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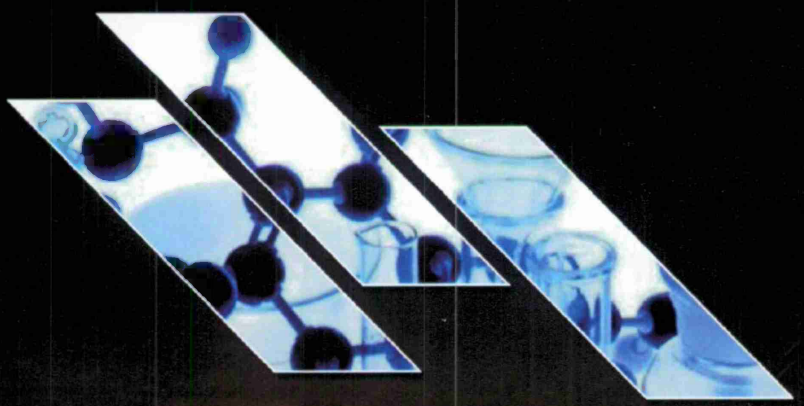
SMALL-ITEM VAPOR TEST METHOD FY11 RELEASE



Teri Lalain

RESEARCH AND TECHNOLOGY DIRECTORATE

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In the decontamination business area, all emerging decontaminants and technologies are required to demonstrate the ability to meet a specified set of chemical efficacy performance requirements prior to transition into an acquisition program. One of the specific key performance parameters to be demonstrated is that of chemical agent decontamination efficacy. The chemical agent efficacy determination is accomplished through the execution of the standard panel contact and vapor tests, as documented in Test Operations Procedure (TOP) 8-2-061 and the 2007 Chemical Decontaminant Source Document. DTRA project DEC499 extended the test methodology suite by the development of vapor test methods for small items of sensitive equipment. The final vapor test method, originally released in FY09, is being reissued as a public release method as the appendix in this report.

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PREFACE

The work described in this report was authorized under the Defense Threat Reduction Agency (DTRA) Project No. CA07DEC499. This work was started in July 2007 and completed in December 2008.

This report was published through the Technical Releases Office; however, it was edited and prepared by the Decontamination Sciences Branch, Research and Technology Directorate, U.S. Army Edgewood Chemical Biological Center (ECBC).

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Acknowledgments

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SMALL-ITEM VAPOR TEST METHOD
FY11 RELEASE

1. INTRODUCTION TO THE CHEMICAL DECONTAMINANT PERFORMANCE EVALUATION SOURCE DOCUMENT

The Chemical Decontaminant Performance Evaluation Source Document (SD) is a collection of updated procedures and the final product for DTRA projects BA06DEC414 and CA08DEC420. The Source Document received its name based on the intended use of the document by the test and evaluation (T&E) community to either formally update Test Operating Procedure (TOP) 8-2-061[1] or generate a new TOP specific to the evaluation of decontaminant performance on various materials of interest.

One of the original program requests by DTRA was to have a collection of procedures that could be distributed to laboratories, based on the targeted information needed from the testing. These methods would support testing a wide range of technologies, materials, and contaminants; provide context regarding data utilization especially for assessing risk; and enable test-to-test and lab-to-lab data comparisons. When properly utilized, the improved methods would generate higher fidelity data, which would be presented in an appropriate context. The data generated from these updated methods enhanced all components of the decontaminant lifecycle, including research and development (R&D), science and technology (S&T), T&E, and developmental and operational testing (DT/OT) activities, technology readiness assessments (TRA) to determine technology readiness level (TRL), technology comparisons, risk assessments and milestone decisions.

DTRA project CA08DEC499 extended the Source Document methods for the evaluation of small items of sensitive equipment in support of the Joint Service Sensitive Equipment Decontamination Program.

2. DEVELOPMENT AND RELEASE OF THE 2007 SD

To fulfill need for robust methodology, the original SD, titled *2007 Chemical Decontaminant Source Document* was developed by the U.S. Army Edgewood Chemical Biological Center (ECBC) Decontamination Sciences Branch. The 2007 SD contained contact and vapor test methodology that was updated from the TOP 8-2-061 document. During development, the core tests for determining remaining contaminant, contact, and vapor tests underwent major transformations.

The 2007 SD utilized a textbook chapter and section structure focusing on specific topics such the contact test method, vapor test method, etc. Each chapter was divided into individual test methods specific to that topic, such the core tests, positive and negative control tests, and sample analysis. Each test method used a basic research procedure outline that included reagents, materials, test procedures, calculations, and reporting. The basic foundation was augmented by incorporating the elements required by ISO-17025 and ASTM methods, such as procedure summary, terminology, reporting criteria, quality assurance, quality control, and test acceptance criteria. This format facilitated individual method insertion into a performing laboratory's quality system. Each test section carried relevant terminology; references, calculations, and quality assurance/quality control requirements so that each chapter subsection could be used as an independent method.

In the 2007 SD, the contact test method had minimal updates to the general procedure for performing the standard two-touch test, but the procedure was expanded to provide greater detail for test consistency and additional rigor for key variables. The contact test chapter included specific test

methodology for determining the remaining agent and performing the contact test, in addition to providing guidance for chromatographic analysis. The test procedures contained options allowing test modifications and guidance on how those modifications could impact data calculations. The contact test chapter contained detailed data calculations which were further divided into calculated, approximated, or inferred calculations. These divisions were based on the availability of required data and indicated the degree of rigor used to calculate the final test result.

The vapor test underwent a major transformation for the 2007 SD, resulting in a significant improvement to vapor sampling and data analysis as part of this effort. The vapor test method was updated to include the key variables associated with vapor sampling and a vapor-emitting item. The method for calculating whether or not a vapor hazard was present was historically based on the vapor concentration measured in the vapor chamber. The measured chamber vapor concentration does not correspond to the vapor concentration to which unprotected personnel would be exposed. The result is often an overestimation of the hazard. Overestimating the resulting hazard can impact decontamination development, resulting in greater logistical requirements and increased potential for material incompatibilities. In addition, comparing a test chamber vapor concentration to a requirement to determine the occurrence of a toxicological response was not correct. The documented methods were now aligned with the DoD-accepted method for the determination of a vapor exposure using a toxic-load calculation. The new calculations involved the characterization of the emission source. This characterization enabled scale up, specific scenarios calculations, and trade space analyses, further enhancing operational considerations and risk assessments. In order to teach this new calculation procedure, the 2007 SD contained example data to enable the method user to practice and check their calculations.

The 2007 SD test methodology contained sufficient rigor for the control, measurement, and reporting of the key process variables, which enabled comparison of test data. The methodology incorporated options to enable testing at different conditions and using different technologies. Detailed data calculation approaches were also developed. The 2007 SD successfully updated the core panel test methodology. The improved test methodology procedures were released in 2007 and formally published in ECBC-TR-671.[2]

3. DEVELOPMENT OF THE SMALL-ITEM VAPOR TEST METHOD

The development of the updated *Chemical Contaminant and Decontaminant Test Methodology Source Document, Second Edition* (SD2ED) continued after the 2007 SD release and through summer 2011. The primary objective, which was similar to the original document, was to continue the development and documentation of robust test methodologies for chemical decontamination including the small-item vapor test method. The SD methodology focused on testing panels of individual materials. There was an expressed need to enable vapor testing of actual full-scale assets. As a result, project CA08DEC499 was funded to implement the advancements of the SD methodology for full-scale assets.

The primary program objective was to develop methodology to enable vapor testing of small items. This program was a Defense Threat Reduction Agency (DTRA)-funded program, with input from the Product Director, Test Equipment, Strategy, and Support (PDTESS) and the Joint Program Executive Office for Chemical Biological Defense Joint Program Manager (JPEO-CBD JPM) for Decontamination office. A program expectation was that the methods would be able to support the evaluation of the small-item priority list for the Joint Materiel Decontamination Systems (JMDS) acquisition program upcoming testing. A second program expectation was that the methodology would be universal and enable supporting programs of records, such as Joint Service Transportable

Decontamination System - Small Scale (JSTDS-LS). This program built onto the foundation developed through the DTRA effort to improve the lab-scale chemical agent vapor test in program BA06DEC414.

The vapor test characterizes the emission of contaminant from the material after the treatment process to determine a contaminant emission function that can be used to determine the risk to unprotected personnel. The vapor test is typically performed to provide data for comparison against technology transition agreements exit criteria, requirement documents, and other health-based criteria. The small-item vapor test method utilized larger dynamic vapor chambers suitable for the sampling of the small items. The final method was documented in the same format at the 2007 SD, including a detailed data example for the data calculation procedures from a small-item vapor test.

The development of Small-Item Vapor Test Method was performed by the Decontamination Sciences Branch laboratories at ECBC, Aberdeen Proving Ground, MD. The development involved input from DTRA, stakeholders, and research and testing communities. The method, which was originally released in FY09, has been revised slightly for editorial improvements and is being reissued in the report appendix. Since its original release, the Small-Item Vapor test method has been integrated in to TOP 8-2-111.[3]

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LITERATURE CITED

1. *CSTE-DTC-TT-M Test Operations Procedure (TOP) 8-2-061 Chemical and Biological Decontamination Testing*; West Desert Test Center: Dugway Proving Ground, UT, 19 November 2002. UNCLASSIFIED Report (AD-A409 136).

2. Lalain, T.; Mantooth, B.; Lynn, T.; Zander, Z.; Humphreys, P. *Development of the 2007 Chemical Decontaminant Source Document*; ECBC-TR-671; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2009. UNCLASSIFIED Report (AD-A511 356).

3. *Test Operations Procedure (TOP) 8-2-111: Chemical, Biological, and Radiological (CBR) Contamination Survivability, Small Items of Equipment*; U.S. Army Dugway Proving Ground West Desert Test Center (TEDY-DPW): Dugway, UT, 11 January 2011. UNCLASSIFIED Report.

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APPENDIX

Small-Item Vapor Test Method

SUMMARY OF PROCEDURE

The vapor test measures the contaminant (agent) emission from a test item placed into a vapor test chamber. Air is passed over the sample then collected onto a solid-sorbent tube solid-sorbent tube is thermally desorbed and analyzed. The vapor test results and calculation procedures can be used to determine the risk that a contaminated item might pose to unprotected personnel. The test measures an *emission factor* or *emission rate* for single or multiple material evaluations, respectively.

This procedure can support testing R&D lab-scale through developmental large-scale testing. The small-item vapor test can be used to evaluate decontaminant performance against liquid-phase contaminants such as chemical-warfare agents, chemical-warfare agent simulants, and toxic industrial chemicals or materials.

Components of this method may include decontaminant evaluations, baseline, control, and background tests. The method contains guidance for baseline positive control (without decontaminant) and negative control (without contaminant). The positive control tests are recommended to determine the amount of agent lost during the decontamination process to weathering or evaporation. The negative control tests are recommended to evaluate if an item emits chemical vapors that may interfere with contaminant chromatographic analysis.

This method contains the calculation procedures to determine the vapor concentration and toxic-load value for the vapor test chamber and applied scenarios.

This procedure provides the following information:

- The mass of agent, in nanograms, recovered from the solid-sorbent tubes collected during the vapor-sampling period.
- The calculated vapor concentration for the vapor test chamber and applied scenarios.
- The calculated vapor toxic-load value for the vapor test chamber and applied scenarios.
- The calculated emission rate for the item.

The following prerequisites are required for this test procedure:

- Capability for liquid sample and solid-sorbent tube chromatographic analysis.
- Vapor test chamber.

Limitations and other test variations:

- The collection of vapor data is not a direct measure for percent efficacy, percent neutralization, or reduction in starting challenge.
- Contaminant Simulant: Chemical compounds for chemical agents are often used during early screening or at non-surety facilities. A chemical agent simulant is a

chemical compound of lower toxicity than the chemical agent, with at least one property similar to the chemical agent such as certain bonding, functional group, physical property, etc. For the most accurate comparison, simulants should be selected based on the main property being tested. Because simulants do not contain all of the same physical and chemical properties of the live agent, simulant data alone is not sufficient to determine decontaminant performance. It is recommended that the simulant performance be confirmed with agent data for final evaluation of decontaminant performance and risk to unprotected personnel. Requirement values are based on toxicological information regarding the agent. Risk assessments should be made based on the results for the live agent.

- Bagging and sampling methods can indicate whether contaminant offgassing is present; however, these methods do not characterize airflow and lack controlled air volume. Therefore, bagging and sampling methods are not appropriate techniques for this method. The airflow and air volume are key variables required to assess risk.
- Residual agent: Because full-item extraction cannot be performed, it is typically not possible to measure the residual agent. The potential future risk estimation may be limited without that value. For this reason, the vapor-sampling time should be long enough to enable evaluation for the scenario of interest.

TERMINOLOGY

Terminology specific to this test procedure is provided alphabetically in the following list:

- **absorption**: The uptake of a contaminant INTO the volume of a material. The contaminant must transport through the surface into the volume of the material
- **adsorption**: The adhesion of a contaminant on a material. This is limited to the surface of the material and does not include contaminant residing inside the material.
- **agent**: See *chemical agent*. Used interchangeably with *contaminant*.
- **air-change rate:** The ratio of the airflow rate in an environment (e.g., vapor chamber, scenario), Q, to the free-air volume, V, of the environment. The air-change rate, n, is calculated as n = Q/V. Air-change rate is expressed in this method using units of both min^{-1} and h^{-1}.
- **airflow rate, chamber:** The airflow rate through the vapor chamber during the experiment, reported in milliliters per minute (mL/min).
- **airflow rate, sampling:** The airflow rate through the solid-sorbent tube during sample collection, reported in milliliters per minute (mL/min), may be different than chamber airflow rate, depending on chamber configuration.
- **ambient temperature**: The temperature of the surrounding air. In this case, the surrounding air is defined as the temperature in the working environment (i.e., the laboratory/hood). This is the same as room condition.
- **analytical sample:** Liquid extract or vapor tube sample generated during testing for chromatographic analysis (GC and/or LC) or for other quantitative analytical tools.

- **breadboard, brassboard, prototype**: Technology still under development, in differing degrees of configuration, which is not in final form. This can apply to test fixtures, formulations, and/or the decontamination system/applicator.
- **chamber vapor concentration:** The vapor concentration measured from the vapor chamber. This does not correspond to the vapor concentration to which unprotected personnel would be exposed, and should not be compared to requirements documents.
- **chemical agent:** A toxic chemical for use in military operations. A comprehensive listing of chemical agents can be found in FM 3-11.9. The term *agent* is used interchangeably with the term *contaminant*.
- **contaminant:** A chemical compound with harmful effects to humans, which needs to be neutralized or removed from surfaces of interest. Typical contaminants include chemical agents, chemical agent simulants, toxic industrial chemicals, and toxic industrial materials.
- **contamination:** The deposition, adsorption, or absorption of chemical agents on or by structures, areas, personnel, or objects. (Reference FM 3-11.9.)
- **contamination, full item:** Application of contaminant evenly over all item surfaces, as identified by the contamination scenario. This option is best suited for developmental testing (DT).
- **contamination, localized**: Application of the contaminant to selected regions to evaluate specific materials, regions, or surfaces, based on test objective. This option is best suited for R&D testing to evaluate and optimize decontaminant performance.
- **confidence interval:** A calculated range for a data set that future results are likely to fall between.
- **contaminant simulant:** Compounds of lower toxicity that contain at least one property similar to the parent contaminant (e.g., live chemical agent).
- **contamination set:** For dose confirmation, a contamination set is a specific contamination density, drop volume, and deposition pattern combination. Deposition pattern only matters if the contaminant application uses more than one drop.
- **decontaminant:** For these procedures, a substance with the capability to remove and/or neutralize chemical agents on/in surfaces of interest. The decontaminant can be liquid-phase, solid-phase (powders, wipes), or gas-phase (fumigants, including aerosols).
- **decontamination process:** The process of making any person, object, or area safe by absorbing, destroying, neutralizing, making harmless, or removing a contaminant. (Reference FM 3-11.9) More specifically for these procedures, the specific series of treatment tasks performed may include contaminating, aging, decontaminating, rinsing, and drying the surface of interest.
- **detection limit:** The lowest quantity of a substance that can be distinguished from the absence of that substance (a blank value) within a stated level of confidence.

