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13. SUPPLEMENTARY NOTES

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This TOP supersedes TOP 8-2-060 Post-Decontamination Vapor Sampling and Analytical Test Methods, dated 12 August 2015.

Marginal notations are not used in this revision to identify changes, with respect to the previous issue, due to the extent of the changes.

14. ABSTRACT

This TOP contains the procedures for the measurement of contaminant vapor present after an item is decontaminated that could pose an exposure hazard to unprotected personnel. The chemical contaminants may include chemical warfare agents (CWAs) or their simulants, nontraditional agents (NTAs), toxic industrial chemicals (TICs), and toxic industrial materials (TIMs). A vapor collection system is used to capture coupon off-gassing. Concentration is measured using chromatographic analytical methods. This laboratory-scale test method uses a range of test articles from coupons, panels, and small fielded equipment items.

15. SUBJECT TERMS

Vapor hazard; vapor sampling; chemical warfare agent; CWA; simulants; nontraditional agent; NTA; toxic industrial chemical; TIC; toxic industrial material; TIM; coupon; contamination; decontamination; toxic load

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US ARMY TEST AND EVALUATION COMMAND
TEST OPERATIONS PROCEDURE

*Test Operations Procedure 08-2-060A 9 August 2021
DTIC AD No.

POST-DECONTAMINATION VAPOR SAMPLING AND ANALYTICAL TEST METHODS

Page

PARAGRAPH

APPENDIX

* This TOP supersedes TOP 08-2-060 Post-Decontamination Vapor Sampling and Analytical Test Methods, dated 1 July 2015.

1. SCOPE.

a. The Post-Decontamination Vapor Sampling and Analytical Test Methods Test Operations Procedure (TOP) covers the procedures necessary to collect residual contaminant vapor from the test article after decontamination, analyze the contaminant concentration, and perform calculations and present the data.

b. These procedures describe a vapor collection system to contain and sample off-gassing vapor. Appendices C, D, and E describe procedures for analyzing the residual vapor data collected to determine human toxicological effects. These laboratory-scale test methods use a range of test articles from small coupons, larger coupons or panels, and small fielded equipment items. Sizes can range from 1 centimeter (cm) in diameter through 50 × 20 × 15 cm and weighing 6.8 kilograms.

c. There are two types of vapor collection systems for conducting vapor sampling:

(1) The first type of vapor collection system is the vapor microchamber, which is constructed of a base material and a lid. The lid is sealed before each test to ensure there is no cross-contamination between tests. The lid is held in place with a pressure-tuned armature that ensures a complete seal between the lid and base when engaged. The microchamber was designed to facilitate a laminar single-pass airflow configuration where the air is swept across the coupon and immediately directed to the vapor sampler.

(2) The second type of vapor collection system is the small item vapor (SIV) chamber. The SIV is a larger chamber up to 1 cubic meter (m^3) with turbulent airflow that creates a well-mixed environment. The vapor sampler then samples a small portion of the chamber volume. The SIV has a door allowing placement of the test article. The door must be sealed before vapor sampling.

d. Commonly used bag-and-sample vapor hazard test methods (not described in this TOP) are not appropriate to determine the presence of a vapor hazard. Bag-and-sample test methods provide an indication that off-gassing may be present, but because the airflow rate is uncharacterized and the air volume is uncontrolled, the data collected cannot provide an accurate analysis of the vapor hazard.

e. The acronym, chemical and biological (CB), is used in this document, rather than nuclear, biological, and chemical (NBC), to reflect current terminology in use within the Department of Defense (DOD).

1.1 Purpose.

a. This TOP contains the procedures for collecting and measuring residual contaminant vapor present after the decontamination process that could pose a vapor exposure hazard to unprotected personnel. The agency that establishes the vapor exposure hazard levels is the U.S. Army Public Health Center (APHC). These procedures use a vapor collection system to capture samples of off-gassed contaminant vapor and chromatography to analyze the concentrations of the samples. Analyzing residual contamination enables testers to characterize the vapor source terms (emission factor and emission rate) of a test article after conducting a decontamination process. Chemical contaminants can include chemical warfare agents (CWAs) or their simulants, nontraditional agents (NTAs), toxic industrial chemicals (TICs), and other hazardous chemicals.

b. Residual vapor data are used to characterize the emission profile of the test article and calculate toxic load (TL) values. The TL values can be applied to a range of operational scenarios to determine the vapor exposure hazard to unprotected individuals. The operational scenarios are provided by organizations that require the use of decontaminated equipment or infrastructure to perform mission-essential (ME) functions.

c. Residual vapor data are used to calculate the vapor source terms after treatment with a decontamination system (decontaminant and/or applicator) used against CWAs, simulants, NTAs, TICs, or other hazardous chemicals.

1.2 Limitations.

a. The procedures contained in this TOP only provide an analysis of the ability of a decontamination system (decontaminant and/or applicator) to reduce a vapor hazard. Residual liquid testing is addressed in TOP 08-2-061A[1][**].

b. This TOP includes procedures for analyzing the decontamination of equipment and infrastructure. Systems and procedures intended for skin or personnel decontamination are not included.

c. This vapor test method applies only to decontaminated items that can subsequently be placed in a vapor collection system (with a characterized airflow, temperature control, and controlled volume) and will produce a vapor. Outdoor scenarios or poorly mixed indoor scenarios will require other modeling methods which are beyond the scope of this TOP.

[**] Superscript numbers and letters correspond to those in Appendix F.

2. FACILITIES AND INSTRUMENTATION.

2.1 Facilities.

Item	Requirement
Chemical agent storage facility.	Constructed to ensure safe and secure storage, handling, analysis, and decontamination of chemical agents.
Chemical agent test facility or chamber.	Constructed to allow test item contamination and decontamination and extended residual hazard sampling of small items of equipment deliberately contaminated with chemical agent/simulant in a temperature and humidity controlled environment. The chamber must have sufficient volume to allow free air circulation around the test item. Ability to control temperature, relative humidity (RH), and wind speed is required.
Chemical agent test laboratory.	Equipment, interior surfaces, tools, and waste must be easily decontaminable. Must have certified fume hoods for the containment of toxic chemicals. All exhaust air must be filtered and monitored to prevent any chemical release to the environment. The facility design should ensure safe transfer, handling, challenge, and disposal of chemical contaminants, decontaminating solutions, and solvents.
Off-gas box (microchamber or SIV)	Off-gas boxes are rigid containers for holding test articles for agent vapor desorption and sampling (precise interior dimensions must be known). Off-gas boxes larger than 30.5 × 30.5 × 25.4 cm require mixing fans to ensure adequate airflow across the test article. All off-gas boxes require, at a minimum, controllable temperature (humidity control is also desirable). The off-gas box must be made of inert materials, e.g., stainless steel, T6061 aluminum, or equivalent. The construction material may be treated to further minimize interactivity with contaminant vapors [e.g., Sulfinert® treatment (Restek Corporation, Bellefonte, Pennsylvania)]***

*** 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.

2.2 Instrumentation.

These values are minimum requirements. Actual instrumentation may have greater precision or accuracy, and actual values must be reported.

Parameter	Measuring Device	Permissible Error of Measurement
Residual contaminant vapor.	MINICAMS® (OI Analytical, College Station, Texas) or other equivalent near real-time (NRT) sampler.	± 15 percent of the contaminant vapor concentration in milligram per cubic meter (mg/m^3), or ± 25 percent of the concentration at the minimum quantification limit (MQL).
Contamination droplet volume.	Calibrated repetitive pipette, syringe, or computerized dispensing system.	± 10 percent of the droplet volume (within the range specified for the contaminant and applicator).
Residual contaminant in samples from solid sorbent tubes (SSTs), or equivalent.	Gas chromatograph (GC), liquid chromatograph (LC), flame ionization detection (FID), flame photometric detection (FPD), or equivalents.	± 15 percent of the mass/volume of the contaminant per sample, or within ± 25 percent of the device MQL.
Temperature.	Thermocouple, remote temperature device, thermometer, or equivalent.	± 3°Celsius (°C).
RH.	Hydrometer or equivalent.	± 5 percentage points.
Differential pressure (ΔP) for test chambers or fixtures only.	Pressure transducer.	± 0.09 millimeters mercury (Hg), or ± 12 Pascal (± 0.05 inch of water gauge (iwg)).
Air velocity (chamber, fixture, or off-gas box).	Hotwire anemometer or equivalent.	± 0.1 meters per second (m/sec).
Airflow rate (off-gas box).	Mass flow controller.	± 1 percent of full scale.
Contaminated surface area.	Digital color camera.	Image resolution adequate to capture surface area. Pixels must be convertible to cm^2.

Parameter	Measuring Device	Permissible Error of Measurement
Visual record (still).	Digital color camera.	Image resolution and frame capture rate adequate to document details of testing.
Visual record (motion).	Digital video camera.	Resolution adequate to document details of testing.
Contamination density or challenge level (grams per square meter (g/m^2)).	A control coupon will be used for the measurement of the actual contamination density applied.	Sample must be ± 15 percent of challenge target.
Chemical agent mass from vapor samples (microgram (µg)).	MINICAMS®, GCs, high-performance liquid chromatography (HPLC), liquid chromatography (LC), spectrophotometer, or equivalent.	Sample must be ± 15 percent of calibration standard.
Chemical agent mass from liquid samples (µg).	GC, HPLC, LC, spectrophotometer, or equivalent.	Sample must be ± 15 percent of calibration standard.

2.3 Test Controls.

These values are minimum requirements. Actual values must be reported.

Item	Requirement
Quality control (QC) check shots for MINICAMS® or equivalent.	Concentration, in mg/m^3, ± 15 percent, or at the MQL ± 25 percent.
Process quality samples for GC, LC, or equivalent. These may be samples of a known mass or periodic calibration standards.	Contaminant in mass/volume, ± 15 percent, or at the MQL ± 25 percent.

3. REQUIRED TEST CONDITIONS.

3.1 Test Planning.

a. 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 and reduce the scope of the current test effort. Review relevant Standing Operating Procedures (SOPs) and other procedures for applicability, completeness, and adequacy.

b. Based on the requirements in the test planning documentation [e.g., Test and Evaluation Master Plan (TEMP), System Evaluation Plan (SEP), etc.], develop a test plan that will include at minimum, a test design, requirements or criteria, execution matrix, detailed procedures, quality assurance (QA)/QC measures, data management, data analysis, and results presentation.

c. The test plan must be prepared, coordinated, and approved before any testing begins. The test procedures described herein must be used as the basis for the test plan. The procedures may require modification for unique items or materials to satisfy specific testing requirements in the TEMP, SEP, or other program documentation. Deviations from these procedures will be coordinated and approved with all concerned organizations in advance of any testing, after 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.

d. Test Design. A design of experiment (DOE) is recommended based on requirements and coordination with the test sponsor. The DOE will specify the necessary sample size for statistical analysis. If a DOE is not developed, then the recommended number of replicates is five coupons per test condition and five dose confirmation samples per contamination set for minimal statistical reliability. This recommendation is based upon how fast a test matrix can grow when there multiple surface types, multiple agents, multiple environmental test conditions, and finally trial replicates. A test with three chemical agents, five different materials (with five replicate samples for each material), three environmental conditions, and three trial replicates will require 3x5x5x3x3 or a total of 675 coupons. Requiring any more than a minimum of five replicates will exponentially increase the amount of testing with subsequent increases in test cost and schedule length. A test matrix will be designed to maximize available resources to meet stated objectives and criteria. If the sample size is less than recommended, statistical confidence limits will be calculated, and reported as a test limitation.

e. Security. Security considerations will be adequately determined and provided for, as applicable to each test program. The security classification guide (SCG) and the installation operations security (OPSEC) requirements will be followed.

3.2 Safety.

Test site specific SOPs and/or other safety documents applicable to the specific item and tests being conducted must be reviewed.

3.3 Environmental.

All test site specific environmental requirements for local, state, and federal approvals will be met and documented.

3.4 QA/QC.

a. A QA plan, as required by the test site, must be prepared for each test program to ensure that all variables that can be controlled are controlled and that appropriate records are kept throughout the duration of testing. Variables that cannot be controlled must be identified in the test plan. Test variables include, but are not limited to: purity and stability of CB agents and simulants used, purity and stability of decontaminants, calibration and maintenance of instrumentation and disseminators, accuracy and precision of the laboratory instruments, and quality and uniformity of all test samples.

b. The condition of the test item at the time of testing is an important test variable. The test item must be inspected. Inspection data, certificates of compliance, or similar documentation must be reviewed to ensure that exterior surfaces, finishes, and packaging meet specifications. The item must be tested in as-received condition, matching its condition when issued to Warfighters in the theater of operations as closely as possible.

c. Chemical agent must have a purity of 90 percent or greater (VX may only achieve a purity between 80 and 90 percent), and a purity certificate must be available. 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 VX, which must be purity-analyzed within 3 months). Purity certification will use one of the following methods: freezing point depression, nuclear magnetic resonance (NMR) analysis (preferred method), or GC analysis documented for each lot.

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

4. TEST PROCEDURES.

The contaminated surface area (i.e., the area wetted by the contaminant) must be measured as accurately as possible using a camera, or may be determined by estimation. Estimating the contaminated surface area (based on the coupon and relative coverage sizes) introduces additional errors in subsequent calculations. Any estimating procedures must be documented. For camera measuring, the highest resolution that can be graphically resolved should be determined and documented. The camera should be calibrated for software calculation of the contaminated area (e.g., pixels/cm^2). This value is used in the data analysis calculations to determine loading factors.

4.1 Test Method Outline.

a. Receipt inspection (Paragraph 4.2).

b. Trial preparation (Paragraph 4.3).

c. Preconditioning procedure (Paragraph 4.4).

d. Contamination procedure (Paragraph 4.5).

e. Weathering/exposure procedure (Paragraph 4.6).

f. Decontamination process (Paragraph 4.7).

g. Vapor test procedure (Paragraph 4.8).

h. Residual contaminant test procedure (Paragraph 4.9).

i. Chromatographic analysis for contaminant (Paragraph 4.10).

4.2 Receipt Inspection.

a. The test articles (which may include coupons, panels, or small items of equipment) will be subjected to a visual receipt inspection in accordance with (IAW) applicable directives such as equipment user/technical manuals after arrival at the test site. Evidence of damage or irregularities to the test articles will be recorded in the laboratory recordkeeping 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 materials.

