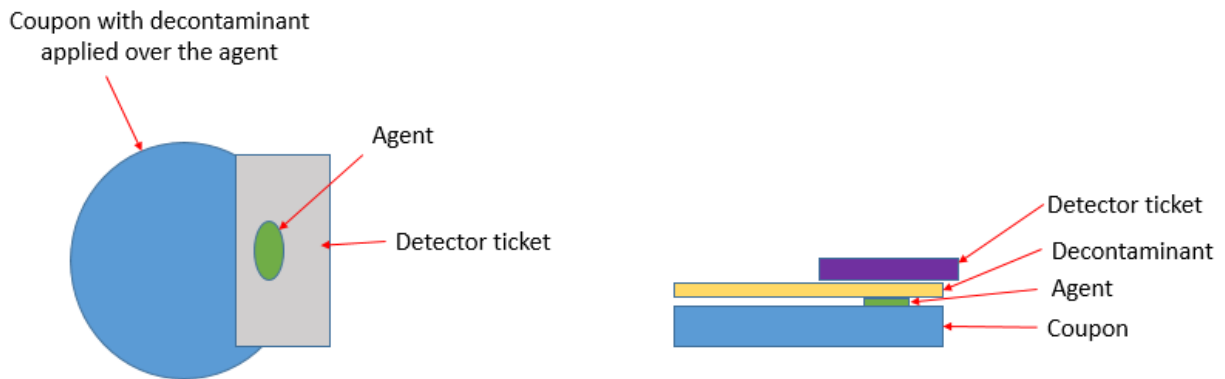


Figure 1. Nominal representation of detector ticket on coupon.

- (d) Record any response after the appropriate time for the detector ticket being tested.
- (e) If the response was positive, repeat the steps in Paragraphs 4.11.c(2)(a) through 4.11.c(2)(d) with the next agent/coupon material combination.
- (f) If the response was negative or the incorrect color was observed repeat the steps in Paragraphs 4.11.c(2)(a) through 4.11.c(2)(d) with a new piece of paper. These repetitions will be conducted at intervals (of additional coupon drying time) outlined in the test plan, with a maximum recommended coupon drying time of 30 minutes.
- (g) For subsequent samples, the detector paper is applied to a portion of the coupon that has not yet been sampled.
- (h) Repeat all steps to achieve the required number of replicates for each agent/decontaminant/substrate outlined in the test plan.

4.12 Pot Life Test.

- a. Pot life is defined as the period of time the decontaminant remains effective 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.
- b. 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 caused by changes in the product's physical state or chemical composition during a typical mission period. The test duration shall be 12 hours, as required by the TEMP, or as otherwise specified by the manufacturer because of product limitations.

(2) The test will be conducted at low temperature (~-32 °C), ambient temperature (21 °C), and high temperature (~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, other than the decontaminant.

c. Procedure.

(1) Properties or performance characteristics used to evaluate continued decontaminant efficacy will be identified in the test plan. A quantitative measure of the 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). Samples will be taken at time intervals outlined in the test plan, 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). Samples will be taken at time intervals outlined in the test plan, 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 hours) 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 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. Procedures.

(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) One hundred five samples, five replicates for each condition and sample period, 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. Each test sample container must contain sufficient product to perform all characterization testing prescribed herein.

(3) Thirty-five 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 seven months or other time period as coordinated with the customer, five product samples will be drawn from the original unopened test containers (packaging), and the decontaminant's 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) of the selected quantitative measure. 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. DATA REQUIRED.

Data required are listed under the individual subtests in Section 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 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.

A.1 MATERIAL SELECTION.

Coupons will be 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 the system evaluators. 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 materiel 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.

A.2 SAMPLE LISTING OF HIGH-PRIORITY MATERIALS.

NOTE: Coupon materials are not limited to the following.

- a. Chemical Agent-Resistant Coating (CARC) on steel (tactical vehicles).
- b. Aircraft topcoat paint on aluminum (aircraft).
- c. Low-infrared paints on aluminum or steel (aircraft and ships).
- d. Ship deck antiskid coating on steel.
- e. Polyurethane, epoxy, and alkyd paints on metals (commercial vehicles).
- f. Aluminum alloys, forged and cast (aircraft surfaces and 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[®] (SABIC Innovative Plastics, Pittsfield, Massachusetts (aircraft canopy/window materials, tactical vehicles)).

APPENDIX A. COUPON MATERIAL.

- p. Glass (commercial vehicles, tactical vehicles).
- q. Butyl rubber (mask, gloves/boots).
- r. Silicon rubber (M40 mask).

APPENDIX B. ABBREVIATIONS.

ACADA	Automatic Chemical Agent Detector and Alarm
AD No.	accession number
AED	atomic emission detection
AH	absolute humidity
AR	Army Regulation
ASTM	American Society for Testing and Materials
AT	advanced threat
ATEC	US Army Test and Evaluation Command
ATP	Army Technical Publication
°C	degrees Celsius
CARC	chemical agent-resistant coating
CAS®	Chemical Abstracts Service®
cm	centimeter
CWA	chemical warfare agent
DA	Department of the Army
DED	detailed equipment decontamination
DTIC	Defense Technical Information Center
ECBC	Edgewood Chemical and Biological Center
FID	flame ionization detector
FPD	flame photometric detector
g/m ³	grams per meter cubed
GC	gas chromatograph
GD	soman
HD	distilled mustard
IAW	in accordance with
ICAM	Improved Chemical Agent Monitor
IPDS	Improved Point Detection System
ISO	International Organization for Standardization
kg	kilogram
LC	liquid chromatograph
M	molarity
m/s	meters per second
mL	milliliter

APPENDIX B. ABBREVIATIONS.

mm	millimeter
MPA	methylphosphonic acid
MQL	minimum quantification limit
MS	mass spectrometer
NTA	non-traditional agent
ORI	operational readiness inspection
OTA	Operational Test Agency
PAM	pamphlet
QA	quality assurance
QC	quality control
R ²	correlation coefficient
RH	relative humidity
RPD	relative percent deviation
SCG	Security Classification Guide
SDS	safety data sheet
SEP	System Evaluation Plan
SICN	sample item control number
SOP	standing operating procedure
SST	solid sorbent tube
t	time
TEMP	Test and Evaluation Master Plan
TICN	test item control number
TIR	test incident report
TO	Test Officer
TOP	Test Operations Procedure
TRR	test readiness review
TTOP	Test and Evaluation Capabilities and Methodologies Integrated Process Team (TECMIPT) TOP
VX	persistent nerve agent

APPENDIX C. REFERENCES.