- **dose-confirmation sample:** A sample providing the mass of contaminant delivered during a test session. It cannot be assumed that contaminant delivery tools, such as pipettors and syringes, will always perform at the manufacturer's specifications, especially for viscous or highly volatile materials. The dose-confirmation sample is used to provide confidence in the amount of agent applied to the sample by the tool during that test session. This value is needed for calculations such as percent neutralization or reduction in starting challenge, which require accurate measurement of the starting contamination.
- **emission rate:** The flux of the agent from the item under test, expressed as mass emitted in milligrams per item per minute (mg $item^{-1}$ min^{-1}).
- **hazard:** A condition with the potential to cause injury, illness, or death of personnel; damage to or loss of equipment or property; or mission degradation. (Reference FM 3-11.9.)
- **item:** A sample used for testing, which can include small items of sensitive equipment.
- **item handling**: Treatment of the test item upon leaving inventory through disposal. Handling may include contamination, decontamination, extraction, etc.
- **limit of detection:** LOD see *detection limit.*
- **limit of quantitation**: LOQ see *quantitation limit.*
- **loading factor:** The ratio of the number of items in an environment (Z) to the free-air volume (V) of the environment. The loading factor (I) is calculated as I=Z/V.
- **moderate condition:** Test condition in the middle of the testing range that is the standard indoor office/laboratory condition at 19–21 °C and 50–60% relative humidity.
- **nonsorptive materials:** A material that does not retain a significant amount of contaminant by absorption, though there may be a minute quantity of adsorption. Sorption is dependent on material-contaminant interactions. A material that is sorptive with one contaminant may or may not be sorptive with another contaminant. Generally, bare metals and glass are nonsorptive materials for some agents.
- **operational decontamination**: Decontamination carried out by an individual and/or a unit, restricted to specific parts of operationally essential equipment, materials, and/or working areas in order to minimize contact, transfer hazards, and to sustain operations. This may include decontamination of the individual beyond the scope of immediate decontamination, as well as decontamination of mission-essential spares and limited terrain decontamination. (Reference FM 3-11.9.)
- **pull schedule:** The schedule that identifies when samples are collected. The pull schedule includes the midpoint and pull times for each sample (tube).
- **quantitation limit:** The lowest amount of analyte in a sample that can be quantitatively determined with suitable precision and accuracy.
- **relative standard deviation (RSD)**: The standard deviation of a data set divided by the mean of the data set.

- **requirement levels**: The documented amount of permissible agent remaining after a decontaminant process, typically expressed as a vapor concentration in milligrams per meter cubed (mg/m^3) or as a surface concentration in milligrams per meter squared (mg/m^2).
- **residual agent**: The amount of contaminant present in or on the material of interest, after the decontaminant process and hazard test has been conducted.
- **rinsate:** The collected rinse from the decontamination process. The sample may include residual decontaminant, agent, or agent byproducts in water.
- **room condition**: The temperature and relative humidity of the test location on the specific test day.
- **scenario:** The scenario is the specific information regarding the environment that the item is placed in. The scenario includes how many items are in the environment, the free-air volume of the environment, and the air-change rate in the environment. The use of scenarios is recommended for risk assessments.
- **scenario vapor concentration:** The calculated vapor concentration using the item's emission model and the scenario's airflow properties and free-air volume. This vapor concentration better represents the vapor concentration to which unprotected personnel would be exposed.
- **sessile drop**: A liquid droplet that is firmly attached to a surface. If the droplet significantly spreads across the surface, it is better described as a thin film.
- **sorptive or porous materials**: A material that absorbs or adsorbs a contaminant. Sorption is dependent on material-agent interactions. A material that is sorptive with one agent may or may not be sorptive with another agent.
- **starting challenge footprint:** Starting challenges are reported as mass per unit area. The area is determined using the item footprint in this method. Alternate interpretations for area can be used with this method.
- **test condition:** For a specific agent-material-decontaminant set, the combined contamination, aging, decontaminant process, environmental, and test sampling process (i.e., contact, vapor, remaining, residual) variables.
- **time, tube pulling:** The length of time that air was flowing through a solid-sorbent tube.
- **time, midpoint:** The time representing when a sample was collected as calculated by the tube initial time plus one half of the tube pull time.
- **time, initial:** The time representing the start of airflow (sample collection) for a tube.
- **vapor chamber:** A vapor microchamber that fully encloses the test item to enable vapor emission analysis. The chamber must facilitate the ability to control airflow and mixing, collect vapor samples, and measure environmental conditions such as temperature and relative humidity.
- **vapor cell**: A vapor enclosure that is placed over the surface to be tested for vapor emission analysis. The tested surface serves as one of the "walls" of the enclosure. The use of a vapor cell is not within the methods described here.

- **vapor hazard:** A value specified in requirements documents, usually specified as a concentration in milligrams per meter cubed (mg/m^3), that should have an accompanying exposure time. The value corresponds to an exposure that presents an acceptable risk level for unprotected personnel exposed to the vapor concentration. The toxic-load model should be applied to calculate a vapor hazard.

REFERENCED DOCUMENTS

- *Multiservice Tactics, Techniques, and Procedures for Chemical, Biological, Radiological, and Nuclear Decontamination Nuclear, Biological, and Chemical (NBC) Decontamination*, Field Manual (FM) 3-11.5, Headquarters, Department of the Army (DA), Washington, DC, 2006.
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- American Society for Testing and Materials (ASTM), *Standard Practice for Selection of Sorbents, Sampling, and Thermal Desorption Analysis Procedures for Volatile Organic Compounds in Air*, ASTM Document Number D 6196, West Conshohocken, PA.
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REAGENTS, EQUIPMENT AND MATERIALS

REAGENTS

- **Contaminants:** The specific contaminants for evaluation are dependent on the test objectives and specific test facility capabilities. Chemical decontaminant evaluation contaminants typically fall into one of three categories.
    - Chemical Agent: Work with chemical agents can only be conducted in approved facilities by specially trained personnel. The types of chemical agents tested include, but are not limited to those documented in FM 3-11.9 "Potential Military Chemical/Biological Agents and Compounds."
    - Chemical Agent Simulant: Chemical agent simulants are compounds with at least one similarity to live chemical agent, but are always lower in toxicity. These compounds do not contain all of the same physical and chemical properties of live agent, but are selected because of similarities in the main property of reference for a specific test.

- Toxic Industrial Chemicals (TICs) and Materials (TIMs): TICs and TIMs are chemicals produced for industrial applications that are toxic to humans. Testing may include, but is not limited to, the published listing from "Task Force 25: Hazard from Industrial Chemicals Final Report" dated April 1998.

- **Decontaminants:** The specific decontaminants used for evaluation are dependent on the test objectives and specific test facility capability. Chemical decontaminants can be liquid, solid, or vapor phase, and may contain a reactive functionality for neutralizing chemical contaminants.
- **Analytical Solvents**: The dose-confirmation samples and liquid chromatography analytical standards require the use of solvents. Solid-sorbent tube analysis also requires analytical standards prepared in solvent for solid-sorbent tube spiking. Typical solvents may include, but are not limited to chloroform, hexane, isopropyl alcohol, methylene chloride, and solvent blends.
- **Water:** Decontamination processes for small items will not typically involve rinsing or the use of water-based decontaminants. However, if water is needed, it is recommended that distilled or deionized water be used, unless otherwise instructed by the test sponsor.

EQUIPMENT

The equipment required for this method includes tools for delivering the contaminant and decontaminant, maintaining environmental control, and preparing analytical samples. Several equipment options exist, ranging in accuracy and complexity. The appropriate tool should be selected based on the test requirements and acceptable measurement uncertainty. The listed equipment is based on commercial items with known accuracy, precision, and/or repeatability. Other equipment may be used, but should be evaluated to determine the impact on test measurement uncertainty. All equipment should be calibrated regularly, and calibration records should be maintained. The types of tools required are listed with primary bullets. Sub-bulleted items provide a list of options that meet the primary bullet requirements.

- **Contaminant Delivery Tool:** the tool used to apply a specified amount of agent to the surface of interest. Tools with repeater capabilities are recommended to ensure identical replicate samples. The typical delivery drop volumes can range from 1 to 20 µL, based on starting challenge interpretation contamination density. The drop volumes most commonly used range from 1 to 5 µL.
    - Pipette: The tool with the largest range of delivery volumes. Positive-displacement pipettes with disposable tips are preferred to prevent cross-contamination if the tool is used for multiple procedure steps, dosing solutions, or contaminants. Positive-displacement pipettes are also recommended for highly viscous materials because the positive wiping action of the piston against the capillary wall assures accurate dispensing, and avoids any carryover. These are also best suited for pipetting volatile liquids. The smallest delivery volume, based on a survey of commercial items with repeater capability, is about 1 µL. Pipettes used for the purpose of contaminant delivery should be compliant with the required performance specifications listed in the most current versions of ISO 8655 Parts 1 and 2 and/or ASTM E 1154 for the volume being measured.

- Syringe: Positive-displacement tool best suited for the delivery of smaller drop volumes. The smallest delivery volume, based on survey of commercial items with repeater capability, is about 0.2 µL. Syringes to be used for the purpose of contaminate delivery should have a maximum inaccuracy of 1%, and a maximum imprecision of 1% of the volume being measured.
- Computerized Dispensing System: An automated tool with ability to deliver specific drop volumes and surface coverage patterns. Based on a review of commercial items, the smallest delivery volume is approximately 0.35 nL, with repeatability <1%. The manufacturer's performance specifications should be reported and evaluated against acceptable measurement uncertainty for the particular test.
- Aerosol Contamination System or Other Applicators: Some applications may use custom-designed tools to deliver contaminant in order to mimic specific scenarios. Tools obtained or developed by the testing laboratory, which have no performance-specification standard or vendor-provided performance information should be tested to determine their accuracy and precision. At a minimum, the tool should be used reproducibly from test to test. The exact usage should be documented, and the test results compared to baseline (agent, no decontaminant) test data to evaluate improvement gained using decontaminant.

- **Decontaminant Delivery Tool:** the tool used to deliver a specific volume of decontaminant to the item-contaminated surface. The specific decontaminant under evaluation will typically determine the delivery tool and decontaminant volume.
  - Pipette: The tool with the largest range of delivery volumes. Positive-displacement pipettes with disposable tips are preferred for work with chemical contaminants to prevent cross-contamination. Pipettes used for the purpose of decontaminant delivery should be compliant with the required performance specifications listed in the most current versions of ISO 8655 Parts 1 and 2 and/or ASTM E 1154, for the volume being measured.
  - Spray Bottle: Some applications will mimic a spray application using a spray bottle. The tool should be evaluated to determine the number of pumping actions required to achieve target decontaminant application. Tools obtained or developed by the testing laboratory, which have no performance-specification standard or vendor-provided performance information, should be tested to determine their accuracy and precision. At a minimum, the tool should be used reproducibly from test-to-test and the exact usage should be documented.
  - Developmental Breadboard, Brassboard or Prototype Technology: These are technologies under development, which are not in final configuration. The decontaminant generation and delivery may not be known. Tools obtained or developed by the testing laboratory, which have no performance-specification standard or vendor-provided performance information should be tested to determine their accuracy and precision. At a minimum, the tool should be used reproducibly from test to test, and the exact usage should be documented.

- Vendor-Provided Technology: This is equipment provided from a vendor that may be breadboard, brassboard, prototype, or commercial in configuration. The technology is operated with vendor guidance. Tools obtained or developed by the testing laboratory, which have no performance-specification standard or vendor-provided performance information should be tested to determine their accuracy and precision. At a minimum, the tool should be used reproducibly from test to test, and the exact usage should be documented.

- **Analytical Standard Preparation Tools:** These are the tools used to prepare sample dilutions. The tool must be capable of delivering the specified liquid volume. Single-dispensing tools (i.e., not repeater tools) are preferred because these typically have higher accuracy and precision. Fresh tips must be used for each sample to prevent cross-contamination.
    - Pipette: The tool with the largest range of delivery volumes. Positive-displacement pipettes with disposable tips are preferred for work with chemical contaminants to prevent cross-contamination. Pipettes used for the purpose of sample dilution or analytical standard preparation should be compliant with the required performance specifications listed in the most current versions of ISO 8655 Parts 1 and 2 and/or ASTM E 1154 for the volume being measured.
    - Volumetric Glassware: Volumetric flasks should be Class A and meet the specifications in the most current version of ASTM standards E288 and E69.
- **Environmental Chamber (optional):** The environmental chamber is a temperature- and relative humidity-controlled chamber for the preconditioning and aging items. The fixture should be able to maintain test specific environmental conditions (e.g., temperature and relative humidity), even when adding or removing samples. The system must have the ability to log temperature and relative humidity data, and be able to store and download temperature and humidity data and traces to a computer for further analysis. The system must be able to maintain temperature and relative humidity. The system operation and range should be known.
- **Vapor Test Chamber:** The vapor test chamber is an enclosed structure of sufficient size to completely contain the item, with the following requirements. General guidance for vapor chamber construction can be located in ASTM D 5116-06 "Standard Guide for Small-Scale Environmental Chamber Determinations of Organic Emissions from Indoor Materials/Products." Several considerations for decontaminant evaluations are provided in the following list.
    - The chamber should be constructed of inert materials.
    - The chamber should ideally run under positive pressure to minimize contamination inside the chamber.
    - The vapor chamber must have a clean air supply with tight control of the chamber airflow rate (±5% minimum).
- **Mass flow controllers** or mass flow meters are preferred over volumetric flow meters (requires standard temperature and pressure [STP] correction).
    - The chamber should have the ability to measure temperature and humidity. Control of temperature and humidity are ideal.

- The chamber should provide a well-mixed environment.
- The volume of the chamber must be known.
- The chamber must have an exhaust port to enable collection of vapor samples.
- The sampling airflow must be known.

- **Analytical Chromatography Equipment:** The method produces liquid samples and solid-sorbent tube samples for chromatographic analysis. The test facility must have the capability to quantitatively analyze the samples immediately after testing. Gas and liquid chromatography equipment, fitted with mass selective detectors is preferred. Other quantitative detectors may be used.

MATERIALS AND SMALL EQUIPMENT

- **Aluminum foil:** Aluminum foil is typically used to line workspace.
- **Anemometer:** Used to measure the force, speed, and sometimes direction of air movement. A wind gauge.
- **Analytical vials and caps**: Appropriate analytical vials for use on the chromatographic equipment. The vial cap should be lined with an inert material. PTFE/Teflon is the preferred material to prevent extraction of plasticizers or other impurities into the sample.
- **Decontaminant bath**: Used to collect spent disposable test items (e.g., pipette tips, analytical vials, and caps, etc.) in a solution that will neutralize any agent left on the material. For decontaminating most chemical agents, this bath contains an excess volume of household bleach to submerge items.
- **General laboratory items:** Items may include glassware (vials, flasks, cylinders, bottles, beakers, jars, etc.), paper towels, beakers, vials, spatulas, parafilm, etc.
- **Items:** The test sample for evaluation.
- **Sample handling tools:** The tools for handling items during testing, which may include forceps, tweezers, or tongs.
- **Sample tray:** Optional item for the handling and movement of items during testing.
- **Standard laboratory record-keeping items:** Record-keeping items may include computer, data test form, laboratory notebooks, and writing utensils.
- **Timing device(s):** Test method requires accurately timing key steps. Digital timers reporting in minutes and seconds are preferred.
- **Solid-sorbent tubes:** A tube such as a depot area air-monitoring (DAAM) tube containing a solid sorbent that absorbs the contaminant. Typical solid sorbents include Tenax, Chromasorb, or Haysep. The appropriate sorbent should be used for the contaminant being tested. ASTM method D 6196 "Practice for Selection of Sorbents, Sampling, and Thermal Desorption analysis Procedures for Volatile Organic Compounds in Air" provides detailed guidance for the selection of the appropriate sorbent tube.

ADDITIONAL MATERIALS AND EQUIPMENT: The testing facility decontaminant preparation and application processes may require additional materials and equipment. Additional materials and equipment may include, but are not limited to, analytical balance, stir plate, stir bars, vortexer, pH meter, transfer pipettes, sample trays, and sample transport containers.

SAFETY PRECAUTIONS

This document does not claim to address all of the safety concerns associated with chemical decontaminant testing because the requirements may vary based on facility, state and other regulatory requirements. It is the responsibility of the user of this method to establish appropriate environmental, health, and safety practices for the use of this method and handling of generated wastes in compliance with applicable regulations prior to use. Users of this method should conduct testing in appropriate facilities and follow proper laboratory practices, including the use of appropriate personal protective equipment and material safety data sheets.

PROCEDURES

Execution of a vapor test requires determining the appropriate settings for all experimental variables including the sampling plan, item treatment, and vapor-sampling method, vapor test chamber settings (air-change rate, air velocity, and temperature), air-mixing evaluation, and analytical detection limits to ensure an emission factor can be calculated that meets the requirements of the program. It is advisable to start by reviewing the calculation section to understand how each of these variables may impact the results and what type of results can be generated. The procedures to execute a small-item vapor test are documented in this section.

PROCEDURE 1: VAPOR CHAMBER SELECTION

The test chamber significantly contributes to the test results. Some considerations for chamber selection are described in the following list:

- **Volume:** The volume of the test chamber is inversely proportional to the observed concentration for the evaluation of the same item. Larger chambers will result in lower concentrations (higher/poorer detection limits). Selection of the smallest chamber that is able to accommodate the test item is recommended. However, there is an operational limit to the capacity of a chamber. An item should not displace more than 25% of the chamber volume to facilitate mixing in the chamber.
- **Air mixing:** Good air mixing inside the chamber is vital to accurately measure the vapor concentration and calculate the emission rates. Chamber mixing is a function of chamber geometry, item geometry, and internal air velocity. The internal air velocity is often controlled by variable speed fans located inside the chamber. Higher fan speeds usually generate turbulent flow conditions, which provide good mixing. If the vapor emission mechanism is evaporative, the air velocity may impact emission rates such that higher air velocity may produce more emission. For this reason, it is advisable to balance the air velocity so that it is similar to the expected scenario and provides good mixing (typical indoor scenarios range from 0.05–0.2 cm/s).
- **Cleaning:** The chamber must have the ability to be cleaned between tests.