(5) Discoloration.

(6) Evidence of deterioration.

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 their reverse side with the TICN. The preferred method for marking the TICN is engraving. Other methods for labeling must consider the potential for analytical interference and record results of the methodology used to determine that no interference exists. The TICN and other pertinent information about the test article must be linked in the laboratory recordkeeping system.

c. If any items are determined to be unfit for testing, they will be rejected and replaced with items that are in suitable condition for testing.

4.3 Trial Preparation.

a. 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.

b. Candidate decontaminants will be prepared IAW the manufacturer's instructions. Fielded decontaminants will be prepared IAW applicable military technical manuals. Quality checks will be performed as necessary by routine analytical methods [such as pH (hydrogen-ion-concentration) measurement, titration, etc.]. The pot life as specified by the manufacturer will not be exceeded. This may require frequent preparation of the decontaminant during trial conduct.

c. Test fixtures and off-gas boxes will be turned on and allowed to equilibrate at the specified test conditions. All equipment will be operational before the start of test. Off-gas boxes should be monitored to ensure that no residual vapor remains to create a positive bias in data collected. The procedure for determining/verifying that the interior of the off-gas box is a well-mixed environment with the test item in place will be performed IAW the Chemical Contaminant and Decontaminant Test Methodology Source Document, Second Edition, ECBC-TR-980[1].

d. The test setup, labeling of vials, trays, jars, etc., and other associated pretest tasks will be completed.

e. Coupons/panels may require cleaning before testing to remove cutting oils or other preparation contaminants. The marked 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.

f. All calibrated instrumentation will have a current calibration date.

g. Timing charts for staggering contamination, decontamination, and other test events may be required. The timing charts will assist in minimizing data scatter that may be caused by subtle differences in coupon treatment.

h. The total length of vapor sampling [when using cumulative vapor samplers (e.g., SSTs)] will reflect a mission profile for the test program and will be delineated in the test plan. The default sampling length will be 12 hours. A vapor sampling plan will be developed to establish when vapor samplers should be replaced.

i. Positive and negative controls will be prepared as described in TOP 08-2-061A[2].

4.4 Preconditioning Procedure.

a. Any required preconditioning of the test articles described in the test planning documentation will be performed.

b. The positive and negative controls will be preconditioned using the same method for the test articles (as described in the test planning documentation).

c. The test articles will remain until ready to execute the next step, Paragraph 4.5, Contamination Procedure.

4.5 Contamination Procedure.

a. Test article contamination will be conducted as described in the test planning documentation.

b. Test articles will be photographed to record and determine the contamination surface area coverage. A dye may need to be added to the contaminant to allow for better visibility and determination of contaminant coverage on some test article materials. When photographs cannot be taken, especially for complex surface test articles, an estimate of the surface coverage will be made and recorded based on the size of the test article and relative size of the coverage.

4.6 Weathering/Exposure Procedure.

a. After contamination, weathering procedures will be conducted as described in the test planning documentation.

b. For testing using coupons/panels, each coupon/panel will be photographed to record and determine the contaminated surface area remaining after weathering/exposure. When photographs cannot be taken, especially for complex surface test articles, an estimate of the surface coverage will be recorded.

c. The test articles will be moved to the decontamination area at the end of the weathering/exposure period.

4.7 Decontamination Process.

a. The decontamination process will be conducted as described in the test planning documentation, based upon manufacturers' instructions for candidate decontaminants and IAW applicable tactics, techniques, and procedures for fielded decontaminants.

b. After decontamination, the test articles will be allowed to dry until no visible moisture is present.

c. Any visible degradation of the test article surface will be recorded.

4.8 Vapor Test Procedure.

a. The off-gas box temperature will be set if temperature conditioning is required. The off-gas box will be conditioned until the required temperature conditions are achieved before placing the test articles inside.

b. The test articles will be placed in the vapor off-gas boxes specifically designed for containing and collecting residual vapor.

c. The off-gas box will be sealed and the airflow initiated based on the vapor sampler being used (MINCAMS®, SST, or equivalent). The airflow rate to be used will be specified in the test plan. Initiation of the airflow through the off-gas box begins the sample collection time and is recorded as time zero (t_0).

d. When using SSTs, the vapor sampling plan outlined in the test plan (e.g., change-out at 30-minute, 1-hour, or 2-hour intervals) will be followed. Dual SSTs may be desirable to minimize loss of data. When dual SSTs are used, the SSTs will be sampling in parallel. Collected SSTs will be taken to an analytical laboratory for thermal desorption or liquid extraction of the contaminant from the sorbent. The desorbed or extracted sample will then be analyzed by GC / mass spectrometer (MS), LC/MS, or other analytical instrumentation, depending upon the nature of the contaminant.

e. The NRT sampling instrumentation calibration range and sampling cycle time will be determined based on the expected concentration of the vapor off-gassed from the test article. Concentrations from the higher end of the expected range will require a shorter sampling time to avoid detector saturation, which would result in a loss of data.

4.9 Residual Contaminant Test Procedure.

a. Additional information may be collected from coupons or panels after vapor sampling using an extraction process for residual contaminant.

b. Each coupon/panel will be placed in a container with extraction solvent. The size of the coupon/panel is an extremely important consideration in this procedure. The larger the test article size, the more solvent that will be required to extract the contaminant. The more extraction solvent used, the more diluted the contaminant will become, making it less likely to be detected. For most materials, the contaminated side will be placed face-up; however, if the material being tested floats, the sample will be placed face-down so that solvent contact occurs. Complete immersion of the test article will provide the best data.

c. The container will be sealed with a lid lined with Teflon® polytetrafluoroethylene [(PTFE) DuPont™, E.I. du Pont de Nemours and Company, Wilmington, Delaware].

d. In order to facilitate contaminant extraction, agitate the container by swirling for 30 seconds.

e. The test article will remain in the extraction solvent for 60 minutes. Other extraction durations may be used but the change from the TOP must be documented in the test plan and the rationale described.

f. At the end of the extraction period, the container will be swirled for 30 seconds before the lid is opened. After swirling, an analytical vial will be opened, and a clean pipette tip will be used to place an aliquot into the vial for analysis.

4.10 Chromatographic Analysis for Contaminant Procedure.

a. Samples will be analyzed following existing chromatographic procedures.

b. The samples may need to be diluted to concentrations within the analytical method calibration range. The dilution factor must be recorded to allow calculation of the sample concentration.

5. DATA REQUIRED.

a. Receipt inspection data (test article information, photographs, etc.).

b. TICN assigned to each test article.

c. Surface area and contaminated surface area of each test article (cm^2).

d. Contaminant (agent, simulant, or other) purity (percent).

e. Solvent purity (percent).

f. Data required from the preconditioning, contamination, weathering, and decontamination procedures outlined in TOP 08-2-061A[1].

g. Sampling time intervals (minutes:seconds).

h. Flow rate of samplers (milliliter per minute (mL/min)).

i. NRT sampler concentration range (mg/m^3).

j. Results of NRT quality check shots (mg/m^3).

k. Trial date, start time, and end time.

l. Residual contaminant vapor by NRT instrument (mg/m^3).

m. Mass of residual contaminant by SST (nanogram (ng)), if thermal desorption of the SST is performed.

n. Mass per volume of residual contaminant (ng/mL) if liquid extraction of the SST is performed.

o. Measured off-gas box temperature (°C) over time.

p. Measured off-gas box RH (percent) over time.

q. Air velocity above the test item (m/sec).

r. Decontaminant and decontamination procedures used.

s. Analytical instrument MQL, calibration range, and quality sample results (spikes, blanks, and standard) (ng).

t Any deviations from existing procedures in the test plan must be documented in the test report.

u. Contaminant delivery applicator data:

(1) Manufacturer name, model number, and volume dispensing range.

(2) Applicator specifications, including accuracy and any other conformance specifications.

(3) Calibration status (if applicable), including the last date calibrated.

v. Decontamination system data to include nomenclature, operational parameters used (e.g., flow rates, pressure, etc.), etc.

w. Decontaminant used, lot number, date of manufacture, and mixing procedures.

x. Off-gas chamber data:

(1) Description of the off-gas box.

(2) Off-gas box volume.

(3) Off-gas box temperature.

(4) Off-gas box humidity (as applicable).

y. Contaminated Surface Area Measuring Data (as applicable). Camera manufacturer, model number, resolution, and description of area measurement calculation and associated error with calculation, if known.

z. Sorbent Tubes Data (as applicable). SST supplier, description, part number, and sorbent used.

aa. Coupon or Panel Material Data.

(1) Stock material description (e.g., bar stock, sheet, etc.), manufacturer and/or supplier, and part number/lot number (as applicable).

(2) Any painting or coating applied to the material, including a description of the surface preparation method, the paint or coating used with manufacturer name and lot number, any primer used, method of application, resulting thickness on the material, date of preparation, and date of use (as applicable).

bb. Test Article Data. Description of each article, as applicable, including manufacturer and/or supplier, model number/serial number, article condition (new or used), if used, any history of use (if known).

6. PRESENTATION OF DATA.

6.1 Data Review.

a. The data may be tested for outliers during data analysis. A standardized method such as American Society for Testing and Materials (ASTM) Standard E0178[3], will be used. Although ASTM Standard E0178 discusses multiple methods to test for outliers, the method discussed in Paragraphs 6.1 through 6.2 of ASTM Standard E0178 is recommended. Data points determined to be outliers may be excluded from statistical data analysis and should be flagged as being excluded, but must be reported along with the appropriate data set.

b. Large data variances (high error percentages) are not always an indication of a poorly executed test and must be investigated in conjunction with the test sponsor before the data are rejected.

6.2 Data Acceptance Parameters.

a. The following are the set point tolerance ranges in which trials should be conducted in order for the data generated to be acceptable for use.

b. SST sampling time: 90 percent of the SST replacement intervals will be within ± 2 percent of the target sampling time.

c. Sampling flow rate: 90 percent of flow rate measurements will be within ± 5 percent of the target flow rate.

d. Temperature: 90 percent of temperature measurements will be within ± 5 °C of the target temperature.

e. RH: 90 percent of RH measurements will be within ± 5 percentage points of the target RH.

f. A summary will be presented for each vapor sampler data package by trial, which will include but is not limited to calibration values, tabular test sample results, QC process sample results, outliers, method used for outlier determination, graphical presentation of test sample results (concentration versus time), etc.

g. A graphical representation or summary table of each off-gas box temperature and RH versus time by trial will be presented.

h. The emission factor calculations will be performed as described in Appendix B. A table of these results will be produced.

i. TL calculations (Appendix E) will be performed. Using operational scenarios provided by the sponsor, and/or the Operational Test Agencies (OTAs), the TL calculations will be integrated to provide vapor hazard predictions.

j. Photographs will be used to document the agent spread and contaminated area on the test articles, and will be included in the report.

k. Examples of various data formats are provided in Appendices C, D, and E.

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APPENDIX A. GLOSSARY.

Term	Definition
Analytically Integrable	Using the rules of calculus to find the value of an integral.
Decontamination	The process of making material safe by absorbing, destroying, neutralizing, rendering harmless, or removing chemical contamination.
Gaussian Quadrature	A numerical method to find the value of an integral. Evaluation points of the function are chosen to minimize the error.
Heteroscedasticity	The quality of data having different variances through time.
Kinetics	Study of the rate at which events occur in a system of chemical reactions in relationship to surrounding environmental conditions.
Levenberg-Marquardt	A numerical method to perform least-squares curve fitting.
Mass Balance Model	A model that is developed using an equation that balances the mass entering and exiting the system.
Microchamber	A chamber with a small free-air volume that is used to collect data about the off-gassing of test articles after decontamination.
Numerical Integration	A method of calculating the value of an integral using a family of algorithms.
Off-gas Box	A fixture used to contain a test article. The box will have an associated vapor sampler or detector to sample any residual vapor off-gassing from the test article after decontamination.
Residual Contamination	Contamination left on a surface that has been decontaminated.
Simpson's Rule	A numerical method to find the value of an integral. A series of parabolas are fit to the evaluation points to calculate the area under the curve.
Simulant	Compound that has physical or chemical properties that are similar to a CWA and can be used to simulate the CWA for testing purposes.

APPENDIX A. GLOSSARY.

Term	Definition
Sponsor	The organization responsible for drafting, staffing, and revising capabilities documents. For this TOP, sponsors include Combat Developers.
ten Berge	A model of chemical exposure that accounts for toxicological effects.
Toxic Load	Total toxicity of an exposure to a chemical that can be used to determine likely toxicological effects to the individual. Model for quantifying exposed dose for chemicals that may not follow Habers Rule and that can handle concentration varying exposures.

APPENDIX B. ABBREVIATIONS.

ΔP	differential pressure
AD No.	DTIC accession number
APHC	U.S. Army Public Health Center
ARD	average relative difference
ASTM	American Society for Testing and Materials
ATEC	U.S. Army Test and Evaluation Command
ATTN	attention
°C	degrees Celsius
CB	chemical and biological
CFD	computational fluid dynamics
cm	centimeter
CWA	chemical warfare agent
DA	Department of the Army
DOD	Department of Defense
DOE	design of experiment
DTIC	Defense Technical Information Center
ECBC	U.S. Army Edgewood Chemical and Biological Center
FID	flame ionization detection
FM	Field Manual
FPD	flame photometric detection
g/m^2	grams per square meter
GC	gas chromatograph
GD	soman
HD	distilled mustard
Hg	mercury
HPLC	high- performance liquid chromatography
IAW	in accordance with
IH	inhalation
iwg	inch of water gauge
LC	liquid chromatograph
m^3	cubic meter
m/sec	meters per second

APPENDIX B. ABBREVIATIONS

µg	microgram
ME	mission-essential
mg/m^3	milligram per cubic meter
mL/min	milliliter per minute
MQL	minimum quantification limit
MS	mass spectrometer
NA	not applicable
NBC	nuclear, biological, and chemical
ng	nanogram
NMR	nuclear magnetic resonance
NRT	near real-time
NSWC	Naval Surface Warfare Center
NTA	nontraditional agent
OC	ocular
OPSEC	operations security
OTA	Operational Test Agency
pH	hydrogen-ion concentration
PTFE	polytetrafluoroethylene
QA	quality assurance
QC	quality control
RH	relative humidity
SAR	same as report
SAS	statistical analysis system
SCG	security classification guide
SCIPUFF	Second Order Integrated PUFF
SEP	System Evaluation Plan
SIV	small item vapor
SOP	Standing Operating Procedure
SST	solid sorbent tube
TEMP	Test and Evaluation Master Plan
TIC	toxic industrial chemical
TICN	test item control number
TIM	toxic industrial material
TL	toxic load
TLE	toxic load exponent
TOP	Test Operations Procedure

APPENDIX B. ABBREVIATIONS

U.S.	United States
USACHPPM	U.S. Army Center for Health Promotion and Preventive Medicine
VCSC	Vapor Composite System Calculations
VLSTRACK	Vapor, Liquid, and Solid Tracking
VX	persistent nerve agent

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APPENDIX C. VAPOR CALCULATIONS FOR A SINGLE MATERIAL SYSTEM.