1. TOP 08-2-060, Post-Decontamination Vapor Sampling and Analytical Test Methods, 12 August 2015.
2. TOP 08-2-065, Developmental Testing of Liquid and Gaseous/Vaporous Decontamination on Bacterial Spores and Other Biological Warfare Agents on Military-Relevant Surfaces, 11 February 2016.
3. AR 50-6, Chemical Surety, 16 April 2018.
4. AR 190-59, Chemical Agent Security Program, 10 April 2012.
5. DA PAM 385-61, Toxic Chemical Agent Safety Standards, 13 November 2012.
6. ECBC-TR-980 The Chemical Contaminant and Decontaminant Test Methodology Source Document, Second Edition, July 2012.
7. ATP 3-11.32, Multiservice Tactics, Techniques, and Procedures for Chemical, Biological, Radiological, and Nuclear Passive Defense, December 2015.
8. ATEC Regulation 73-1, Test and Evaluation: Developmental Test Policy, 13 May 2019.
9. DA PAM 73-1, Test and Evaluation: Test and Evaluation in Support of Systems Acquisition, 11 March 2013.
10. MIL-HDBK 310, Global Climatic Data for Developing Military Products, 23 June 1997.
11. ISO 17025:2017, General Requirements for the Competence of Testing and Calibration Laboratories, November 2017.
12. ISO 5725, Accuracy (trueness and precision) of Measurement Methods and Results, 1994.
13. AR 385-10, The Army Safety Program, 24 Marc 2017.
14. TOP 08-2-500A, Receipt and Inspection of Chemical - Biological (CB) Materiel, 31 August 2017.
15. ASTM G31-72(2004), Standard Practice for Laboratory Immersion Corrosion Testing of Metals, 1972, revised 2004.
16. ASTM D4762-11a, Standard Guide for Testing Polymer Matrix Composite Materials, 2011, revised 2011.
17. ASTM D471-12, Standard Test Method for Rubber Property - Effect of Liquids, 2012.

APPENDIX C. REFERENCES.

18. ASTM D2240-05(2010), Standard Test Method for Rubber Property - Durometer Hardness, 2005, revised 2010.
19. ASTM D543-06, Standard Practices for Evaluating the Resistance of Plastics to Chemical Reagents, 2006.
20. ASTM D785-08, Standard Test Method for Rockwell Hardness of Plastics and Electrical Insulating Materials, 2008.
21. ASTM D1003-11e1, Standard Test Method for Haze and Luminous Transmittance of Transparent Plastics, 2011.
22. ASTM F502-08, Standard Test Method for Effects of Cleaning and Chemical Maintenance Materials on Painted Aircraft Surfaces, 2008.
23. ASTM D3363-05(2011)e2, Standard Test Method for Film Hardness by Pencil Test, 2005, revised 2011.

APPENDIX D. APPROVAL AUTHORITY.

CSTE-CI

7 January 2021

MEMORANDUM FOR

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

SUBJECT: Test Operations Procedure 08-2-061B, Chemical Decontaminant Testing,
Approved for Publication

1. Test Operations Procedure (TOP) 08-2-061B, 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.

2. Scope of the document. 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 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) and advanced threat agents. 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.

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

4. Comments, suggestions, or questions on this document should be addressed to U.S. Army Test and Evaluation Command (CSTE-CI), 6617 Aberdeen Boulevard-Third Floor, Aberdeen Proving Ground, MD 21005-5001; or e-mailed to usarmy.apg.atc.mbx.atc-standards@mail.mil.








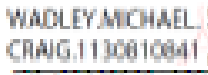




ZWIEBEL MICHAEL J.
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MICHAEL J. ZWIEBEL
Director, Directorate for Capabilities
Integration (DCI)

APPENDIX D. APPROVAL AUTHORITY.

TECMIPT Concurrence Sheet for the TECMIPT Test Operations Procedure (TOP) 08-2-061B, Chemical Decontaminant Testing

The Contamination Mitigation CAPAT recommends approval of the TTOP 08-2-061. If a representative non-concurs, a dissenting position paper will be attached.

Organization	Signature	Date
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Joint Requirements Office for Chemical, Biological, Radiological and Nuclear Defense (JRO-CBRND)	 Lt Col Paul M. McManus, Army	15 Oct 19
Joint Science and Technology Office (JSTO)	 Tom Yuzuk	09 Oct 17
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Operational Test and Evaluation Force (OPTEVFOR)	 CAPT M. R. Struers	23 Nov 19
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Forward comments, recommended changes, or any pertinent data which may be of use in improving this publication to the following address: Policy and Standardization Division (CSTE-CI-P), U.S. Army Test and Evaluation Command, 6617 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: <https://www.atec.army.mil/publications/documents.html>, or through the Defense Technical Information Center, 8725 John J. Kingman Rd., STE 0944, Fort Belvoir, Virginia 22060-6218. This document is identified by the accession number (AD No.) printed on the first page.