- **Test temperature:** Vapor emission is a result of mass transport of agent out of the material. Most mass transport processes (e.g., vapor emission) are influenced by temperature, and some material-agent combinations may be influenced more than others. Usually, higher temperatures increase vapor pressures and transport/diffusivity rates, resulting in higher emissions (and higher vapor concentrations). The chosen test temperature should be as similar to the scenario as reasonable.

PROCEDURE 2: DETERMINATION OF ANALYTE BREAKTHROUGH ON SOLID-SORBENT TUBES

Breakthrough is the result of weak analyte and sorbent interactions which, as a function of air volume, temperature, and flow rate, could result in lack of analyte retention in the sorbent. It is vital to ensure that the collection of samples with the potential for breakthrough is avoided in the methodology because this would result in underestimation of the vapor concentration (and consequently underestimation of the hazard). Even with strong analyte-sorbent interactions, breakthrough can occur as a function of the air volume passed through the solid-sorbent tube, the temperature, and airflow rate.

This procedure should be conducted for each analyte-sorbent pair of interest using the selected sampling method. Ideally, the breakthrough test should be conducted at the harshest condition to be studied (e.g., highest temperature and flow rate). If breakthrough does not occur at the harshest condition, then breakthrough will not occur at ambient conditions. This procedure only needs to be repeated when new analytes, different sorbents, or different sampling methods (e.g., air volume, airflow rate, and higher temperatures) are used. This procedure will identify the Safe-Sample Volume (SSV), which indicates the maximum volume of air that should be sampled during an experiment. A single sample evaluation is presented in this section. Multiple sampling times can be used, and the results can be analyzed using linear regression.

When using this method, refer to the following documents for detailed background and guidance regarding breakthrough determinations. A general procedure is provided in this section specific to typical decontaminant vapor testing.

- American Society for Testing and Materials (ASTM), *Practice for Selection of Sorbents, Sampling, and Thermal Desorption Analysis Procedures for Volatile Organic Compounds in Air;* ASTM method D 6196, West Conshohocken, PA.
- EPA *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air;* Compendium Method TO-17, Section 10.8, Second Edition, 1999.

This test uses two solid-sorbent tubes (see Figure 1). The first tube in line is referred to as Tube 1. Tube 1 is spiked with a known mass of agent. Tube 2 is connected downstream of Tube 1 using an appropriate union fitting. The tubes are connected to the sampling system used for experiments, and air is pulled through the tubes at a measured flow rate and time.

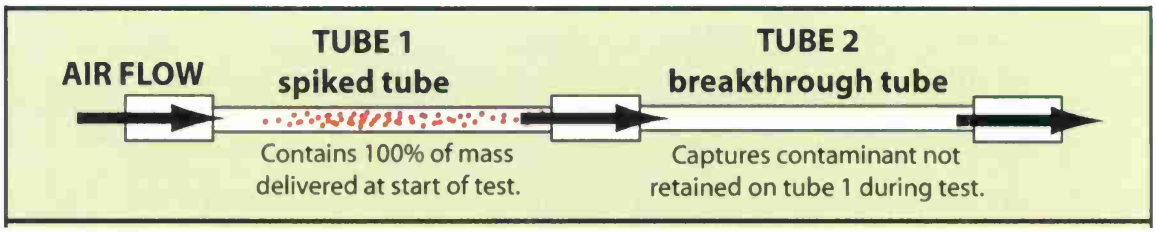

Figure 1. Breakthrough test Tube 1 and Tube 2 representation.

The mass of analyte detected on Tube 2 is referred to as the *breakthrough mass*. The *breakthrough volume* is defined as the volume of air passed through Tube 1, which results in a breakthrough mass that is approximately 5% of the delivered mass. The *safe-sample volume* is defined as 70% of the breakthrough volume per ASTM D 6196.

To perform a general breakthrough test procedure, follow these steps:

1. Spike Tube 1 with a known mass. This mass on tube should represent the high end of the method calibration curve used to analyze samples. If a laboratory has multiple methods, this test should be performed using the highest range curve.
2. Connect Tubes 1 and 2 to the vapor chamber where samples are typically collected.
3. Run the empty vapor chamber at the desired operating chamber airflow rate, sampling flow rate, and temperature for a specified period of time. A sampling time twice the length of the desired sampling time is recommended.
4. Analyze both tubes using appropriate chromatographic technique. The analytical method used for Tube 2 must be capable of quantifying 5% of the spike mass.
5. If 5% of the delivered mass is recovered on Tube 2, breakthrough has occurred. A safe-sampling volume would be 70% of the air volume used. For example, if the breakthrough test used a 120 min pull time, resulting in 5% breakthrough, then an 84 min pull time can be used for testing without changing the airflow rate or temperature.
    - Note: If greater than 5% breakthrough is observed, the safe-sampling volume will be less than 70% of the air volume used. A second test with adjusted parameters would be required to determine the parameters generating a 5% breakthrough so that the SSV could be determined.
6. If the SSV is significantly small, the following guidance should be considered to enable decontaminant vapor testing and emission-factor calculations:
    - Consider using a different sorbent.
    - Consider using a lower amount of contaminant mass for the tube loading (i.e., less mass on tube).
7. To ensure that breakthrough has not occurred at the final airflow rate, air volume, and temperature operating conditions, a final test at the desired pull time should be performed to confirm that breakthrough does not occur.

PROCEDURE 3: DETERMINATION OF CHAMBER FREE-AIR VOLUME

The chamber free-air volume is required for the data calculations to determine the air-change rate and loading factor. The chamber free-air volume is calculated as the chamber total interior volume minus the volume of supporting hardware (e.g., fans, bake-out heater) and test items.

The volume of complex items may be calculated using Computer Aided Design (CAD) software or by simplifying the geometry of basic objects such as cylinders, boxes, or spheres. Error in the free-air volume can have a significant impact in the calculated emission value. The impact of free-air volume calculation error should be evaluated before calculating chamber hardware and test item volumes. The detailed texture in some items may influence only 0.0001% of the free-air volume. The free-air volume is calculated to three significant figures. The laboratory process for determining free-air volume, including chamber and item volume calculations, should be provided with the technical data package.

PROCEDURE 4: VAPOR-SAMPLING PLAN DEVELOPMENT

The vapor-sampling plan is the schedule for when and how long a vapor sample is collected during the vapor test. The vapor-sampling plan's goal is to provide a sampling schedule that will load each tube with an analyte mass that can be detected without saturating the detector during long sampling times. In addition, the sampling schedule should not result in the loading of a mass that is below the analytical method's detection during short sample times. The selection of tube midpoint times and tube pull times is a combination of the following items:

- Vapor chamber operating parameters (e.g., airflow rate, air volume, and temperature)
- SSV (determined by the breakthrough test)
- Sample under investigation
- Analytical method calibration range

The vapor test solid-sorbent tube results are used to calculate the emission value, which varies as a function of time. Sample collection timing is determined by the emission characteristics, which can vary for different materials. Sampling duration is determined by the item's vapor off-gassing concentration, solid-sorbent tube SSV, and the dynamic range of the analytical instrumentation.

The planning process requires a bit of trial-and-error. The steps provided are general guidance that can be used to determine the system schedule. Anyone highly skilled in the art of vapor testing may execute testing using procedures already established at their facility. Requirements should not be ignored. Constructing a test outside of requirements could severely impact and may invalidate test results.

The following procedure provides guidance for the construction of a successful sampling plan with an example from small-item evaluation, using portable DVD players contaminated with a dilute HD solution.

1. **Determine vapor test airflow settings to include the air-change rate and loading factor.**

The key variables for indoor scenarios are the room volume, the air-change rate, and the number of items in the scenario. There are cases where the air velocity inside the room may influence emission rates. The same variables affect the vapor concentration inside a vapor chamber. The emission calculation normalizes these variables; however, the test values should be carefully considered and aligned with the scenario, if possible.

The air-change rate is the ratio of the chamber airflow rate to the chamber free-air volume, reported in units of 1/time. Some facts regarding air-change rate selection are detailed in the following list.

- Air-change rates are inversely proportional to vapor concentration; doubling the air-change rate will decrease vapor concentration by a factor of two.
- Large air-change rates, 0.08–0.167 min^{-1} (5–10 h^{-1}), will occur on occasion in certain scenarios.
- Typical air-change rates in indoor scenarios will be in the range of 0.008–0.08 min^{-1} (0.5–5 h^{-1}).
- It is advisable to use air-change rates similar to the scenario to be modeled.
- Large air-change rates provide "dilution" that will generate lower vapor concentrations (high/poor detection limits), but the rate of change of the emission factor can be well characterized.
- Small air-change rates will increase vapor concentrations (enabling lower/better detection limits), at the expense of the ability to characterize how the emission rate changes as a function of time.
- A recommended air-change rate minimum of 0.08 min^{-1} (0.5 h^{-1}), provides a balance of detection limits and time resolution. Typical tests may execute with air-change rates in the range of 0.016–0.03 min^{-1} (1.0–2.0 h^{-1}).
- Chamber mixing is typically optimal at low air-change rates and decreases as air-change rates are increased. The test airflow properties should be aligned with scenarios, if available.

2. **Determine the test duration.**

The test duration should be aligned with the scenario of interest. If no scenario is available, the recommended test duration is 12 h. Test durations should not be shorter than 6 h because the use of extremely short experiments could limit the ability to properly characterize the emission source.

3. **Determine the number of solid-sorbent tubes.**

Characterization of the emission source should use no less than six solid-sorbent tubes.

4. Determine the midpoint times (t_m) for all tubes.

The initial sampling should start no sooner than 2.3/air-change rate. This allows the chamber to mix and produce a measurable concentration. The last tube should be sampled at the end of the test duration. Most items have nonlinear decay characteristics where the concentration changes rapidly early in the experiment. To capture this characteristic, and accurately measure an emission factor/rate, more samples are collected early in the experiment. Examples of midpoint times for the DVD player test are shown in Table 1.

Table 1. Example data set midpoint time values.

Tube #	Midpoint Time (min)
1	10.1
2	30.1
3	60.1
4	90.1
5	150.1
6	250.1
7	400.1
8	720.1
9	1380.1

5. Determine maximum pull time for each tube.

5.1 The pull time for a tube has design rules that must be observed as follows:

- Requirement: The SSV of the sorbent, determined in Procedure 2 cannot be exceeded.
    - The volume of sampled air is calculated as the pull time multiplied by the sampling flow rate.
    - The sampling flow rate is usually constant during a test to facilitate this procedure. Calculate the maximum pull time that would result in sampling the SSV at the selected sampling flow as shown in Equation 1.

$$\text{max pull time} = \frac{\text{SSV}}{\text{F}} \qquad \textbf{Equation 1}$$

where

max pull time	=	pull time not to exceed (min)
SSV	=	safe-sample volume (mL)
F	=	sampling flow rate (mL/min)

- Requirement: The total sampling flow cannot exceed 50% of the chamber flow rate.

- Requirement: The minimum pull time must be reproducible. The sampled air volume error, resulting from sample flow controllers establishing set-point flows, should be <2%. Guidance: 30 s is usually a safe lower bound for PID-controlled mass flow controllers. This value should be checked for the testing hardware.

5.2 Selecting a pull time should produce a sample with an agent mass in the calibration range of the analytical method. If there is prior data or an estimation of the observed test vapor concentration, an ideal pull time can be calculated using the following method. An example total pull time schedule for the DVD test is provided in Table 2.

5.2.1 Identify a target mass (M_t) to load on the tube. Guidance: Select a target mass of 33% of the maximum mass on tube. For example, if the HD high-level method, with calibration ranges of 50–1500 ng on tube is used, 33% of the maximum mass is 495 ng.

5.2.2 Identify the expected vapor concentration (C) in milligrams per cubic meter (mg/m^3) and the sampling flow (F) in cubic meters per minute (m^3/min).

5.2.3 Calculate the pull time ($t_{pull,\,i}$) for tube *i*, using the midpoint time for tube *i* ($t_{m,i}$) as shown in Equation 2.

$$t_{pull,i} = \frac{M_t}{C(t_{m,i}) \times F} \qquad \textbf{Equation 2}$$

5.2.4 If the calculated pull time exceeds the SSV (or maximum pull time), consider using an analytical method with lower detection limits, select a lower target mass, and repeat this procedure.

- If the lowest analytical method is used, and the pull time requires sampling more than the SSV, obtain samples at the SSV.
- If a lower emission factor/rate detection limit is desired, and it is acceptable within the test program, the air-change rate can be decreased, which increases the chamber vapor concentration.

Table 2. Example data set midpoint and total pull time values.

Tube #	Total Pull Time (min)
1	4.0
2	6.0
3	8.0
4	10.0
5	10.0
6	15.0
7	25.0
8	35.0
9	50.0

6. Calculate the start and end time for each tube.

6.1 The start time for each tube is calculated using Equation 3.

$$\text{start time}_i = t_{m,i} - \frac{t_{pull,i}}{2} \qquad \textbf{Equation 3}$$

6.2 The end time for each tube is calculated using Equation 4.

$$\text{end time}_i = t_{m,i} + \frac{t_{pull,i}}{2} \qquad \textbf{Equation 4}$$

6.3 Ensure that the start and end times do not overlap between tubes. If there is overlap, consider increasing the sampling flow rate to decrease pull time. An example pull schedule is provided in Table 3.

Table 3. Example data set sampling time values.

Tube #	Total Pull Time (min)	Midpoint Time (min)	Start Time (min)	End Time (min)
1	4.0	10.1	8.1	12.1
2	6.0	30.1	27.1	33.1
3	8.0	60.1	56.1	64.1
4	10.0	90.1	85.1	95.1
5	10.0	150.1	145.1	155.1
6	15.0	250.1	242.6	257.6
7	25.0	400.1	387.6	412.6
8	35.0	720.1	702.6	737.6
9	50.0	1380.1	1355.1	1405.1

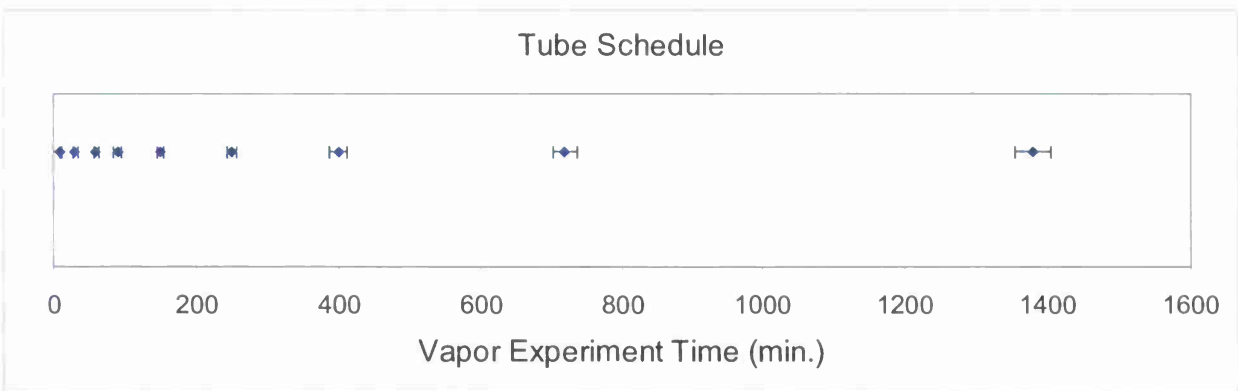

Figure 2. Graphical representation of the tube-pulling schedule.

7. **Document the sampling plan in the test report.**

PROCEDURE 5: DETERMINATION OF CHAMBER MIXING (TRACER GAS DECAY)

A requirement for the application of the mass balance equation, which is used to calculate an emission factor, is that the chamber must have a well-mixed environment so that the exhaust vapor concentration sampled is representative of the chamber vapor concentration. If the chamber has a poorly mixed environment, the calculated emission factor may have significant errors, and measured vapor concentrations may be erratic and not reproducible. For this reason, the mixing for each chamber and item configuration should be evaluated.

Chamber mixing is evaluated by establishing a uniform concentration of a tracer gas in the chamber and measuring the decay as a function of time. If a sensor with near real-time response for the concentrations generated in the chamber is available, any tracer gas can be used. Chambers used for this testing should have the ability to control and measure humidity. Therefore, water vapor is used as the tracer gas, and the environmental relative humidity sensor is used to characterize the tracer gas decay. This requires the ability to produce a controlled humidity in the chamber, and the ability to quickly reduce the humidity of the chamber airflow to a minimal value. The air supply to the chamber should have a dew point of less than or equal to -30 °C. This corresponds to supplying a 20 °C chamber with 0.33 g water/m^3 air, while the calculation assumes 0 g water/m^3 air.

Two correction methods are provided to account for humidified chamber supply air. (1) Mixing (η) is evaluated by comparing the measured decay rate to that of an ideal chamber. The mixing value compares the integral of the measured response to the integral of the ideal response. (2) The test is conducted for periods in relation to the air-change rate. The time for one air change (t_n) is defined as $t_n = 1/n$, where n is the air-change rate. Air-change rate is defined as the chamber airflow rate (Q) divided by the chamber free-air volume (V). Mixing can be affected by the air-change rate or the air velocity in the chamber, which is determined by the mixing fans.