TABLE LIST

FIGURE LIST

APPENDIX C. VAPOR CALCULATIONS FOR A SINGLE MATERIAL SYSTEM.

C.1. CALCULATING VAPOR CONCENTRATION.

To quantify the vapor emitted from residual contamination, the mass data collected must first be converted to a vapor concentration. NRT detection systems perform this conversion internally, but data from other systems, such as SSTs, must be converted by calculation. The vapor concentration is then used with a mass balance model to calculate the rate at which vapor is emitted from the residual contamination. The rate of vapor emission is used in the toxic load calculations described in Appendix B to scale the hazard to larger items and operationally relevant scenarios. **NOTE**: Calculations described in the following paragraphs are based on those in the U.S. Army Edgewood Chemical Biological Center (ECBC) Chemical Contaminant and Decontaminant Test Methodology Source Document, Second Edition, Document Number ECBC-TR-980. The integrating factor method[6] was used to solve the equations instead of the numerical method used in ECBC-TR-980. Numerical methods are estimations and can introduce calculation errors; therefore, the analytical methods described were used.

C.2. RESIDUAL CONTAMINATION VAPOR COLLECTED.

a. When an NRT detector is used to collect data, the vapor concentration will be calculated within the detection system. When SSTs are used, the collected mass data will be converted to a vapor concentration using Equation C-1. Conversion of units is not required in Equation C-1 because a value given in mg/m^3 is equivalent to a value given in ng/mL.

$$c(t) = \frac{m(t)}{t_s F} \qquad \textit{(Equation C-1)}$$

where:

$c(t)$ = time-dependent vapor concentration from an SST (mg/m^3)
t = elapsed time (min)
$m(t)$ = time-dependent analyte mass on the tube (ng)
t_s = SST sampling duration (min)
F = flow rate through the SST (mL/min)

b. For example, Equation C-1 can be used to calculate distilled mustard (HD) vapor concentrations from the example SST masses shown in Table C-1 and the associated sampling periods shown in Table C-2. In this example, the flow rate for each SST was 500 mL/min. Based on the values in Tables C-1 and C-2, Equation C-1 yields the HD vapor concentrations shown in Table C-2.

APPENDIX C. VAPOR CALCULATIONS FOR A SINGLE MATERIAL SYSTEM.

TABLE C-1 EXAMPLE OF DISTILLED MUSTARD (HD) MASS COLLECTED FROM CONTAMINATION OFF-GASSING

TEST ARTICLE NUMBER	HD MASS (ng) COLLECTED AT THE SORBENT TUBE CHANGE OUT INTERVAL					
	5 min	15 min	25 min	45 min	90 min	150 min
1	35	50	46	136	231	173
2	32	46	52	136	254	202
3	34	49	53	164	217	166
4	33	46	54	154	222	222
5	41	48	57	151	241	212
6	41	45	47	158	219	194
7	32	55	54	142	257	205
8	38	53	45	133	256	199

TABLE C-2. EXAMPLE DATA FOR DISTILLED MUSTARD (HD) VAPOR CONCENTRATIONS CALCULATED WITH EQUATION C-1

TEST ARTICLE NUMBER	HD VAPOR CONCENTRATION (mg/m^3) PER SORBENT TUBE CHANGE-OUT INTERVAL					
	5 min	15 min	25 min	45 min	90 min	150 min
1	0.0233	0.0111	0.0102	0.0094	0.0078	0.0059
2	0.0213	0.0102	0.0116	0.0094	0.0086	0.0068
3	0.0227	0.0109	0.0118	0.0113	0.0074	0.0056
4	0.0220	0.0102	0.0120	0.0106	0.0075	0.0075
5	0.0273	0.0107	0.0127	0.0104	0.0082	0.0072
6	0.0273	0.0100	0.0104	0.0109	0.0074	0.0066
7	0.0213	0.0122	0.0120	0.0098	0.0087	0.0069
8	0.0253	0.0118	0.0100	0.0092	0.0087	0.0067

NOTE: A solid sorbent tube (SST) flow rate (*F*) of 500 mL/min was used to calculate these example data using the mass concentrations in Table C-1 and the sampling periods in Table C-2.

C.3. EMISSION FACTOR MODEL.

a. Equation C-2 is a mass balance equation that describes the relationship among the off-gas box vapor concentration, vapor emission from residual contamination on a single test article, and the test conditions, including the enclosure or room volume and airflow rate. Equation C-2

APPENDIX C. VAPOR CALCULATIONS FOR A SINGLE MATERIAL SYSTEM.

assumes that the air in the enclosed space (room, test, fixture, etc.) is well mixed. Also, the mass balance equation does not account for any test surfaces that could adsorb or react with the vapor. The emission factor model is temperature and humidity dependent.

$$\frac{dc}{dt} = \frac{A}{V}E(t,\beta) - \frac{Q}{V}c(t) \qquad \textit{(Equation C-2)}$$

where:

$\frac{dc}{dt}$ = derivative of concentration with respect to time [mg/(m^3·min)]
$c(t)$ = time-dependent off-gas box vapor concentration (mg/m^3)
V = available off-gas box volume (off-gas box volume minus the test article volume) (m^3)
Q = mean airflow rate (m^3/min)
A = contaminated area (m^2)
$E(t,\beta)$ = time-dependent emission factor of test article [mg/(m^2 min)]
β = the vector of regression coefficients for each emission function (β_0, β_1 and β_2)

b. The mass balance equation (Equation C-2) can be simplified by substituting the loading factor ($l = A/V$), and air exchange rate ($n = Q/V$) and rearranging (Equation C-3).

$$\frac{dc}{dt} + nc(t) = lE(t,\beta) \qquad \textit{(Equation C-3)}$$

where:

$\frac{dc}{dt}$ = derivative of concentration with respect to time [(mg/(m^3·min)]
$c(t)$ = time-dependent off-gas box vapor concentration (mg/m^3)
$E(t,\beta)$ = time-dependent emission factor of test article [mg/ (m^2 min)]
β = the vector of regression coefficients for each emission function (β_0, β_1 and β_2)
l = loading factor (1/m)
n = air exchange rate in (min^{-1})

c. For a known emission function, Equation C-3 can be analytically solved using the integrating factor method for many emission functions[4] as shown in Equation C-4. When there is not an analytical solution, Equation C-4 can be numerically integrated using methods including and Simpson's rule[5]. Another approach is to numerically solve Equation C-3 using implicit Runge-Kutta or Backward Euler[7]. These methods can be implemented in Matlab (MathWorks, Natick, Massachusetts), Octave (Free Software Foundation Inc., Boston, Massachusetts), SAS

APPENDIX C. VAPOR CALCULATIONS FOR A SINGLE MATERIAL SYSTEM.

[(statistical analysis system) SAS Institute Inc., Cary, North Carolina], R (R Foundation, Vienna, Austria), and other data analysis software.

$$c(t) = le^{-nt} \int_0^t e^{nt'} E(t', \beta) dt' \qquad \textit{(Equation C-4)}$$

where:

$c(t)$ = time-dependent off-gas box vapor concentration (mg/m^3)
$E(t',\beta)$ = time-dependent emission function of test article [1/(mg × min)]
β = the vector of regression coefficients for each emission function (β_0, β_1 and β_2)
l = loading factor (1/m)
n = air exchange rate in (min^{-1})
t' = variable of integration
e = Euler's number (approximately 2.7182)
dt' = differential of time

d. The emission function models the off-gassing process. It is not always possible to know the exact physics that will occur, so regression must be done using several different models.

e. Three basic models will cover the most likely mechanisms. The first model approximate off-gassing occurring at a rate proportional to the surface loading (first order kinetics, Equation C-5). The second model approximates off-gassing as caused by a reaction of two molecules (second order kinetics, Equation C-6). The third model approximates off-gassing only after a contaminant that had soaked into a porous/permeable material diffuses to the surface (Equation C-7).

$$E(t, \beta) = \beta_0 e^{(-\beta_1 t)} \qquad \textit{(Equation C-5)}$$

$$E(t, \beta) = \frac{\beta_0}{1+\beta_0 \beta_1 t} \qquad \textit{(Equation C-6)}$$

$$E(t, \beta) = \beta_0 \left(e^{-\beta_1 t} - e^{-\beta_2 t}\right) \qquad \textit{(Equation C-7)}$$

where:

$E(t,\beta)$ = the emission function [mg/(m^2 min)]
t = the elapsed time (min)
β = the vector of regression coefficients for each emission function (β_0, β_1 and β_2)

f. When emission models are not derived a physical mechanism can be used. It is preferable that models be chosen so that Equation C-3 can be solved analytically to avoid using a numerical method. The simplest function that could be chosen is a line (Equation C-8). Sums of these function may be used together. It may be useful to add one or both terms of Equation C-8 to one of the other functional forms.

$$E(t,\beta) = \beta_0 t + \beta_1 \qquad \textit{(Equation C-8)}$$

where:

$E(t,\beta)$ = the emission function [mg/ (m^2 min)]
t = the elapsed time (min)
β = the vector of regression coefficients for each emission function (β_0 and β_1)

g. Using the log normal distribution found in Equation C-9 as the emission function has been successful at fitting vapor sampling data. Unfortunately, Equation C-3 is not analytically solvable with this emission function. However, Equation C-4 can be integrated numerically and Equation C-3 can be solved numerically with this function.

$$E(t,\beta) = \beta_2 \exp\left(-\frac{(\ln(t)-\beta_1)^2}{\beta_o}\right) + \beta_3 \qquad \textit{(Equation C-9)}$$

where:

$E(t,\beta)$ = the emission function [mg/(m^2 min)]
t = the elapsed time (min)
β = the vector of regression coefficients for each emission function (β_0 and β_1)

h. If a new emission function is introduced it must have four or fewer β parameters. If too many parameters are allowed to vary, the model could be very close to all of the data points without actually representing the physical process. If more parameters are desired, more sorbent tubes will need to be collected in each replicate.

i. When the air in the off-gas box (especially in a microchamber with a small free-air volume) is exchanged more than 50 times before the first data point is collected (i.e., when n multiplied by the time to the first data point collection is more than 50), solve Equation C-3. In these cases the air in the fixture is being exchanged faster than can be detected by the instrumentation and the concentration can be modeled as in Equation C-10.

$$c(t) = \frac{l}{n} E(t,\beta) \qquad \textit{(Equation C-10)}$$

where:

$c(t)$ = the concentration as a function of time (mg/m^3)
t = the elapsed time (min)
l = loading factor (1/m)
n = air exchange rate (min^{-1})
$E(t,\beta)$ = time-dependent emission function of test article [mg/(m^2·min)]
β = vector of regression coefficients for each emission function (β_0, β_1 and β_2 from Equations C-5 through C-8)

j. When the number of air exchanges before the first data point is collected is less than 50 (i.e., when n multiplied by the time to the first data point collection is less than 50), the mass balance equation (Equation C-3) should be solved. Equations C-11 through C-14 are solutions to the mass balance equation (Equation C-3). These solutions were found by substituting Equations C-4 through C-8, respectively, into Equation C-3, and integrating. These solutions can be added together if necessary. In particular, Equation C-14 may be useful when added to these other solutions.

$$c(t) = l\beta_0 \frac{e^{-\beta_1 t} - e^{-nt}}{n - \beta_1} \qquad \textit{(Equation C-11)}$$

$$c(t) = \frac{l e^{-\frac{n}{\beta_0 \beta_1} - nt}}{\beta_1} \left(Ei\left(-\frac{n}{\beta_0 \beta_1}\right) - Ei\left(-\frac{n}{\beta_0 \beta_1} - nt\right) \right) \qquad \textit{(Equation C-12)}$$

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$$c(t) = l\beta_0\left(\frac{e^{-\beta_1 t}-e^{-nt}}{n-\beta_1} - \frac{e^{-\beta_2 t}-e^{-nt}}{n-\beta_2}\right) \qquad \text{(Equation C-13)}$$

$$c(t) = \frac{l}{n}(1-e^{-nt})(\beta_0 t + \beta_1) \qquad \text{(Equation C-14)}$$

where:

$c(t)$ = concentration as a function of time (mg/m^3)
t = elapsed time (min)
β = vector of regression coefficients for each emission function (β_0, β_1 and β_2 from Equations C-5 through C-8)
e = Euler's number, approximately 2.7182
l = loading factor (1/m)
n = air exchange rate in (min^{-1})
Ei = integrated exponential function

C.4. REGRESSION AND SELECTION OF EMISSION FUNCTION.

a. Several aspects of this system must be addressed to successfully implement regression techniques for this purpose. First, vapor data are usually heteroscedastic (the variance differs through time). Second, all of the analytical equations are nonlinear.

(1) To account for heteroscedasticity, weighting should be applied to the regression. The error in measurement is expected to be inversely proportional to concentration. It usually makes sense to apply the weight using Equation C-15.

$$w(t) = \frac{1}{s(t)^2} \qquad \text{(Equation C-15)}$$

where:

$w(t)$ = weight function
$s(t)$ = standard deviation at each time interval
t = time

(2) An alternate approach to weighting would be to conduct a transformation on the data (e.g., logarithmic transform). If this approach is used, the process and rationale must be documented in the test report.