1. **Perform the mixing procedure.**
   - 1.1 Set the chamber conditions to the following test conditions.
     - 1.1.1 Calculate the chamber free-air volume (V) in cubic meters (m^3).
     - 1.1.2 Select desired air-change rate (n) in reciprocal minutes (min^{-1}).
     - 1.1.3 Calculate chamber flow rate (Q) in milliliters per minutes (mL/min) to achieve the desired air-change rate, Q = nV/1000000.
     - 1.1.4 Place a sample item inside the chamber.
     - 1.1.5 Position any mixing fans as they would be used in a test.
     - 1.1.6 Set mixing fans to the air velocity to be used during testing.
     - 1.1.7 Set chamber airflow.
     - 1.1.8 Seal chamber door.
     - 1.1.9 Measure air velocity over the sample using an appropriate tool such as a hot wire anemometer.

1.2 Charge the chamber with tracer gas.

1.2.1 Log the chamber tracer gas concentration at an interval of at least once every 30 s.

1.2.2 Guidance: Ensure the chamber is equilibrated by waiting for the chamber concentration to remain constant for at least 0.5 t_n.

1.3 Stop the tracer gas injection. Time zero for tracer gas decay testing is defined as the first measurement point with a value below the equilibrium value.

1.4 Log the tracer gas concentration (RH), chamber temperature, and chamber airflow for 1.5 t_n.

2. Calculate the air-change rate.

Calculate the measured air-change rate as shown in Equation 5.

$$n = \frac{\sum_{j=1}^{J}(Q_j)}{1{,}000{,}000 \cdot J \cdot V} \qquad \textbf{Equation 5}$$

where
n = air-change rate (min^{-1})
Q_j = chamber airflow at log point j (mL/min)
V = chamber free-air volume (m^3)
j = log index
J = number of log points corresponding to t_n length of time

3. Convert the relative humidity to absolute humidity.

Convert relative humidity, RH, to absolute humidity, H, (i.e., water vapor concentration) for each log point. Several equations exist, one has been provided in Equation 6.

$$\mathrm{H}(RH,T) = \frac{RH \cdot 13.2238 \cdot \exp\left(\frac{17.27 \cdot T}{T + 237.3}\right)}{T + 273.16} \qquad \textbf{Equation 6}$$

where
H = absolute humidity (g water/m^3)
RH = relative humidity (%)
T = temperature (°C)

4. Calculate the mixing factor (η).

Calculate the mixing factor (η) for the first air change, following the option that best aligns with the air source.

Option A: Dry air assumption. This calculation assumes that the chamber airflow supply has a tracer gas concentration of 0 (g/m^3) and that there are no sinks or sources in the system. If there are tracer gas sources (i.e., emitters) or the chamber airflow supply has a finite concentration, the mixing test will fail faster (i.e., it is harder to pass mixing criteria).

$$\eta = \left(1 - \frac{\sum_{i=1}^{J} \left| C(t_i) - C_0 e^{-nt_i} \right|}{\sum_{i=1}^{J} C_0 e^{-nt_i}}\right) \times 100\% \qquad \textbf{Equation 7}$$

where

η	=	mixing level
n	=	air-change rate = Q/V = chamber flow/chamber volume
t_i	=	time at measurement point i
i	=	log index number
t_{i-1}	=	time at a previous measurement point
C_0	=	concentration at time zero
j	=	number of log points that correspond to t_n length of time

Option B: Chamber air supply dew-point correction. If the chamber air supply has a finite concentration of the tracer gas, this should be accounted for in the mixing equation. Calculate an estimate of the steady-state chamber air supply concentration (C_s) from the dew-point temperature (T_d) of the air supply (Equation 8). For example, commercial air driers may provide air with T_d = -30 °C. Calculate the mixing level for the chamber using Equation 9.

$$C_s = \mathrm{H}(T_d) = \begin{cases} \dfrac{1322.38 \cdot \exp\left(\dfrac{17.27 \cdot T_d}{T_d + 237.3}\right)}{T_d + 273.16} & T_d > 0\,^{\circ}\mathrm{C}\ \text{(dew point)} \\ \dfrac{1322.38 \cdot \exp\left(\dfrac{21.875 \cdot T_d}{T_d + 265.5}\right)}{T_d + 273.16} & T_d < 0\,^{\circ}\mathrm{C}\ \text{(frost point)} \end{cases} \qquad \textbf{Equation 8}$$

$$\eta = \left(1 - \frac{\sum_{i=1}^{J} \left| C(t_i) - \left(C_s - (C_s - C_0) \cdot e^{-nt_i}\right) \right|}{\sum_{i=1}^{J} C_s - (C_s - C_0) \cdot e^{-nt_i}}\right) \times 100\% \qquad \textbf{Equation 9}$$

where

η	=	mixing level
n	=	air-change rate = Q/V = chamber flow/chamber volume
t_i	=	time at measurement point i
i	=	log index number

Option C: Chamber air supply continuous correction. If the chamber air supply has a finite concentration of the tracer gas that can be measured during the test, the following procedure can be used. The humidity probe must be located in the chamber air supply stream and provide accurate measurements for low humidity. Calculate the humidity of the chamber air supply for each log point using Equation 10. Calculate the mixing level for the chamber using Equation 11.

$$C_s(t_i) = \mathrm{H}(RH_i, T_i) = \frac{RH_i \cdot 13.2238 \cdot \exp\left(\frac{17.27 \cdot T_i}{T_i + 237.3}\right)}{T_i + 273.16} \qquad \textbf{Equation 10}$$

$$\eta = \left(1 - \frac{\sum_{i=1}^{J} \left| C(t_i) - \left[C_s(t_i) - \left(C_s(t_i) - C_0\right) \cdot e^{-nt_i}\right]\right|}{\sum_{i=1}^{J} \left[C_s(t_i) - \left(C_s(t_i) - C_0\right) \cdot e^{-nt_i}\right]}\right) \times 100\% \qquad \textbf{Equation 11}$$

where

η	=	mixing level
n	=	air-change rate = Q/V = chamber flow/chamber volume
t_i	=	time at measurement point i
i	=	log index number

5. Assess the mixing.

If η > 80%, mixing is considered adequate.

6. Report the following information.

- chamber V
- measured n
- chamber flow
- fan settings
- air velocity (and how measured)
- item description and configuration
- mixing level
- mixing result graphs and item configuration pictures are encouraged to enhance data report

PROCEDURE 6: ITEM TREATMENT AND VAPOR TEST

Procedure 6 specifies the sample treatment and vapor sampling actions. Additional steps for moving samples between workspaces (i.e., sample containment and transfer between engineering controls/hoods), sample decontamination, and waste disposal steps are not presented here. Those steps should be added as appropriate, based on the method user's

facility safety and regulatory requirements. Several steps have options to enable evaluation of different types of decontaminants and control tests.

1. **Prepare for the test.**

Ensure that all necessary equipment, materials, reagents, and analytical capabilities are available for the test. All equipment should be confirmed as operational before starting the test. Preparation tasks for this method may include:

- Turn on any equipment that will need to thermally equilibrate (e.g., environmental chamber, vapor chamber).
- Select the contamination method, complete preparatory calculations, and plan sample contamination.
- Obtain documented decontaminant application method.
- Complete test area set-up tasks, including any labeling (e.g., vials, trays) and other associated pre-test tasks that can be performed.
- Prepare items for testing,
- Prepare the decontaminant.
- Check to make sure that the necessary equipment is operational and that all calibrations are current (if applicable).
- Obtain the contaminant. The contaminant may require thermal equilibration to room temperature (or other sponsor-specified temperature) prior to use.

This procedure can be applied to multiple items during a single test session. In that case, it is important to treat each item identically. The use of timing charts to stagger major steps is strongly encouraged because subtle differences in item treatment may contribute to data scatter. The vapor test timeline is depicted in Figure 3.

The number of replicate items is dependent on the test objectives and item availability. Reuse of items is not recommended as items may contain entrapped agent that could resurface over time, creating a false positive test interference. Three dose-confirmation samples should be used per contamination set.

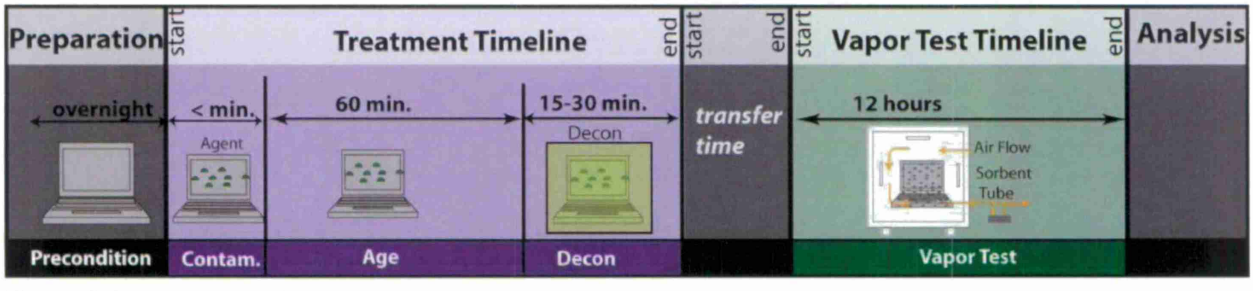

Figure 3. Vapor test timeline representation.

2. Precondition the items.

Identify and execute the desired conditioning method. Items should be conditioned at the desired test temperature for at least 60 min. The recommended conditioning time period is at least 12 h.

OPTION A: At the test site/laboratory/room conditions. Items should be covered if there is a risk of contamination from foreign material at the conditioning location. The environmental conditions should be recorded.

OPTION B: At a specific temperature using an environmental chamber. The preferred moderate condition case temperature is 21 ± 3 °C (70 ± 5 °F), with ±5 °C maximum. Other temperature settings can be used. Temperature control should be within ±5 °C because spans greater than ±5 °C may introduce significant scatter for some materials. At a minimum, relative humidity should be measured and reported. If relative humidity can be controlled, then relative humidity can be specified. The environmental chamber should be operated in accordance with manufacturer's specifications, if available. A generalized procedure for item preconditioning, using an environmental chamber, could include:

2.1 Set the environmental chamber to the specified test condition.

2.2 Allow the environmental chamber to equilibrate at the set-point temperature and relative humidity. The time to reach set-point equilibration may vary based on equipment and set-point conditions. Temperature and humidity should be maintained at the setpoint for at least 30 min before the start of conditioning.

2.3 Place the items in the chamber with the test surface facing upwards.

2.4 Condition the items for at least 60 min. If possible, items should be preconditioned overnight.

- Note: some materials may require special preconditioning treatments. For example, cellulose-based materials and concrete contain significant moisture. These types of materials do not typically achieve moisture equilibrium in less than 24 to 48 h. Longer preconditioning times may be required for certain materials. An example procedure for wood is ASTM D4442.

2.5 Minimize temperature fluctuations by removing samples from the environmental chamber immediately before executing Step 3: Contaminate the items.

3. Contaminate the items.

3.1 Select the contamination option from the following choices:

- Full contamination
- Localized contamination
- No contaminant: This is a negative control test to evaluate whether the item or decontaminant-item pair emits vapors that may interfere with the chromatographic analysis of the desired analyte. Proceed to Step 5: Allow the item to age.

3.2 Determine the contaminant volume and drop size as follows:

3.2.1 Select the target starting challenge (i.e., contamination density), in units of grams per square meter (g/m^2).

3.2.2 Determine the item footprint in square meters (m^2).

3.2.3 Determine the mass of the contaminant.

3.2.4 Determine the volume of the contaminant to be applied, using the calculated mass, and contaminant density.

3.2.5 Select the drop volume(s) to be used.

3.2.6 Determine the number of drops to be applied.

3.3 Identify the contamination regions:

3.3.1 Identify the test item configuration. This is how the item will be placed for contamination, decontamination, and vapor testing. For example, a laptop may have several configurations including closed and open, as shown in Figure 4.

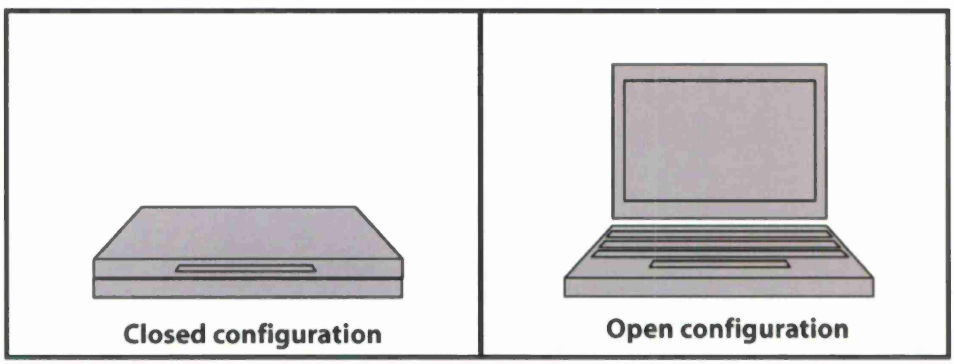

Figure 4. Example test item configurations for laptop.

3.3.2 Select the contamination areas. A test item may have several different contamination options, based on the test objective and procedures for handling the contaminated item safely. Three potential contamination areas for a laptop, with contaminated surfaces highlighted in red, are shown as an example in Figure 5.

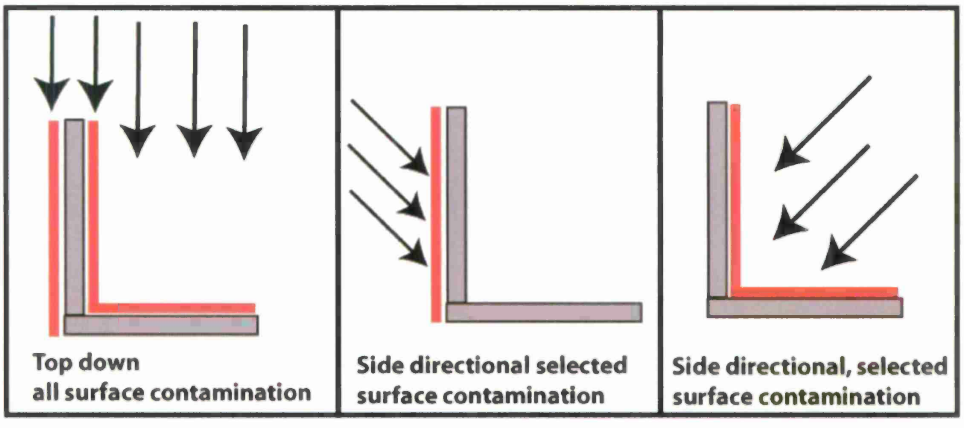

Figure 5. Example test item contamination areas for a laptop in the open configuration.

3.4 Identify the specific locations for contaminant placement:

3.4.1 Divide the selected surface into approximately 3 x 3 in. regions. An illustration of the laptop example is provided in Figure 6. The regions to be prepared should be marked on paper rather than mark the actual item because the contaminant mass transport may be affected by creating artificial barriers.

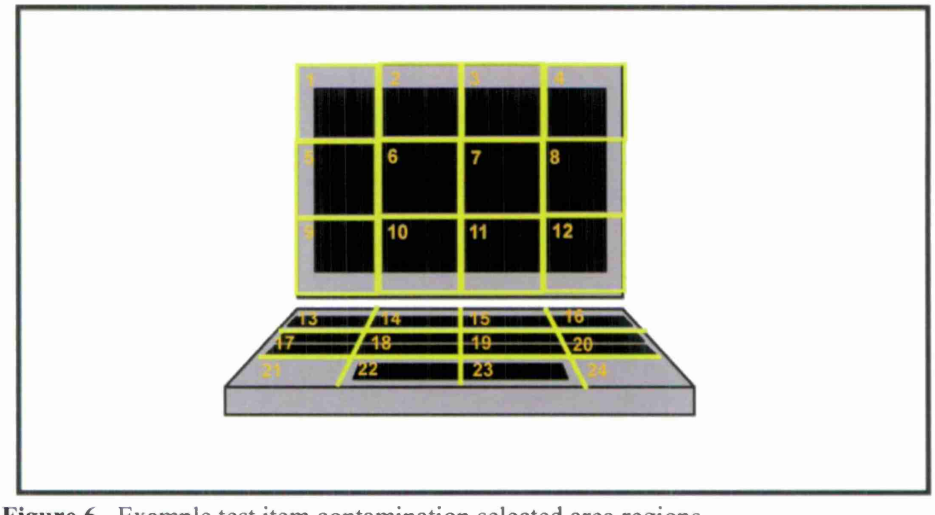

Figure 6. Example test item contamination selected area regions.

3.4.2 Select and document the contamination locations as follows:

- Full contamination: The contaminant should be evenly spaced over the entire selected surface area, contacting the different material types and interfaces. An illustration showing a laptop contaminated with a starting challenge of approximately 1 g/m^2 HD, applied as 29 drops that are each 2 µL in volume, is shown in Figure 7.