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(3) All of the solutions to Equation C-3 require nonlinear techniques to fit them to data. Regression using each of these solutions can be performed using the Levenberg-Marquardt algorithm. The convergence of the Levenberg-Mardquardt algorithm can be enhanced for Equations C-11 through C-14 by providing the partial derivatives of the functions with respect to the parameters (*β*). As an example, the derivatives of Equation C-11 are provided in Equations C-16 and C-17. The Levenberg-Marquardt algorithm is available in Matlab, Octave, SAS, R, or other data analysis software.

$$\frac{\partial c}{\partial \beta_o} = l\frac{e^{-\beta_1 t} - e^{-nt}}{n - \beta_1} \qquad \text{(Equation C-16)}$$

$$\frac{\partial c}{\partial \beta_1} = -l\beta_0\beta_1\frac{e^{-\beta_1 t}}{n - \beta_1} \qquad \text{(Equation C-17)}$$

where:

∂ = partial differential operator
β = vector of regression coefficients for each emission function (β_0, β_1 and β_2 from Equations C-5 through C-9)
t = elapsed time (min)
e = Euler's number (approximately 2.7182)
l = loading factor (1/m)
n = air exchange rate (min^{-1})

(4) The regression is calculated on all replicates of a trial (conducted on the same test article using the same challenge, challenge concentration, and trial conditions) at the same time.

b. After regression is completed on all of the concentration models, best-fit analysis can be performed to determine which model best represents the data. The best representation of the data will have small and randomly distributed standardized residuals (residual adjusted by the actual data standard deviation). To check the size of the residuals (the difference between the model output and the actual data), the average relative difference (ARD) should be calculated using Equation C-18[8].

$$\bar{R} = \frac{1}{N}\sum\frac{|c(t_i) - c_m(t_i)|}{c(t_i)} \qquad \text{(Equation C-18)}$$

where:

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$\bar{R}$ = average relative difference
$c(t_i)$ = mean experimental concentration (mg/m^3) at the ith time
$c_m(t_i)$ = model concentration (mg/m^3) at the ith time
N = number of data points

c. To check the randomness of the residuals, the standardized residual plots should be examined. The standardized residuals should be randomly distributed above and below zero (i.e., not exhibit any systematic error).

d. When two models perform similarly, the model with the fewest parameters should be chosen. This maximizes the degrees of freedom and will improve the results.

e. After a model is selected, then the error bounds on the emission function need to be obtained. First the error bounds on the concentration model will be calculated, then these will be converted to emission function error bounds.

(1) When the Levenberg-Marquardt algorithm is performed, the software usually provides either a variance of the residuals or the standard deviation of the residuals. To generate concentration error bounds, five replicates (recommended) should be conducted per trial (more replicates will provide better confidence intervals). For simplicity, the largest value of *s* will be used to generate the concentration 95 percent confidence intervals (Equations C-19 and C-20). A tighter confidence interval can be calculated, but would need more rigorous justification.

$$\epsilon_c = 1.96 \max s \qquad \textit{(Equation C-19)}$$

$$CI = (\max(c_m(t) - \epsilon_c, 0), c_m(t) + \epsilon_c) \qquad \textit{(Equation C-20)}$$

where:

ϵ_c = concentration 95 percent confidence bound (mg/m^3)
s = sample standard deviation of the concentration at each time point (mg/m^3)
CI = confidence interval of the concentration at each time point (mg/m^3)
$c_m(t)$ = mean concentration at each time point (mg/m^3)

(2) The confidence interval on the concentration is not equal to the confidence interval on the emission function. A bound on the emission function error can be calculated using Equation C-21. Equation C-21 can be proved directly from Equation C-4. There are more

sophisticated ways of calculating this bound that will have to be described in the test plan if they are used.

$$\epsilon_E = \max \frac{1.96ns}{l(1-e^{-nt})} \qquad \textit{(Equation C-21)}$$

where:

ϵ_E = emissions error bound [mg/(m^2·min)]
s = sample standard deviation of the concentration at each time point (mg/m^3)
n = air exchange rate (1/min)
l = loading factor (1/m)
t = is the elapsed time at which each sample was collected (minutes)

f. Example of regression procedures using microchambers.

(1) The microchambers developed by ECBC are examples of fixtures that can be modeled by substituting Equations C-5 through C-9 into Equation C-10. The microchamber has an air change rate of n = 9.36 per minute and a loading factor of l = 63.06/m. From this fixture, five replicates were collected from a single trial. The example data are in Table C-3.

TABLE C-3. MICROCHAMBER EXAMPLE LEAST SQUARES PARAMETERS

MODEL	EQUATION	FIRST PARAMETER	SECOND PARAMETER	THIRD PARAMETER[a]	AVERAGE RELATIVE DIFFERENCE (ARD)
First order	C-5	4.51×10^{-2}	1.04×10^{-3}	NA	0.144
Second order	C-6	4.56×10^{-2}	3.17×10^{-2}	NA	0.149
Linear	C-7	-3.29×10^{-5}	4.43×10^{-2}	NA	0.139
Dual exponential	C-8	5.11×10^{-2}	1.28×10^{-3}	1.37×10^{-1}	0.134

[a] Some models do not have a third parameter. These are marked not applicable (NA).

(2) For the example dataset (Table C-4), the first time point is at 10 minutes. For the microchamber, the air exchange rate of 96/min is greater than the threshold value of 50

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(Paragraph C.3.h). To find the emission function, the concentration will be modeled by substituting Equations C-5 through C-9 into Equation C-10. Parameters were calculated using the Levenberg-Marquardt algorithm. These models are shown in Figure C-1 and the parameter values are in Table C-4.

TABLE C-4. LARGE OFF-GAS BOX EXAMPLE LEAST SQUARES PARAMETERS

MODEL	EQUATION	FIRST PARAMETER	SECOND PARAMETER	THIRD PARAMETER[a]	AVERAGE RELATIVE DIFFERENCE (ARD)
First Order	C-10	1.69×10^{-3}	2.30×10^{-1}	NA	0.074
Second Order	C-11	8.73×10^{7}	4.07×10^{3}	NA	0.133
Linear	C-12	-1.12×10^{-6}	1.97×10^{-4}	NA	0.432
Dual Exponential	C-13	1.69×10^{-3}	2.30×10^{-1}	1.55×10^{25}	0.074

[a] Some models do not have a third parameter. These are marked not applicable (NA).

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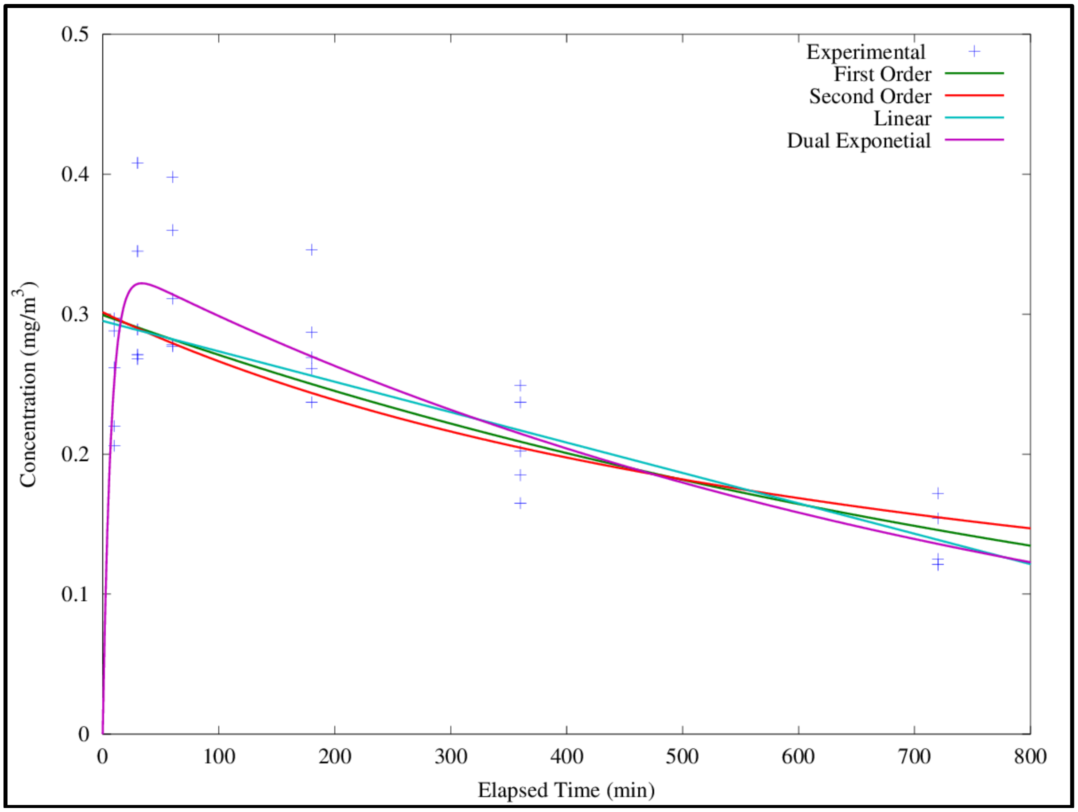

Figure C-1. Microchamber example of least squares results.

(3) All of the models have small ARDs with the dual exponential doing the best for this data set. Figure C-2 shows the residuals for these models. From Figure C-2 it can be seen that the dual exponential model has the most consistent residuals. For this example the choice is clear, and the dual exponential model is chosen with the parameters given in Table C-5.

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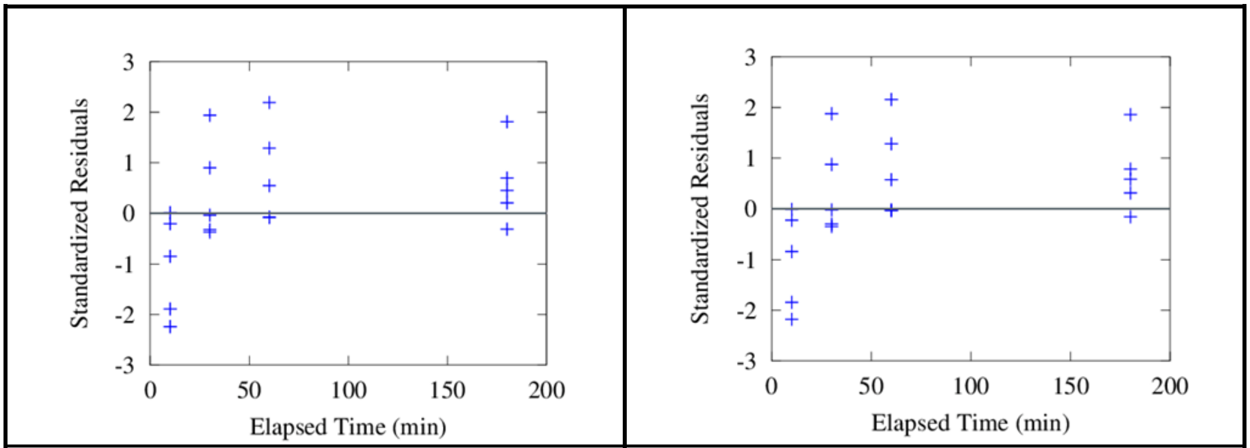
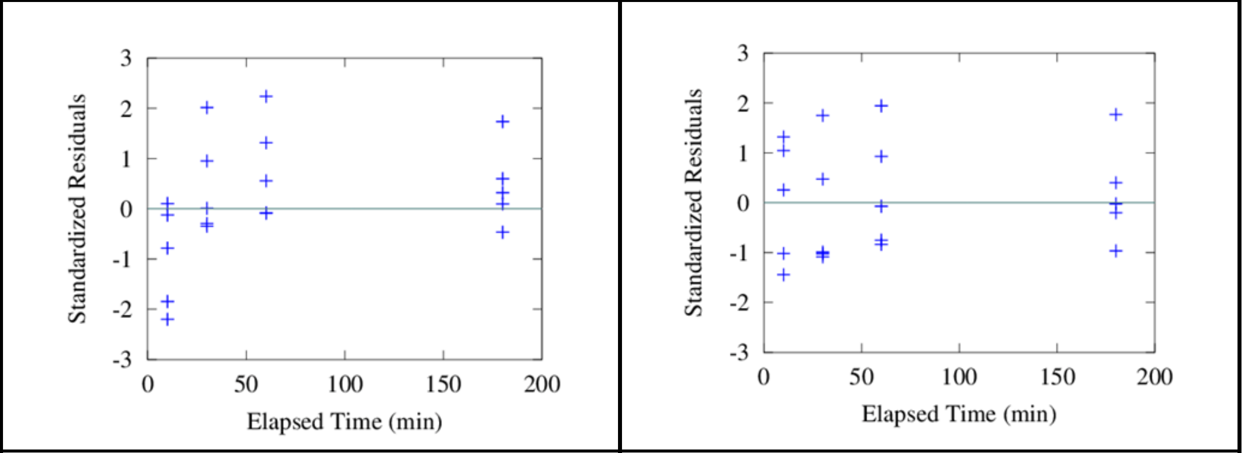


A. First Order

B. Second Order



C. Linear

D. Dual Exponential

Figure C-2. Example residual plot for the microchamber.

TABLE C-5. MICROCHAMBER EXAMPLE OF EMISSION FUNCTION ERROR BOUNDS

TIME (minutes)	SAMPLE STANDARD DEVIATION (*s*) (mg/m^3)	EMISSION FUNCTION ERROR BOUND (ϵ_E) [mg/(m^2·min)]
10	0.0367	0.0107
30	0.0543	0.0158
60	0.0482	0.0140
180	0.0372	0.0108
360	0.0319	0.0093
720	0.0211	0.0061

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(4) The 95 percent emission function error bounds are calculated by using Equation C-21. Table C-6 shows the details of this calculation. The emission function error bound is the value that is maximized in Equation C-21. This value is added and subtracted from the selected model (Equation C-20). Figure C-3 shows the results of this calculation. The emission functions are calculated using Equation C-22.

TABLE C-6. MICROCHAMBER PERSISTENT NERVE AGENT (VX) EXAMPLE CONCENTRATION DATA

TEST ARTICLE NUMBER	VX CONCENTRATION PER SAMPLING CHANGE-OUT INTERVAL (mg/m^3)					
	10 min	30 min	60 min	180 min	360 min	720 min
1	0.297	0.289	0.277	0.261	0.185	0.121
2	0.262	0.345	0.36	0.287	0.237	0.172
3	0.288	0.408	0.398	0.346	0.249	0.154
4	0.220	0.271	0.287	0.237	0.165	0.125
5	0.206	0.286	0.311	0.269	0.202	0.121

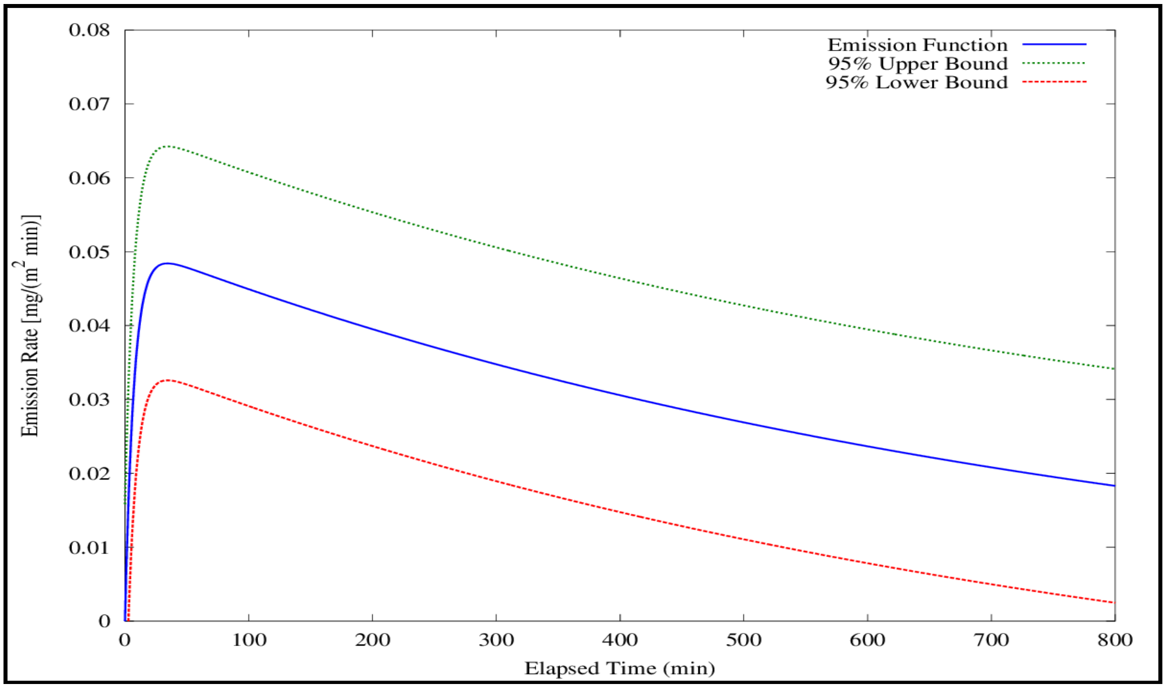

Figure C-3. Emission function for microchamber vapor sampling with error bounds.

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$$E(t) = 0.0511\left(e^{(-1.28\times10^{-3})t} - e^{(-1.37\times10^{-1})t}\right) \pm 0.0158 \quad \textit{(Equation C-22)}$$

where:

E(t) = time-dependent emission function of test article [mg/(m^2·min)]
t = elapsed time (min)

(5) Example of the regression procedure using a large off-gas box:

(a) The large off-gas box developed by ECBC is an example of a fixture that must be modeled with Equations C-11 through C-14. The large off-gas box has a total volume of 0.17 m^3. Removing the volume of instrumentation and a test article from the total volume, results in a free volume of 0.13 m^3 which then equals 127 L (V). For this example, the SSTs have an airflow of 500 mL/min (Q). For this configuration, the exchange rate $n = Q/V = 0.5/127 = 0.004$/minute. The test article being used in this case is a laptop computer with an exposed area of 0.21 m^2. Therefore l = A/V = 0.21/0.13 = 1.6/m. From this fixture, five replicates were collected from a single trial. The example input data are in Table C-6.

(b) For this example dataset, the first data collection time point is at 15 minutes. That means that the air in the fixture was exchanged 0.06 times before the first data point was collected, which is much less than the 50 times exchange threshold (Paragraph C.3.h). To find the emission function, the concentration data were fit to the models in Equation C-11 through C-14. Parameters were calculated using the Levenberg-Marquardt algorithm. These models are shown in Figure C-4 and the parameter values are shown in Table C-7.

APPENDIX C. VAPOR CALCULATIONS FOR A SINGLE MATERIAL SYSTEM.

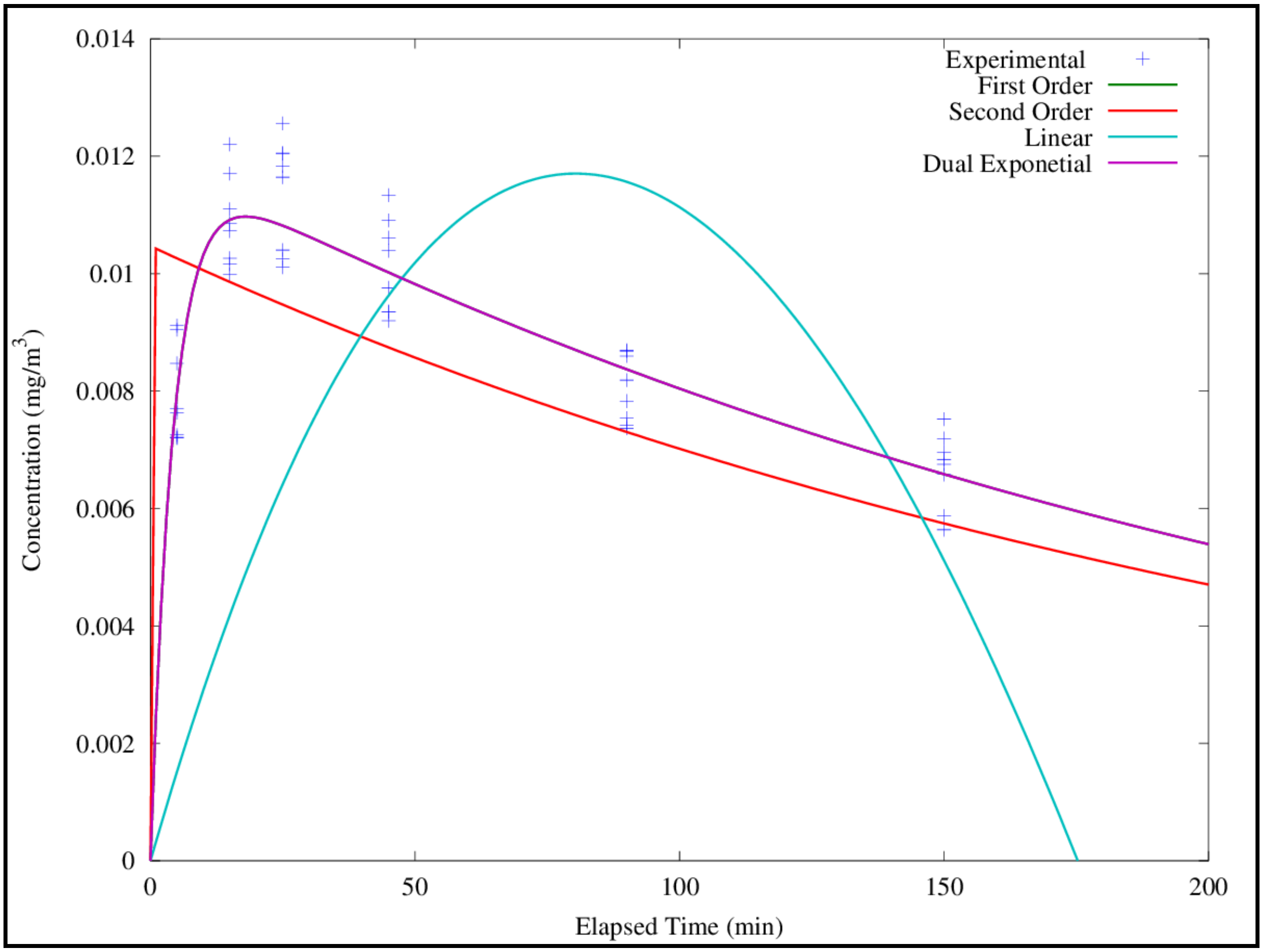


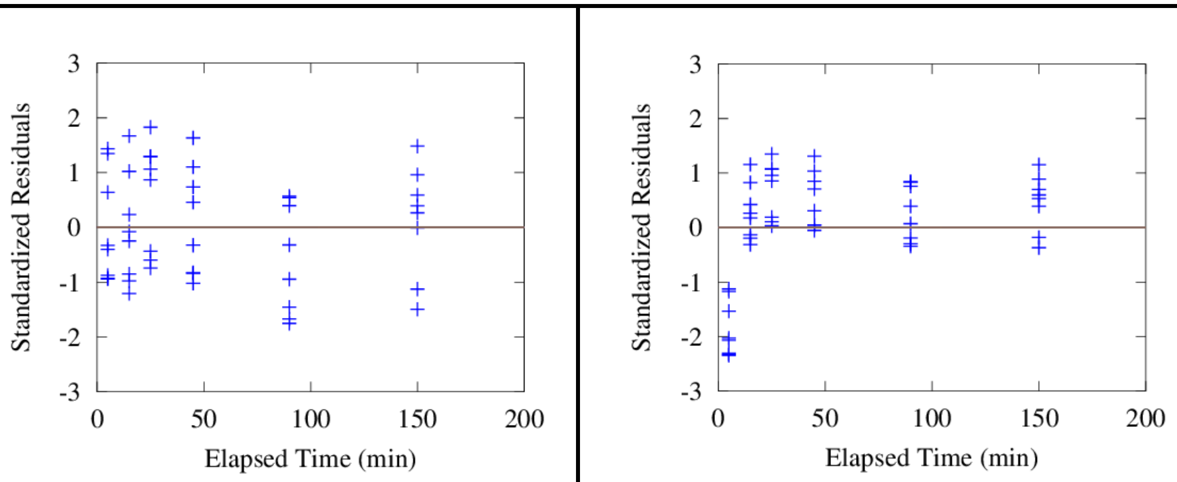
Figure C-4. First order model line is hidden behind the dual exponential model line.

TABLE C-7. LARGE OFF-GAS BOX EXAMPLE OF EMISSION FUNCTION ERROR BOUNDS

TIME (minutes)	STANDARD DEVIATION (s) (mg/m^3)	EMISSION FUNCTION ERROR BOUND (ϵ_E) (mg/m^3)
5	8.10×10^{-4}	1.99×10^{-4}
15	7.74×10^{-4}	6.48×10^{-5}
25	9.57×10^{-4}	4.90×10^{-5}
45	8.10×10^{-4}	2.40×10^{-5}
90	5.75×10^{-4}	9.28×10^{-6}
150	6.35×10^{-4}	6.86×10^{-6}

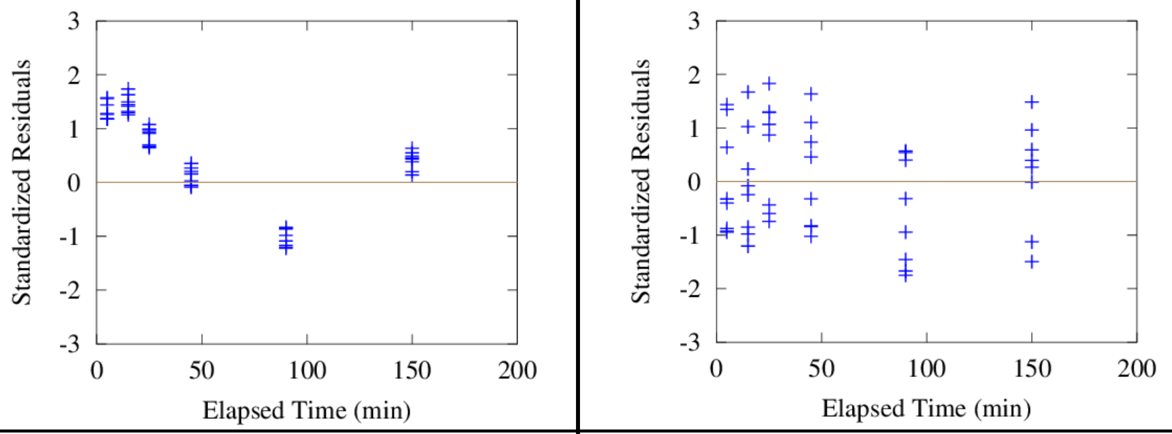
APPENDIX C. VAPOR CALCULATIONS FOR A SINGLE MATERIAL SYSTEM.

(c) The first-order model and the dual-exponential model have the same ARD. In this case, preference is given to the model with the fewest parameters. To select the model, the residuals are analyzed to be sure the correct model is selected. Figure C-5 shows plots of the residuals. The top right and bottom left plots show residuals that are not evenly distributed above and below zero. Figure C-5 demonstrates that the first order model suffers less systematic error than the other models. For these reasons, the first order emission function is chosen with the parameters given in Table C-5 with the equation results shown in Table C-7.



A. First Order

B. Second Order



C. Linear

D. Dual Exponential

Figure C-5. Example residual plots for the large off-gas box.

APPENDIX C. VAPOR CALCULATIONS FOR A SINGLE MATERIAL SYSTEM.