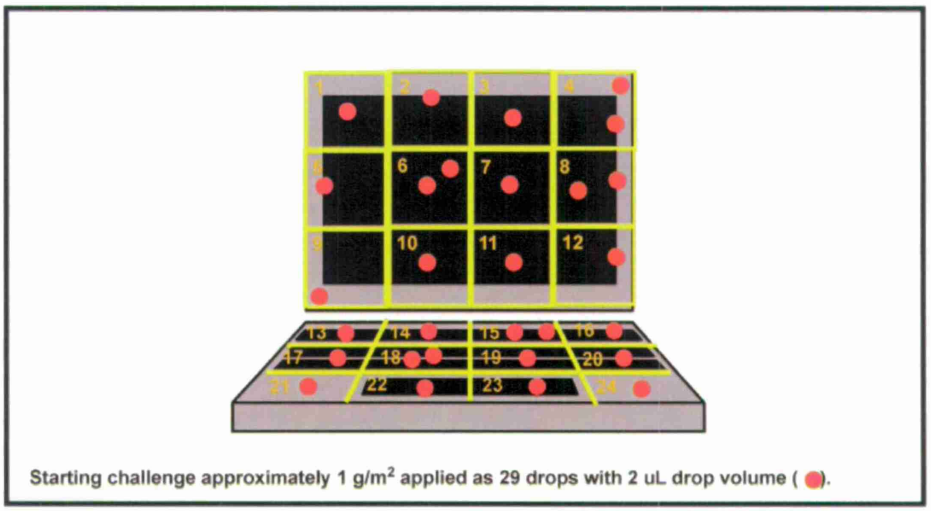

Figure 7. Full item contamination illustration.

- Localized contamination: The contaminant is placed in specified regions, based on the test objective. An illustration showing a laptop contaminated with a starting challenge of approximately 1 g/m^2 HD, applied as 29 drops that are each 2 µL in volume is shown in Figure 8. This example meets a test objective to evaluate the ability to decontaminate the screen-case interface.

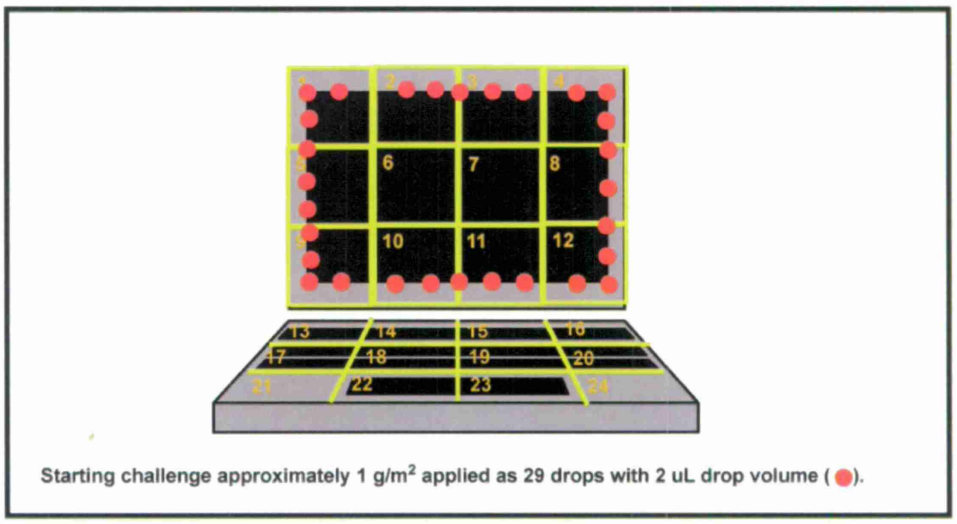

Figure 8. Localized contamination illustration.

3.5 Contaminate the item as directed in this step. Pipette application is used in this example. Other techniques can be used, if specified by the test sponsor. The alternate methods should be documented in the test report.

3.5.1 Set the dispensing tool to the appropriate drop volume.

- Note: The pipette volume should not be changed within a given set of procedures. Tests have shown that changing the tool's dispensing volume can affect delivery. If changes must be made, dose-confirmation samples must be prepared after each change.

3.5.2 Fit the pipettor with a clean, appropriate pipette tip.

3.5.3 Load the contaminant delivery tool in accordance with manufacturer's directions.

3.5.4 Prepare the initial dose-confirmation samples. At least three replicate samples are recommended.

3.5.4.1 Uncap the vial.

3.5.4.2 Deliver the appropriate number of drops to a scintillation vial containing 20 mL of extraction solvent to achieve the contamination density.

3.5.4.3 Cap the scintillation vial.

3.5.4.4 Thoroughly mix contents by inverting the vial three times.

Note: Steps 3.5.4.5, 3.5.4.6, and 3.5.4.7 may be performed later in the test when samples are diluted and prepared for analysis. This delay typically occurs in tests using a large number of panels enabling completion of the staggered timing chart. Note: The samples should be prepared for analysis on the same day as the test. Samples should be run as soon as possible after the end of the test to reduce potential issues due to sample degradation.

3.5.4.5 Uncap the scintillation vial.

3.5.4.6 Using a clean, disposable pipette, load the analytical vial with an aliquot of extractant solution.

3.5.4.7 Cap the analytical and scintillation vials.

3.5.5 Deliver the appropriate number of drops, needed to achieve the contamination density, to the surface. Reload the tool and repeat as needed. Treatment time starts after the item is contaminated. The use of timing charts for multiple samples is recommended.

- Note: If the repeater pipette is at rest for more than a few seconds, the pipette should be cleared by dispensing a drop onto adsorbent paper (M8 paper for surety tests) or equivalent. Solvent and agent evaporation can occur in the tip, affecting the next dose from the tool.

3.5.6 Prepare the final dose-confirmation samples. At least two replicate samples are recommended.

3.5.6.1 Uncap the vial.

3.5.6.2 Deliver the appropriate number of drops to a scintillation vial containing 20 mL of extraction solvent to achieve the contamination density.

3.5.6.3 Cap the scintillation vial.

3.5.6.4 Thoroughly mix contents by inverting the vial three times.

Note: Steps 3.5.6.5, 3.5.6.6, and 3.5.6.7 may be performed later in the test when samples are diluted and prepared for analysis. This delay typically occurs in tests using a large number of panels

enabling completion of the staggered timing chart. Note: The samples should be prepared for analysis on the same day as the test. Samples should be run as soon as possible after the end of the test to reduce potential issues due to sample degradation.

3.5.6.5 Uncap the scintillation vial

3.5.6.6 Using a clean, disposable pipette, load the analytical vial with an aliquot of extractant solution.

3.5.6.7 Cap the analytical and scintillation vials.

4. Observe the post-contamination contaminant-material interaction.

Guidance: When evaluating replicate samples, the lab-scale test utilizes imaging to account for the effect that the contaminated surface area can have on the final test result. This effect is most pronounced for contaminant–material pairs that display the greatest range in surface area coverage for an identical contamination application.

The ability to image real items is not trivial. The method guidance is to document the drop interaction after contamination as accurately as possible, either through visual inspection or using photography.

- Visual inspection: The drop-surface interaction is documented both in words and a hand drawing.
- Digital photography: Fixed-site photographic setup is used to visually capture the agent–contamination surface area coverage after dosing, aging, and any other critical steps in the decontamination process. Photograph resolution of 9 to 25 pixels per droplet measured is recommended, if surface area calculations are performed.
- Contrast enhancement: If further investigation is needed, contrast enhancement using a dye may be used. Adding the dye to the contaminant is not recommended as the dye may change the mass transport and vapor properties of the contaminant under investigation.

5. Allow the item to age.

5.1 Select the desired aging time. The standard aging time for the lab-scale test is 60 min. Different time periods may be used, depending on the test objective.

5.2 Select and execute the item-aging procedure.

OPTION A: At the test site/laboratory/room conditions. Items are aged at test site conditions, with the contaminated surfaces facing upward to minimize the potential for contaminant loss through contact transfer. Items should be covered, if there is a risk of contamination at the conditioning location. Covering the items also reduces potential for contaminant evaporation. Allow the items to age for the desired aging time.

OPTION B: At a specific temperature using an environmental chamber. Items are aged at specified conditions, with the contaminated surfaces facing upward to minimize the potential for contaminant loss through contact transfer. The

temperature preferred for a moderate condition case is 21 ± 3 °C (70 ± 5 °F), with ±5°C maximum. Other temperature settings can be used. Temperature control should be within ±5 °C because spans greater than ±5 °C may introduce significant scatter for some materials. At a minimum, relative humidity should be measured and reported. If relative humidity can be controlled, then relative humidity can be specified. The environmental chamber should be operated in accordance with manufacturer's specifications, if available. A generalized procedure for item preconditioning, using an environmental chamber, could include the following steps:

5.2.1 Set the environmental chamber to the specified test condition.

5.2.2 Allow the environmental chamber to equilibrate at the set-point temperature and relative humidity. The time to reach set-point equilibration may vary based on equipment and set-point conditions. Temperature and humidity should be maintained at the setpoint for at least 30 min before the start of aging.

5.2.3 Place the items in the chamber. Items should be placed with the contaminated test surface facing upwards. Items should be spaced appropriately to minimize contact between items.

5.2.4 Allow the items to age for desired aging time.

5.2.5 Remove samples from the environmental chamber at the end of the aging period.

6. Observe the post-aging contaminant–material interaction.

Guidance: When evaluating replicate samples, the lab-scale test utilizes imaging to account for the effect that the contaminated surface area can have on the final test result. This effect is most pronounced for contaminant-material pairs that display the greatest range in surface area coverage for an identical contamination application.

The ability to image real items is not trivial. The method guidance is to document the drop interaction after contamination either through visual inspection or using photography.

- Visual inspection: The drop-surface interaction is documented both in words and a hand drawing.
- Digital photography: Fixed-site photographic setup is used to visually capture the agent–contamination surface area coverage after dosing, aging, and any other critical steps in the decontamination process. Photograph resolution of 9 to 25 pixels per droplet measured is recommended, if surface area calculations are performed.
- Contrast enhancement: If further investigation is needed, contrast enhancement using a dye may be used. Adding the dye to the contaminant is not recommended as the dye may change the mass transport and vapor properties of the contaminant under investigation.

7. Pre-rinse the items.

The evaluation of small items of sensitive equipment is unlikely to use rinsing because water could have an adverse effect on some items. This section contains the options for rinsing and not rinsing.

OPTION A: No rinse. Rinsing is not performed. Please continue to Step 8.

OPTION B: Rinsing is performed. Before decontamination, contaminated items are rinsed to remove gross contamination. The amount of rinse water used should be identified and documented. Some of the considerations, such as materials and equipment may include:

- Rinse water delivery tool: The tool used to deliver specific volumes of water to remove contaminant from the surface. A repeater tool is recommended, if multiple items are used in each test. The tool used should have the ability to control flow rate to reduce operator-to-operator variations.
    - Bottle-Top Dispenser: These are precision liquid dispensers that can be connected to solvent and rinse water bottles. These tools are available in different configurations, based on liquid to be dispensed. The appropriate tool should be used to dispense organic solvents. Examples are the Dispensette and Brinkman brands. Bottle-top dispensers, to be used for the purpose of solvent delivery, should be compliant with the required performance specifications for the volume being measured. These specifications are listed in the most current versions of ISO 8655, Parts 1 and 5, and/or ASTM E 1154.
    - Pump: Other precision liquid-dispensing systems. The manufacturer's performance specifications should be reported and evaluated against acceptable measurement uncertainty for the particular test. Tools obtained or developed by a testing laboratory that has no performance specification standard or vendor-provided performance information should be tested to determine their accuracy and precision. At a minimum, the tool should be used reproducibly from test to test, and the exact usage should be documented.
    - Commercial water delivery system such as a pressure washer or garden sprayer.
- Rinsate collection container: If rinse water analysis is required, the rinse should be collected in a glass container of sufficient volume for the rinse water and extraction solvent, preferably a wide-mouth jar. The use of funnels or other tools that may uptake agent during collection should be limited. The use of plastic containers is not recommended for chemical agent testing. The container cap should be lined with an inert material to prevent extraction of plasticizers or other impurities into the sample.
- Hot soapy water: The rinse procedure may call for the use of hot soapy water.

OPTION C: Pre-clean step is performed. Pre-cleaning may include wipes used prior to decontamination to remove gross contamination. The type of wipe and application process should be documented in the test report.

8. Decontaminate the items.

8.1 Select the desired decontaminant residence time, based on test objective. Liquid decontaminants typically use 5 to 30 min residence times. Vaporous and other decontaminants may require longer residence times.

8.2 Select the desired environmental conditions for temperature and relative humidity.

- Note: The decontaminant hardware may determine the environmental conditions. For example, vaporous decontaminants typically use a decontamination chamber that is operated at the temperature and relative humidity required for effective decontamination.
- Note: The test location may determine the environmental conditions, if decontamination is not conducted using an environmental chamber.

8.3 Select and execute the decontamination procedure, choosing from the options below:

OPTION A, Vaporous Decontaminants: The item is placed in a decontamination chamber. Then the vaporous decontaminant is introduced into the chamber following a documented procedure. The item remains in the chamber for the specified residence period.

OPTION B: Liquid Decontaminants: FM 3-11.5 recommends using a decontaminant-to-contaminant ratio of 50:1. The decontaminant amount, application method, and environmental requirements may be dependent on the specific technology and test objective. The following list provides information regarding the application method and the use of an environmental chamber.

- Pipette application: The volume of decontaminant needed to achieve the target decontaminant-to-contaminant ratio is used. The decontaminant is evenly dispensed over the entire test surface. Some agent-material interactions could result in significant contaminated surface coverage. Smaller decontaminant volumes may not be able to adequately cover the entire contaminated surface, yielding data scatter due to decontaminant delivery.
- Spray application: The volume of decontaminant is applied using specified hardware. The hardware use is documented. The amount dispensed is measured and reported.
- Environmental chamber: The desired environmental conditions may require the use of an environmental chamber. The preferred moderate condition case temperature is 21 ± 3 °C (70 ± 5 °F), with ±5 °C maximum. Other temperature settings can be used. Temperature control should be within ±5 °C because spans greater than ±5 °C may introduce significant scatter for some materials. At a minimum, relative humidity should be measured and reported. If relative humidity can be controlled, then relative humidity can be specified. The environmental chamber should be operated in accordance with manufacturer's specifications, if available. A generalized procedure for item decontamination using an environmental chamber could include:

  a. Set the environmental chamber to the specified test condition.

b. Allow the environmental chamber to equilibrate at the set-point temperature and relative humidity. The time to reach set-point equilibration may vary based on equipment and set-point conditions. Temperature and humidity should be maintained at the setpoint for at least 30 min before the start of aging.

c. Apply the decontaminant.

d. Place the items in the chamber. Items should be placed with the contaminated test surface facing upwards. Items should be spaced appropriately to minimize contact between items.

e. Wait the desired decontaminant residence time.

f. Remove samples from the environmental chamber at the end of the decontamination period.

OPTION C: Other Decontaminants: Solid decontaminants, sorbent wipes, brushing, or mechanical scrubbing methods may be used in some applications. Solid and wipe technologies may also have reactive properties. These decontaminants should be used following a documented procedure. These tests could also be executed using an environmental chamber, as described in Option B.

OPTION D: Positive Control Test: No decontaminant is used for positive control tests. Positive-control tests may include the determination of the vapor emission baseline for the test treatment process and environmental conditions under investigation.

9. Perform a post-rinse and dry procedure.

9.1 The evaluation of small items of sensitive equipment is unlikely to use rinsing because water could have an adverse effect on some items. This section contains the options for rinsing and not rinsing.

OPTION A: No rinse. Rinsing is not performed. Please continue to Step 10.

OPTION B: Rinsing is performed. Please see Step 7, Option B, for rinsing details. Several factors should be considered for the drying process.

- Drying method: Passive drying is recommended at room conditions, preferably in a chemical fume hood (or equivalent), with an approximate airflow of 100 lfm. Controlled air dryers can be used. Blotting, wiping, or other direct-surface contact methods are not recommended because contaminant may be removed as part of the process.
- Item placement: The items should be positioned to increase the airflow over the surface.
- Dry time: A specific dry time should be selected and applied to all replicate tests. A general recommendation is that the items should not be dried for more than 30 min because contaminant evaporation could occur, resulting in different drying time periods and potential differences in vapor test results.

- Inspection of surface after dry time is complete: The surface should be inspected and documented, including any residual water that may be present.

9.2 Record the date and time then note that the end of the item treatment process timeline is complete.

9.3 Package and move the item to the vapor test location (if needed).

10. Perform the vapor test.

The exact steps for the execution of vapor sampling will be dependent on the vapor test chamber design. A general procedure for chamber operation is provided.