(d) The 95 percent error bounds are calculated using Equation C-21. In this case, the 5-minute quotient is the largest; therefore, the largest bound on the emission function is 1.99×10^{-4}. This value is added and subtracted from the selected model (Equation C-23). Figure C-6 shows the emission function derived from the large off-gas box dataset.

$$E(t) = 1.69 \times 10^{-3} e^{-2.30\times10^{-1}t} \pm 1.99 \times 10^{-5} \qquad \textit{(Equation C-23)}$$

where:

$E(t)$ = time-dependent emission function of test article [mg/(m^2·min)]
t = elapsed time (min)

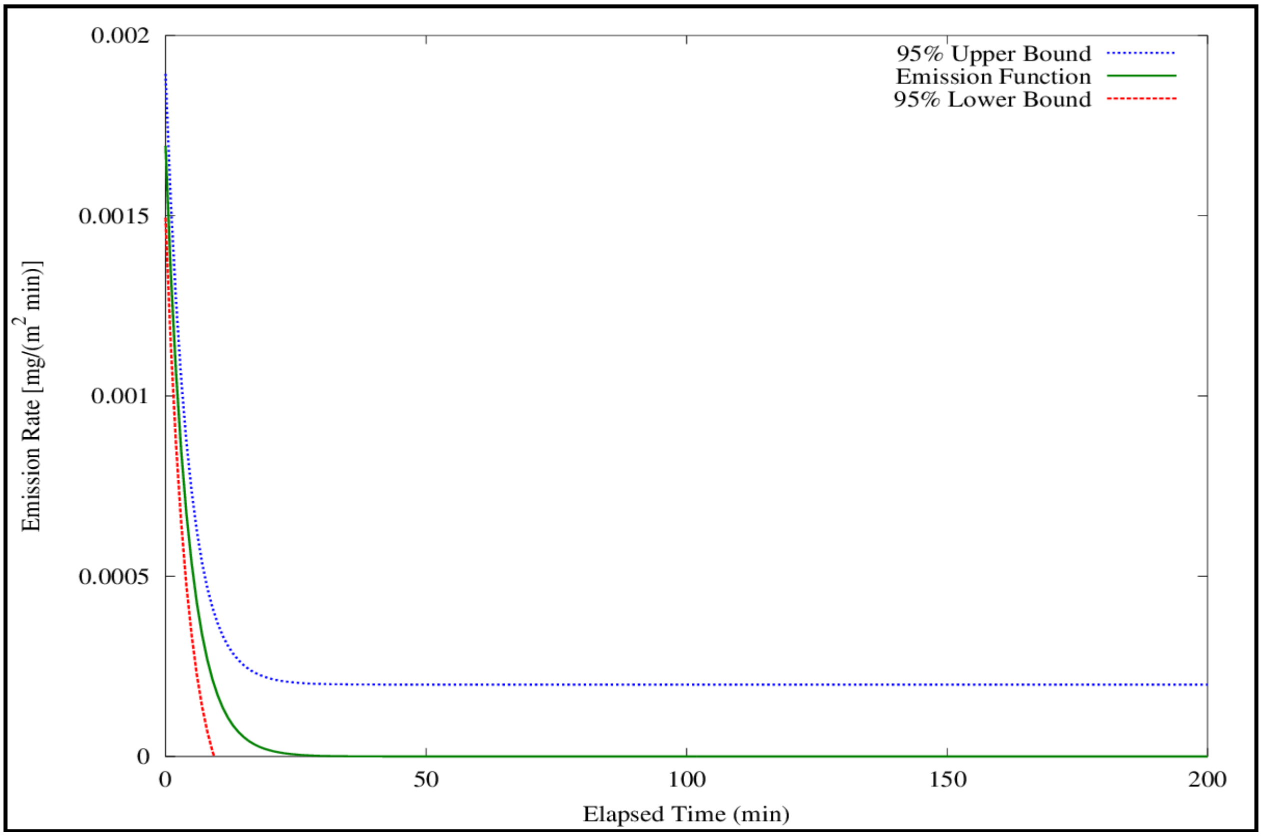


Figure C-6. Selected concentration model for large off-gas box vapor sampling with error bounds.

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APPENDIX D. VAPOR COMPOSITE SYSTEM CALCULATIONS (VCSC).

TABLE LIST

FIGURE LIST

APPENDIX D. VAPOR COMPOSITE SYSTEM CALCULATIONS (VCSC).

D.1. GENERAL.

a. Appendix C covers the calculation of emission functions from single materials. The emission function can be used to predict the concentration inside a room or within another enclosed area. Because most military assets are composed of multiple materials it is also useful to calculate the concentration off-gassed by a composite system, a system composed of multiple materials.

D.2. CONCENTRATION PREDICTION FOR A SINGLE MATERIAL.

a. Using the emission function derived in Appendix C, the concentration in an enclosed volume can be predicted by solving Equation C-3 with the loading factor (l_s) and air exchange rate (n_s) specific to the scenario, using Equations D-1 and D-2. **NOTE**: Outdoor scenarios or poorly mixed indoor scenarios will require other modeling methods which are beyond the scope of this TOP, including Gaussian plume[6], computational fluid dynamics (CFD)[7], Second Order Integrated PUFF [(SCIPUFF), L-3 Titan Corporation, Princeton, New Jersey][8], or Vapor, Liquid and Solid Tracking [VLSTRACK, Naval Surface Warfare Center (NSWC)][9], Dahlgren Division, Dahlgren, Virginia].

$$l_s = \frac{A_s}{V_s} \qquad \text{(Equation D-1)}$$

$$n_s = \frac{Q_s}{V_s} \qquad \text{(Equation D-2)}$$

where:

l_s = scenario loading factor (1/m)
n_s = scenario exchange rate (1/min)
A_s = scenario contaminated surface area (m^2)
V_s = free-air volume of the enclosed volume (m^3)
Q_s = scenario airflow rate (m^3/min)

b. Equation C-3 is solvable with the emission functions described in Equations C-5 through C-8 and has the solutions given in Equations C-10 through C-13, but with $l = l_s$ and $n = n_s$. These solutions and error bounds are in Equations D-3 and D-4. Because the emission function was selected in Appendix C, only the selected model needs to be calculated. These solutions assume that the contaminant vapor is well mixed in the enclosed area. Also, these models do not account for materials that could adsorb, absorb, or react with agent vapor. The scenario temperature and humidity need to match the experimental values to create a valid model of the scenario.

APPENDIX D. VAPOR COMPOSITE SYSTEM CALCULATIONS (VCSC).

$$\epsilon_s = \frac{l_s \epsilon_E}{n_s}(1 - e^{-n_s t}) \qquad \textit{(Equation D-3)}$$

$$c_s(t) = l_s e^{-n_s t} \int_0^t e^{n_s t'} E(t') dt' \pm l_s \epsilon_E (1 - e^{-n_s t})/n_s \qquad \textit{(Equation D-4)}$$

where:

ϵ_E = 95 percent error bound on the emission function calculated in Equation C-18 (mg/m^3)
l_s = scenario loading factor (1/m)
n_s = scenario exchange rate (1/min)
$c_s(t)$ = concentration as a function of time (mg/m^3)
t = elapsed time in minutes
e = Euler's number (approximately 2.7182)
$E(t)$ = emission function found in Appendix A [mg/(m^2·min)]
t' = variable of integration
dt = differential of time

c. A valid assumption for test conditions in vapor off-gas boxes is that they are well mixed. However, as room volumes increase and airflow patterns become more complex, this assumption may not hold, which may decrease the accuracy of the predicted vapor concentrations and the resulting exposure analysis. Error bounds calculated in this process cannot account for the complexity of the airflow.

d. The error bound on the emission function found in Appendix C can be used to derive the error bound on the concentration for the scenario using Equation D-3. The general form of the scenario concentration, given the emission function and its error bounds, are given in Equation D-4. Solutions to Equation D-4 can easily be derived from Equations C-11 through C-14.

D.3. DEVELOPING VCSC FROM EMISSION FUNCTIONS.

a. To predict the off-gassing of a composite system, the procedure in Paragraph D.2 will be generalized. The off-gassing from each contaminated area will be combined into the concentration for the composite system.

APPENDIX D. VAPOR COMPOSITE SYSTEM CALCULATIONS (VCSC).

b. To calculate the off-gassing, a modified version of Equation C-2 is solved using Equation D-5. The modification simply replaces the emission function in Equation C-2 and contaminated area, with the sum of source terms for each contaminated area.

$$\frac{dc_s}{dt} = \sum_i \frac{A_i}{V_s} E_i(t) - \frac{Q_s}{V_s} c_s(t) \qquad \textit{(Equation D-5)}$$

where:

$\frac{dc}{dt}$ = derivative of concentration with respect to time [(mg/(m^3·min)].
$c_s(t)$ = time-dependent vapor concentration (mg/m^3).
A_i = contaminated area of the ith material.
$E_i(t)$ = time-dependent emission rate of ith material [mg/(m^2·min)].
t = elapsed time (min).
A_i = the contaminated area of the material (m^2).
Q_s = mean airflow rate in the scenario (m^3/min).
V_s = available volume in the scenario (m^3).

c. The procedure to solve this equation is analogous to the procedures to solve Equation C-2. First the equation is rearranged in Equation D-6. Then the equation is simplified by substituting the loading factor for each material ($l_i = A_i/V_s$) and the air exchange rate ($n_s = Q_s/V_s$). Finally, the integrating factor and the property of linearity of solution is applied to derive Equation D-7. When there is not an analytical solution, Equation D-6 can be numerically integrated using methods including Simpson's rule[7]. Another approach is to numerically solve Equation D-6 using implicit Runge-Kutta or Backward Euler[7]. These methods can be implemented in Matlab, Octave, SAS, R and other data analysis software.

$$\frac{dc}{dt} + n_s c(t) = \sum_i l_i E_i(t, \beta) \qquad \textit{(Equation D-6)}$$

where:

$\frac{dc}{dt}$ = derivative of concentration with respect to time [(mg/(m^3·min)].
n_s = scenario exchange rate (1/min).
$c(t)$ = time-dependent off-gas box vapor concentration (mg/m^3).
l_i = loading factor for the ith material.
$E_i(t,\beta)$ = time-dependent emission rate of ith material [mg/(m^2·min)].
β = the vector of regression coefficients for each emission function (β_0, β_1 and β_2) l_s = scenario loading factor (1/m)

APPENDIX D. VAPOR COMPOSITE SYSTEM CALCULATIONS (VCSC).

$$c(t) = e^{-n_s t} \int_0^t e^{n_s t'} \sum_i l_i (E_i(t',\beta) \pm \epsilon_{Ei})\ \ dt' \qquad \textit{(Equation D-7)}$$

where:

$c(t)$ = time-dependent off-gas box vapor concentration (mg/m^3).
$E_i(t',\beta)$ = time-dependent emission function of contaminated area [1/(mg × min)].
β = the vector of regression coefficients for each emission function (β_0, β_1 and β_2).
l_i = loading factor for each contaminated area (1/m).
n = air exchange rate in (min^{-1}).
t' = variable of integration.
e = Euler's number (approximately 2.7182).
ϵ_{Ei} = error bound on the emission function for each material.
dt' = differential of time.

d. Equation D-6 can be arranged to demonstrate a useful property of the solutions. In Equation D-8 the sum has been moved out of the integral, which can be done because this is a finite sum. Also, the error term was integrated and represented as a separate sum. Equation D-8 shows that the concentration is the sum of terms in the same form as Equation C-4. The total concentration in the fixture is simply that of the sum of the concentration profiles from each contaminated region. Also, the error is the sum of errors that have been converted by Equation D-3. Each region can be calculated separately and then summed together to generate the VCSC.

$$c(t) = \sum_i l_i e^{-nt} \int_0^t e^{nt'} E_i(t',\beta)\ \ dt'\ \pm \sum_i l_i \epsilon_{Ei} (1 - e^{-n_s t})/n_s \qquad \textit{(Equation D-8)}$$

where:

$c(t)$ = time-dependent off-gas box vapor concentration (mg/m^3).
$E_i(t',\beta)$ = time-dependent emission function of contaminated area [1/(mg × min)].
β = the vector of regression coefficients for each emission function (β_0, β_1 and β_2).
l_i = loading factor for each contaminated area (1/m).
n_s = air exchange rate in (min^{-1}).
t' = variable of integration.
e = Euler's number (approximately 2.7182).
ϵ_{Ei} = error bound on the emission function for each material.
dt' = differential of time.
t = time.

APPENDIX D. VAPOR COMPOSITE SYSTEM CALCULATIONS (VCSC).

(1) When the air in the off-gas box (especially in a microchamber with a small free-air volume) is exchanged more than 50 times before the first data point is collected (i.e., when n multiplied by the time to the first data point collection is more than 50), there is no need to solve Equation D-3. In these cases the concentration can be modeled using Equation D-9.

$$c(t) = \frac{1}{n_s} \sum_i l_i E_i(t, \beta_i) \pm l_i \epsilon_{Ei} \qquad \textit{(Equation D-9)}$$

where:

$c(t)$ = the concentration as a function of time (mg/m^3).
t = the elapsed time (min).
l_i = loading factor of the ith contaminated area (1/m).
n_s = air exchange rate of the scenario (min^{-1}).
$E_i(t,\beta_i)$ = the time-dependent emission function of the ith contaminated area [mg/(m^2·min)].
β_i = vector of regression coefficients for ith emission function (β_0, β_1 and β_2 from Equations C-5 through C-8).
ϵ_{Ei} = error bound on the emission function for each material.

(2) When the number of air exchanges before the first data point is collected is less than 50 (i.e., when n multiplied by the time to the first data point collection is less than 50), the mass balance equation (Equation D-3) should be solved. The solution to Equation D-3 is the sum of the solutions to Equation C-3. Solutions for the emission functions discussed in Appendix C are in Equations C-10 through C-13.

(3) The confidence interval on concentration increases in width through time. This is a consequence of the cumulative nature of the mixing in the fixture. The calculation of the error bound for the emission function in Equation C-21 also accounts for this effect.

D.4. EXAMPLE CONCENTRATION FROM SINGLE MATERIAL.

As an example, the concentration generated from a contaminated item in a conference room will be calculated. The conference room has a free-air volume of 280 m^3 and 17 m^3/min of ventilation. There is 0.25 m^2 of contaminated item in the conference room. In this scenario, l_s = 0.000893/m and n_s = 0.0607/min. Using the parameter values in Equation C-22 with the scenario-specific parameters, Equation D-4 can be used to predict the concentration in the conference room as shown in Equation D-10 and modeled in Figure D-1. Solving Equation D-6 in this scenario is essentially the solution given in Equation C-10 with $l = l_s$ and $n = n_s$.