10.1 Turn off the chamber airflow and fans inside chamber.

10.2 Remove the chamber door.

10.3 Place the sample item in center of chamber in the desired test configuration.

10.4 Replace the chamber door, securing the seal as appropriate for the vapor test chamber being used.

10.5 Turn on the chamber airflow and fans.

10.6 Begin the sampling timeline. This step establishes the vapor test time zero (Figure 9).

10.7 Record the date and time then note the start of the item vapor-sampling process.

10.8 Collect the tubes per the vapor-sampling plan.

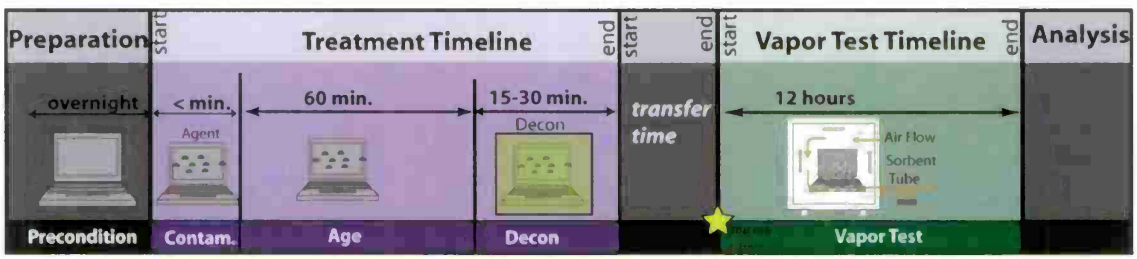

Figure 9. Vapor test timeline representation

11. Perform a chromatographic analysis for the agent.

11.1 Samples are analyzed. This test generates two sample types for analysis:

- Dose-confirmation liquid sample
- Vapor sorbent tube

11.2 Dose-confirmation samples.

11.2.1 Sample dilution may be required for the sample to be within the analytical method calibration range. This is typically true for the dose-confirmation samples.

11.2.2 Obtain the list of analytical results for extracts in units of nanograms per milliliter (ng/mL).

11.2.3 Correct the results for dilutions.

11.3 Solid-sorbent tubes.

11.3.1 Vapor analysis sample queue design should consider the amount of contaminant expected on the solid-sorbent tubes. The chance for analyte carryover is greater with higher contaminant mass loading on the solid-sorbent tube. A potential queue design could include the analysis of the lower contaminant mass-containing tubes first to minimize a positive bias, should a higher contaminant mass tube result in carryover. Sample tubes that are expected to contain higher mass are then analyzed later in the sample queue, if possible. If there is concern that some samples may result in carryover, then blank tubes can be placed between those samples to determine if carryover occurred and to minimize carryover to the next sample. In most cases, a scoping test with a few samples can provide this information before a full test with multiple replicates is performed.

11.3.2 Obtain the list of analytical results for solid-sorbent tubes in units of nanograms per tube (ng/tube).

12. Complete the required reporting for this procedure.

REAGENTS

- Contaminant Information: Provide the contaminant name, source, purity, and lot for each contaminant used.
- Decontaminants: Provide the decontaminant name/description, source, date of preparation, and purchase or expiration date (as applicable) for each decontaminant used. Include a description of the preparation process for materials requiring pre-use preparation, such as dilution or mixing.
- Analytical Solvents: Provide the source, grade, purity, and lot for each extraction solvent used.

EQUIPMENT

- Contaminant Delivery Tool: Provide the following information on the equipment used.
    - Pipettes, Syringes, and Commercial Applicators: Provide the tool identification including the manufacturer, model number, and volume-dispensing range; tool performance specifications including accuracy and any other conformance specifications (e.g., ISO 8655 or ASTM E1154); and confirmation of current calibration.
    - Other Application Systems: Provide the following information for systems without a documented performance-specification standard:

tool description, source, and a description of how the tool's reproducibility from test-to-test is ensured. If the laboratory-determined accuracy and precision is available, then this information is recommended in the final report.

- Decontaminant Delivery Tool: Provide the following information on the delivery tool used.
    - Pipettes and Syringes: See contaminant-delivery tool reporting requirements for pipettes and syringes.
    - Breadboard, Brassboard, and Prototype Equipment: Provide a description of the decontamination system including configuration and identification number/name. If the system uses vendor-provided equipment, then also provide the vendor name, item description, and model number.
- Analytical Standard Preparation Tools: Provide the following information on the preparation tools used.
    - Pipettes and Syringes: See contaminant-delivery tool reporting requirements for pipettes and syringes.
    - Volumetric Glassware: Provide glassware description including the manufacturer, part number, volume, class, and conformance specifications (e.g., ASTM standards E288 and E69).
- Environmental Chamber: Provide a description of the chamber, including the manufacturer and model number for commercial items or a description for fabricated systems. If a data logger is used, include the data logging frequency.
- Vapor Chamber: Provide a description of the chamber, including the manufacturer and model number for commercial items or a description for fabricated systems.
- Contaminated Area Measurement (if performed): Include the tool identification, including the manufacturer and model number, camera resolution, and description of area measurement calculation.
- Analytical Chromatography: Provide a description of the entire unit configuration, including the major components and a description of the component (e.g., detector, injection system, etc.), manufacturer, and model number.

MATERIALS

- Sorbent Tubes: Include the source, description, part number, and sorbent.
- Items: Provide an item description, including description, manufacturer, and model number.
- Precondition Items: Description of how the conditioning was performed, including the following:
    - Location.
    - Precondition length of time with units of hours and minutes.
    - Temperature average with high, low, and standard deviation, for the conditioning period.

- Relative humidity average with high, low, and standard deviation for the conditioning period.
- Identification and discussion of any temperature or humidity excursions to include excursion value, duration, and suspected cause.

- Contamination: Include the following information regarding the agent.
  - Description of how the contamination was performed.
  - Target contamination density in grams per square meters (g/m^2).
  - Total agent volume in microliters (μL) applied per item.
  - Agent drop volume size(s) in microliters (μL) per item.
  - Description of the contamination drop deposition pattern. A drawing or photograph is recommended.
  - Test location temperature and relative humidity during contamination.
  - Contaminant temperature at the time of contamination.
  - Description of the contaminant handling. For example, if the contaminant was applied "cold" or "warm," provide a description of how the contaminant was chilled or heated. For example, if the contaminant was warmed to room temperature before application, note this or any other handling information in the report.
- Dose-Confirmation Sample Preparation: Include the contamination density in grams per square meter (g/m^2), the total agent volume in microliters (μL) per vial, the agent drop volume size(s) in microliters (μL) per vial, the solvent identification, and the solvent volume.
- Contaminant-Material Interaction Observations: Include the following information regarding the observations.
  - Written description of applied drops as they appeared for each sample (e.g., sessile, spread).
  - If available, a photograph or hand drawing capturing drop observation.
- Aging: Provide a description of how the aging was performed, including the following.
  - Aging length of time with the appropriate units.
  - Temperature average with high, low, and standard deviation for the aging period.
  - Relative humidity average with high, low, and standard deviation for the aging period.
  - Identification and discussion of any temperature or humidity excursions to include excursion value, duration, and suspected cause.
  - Description of the item cover (if used), including the size and volume.
- Pre- and Post-Rinse and Drying: Include the following information regarding the rinsing and drying.
  - Description of the rinse solution, including water quality, temperature, and, if used, soap manufacturer and part number.

- Description of the rinse method, including temperature, tool use for delivery, total volume applied, and force and rate of rinse application (if available).
- Description of the drying process, including the location, time, item placement, air velocity (hood), flow rate (dry chamber), temperature, and relative humidity. Provide a description of the visual inspection of the surface at the end of aging, including any residual water on the surface.

- Decontamination: Include the following information regarding the decontamination process.
  - Decontaminant information, including vendors and part numbers for commercial items, and the configuration for developmental items.
  - Document the decontamination method, including the following:
    - Decontaminant residence time.
    - Temperature average with high, low, and standard deviation for the decontamination period.
    - Relative humidity average with high, low, and standard deviation for the decontamination period.
    - Decontaminant temperature at the time of application (e.g., room temperature, chilled, warmed, etc.). If the decontaminant was applied "cold" or "warm" provide a description of how the decontaminant was chilled or heated.
    - Decontaminant amount delivered to item.
    - Vaporous decontaminants: injection rate, flow rate, fumigant concentration, temperature, and relative humidity.
    - Liquid decontaminants: volume delivered.
    - Solid decontaminants: mass delivered.
    - Other decontaminants: amount delivered.
- Vapor Test: Include all of the required calculation tables and graphs, vapor test chamber temperature, and relative humidity observed during testing. Also include the volume of vapor chamber (m^3), chamber airflow rate (mL/min), sampling airflow rate (mL/min), sampling time per tube (min), midpoint time for each sample (min), mass of analyte on tube for each sample (ng), and confirmation statement that the pull times used do not exceed the time determined in the breakthrough test.
- Chromatographic Analysis: Describe the chromatographic analysis including the queue design, the analytical method, CCV sample use and acceptance, calibrated range, method LOD and LOQ, calibration curve-fitting method, correlation coefficient, and goodness-of-fit results.
- Dose-Confirmation Results: The dose-confirmation results should be maintained on file. Report the amount delivered per test.
- Reporting Statement (Recommended): Small-item vapor test results are highly dependent on the test item treatment process. To ensure full context for the

report, providing the test results along with a description of the treatment, including contamination and decontamination is recommended.

PROCEDURE 7: CHAMBER CLEANING

The chamber must be cleaned between uses to minimize the potential of test-to-test cross-contamination that could result in a positive bias in the test results. There are no strict rules for cleaning the chamber. This section provides general cleaning guidance using wipe-down and bake-out methods, in addition to determining chamber cleanliness.

- Wipe-down: A solvent-wetted wipe is used to physically scrub the chamber interior. A solvent, selected for the contaminant of interest, is recommended to ensure contaminant removal. All surfaces should be wiped. After wiping is complete, purging the system overnight with air is preferred to dry the chamber.
- Bakeout: Bakeout is performed by elevating the temperature of the chamber. Air flows through the chamber during this process to remove vaporized contaminant from the chamber. Good mixing is not needed for bakeout, so high air-change rates can be used to expedite cleaning. Typical bakeout conditions use temperatures in the range of 50–70 °C, air-change rates of 5–10 h^{-1}, and time durations in the range of 12–24 h. The bakeout temperature should be high enough to volatilize the contaminant, but should not exceed the maximum operating temperature for chamber components such as fans. Bakeout is recommended after the evaluation of persistent agents, high vapor-emitting samples such as positive control tests, and vapor test chambers with exposed sorptive materials.
- Determination of cleanliness: Cleanliness is determined through the collection and analysis of Tube 0. Tube 0 should be sampled using a minimum of 60 min sampling time. The Tube 0 sampling time should not exceed the longest sampling time used or SSV. The Tube 0 sampling should use the test operation air-change rate and operating temperature. The system is considered clean when the Tube 0 result is significantly below the target mass on tube sampled or below the analytical quantitation limit. If Tube 0 results indicate chamber concentration above the laboratory acceptance criteria, the chamber should be cleaned again and a new Tube 0 collected and analyzed. Each lab should document the method and acceptance criteria for determining chamber cleanliness in the test report.

If the analyte concentration continues to persist, more drastic measures may be needed. Multiple bakeouts or longer bakeout durations may be required. Chamber interior surfaces may need to be wiped down with an active decontaminant to remove the agent (followed by a solvent wipe down to minimize the decontaminant remaining in the chamber during the next test). If these actions do not mitigate the background contaminant concentrations, potentially sorptive surfaces, such as the door gasket, mixing fans, or any other plastics or elastomers in the system, may need to be replaced. Care should be taken to ensure the sampling system is not the source of any background concentrations.

PROCEDURE 8: TUBE CLEANING AND CLEARANCE GUIDANCE

The use of solid-sorbent tubes requires a few additional considerations to ensure optimum performance. The solid-sorbent tubes should be used and analyzed in accordance with the manufacturer's instructions. Guidance for tube conditioning and confirmation are described in this section. The laboratory cleaning, chromatographic confirmation procedure, and acceptance criteria should be documented in the test report.

- New solid-sorbent tube receipt: Most manufacturers recommend an initial conditioning process to remove any contaminants from the sorbent material as a result of the manufacturing or shipment process before the first use.
- Solid-sorbent tube spiking: Tube spiking is the process of making analytical calibration samples for vapor analysis. Tube spiking should be performed on conditioned and verified-clean tubes. Analytical calibration samples are prepared by introducing a known volume of a known concentration contaminant standard solution, using a microliter (μL) syringe to the spike the sorbent material contained within the tube. The solid-sorbent tube must have airflow to pull the contaminant standard solution onto the sorbent bed and to aspirate away the delivery solvent. Solid-sorbent tube spiking and sample collections are always performed on the same end of the tube. Sample analysis must desorb the sample from the same end that the sample was collected or spiked.
- Solid-sorbent tube conditioning: According to the manufacturer, solid-sorbent tubes are typically reusable for about 100 heating cycles. Because they are reused, one challenge is to ensure that reused tubes are clean before being used in another test. Sorbent material that has retained analyte from the previous sample analysis desorption cycle will induce a positive bias in the results of the next sample. The retention of analyte on a tube after analysis is called *carryover*. The solid-sorbent tubes must be conditioned to prevent carryover in subsequent tests. Tube conditioning can be conducted using commercial hardware. The commercial conditioners typically heat the tubes to a specified temperature, usually above the desorption temperature for most analytes, and below the breakdown temperature of the sorbent. Nitrogen or air is purged through the tube to remove residual analyte.
- Chromatographic confirmation of conditioning: In order to ensure that the solid-sorbent tubes are clean, the tubes must be checked analytically. Checking each sorbent tube individually would be labor intensive. The American National Standard Z 1.4 "Sampling Procedures and Tables for Inspection by Attributes" supplies sampling plans that can provide a high level of confidence that batches of solid-sorbent tubes are clean and ready for use, without requiring the analysis of each individual tube. The sampling plans in the standard were designed so that the more "defects or items-failing-the-acceptance-criteria" contained in a batch or lot, the more likely that it will be rejected. In this case, if a batch of solid-sorbent tubes fails to meet the laboratory acceptance criteria, the batch of solid-sorbent tubes would undergo a second cleaning.

PROCEDURE 9: DOSE-CONFIRMATION SAMPLE CALCULATION

This section describes the method used to calculate the sample dose-confirmation value.

1. Calculate the contaminant mass delivered.

1.1 Obtain the raw chromatography results in units of nanograms per milliliter (ng/mL) for the dose-confirmation samples ($\mathbf{DC_E}$).

1.2 Correct the raw result for any dilutions performed between sample collection and analysis. Report the corrected value ($\mathbf{DC_{E\text{-}C}}$) in units of nanograms per milliliter (ng/mL).

1.3 Calculate the contaminant mass delivered (***Del***) in nanograms (ng) for each corrected dose-confirmation sample result ($\mathbf{DC_{E\text{-}C}}$). This is accomplished by multiplying the corrected dose-confirmation sample result ($\mathbf{DC_{E\text{-}C}}$) and the solvent volume (**SV**) in units of milliliters (mL). For the method as written, the solvent volume is 20 mL.

$$Del = DC_E \times SV \qquad \textbf{Equation 12}$$

1.4 The ***Del*** value may be better represented in units of grams (g) for item testing. If different mass units are preferred, then perform the appropriate unit conversion calculation.

1.5 Calculate the average and standard deviation for the set of ***Del*** values.

2. Complete the required reporting for this section.

2.1 Report each ***Del*** value, and the calculated average and standard deviation, including units.

PROCEDURE 10: VAPOR TEST CHAMBER DATA CALCULATION

Procedure 10 contains the data calculation to determine vapor test chamber concentration. This procedure uses real data from DVD players that were contaminated with a HD dilute solution, aged 60 min, and then vapor sampled. This example vapor test data set was for three replicate trials using the same vapor test chamber. Each trial was 12 h in duration with the collection of nine solid-sorbent tubes.

The vapor test chamber concentration (mg/m^3) is calculated from the contaminant mass on the solid-sorbent tube (determined by a validated analytical technique such as TD-GC-MSD), the sampling airflow rate through the tube (note, use the sampling airflow rate, *F*, not the chamber airflow rate, *Q*), and the sampling time. All three values must be accurately measured to ensure accurate calculation of the chamber vapor concentration. The sampling flow rate should be logged. Ideally, the flow rate used here should be the average flow rate observed during the collection of the sample. This concentration corresponds to the concentration of vapor in the chamber at the midpoint sampling time (t_m). This chamber concentration should not be compared to a requirement, and does not correspond to the vapor concentration to which unexposed personnel may be exposed.

1. **Obtain the midpoint and total pull times for each solid-sorbent tube.**

The midpoint and total pull time values for each solid-sorbent tube should be reported. The example data set results are provided in Table 4.

Table 4. Example data set midpoint and total pull time values.

Tube #	Trial 1 Midpoint Time (min)	Trial 1 Total Pull Time (min)	Trial 2 Midpoint Time (min)	Trial 2 Total Pull Time (min)	Trial 3 Midpoint Time (min)	Trial 3 Total Pull Time (min)
1	10.1	4.0	10.1	4.0	10.1	4.0
2	30.1	6.0	30.1	6.0	30.1	6.0
3	60.1	8.0	60.1	8.0	60.1	8.0
4	90.1	10.0	90.1	10.0	90.1	10.0
5	150.1	10.0	150.1	10.0	150.1	10.0
6	250.1	15.0	250.1	15.0	250.1	15.0
7	400.1	25.0	400.1	25.0	400.1	25.0
8	720.1	35.0	720.1	35.0	720.1	35.0
9	1380.1	50.0	1380.1	50.0	1380.1	50.0

2. **Obtain the sampling flow values for each solid-sorbent tube.**

The sampling flow values for each solid-sorbent tube should be reported. The example data set results are provided in Table 5.

Table 5. Example data sampling flow values.

Tube #	Flow Rate for Trial 1 (mL/min)	Flow Rate for Trial 2 (mL/min)	Flow Rate for Trial 3 (mL/min)
1	500.0	500.0	500.1
2	500.0	500.0	500.1
3	500.8	500.0	500.0
4	500.2	500.0	500.0
5	500.2	500.0	500.0
6	500.1	500.0	500.0
7	500.1	500.0	500.0
8	500.0	500.0	500.0
9	500.0	500.0	500.0

3. **Obtain the solid-sorbent tube results.**

The solid-sorbent tube results should be reported. The example data set results are provided in Table 6.

Table 6. Example data set solid-sorbent tube results.

Tube / ID	Analyte Mass for Trial 1 (ng)	Analyte Mass for Trial 2 (ng)	Analyte Mass for Trial 3 (ng)
1	1129.7	1256.1	1026.9
2	810.5	918.9	879.3
3	560.1	706.5	653.3
4	537.1	714.1	613.2
5	383.2	490.6	436.2
6	417.0	495.9	462.0
7	465.3	605.8	546.6
8	425.6	517.7	474.8
9	354.8	398.1	374.6

4. Calculate the vapor test chamber concentration.

The vapor test chamber concentration is determined using Equation 13.

$$C(t_m) = \frac{m/100{,}000}{V_S} = \frac{m}{t_{pull}F/1{,}000{,}000} \qquad \textbf{Equation 13}$$

where

$C(t_m)$ = vapor concentration at mid time t_m (mg/m^3)

m = analyte mass on tube (ng)

V_S = sampled air volume (m^3)

t_t = tube pull time (min)

F = sampling airflow (mL/min)

Calculate the chamber vapor concentration, C (mg/m^3), for each tube. The example data set-calculated concentrations for the vapor test chamber are provided in Table 7.

Table 7. Example data set vapor test chamber concentrations.

Tube / ID	Trial 1 Chamber Concentration (mg/m^3)	Trial 2 Chamber Concentration (mg/m^3)	Trial 3 Chamber Concentration (mg/m^3)
1	0.565	0.628	0.513
2	0.270	0.306	0.293
3	0.140	0.177	0.163
4	0.107	0.143	0.123
5	0.0766	0.0981	0.0872
6	0.0556	0.0661	0.0616
7	0.0372	0.0485	0.0437
8	0.0243	0.0296	0.0271
9	0.0142	0.0159	0.0150

5. Graph the vapor test chamber concentration

The vapor test chamber concentration is graphed. The example data set results are provided on a linear scale in Figure 10 and on a log scale in Figure 11.

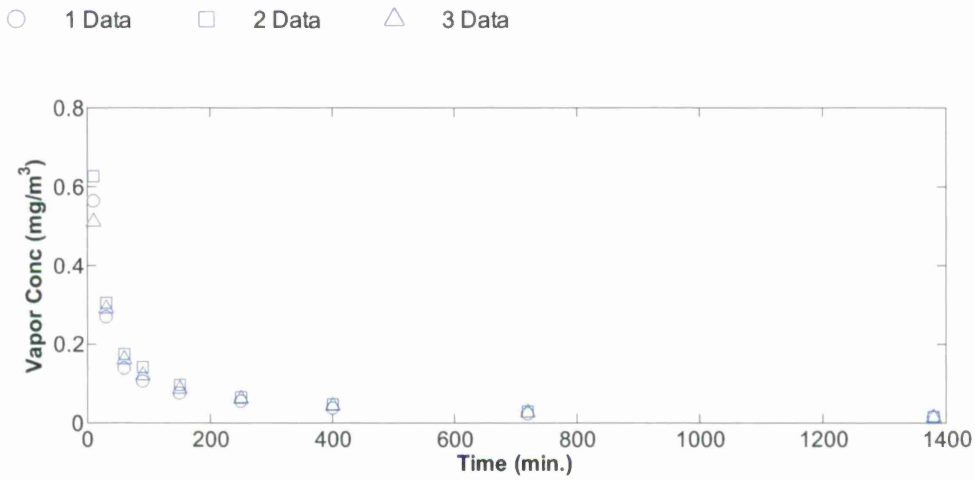

Figure 10. Example data vapor test chamber concentrations, linear scale.

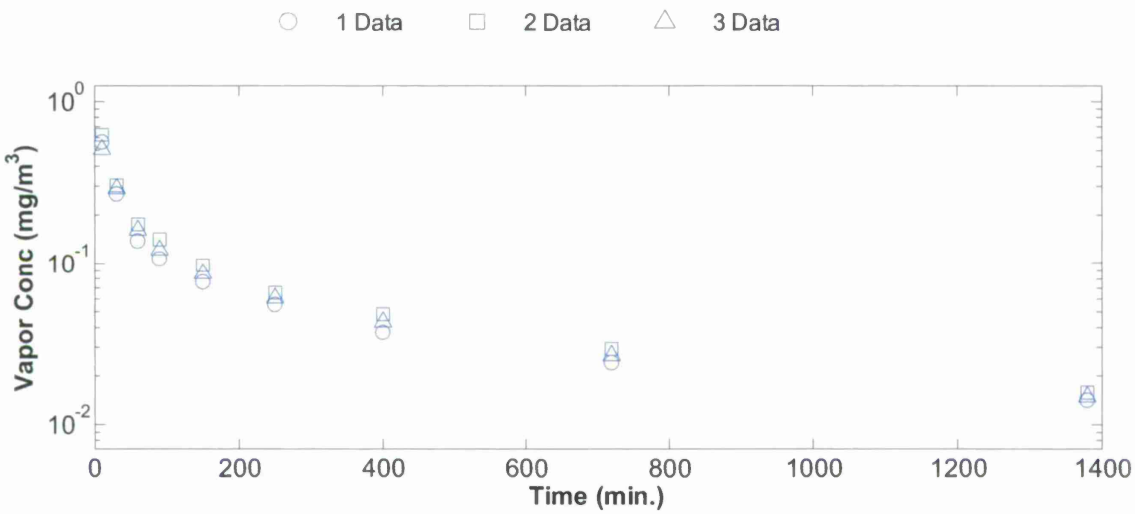

Figure 11. Example data vapor test chamber concentrations, log scale.

PROCEDURE 11: EMISSION VALUE CALCULATION

Procedure 11 contains the vapor test chamber data calculation to determine the emission value. This procedure uses real data from DVD players that were contaminated with a HD dilute solution, aged 60 min, and then vapor sampled. This example vapor test data set was for three replicate trials using the same vapor test chamber. Each trial was 12 h in duration with the collection of nine solid-sorbent tubes.

The emission rate (mg item^{-1} min^{-1}) is used to calculate the mass of agent emitted by a full item (e.g., radio, computer, vehicle, etc.), and is used to evaluate the emission of a full item, which is potentially composed of multiple materials and complex interfaces. The emission rate is scalable by number of items term Z.

$$\frac{dC}{dt} = E(t)\frac{Z}{V} - C(t)\frac{Q}{V}$$ **Equation 14**

$$\frac{dC}{dt} = E(t)l - C(t)n$$ **Equation 15**

where

C(t) = time-dependent chamber vapor concentration (mg/m^3)
V = chamber volume – test item volume (m^3)
Q = airflow rate (m^3 min^{-1})
Z = number of items in free-air volume,
E(t) = time-dependent emission rate of test article (mg item^{-1} min^{-1})
l = loading factor (item / m^3)
n = air-change rate = Q/V (min^{-1})

1. Calculate the air-change rate.

The air-change rate calculation uses the chamber airflow rate and the chamber free-air volume. The average chamber flow rate should be used, if the chamber flow rate is computer logged during testing. The air-change rate, *n,* is calculated using Equation 16. The example data set results are provided in Table 8.

$$n = \frac{\sum_i Q_i / I}{V}$$ **Equation 16**

where

n = air-change rate (min^{-1})
Q_i = chamber airflow rate for log point I (mL min^{-1})
I = total number of log points acquired for test duration
V = chamber free-air volume (m^3)

Table 8. Example data set air-change rate calculation results.

Parameter / ID	Trial 1	Trial 2	Trial 3
Chamber Volume, V (m^3)	0.01848	0.01878	0.01878
Air Change, n (min^{-1})	0.07231	0.07236	0.07306
Air Change, n (h^{-1})	4.339	4.342	4.384
Ave Chamber Airflow, Q (mL/min)	1336	1359	1372
Stdev Q (mL/min)	8.731	9.266	9.353
Min Q (mL/min)	1309	1279	1334
Max Q (mL/min)	1375	1392	1408

2. Calculate the loading factor.

The loading factor is calculated using Equation 17. The example data results are provided in Table 9.

$$l = \frac{Z}{V} \qquad \textbf{Equation 17}$$

where

l = the loading factor (m^{-3})

Z = number of items in the chamber

V = chamber free-air volume (m^3)

Table 9. Example data set loading factor results.

Parameter / ID	Trial 1 (item/m^3)	Trial 3 (item/m^3)	Trial 3 (item/m^3)
Loading	54.11	53.25	53.25

3. Numerically calculate the emission rate or emission factor.

The direct calculation of the emission rate as a function of time is given by Equation 18. ΔC/Δt is calculated using Equation 19.

$$E(t_m) = \frac{\frac{\Delta C_m}{\Delta t_m} + nC_m}{l} \qquad \textbf{Equation 18}$$

$$\frac{\Delta C_m}{\Delta t_m} = \frac{\frac{C_m - C_{m-1}}{t_m - t_{m-1}} + \frac{C_{m+1} - C_m}{t_{m+1} - t_m}}{2} \qquad \textbf{Equation 19}$$

where

$E(t_m)$ = the emission rate at time t_m (mg m^{-2} min^{-1})

C_m = the vapor concentration at t_m (mg m^{-3})

t_m = midpoint tube pull time for concentration measurement (min)

Note that the calculation of ΔC/Δt for time t_m references time t_{m-1}, thus an emission rate cannot be calculated for the first concentration measurement. The same effect is generated for the last concentration measurement. Because ΔC/Δt for time t_m references time t_{m+1}, the emission rate for the last concentration measurement cannot be calculated. This calculation approach provides x-2 emission rate values if *x* chamber vapor concentrations are collected.

Advanced techniques can be used to directly fit the differential equation, rather than use this numerical calculation. If an advanced technique is used, the methods or software used should be documented in the test report. Advanced techniques are highly recommended as they provide more degrees of freedom in the model. SSE and NMSE values can be improved by orders of magnitude using the direct calculation.

There are instances when the direct calculation ($\Delta C/\Delta t$) will not converge for a model, although advanced techniques will quickly converge and provide superior results. These advanced techniques use numerical techniques that are beyond the scope of this method. Interested users should consult texts such as "Numerical Recipes, the art of scientific computing, third edition" by Press, et al. Cambridge University Press 2007, for guidance on these techniques.

Report the emission rate, $E(t)$, (mg m^{-2} min^{-1}) and the time for which the emission factor was calculated, t_m (min). The calculated emission factor, vapor concentration, and midpoint sample times, such as those shown in Table 10, should be included as a report appendix to enable re-evaluation of the data, if it is needed at a later time.

Table 10. Numerical emission rate (mg $item^{-1}$ min^{-1}) for dilute dose DVD player, chamber Sc3.

Concentration Term	Trial 1 Emission Factor, $E(t_m)$	Trial 2 Emission Factor, $E(t_m)$	Trial 3 Emission Factor, $E(t_m)$
$C_{Tube\ 1\ to\ 3}$	0.0001848	0.0002247	0.0002581
$C_{Tube\ 2\ to\ 4}$	0.0001366	0.0001888	0.0001707
$C_{Tube\ 3\ to\ 5}$	0.0001288	0.0001765	0.0001500
$C_{Tube\ 4\ to\ 6}$	0.00009567	0.0001233	0.0001117
$C_{Tube\ 5\ to\ 7}$	0.00007120	0.00008574	0.00008098
$C_{Tube\ 6\ to\ 8}$	0.00004823	0.00006421	0.00005839
$C_{Tube\ 7\ to\ 9}$	0.00003198	0.00003946	0.00003657

4. Graph the emission value results.

The emission value results should be plotted to enable inspection of the results, especially when replicate samples are compared. The example data set emission rate results are provided on a linear scale in Figure 10 and on a log scale in Figure 11.

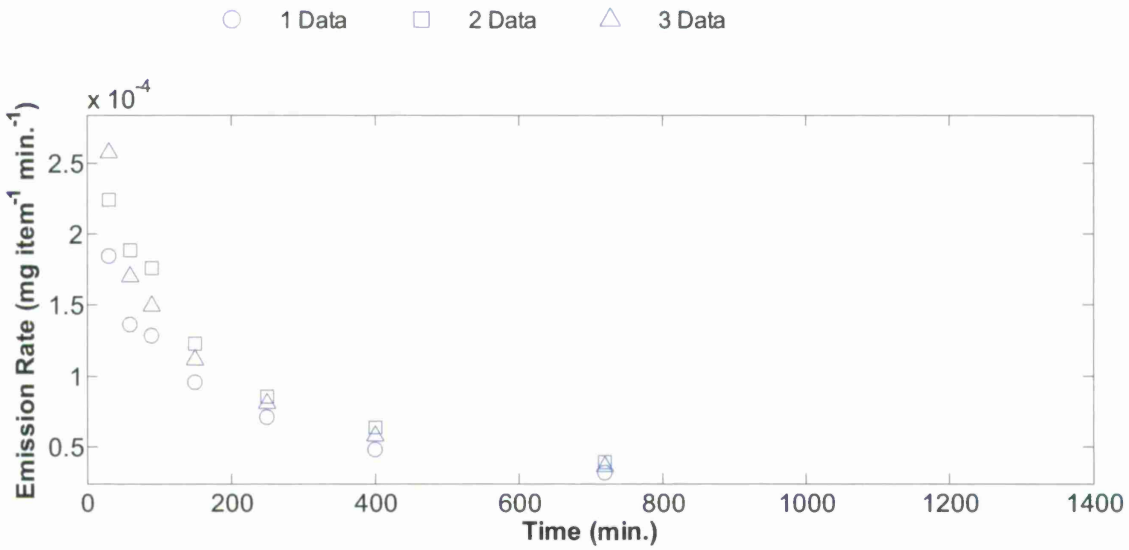

Figure 12. Example data vapor test chamber emission rates, linear scale.

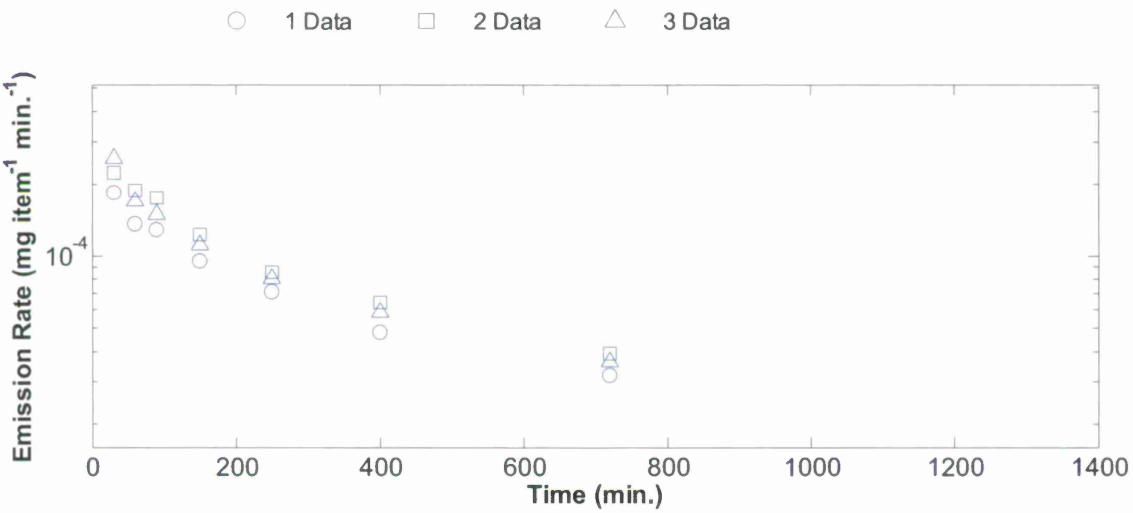

Figure 13. Example data emission rates, log scale.

5. Calculate the emission model.

The emission model is an empirical model (i.e., equation) that represents the emission data as a function of time. The emission model is required to calculate the vapor concentration that would be present in a specified scenario, and enables the comparison of data acquired using different sampling plans. The emission model is evaluated for each item because real items may have some variability, even for the same treatment and sampling process. The emission characterization does account for test variables such as chamber volume and the air-change rate, which can typically enable comparison of items tested at different air-change rates and in different chambers.

Grouping test item results as replicates should be limited because sample treatment (contamination or decontamination) may not always be identical for real items. This may result in different emission rates. The item method does not correct for contaminated surface area or multiple material effects.