APPENDIX D. VAPOR COMPOSITE SYSTEM CALCULATIONS (VCSC).

$$c_s(t) = 9.29 \times 10^{-6} \times (e^{-0.23t} - e^{-0.067t}) \pm 2.66 \times 10^{-6}(1 - e^{-0.067t}) \quad \textit{(Equation D-10)}$$

where:

$c_s(t)$ = the concentration in the room (mg/m^3).
e = Euler's number (approximately 2.7182).
t = elapsed time (min).

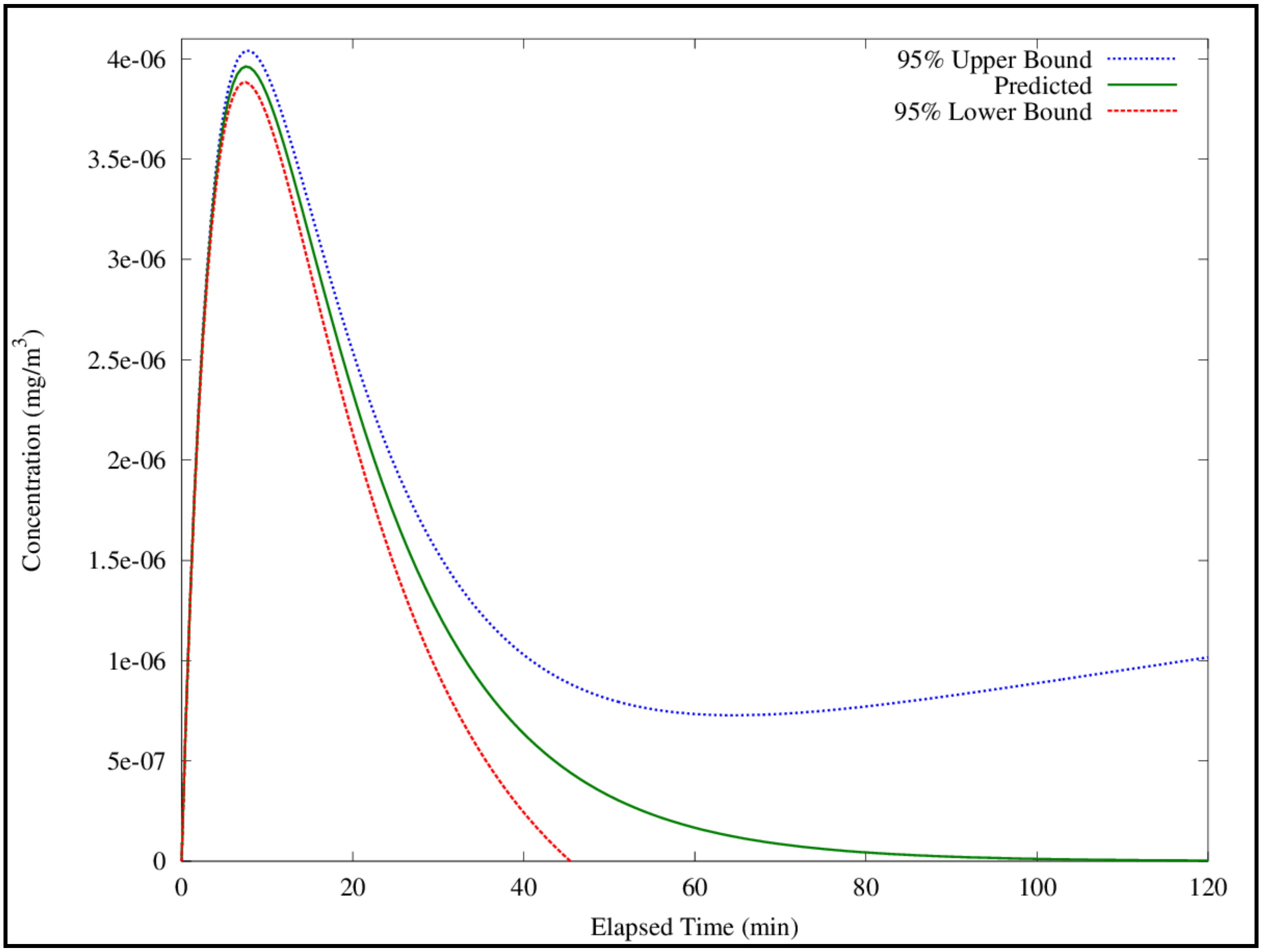

Figure D-1. Conference room concentration modeled from the emission function.

APPENDIX D. VAPOR COMPOSITE SYSTEM CALCULATIONS (VCSC).

D.5. EXAMPLE VCSC SCENARIO.

a. An emission function for each material is required to conduct a VCSC scenario. Assume we have a system comprised of three components made of different materials. The emission function for a chemical agent and the contaminated area for each material is in Table D-1. The total error for the emissions function is 143.23 mg/(m^2 min).

TABLE D-1. EXAMPLE PARAMETER VALUES FOR EMISSION FUNCTIONS AND AREA OF CONTAMINATION

MATERIAL	EMISSION EQUATION	β_o	β_1	β_2	β_3	AREA OF CONTAMINATION (MEAN ± STANDARD DEVIATION) (cm^2)	LOADING FACTOR[a] (1/m)
Antenna	C-9	4.456	1.935	1145	NA[a]	0.127± 0.029	6.01×10^{-4}
Body	C-9	2.652	2.226	1712	6.375	0.123± 0.005	5.82×10^{-4}
Screen	C-6	9.863×10^8	0.0011	NA	NA	0.365± 0.040	1.73×10^{-3}

[a] Not applicable

b. VCSC is calculated using Equation D-11 and shown in Figure D-2. Figure D-3 shows the VCSC with error bounds.

$$c(t) = e^{-0.0955t}\int_0^t e^{0.0955t'}\left(145.4exp\left(-\frac{(ln(t)-1.935)^2}{4.456}\right) + 210.6exp\left(-\frac{(ln(t)-2.226)^2}{2.652}\right) + 0.7841 + \frac{3.60\times10^{-8}}{1+1.08\times10^{-10}t} \pm (6.01\times10^{-4} + 5.82\times10^{-4} + 1.73\times10^{-3})143.23\right)\,dt' \qquad \text{(Equation D-11)}$$

where:

c(t) = the concentration in the room (mg/m^3).
t = time.
ln = natural log.
dt' = differential of time.

APPENDIX D. VAPOR COMPOSITE SYSTEM CALCULATIONS (VCSC).

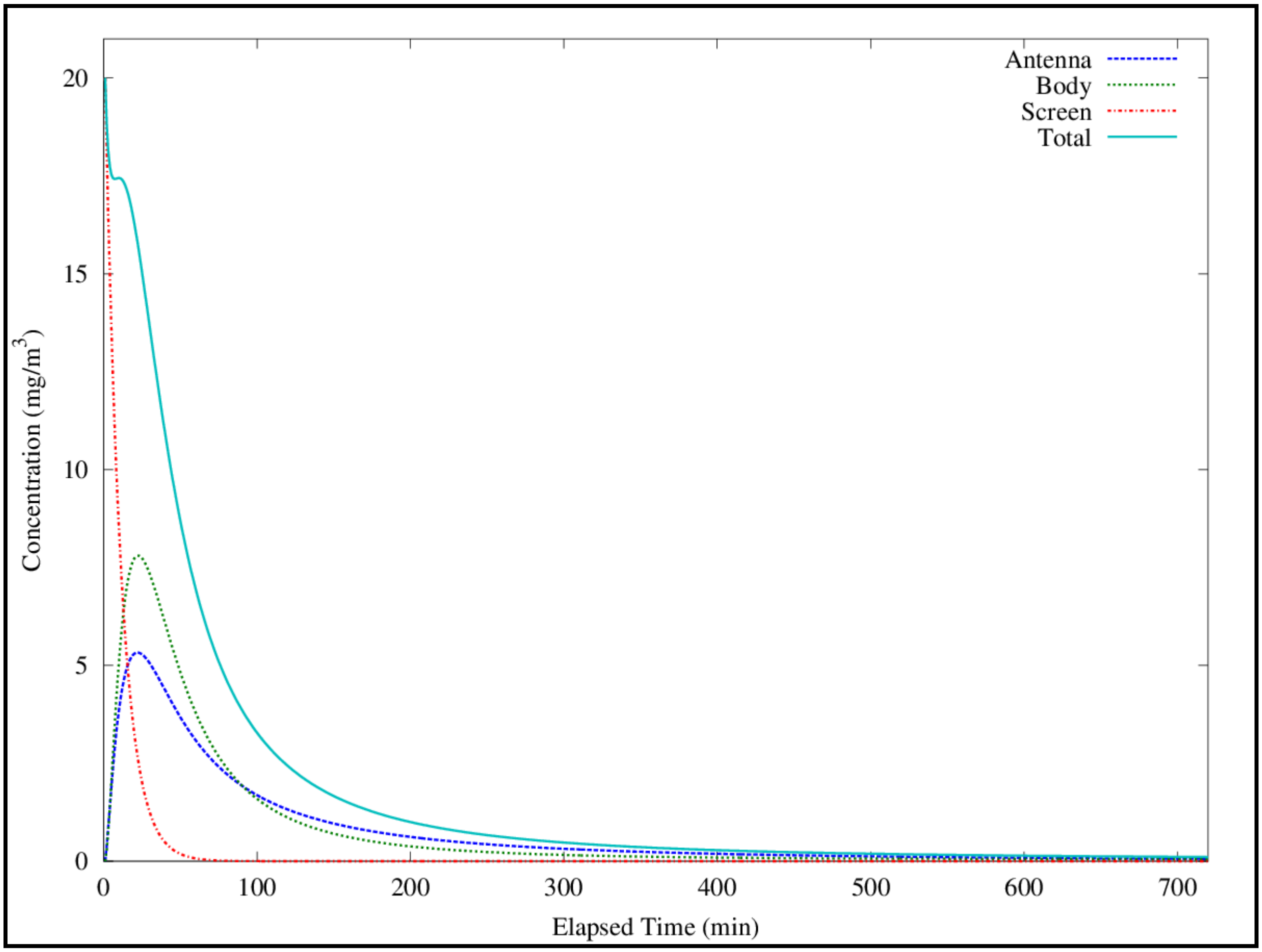

Figure D-2. Example vapor composite system calculation.

APPENDIX D. VAPOR COMPOSITE SYSTEM CALCULATIONS (VCSC).

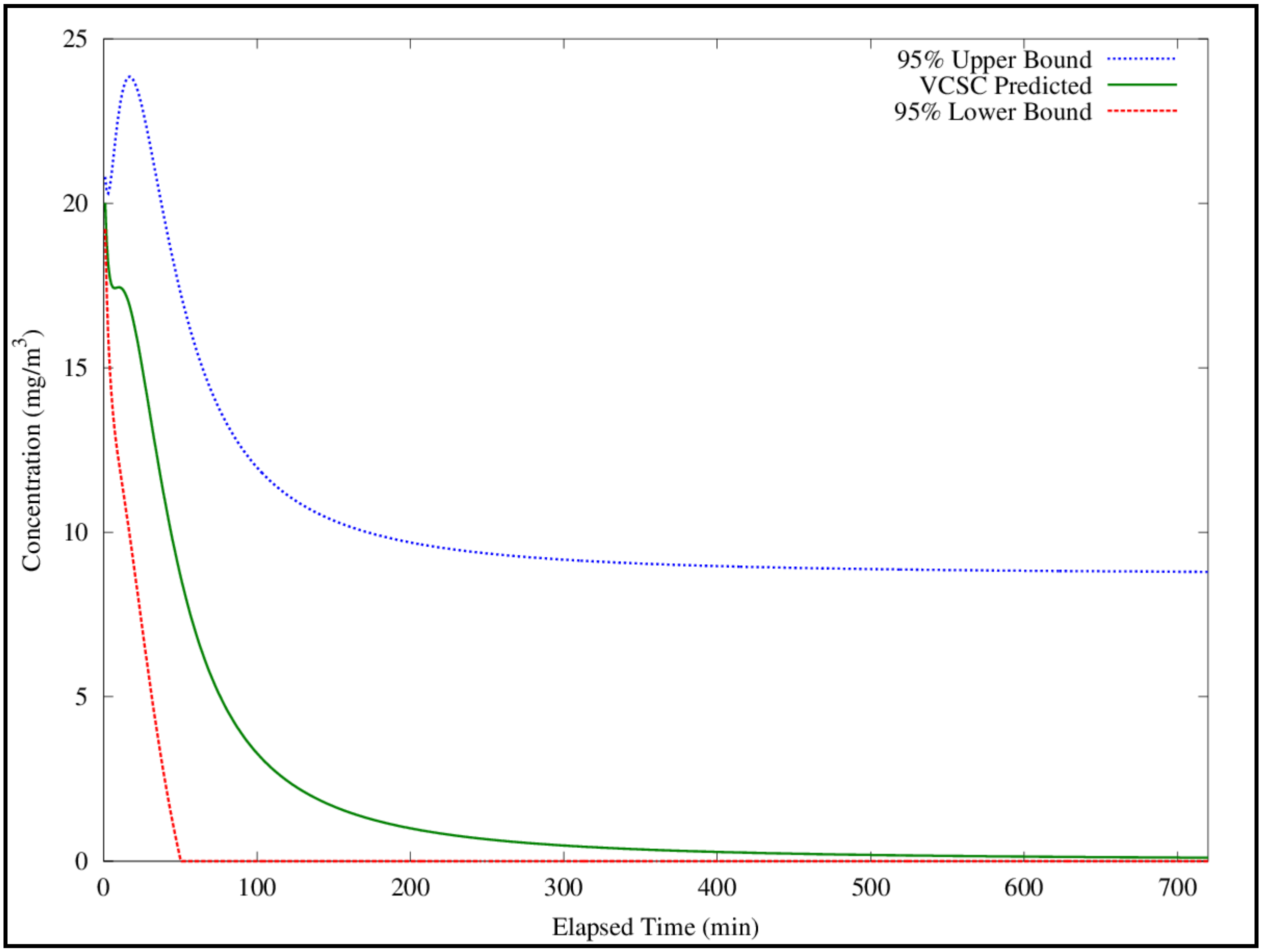

Figure D-3. Vapor Composite System Calculations (VCSC) with error.

APPENDIX E. CONCENTRATION AND TOXIC LOAD (TL) PREDICTION.

TABLE LIST

FIGURE LIST

APPENDIX E. CONCENTRATION AND TOXIC LOAD (TL) PREDICTION.

E.1. SCENARIO-SPECIFIC CONCENTRATION MODEL.

The emission function selected using the procedures in Appendix C predicts the rate of contaminant emission per unit surface area (m^2). Appendix D describes the VCSC that takes these emission functions and calculates a concentration from multiple materials. To predict the effect of this exposure on personnel, the toxic load (TL) for a particular operationally relevant scenario can be calculated.

E.2. CALCULATION OF TL.

a. To calculate a TL, the toxic load exponent (TLE) must be selected based on the source of the derived requirements. The example TLE is a function of the contaminant and can be found in Table E-1. If the data correspond to the agent soman (GD), a TLE of 1.4 is selected. The inhalation (IH) or ocular (OC) exposure mild effect level should always be selected as the TLE for decontaminated items using the assumption that a decontaminated item is expected to be clean, but may not be clean[3].

TABLE E-1. TOXIC LOAD (TL) EXPONENTS (TLE) FROM INHALATION TOXICITY PARAMETERS.

Agent	Effect Level[a]	Ct_{50} (mg-min/m^3)	Probit Slope	TLE	Time basis (min)
GB	Lethal	33	12	1.5	2
	Severe	25	12	1.5	2
	Mild	0.4	10	1.4	2
GD	Lethal	33	12	1.5	2
	Severe	25	12	1.5	2
	Mild	0.2	10	1.4	2
VX	Lethal	12	12	1	2
	Severe	9	12	1	2
	Mild	0.04	4	1.4	2
HD	Lethal	1000	6	1.5	2
	Severe[b]	75	3	1	2
	Mild[b]	25	3	1	2

[a] Values from Romano Jr., J.A., Salem, H., & Lukey, B.J. (Eds.). (2019). Chemical Warfare Agents: Biomedical and Psychological Effects, Medical Countermeasures, and Emergency Response (3rd ed.). CRC Press. https://doi.org/10.1201/9781498769235[10]

[b] HD Severe and Mild value from ECBC-TR-349[11], and represent occular effect endpoints for unprotected individuals exposed to HD vapor.

b. The TL for a scenario is calculated using the scenario vapor concentration (Equation D-4). This calculation will generate a single number that can be compared with a criterion to determine if a scenario would induce a toxicological response. The TL is calculated using the ten Berge model[12] (Equation E-1).

$$TL = \int_{t_0}^{t_f} c_s(t)^{\eta} dt \qquad \textit{(Equation E-1)}$$

where:

TL = the TL (mg min/m^3).
$c_s(t)$ = scenario concentration as a function of elapsed time (mg/m^3).
η = TL exponent.
t_0= start of personnel exposure.
t_f = end of exposure of personnel.
dt = differential of time.

c. The scenario error bound for the concentration is contained within the integral in Equation E-1. Being inside the integral causes the error term to increase with time. The size of the error term will limit the length of time that can be modeled accurately. When using Equation E-1, all three integrals should always be calculated (predicted TL, 95 percent upper bound, and 95 percent lower bounds). The usual use of this data is to determine if the TL was less than some toxicological level on some time interval. The TL can be calculated for various time intervals within the scenario duration by selecting different values for t_0 and t_f.

d. Equation D-5 is numerically integrable using basic numerical integration, such as Gaussian quadrature, an adaptive method, Simpson's rule, or trapezoid rule[7]. Trapezoid rule[7] is the least technically challenging of these, but may have significant error unless small time steps are chosen. Numerical methods to perform the integration are available in commercial and open-source software (e.g., Matlab, Octave, SAS).

e. As an example, the TL will be calculated for the selected emission function in Equation C-22. There are five people (Attendees 1 through 5) in the meeting in a conference room. Attendee 1 attends the full 2 hours of the meeting. The rest of the attendees either arrive late or leave early. Attendee 1 executes the procedures in Paragraphs 4.7 through 4.10 using VX. However, Attendee 1 brings one item to the meeting instead of placing it in the fixture for vapor sampling. The item is the same material used in the examples in Paragraph C.4.f of Appendix C. They will be exposed to the concentration calculated in Paragraph D.5.

f. From the concentration in the conference room (Equation E-1), the TL for personnel that enter the room is calculated for the time intervals from Table E-2 and the TLE from Table E-1.

TABLE E-2. EXAMPLE TOXIC LOAD (TL) VALUES FOR THE MEETING ATTENDEES (EXAMPLE CALCULATION SCENARIO)

ATTENDEE	EXPOSURE (min)			TL $[(mg/m^3)^{TLE}$ min]			ERROR (%)
	Start	End	Duration	Lower Confidence Bound	Predicted	Upper Confidence Bound	
1	0	120	120	4.71×10^{-7}	5.44×10^{-7}	8.27×10^{-7}	52
2	0	60	60	4.71×10^{-7}	5.40×10^{-7}	6.39×10^{-7}	18
3	0	15	15	3.08×10^{-7}	3.19×10^{-7}	3.30×10^{-7}	3
4	30	90	60	1.93×10^{-8}	5.72×10^{-8}	1.98×10^{-7}	245
5	60	120	60	0.00×10^{-9}	3.44×10^{-9}	1.88×10^{-7}	5350

g. Figure E-1 illustrates that the error bounds get wider as the duration of exposure increases using Attendee 1’s TL. For Attendee 2, the error is 18 percent of the predicted value over a 60 minute period of time (Table E-2). This is early in the trial so the error is small. For Attendee 5 the error is 5350 percent of the predicted value over a different 60 minute period (Table E-2). This illustrates the importance of calculating the error. Time periods selected can have drastically different errors even if they have the same duration.

APPENDIX E. CONCENTRATION AND TOXIC LOAD (TL) PREDICTION.

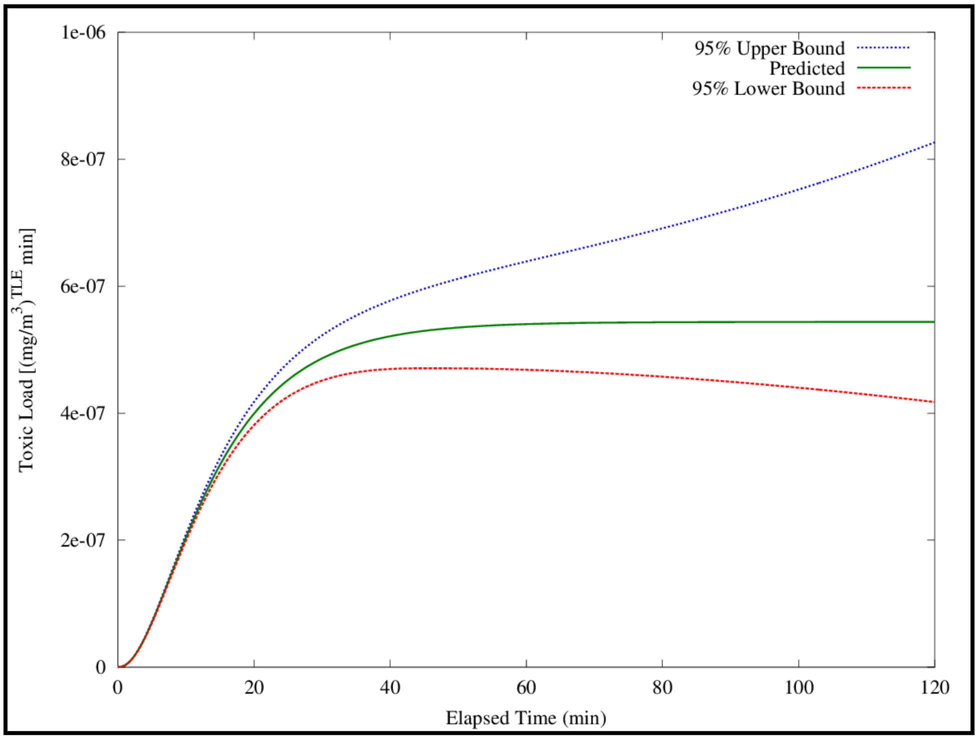

Figure E-1. Toxic Load (TL).

h. The value of most interest here is the upper confidence bound. Even with the large errors, the upper confidence bound can be used to draw some conclusions. All of our attendees were below the mild effects level for 1 percent of the population, IAW Table E-4 of USACHPPM, Report 47-EM-5863-04[4]. Even with the decontaminated item in the room, the meeting attendees (all with an IH rate assumption of 15L/min) are not expected to have any ill effects from exposure to VX.

i. i. Using equation E-2 the toxic load exposure values calculated with Equation E-1 may be converted into population response percentages typically found in requirements. For example a population response percentage of 12% for mild effects would be below a typical health based requirement specification of ECt16 (mild).

APPENDIX E. CONCENTRATION AND TOXIC LOAD (TL) PREDICTION.

$$Response_{\%} = N\left(\log\left(\frac{\left(\frac{TL}{t_b}\right)^{\frac{1}{TLE}} t_b}{Ct_{50}}\right) slope\right) \qquad \textit{(Equation E-2)}$$

where:

$\text{Response}_{\%}$ = Percentage of exposed population that would experience effects
N() = Normit function
TL = Toxic Load From Equation E-1
TLE = Toxic Load Exponent (unitless)
t_b = ECt data timeframe basis (min)
Ct_{50} = Estimated Ct value for 50% response (mg-min/m3)
slope = probit slope

APPENDIX F. REFERENCES.

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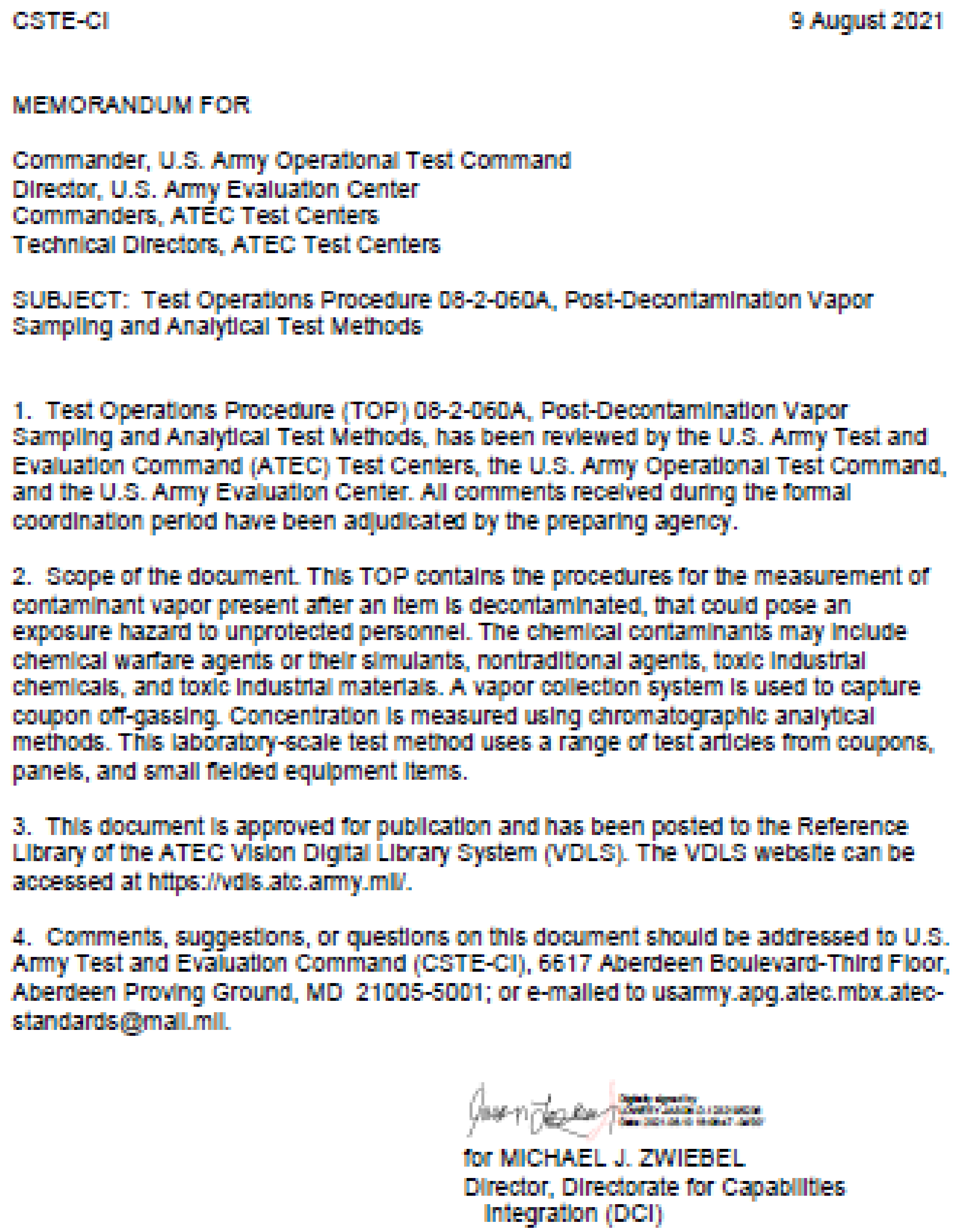
APPENDIX G. APPROVAL AUTHORITY.



CSTE-CI 9 August 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 08-2-060A, Post-Decontamination Vapor Sampling and Analytical Test Methods

1. Test Operations Procedure (TOP) 08-2-060A, Post-Decontamination Vapor Sampling and Analytical Test Methods, 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 contains the procedures for the measurement of contaminant vapor present after an item is decontaminated, that could pose an exposure hazard to unprotected personnel. The chemical contaminants may include chemical warfare agents or their simulants, nontraditional agents, toxic industrial chemicals, and toxic industrial materials. A vapor collection system is used to capture coupon off-gassing. Concentration is measured using chromatographic analytical methods. This laboratory-scale test method uses a range of test articles from coupons, panels, and small fielded equipment items.

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.atec.mbx.atec-standards@mail.mil.

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

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Forward comments, recommended changes, or any pertinent data, which may be of use in improving this publication to the 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: Commander, West Desert Test Center, US Army Dugway Proving Ground, ATTN: TEDP-WD, 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, VA 22060-6218. This document is identified by the accession number (AD No.) printed on the first page.