The emission rate will change as a function of time due to the various mass transport mechanisms of the contaminant in a given material and how the decontaminate neutralized or removed the contaminant. The physical mechanism that drives the mass transport could be evaporative, diffusion limited, or other mechanism, and is highly dependent on the material structure and the material-agent interactions. There are many empirical models that can be used to fit the emission factor including, but not limited to, the examples shown in Equation 20 through Equation 24.

Constant emission source model

$$E(t) = A \qquad \textbf{Equation 20}$$

Power Law model

$$E(t) = At^{B} \qquad \textbf{Equation 21}$$

First-order exponential decay model

$$E(t) = A\exp(-Bt) \qquad \textbf{Equation 22}$$

Second-order decay model

$$E(t) = \frac{E_0}{1 + ktE_0} \qquad \textbf{Equation 23}$$

Log-normal model

$$E(t) = C\exp\left[\frac{-(\log(t) - B)^2}{A}\right] \qquad \textbf{Equation 24}$$

where

A, B, E_0, k, and t_0 = the fitting coefficients (units vary by model)

t = time (min)

In most cases, it is expected that the first- or second-order decay model will fit most materials, with possible application of the peak model when applied to some elastomers. If the calculated emission value goes to zero, the first instance of this occurrence should be used in the data fitting; subsequent data points below detection should not be used for model fitting. The emission factor can be assigned to a value of zero for time points after the first occurrence of below detection.

The emission value results are fit to the empirical models, using an appropriate statistical tool such as Excel, Matlab, or Sigma Plot to determine the emission model.

6. Evaluate the emission model.

The emission model best-fit determination should use statistical parameters such as, but not limited to, the Sum of the Square of the Error (SSE), Root Mean Square Error (RMSE), and R^2. The best fit will provide smaller SSE and RMSE values and an R^2 near 1.0. If no model presents a good fit for the data, the calculation of a scenario vapor concentration may be inaccurate. Report the emission model used, the coefficients of the model, and the statistical parameters used to identify the model (e.g., SSE, RMSE, R^2).

The example data sets were individually fit. The best model and summary statistics are provided in Table 11. The summary statistics for the four fit models used for trial 1, trial 2, and trial 3 are provided in Table 12, Table 13, and Table 14, respectively.

Table 11. Goodness-of-fit values for example data set.

GOF Param. / ID	Trial 1	Trial 2	Trial 3
Model	Power Law	Power Law	Power Law
Coef. / Eqn.	$E(t) = A * t^B$	$E(t) = A * t^B$	$E(t) = A * t^B$
A	0.0008685	0.0008635	0.001739
B	-0.4497	-0.3887	-0.5584
Corr. Coef.	0.8939	0.8617	0.9765
SSE	0.1583	0.2065	0.04146
RMSE	0.1326	0.1515	0.06787
Sum RPD (%)	55.3276	67.5704	27.9469
Ave. RPD (%)	6.14751	7.50783	3.10521

Table 12. Model-fit statistical results for example data Trial 1

Model	Total SSE	Ave RMSE	Total RPD	Ave RPD	Ave Rank	Selected Model
Power Law	0.158 (1)	0.133 (1)	55.3 (1)	6.15 (1)	1.33	x
Exponential	0.277 (3)	0.176 (3)	74 (2)	8.22 (2)	2.67	
Second Order	0.271 (2)	0.174 (2)	135 (3)	15 (3)	2.50	
Log-normal	8.16E+005(4)	301 (4)	1.12E+005(4)	1.24E+004 (4)	3.67	

Table 13. Model-fit statistical results for example data Trial 2.

Model	Total SSE	Ave RMSE	Total RPD	Ave RPD	Ave Rank	Selected Model
Power Law	0.206 (1)	0.151 (1)	67.6 (1)	7.51 (1)	1.33	x
Exponential	0.329 (3)	0.191 (3)	82.3 (2)	9.15 (2)	2.67	
Second Order	0.275 (2)	0.175 (2)	129 (3)	14.4 (3)	2.50	
Log-normal	8.07E+005(4)	299 (4)	1.11E+005(4)	1.23E+004 (4)	3.67	

Table 14. Model-fit statistical results for example data Trial 3.

Model	Total SSE	Ave RMSE	Total RPD	Ave RPD	Ave Rank	Selected Model
Power Law	0.0415 (1)	0.0679(1)	27.9 (1)	3.11 (1)	1.00	x
Exponential	0.202 (2)	0.15 (2)	65.6 (2)	7.28 (2)	2.33	
Second Order	0.22 (3)	0.156 (3)	124 (3)	13.8 (3)	2.83	
Log-normal	8.12E+005(4)	300 (4)	1.11E+005 (4)	1.23E+004 (4)	3.83	

7. Graph the best emission model.

The emission model best-fit results should be plotted. The emission model should be reported in units of milligrams per item per minute (mg item^{-1} min^{-1}). An emission model was calculated for each trial from the sample data. The Power Law model was the best fit for trial 1 (Figure 14), trial 2 (Figure 15), and trial 3 (Figure 16).

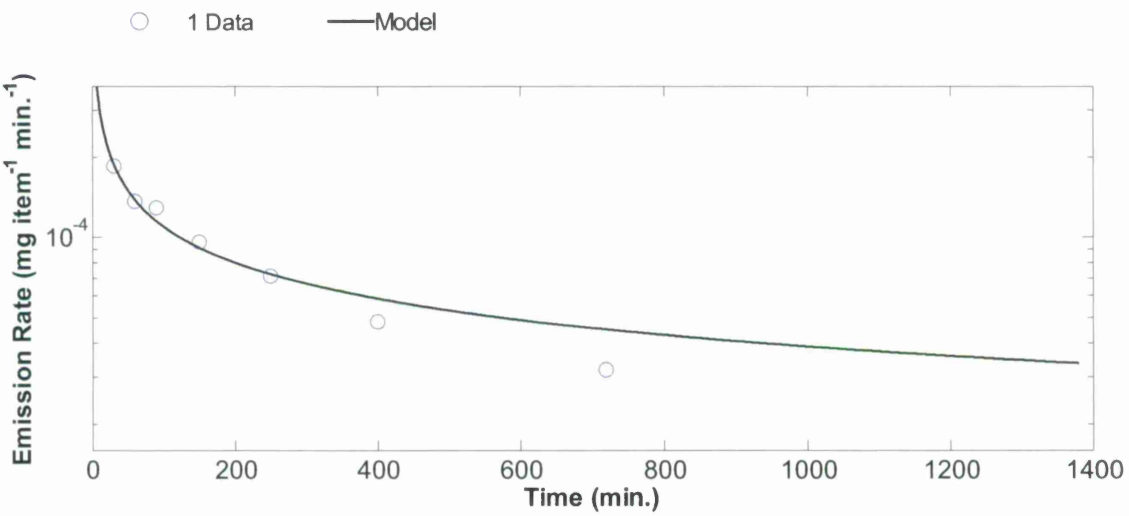

Figure 14. Example data numerically calculated emission rate for trial 1.

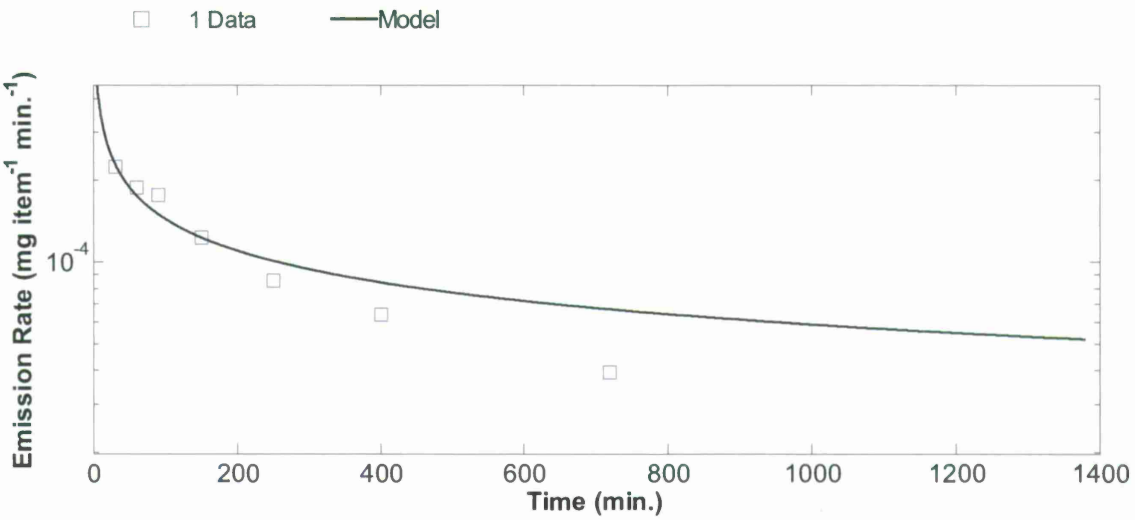

Figure 15. Example data numerically calculated emission rate for trial 2.

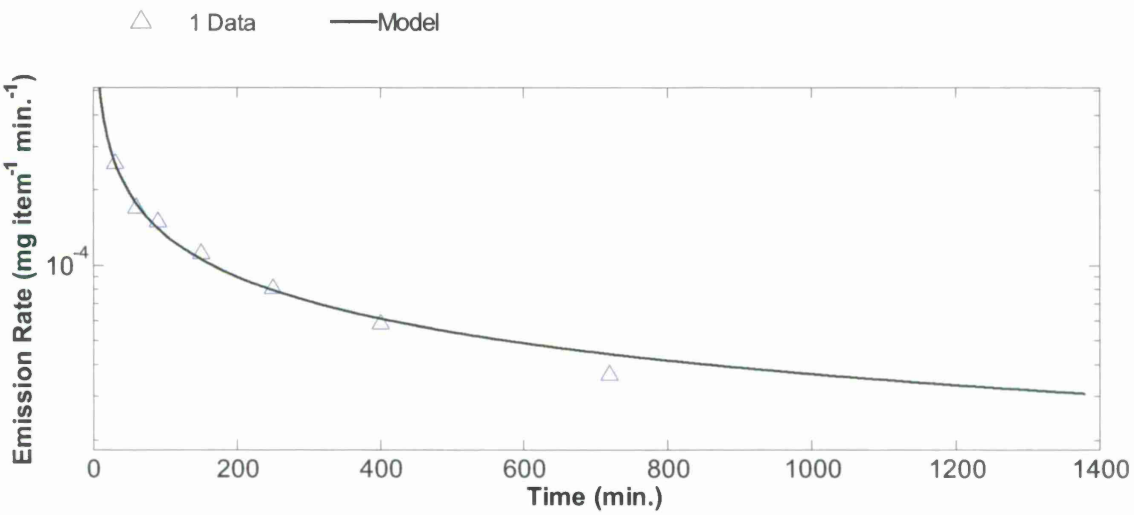

Figure 16. Example data numerically calculated emission rate for trial 3.

PROCEDURE 12: VAPOR TEST CHAMBER TOXIC-LOAD CALCULATION

Procedure 12 contains the data calculation to determine vapor test chamber toxic-load values. This procedure uses real data from DVD players that were contaminated with a HD dilute solution, aged 60 min and then vapor sampled. This example vapor test data set was for three trials using the same treatment approach, vapor test sampling process, and the same vapor test chamber. Each trial was 12 h in duration, with the collection of nine solid-sorbent tubes.

To establish whether the results of a given vapor test would pose a risk for unprotected personnel, the toxic-load value can be calculated. Historically, time-weighted-average vapor concentrations were used to compare to a requirement to determine technology performance. Guidance from toxicology experts and FM 3-11.9 suggest that using a toxic-load (TL) model to calculate an exposure provides a more accurate risk determination. It should be noted that the test chamber is a scenario, and can provide a different toxic-load value than what may occur in a building, aircraft, and other field scenarios.

1. Select the appropriate toxic-load exponent.

To calculate a toxic-load exposure, the toxic-load exponent must be selected. The typical variable used to describe the toxic-load exponent is, *n*, not to be confused with the air-change rate using the same variable name. The toxic-load exponent is a function of the agent and can be found in FM 3-11.9. The current values are presented in Table 16. In recent documents, various organizations have used different toxic-load exponents. Decontamination testing should compare the use of the toxic-load exponents for mild effects, which are highlighted in yellow in Table 16. For example, if the data corresponds to the agent GD, a toxic-load exponent of $n = 1.4$ is selected.

Table 15. Toxic-load exponents used by FM 3-11.9 and by CHPPM.

Route of Exposure	Effect	GD		VX		HD	
		FM 3-11.9	CHPPM	FM 3-11.9	CHPPM	FM 3-11.9	CHPPM
IH/OC	Lethal	1.25	2	1	2	1.5	1
IH/OC	Severe	1.25	2	1	2	1	1
IH/OC	Mild	1.4	2	1	2	1	1
PC	Lethal	1	N/A	1	N/A	1	N/A
PC	Severe	1	N/A	1	N/A	1	N/A
PC	Mild	1	N/A	1	N/A	1	N/A

IH/OC – inhalation/ocular exposure
PC – percutaneous (through the skin)
Yellow indicates the value that should be used for toxic-load calculations in this report.

2. Calculate the toxic-load value.

The toxic-load value (*TL*) is calculated using the vapor test chamber concentration [C(t)]. This calculation will generate a single number that can be compared with a requirement to determine if a scenario would induce a toxicological response. The toxic-load value is calculated using the ten Berge equation (Equation 25).

$$TL = \int C(t)^n \, dt$$ **Equation 25**

Because the vapor concentration was calculated numerically using discrete time steps, the toxic-load value for any time duration from t_{start} to t_{end} is expressed as the summation shown in Equation 26.

$$TL = \sum_{t_{start}}^{t_{end}} C(t)^n \cdot \Delta t$$ **Equation 26**

Using a notation similar to the numerical method to calculate the vapor concentration, and setting the boundary conditions of $t_{start} = 0$ and $TL(t_{start}) = 0$, the toxic-load value can be calculated as shown in Equation 27.

$$TL(t) = TL(t - \Delta t) + C(t)^n \, \Delta t$$ **Equation 27**

where

TL(t)	=	toxic-load value at time t (mg^n min/m^{3n})—note units vary with *n*
TL(t-Δt)	=	toxic-load value at the previous time step
C(t)	=	vapor test chamber concentration at time t (mg/m^3)
n	=	toxic-load exponent (unitless)
t_{start}	=	integration start time (min)
t_{end}	=	integration end time (min)
Δt	=	time step size (min)

The end time should correspond to the test duration (t_{test}). The time step size (Δt) must be the same as that used to calculate the scenario vapor concentration. The quantity $C(t)^n\Delta t$ corresponds to the toxic-load exposure for a time step (Δt). The toxic-load value for the test duration [$TL(t_{test})$], would provide the result for the vapor test chamber. It is not recommended that this value be compared to a requirement value; however, it can be useful at the lab-scale for data comparisons.

3. **Graph the vapor test chamber toxic-load value.**

Plot the calculated toxic-load value as a function of time. The example vapor test chamber data toxic-load graphs are provided in Figure 17, Figure 18, and Figure 19, for example data test trials 1, 2, and 3, respectively.

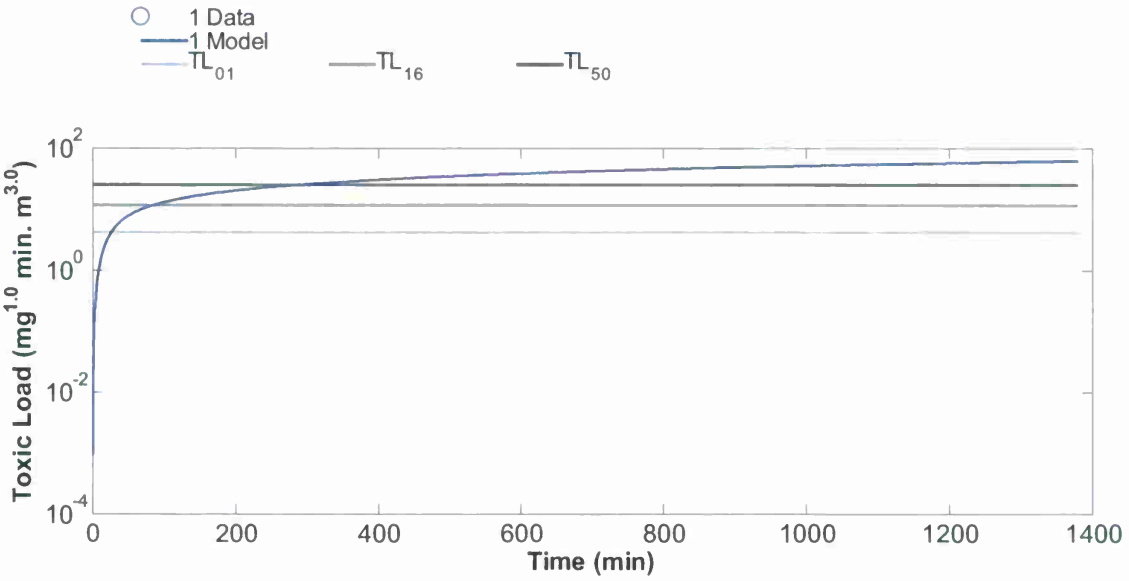

Figure 17. Example toxic-load graph for example data result 1.

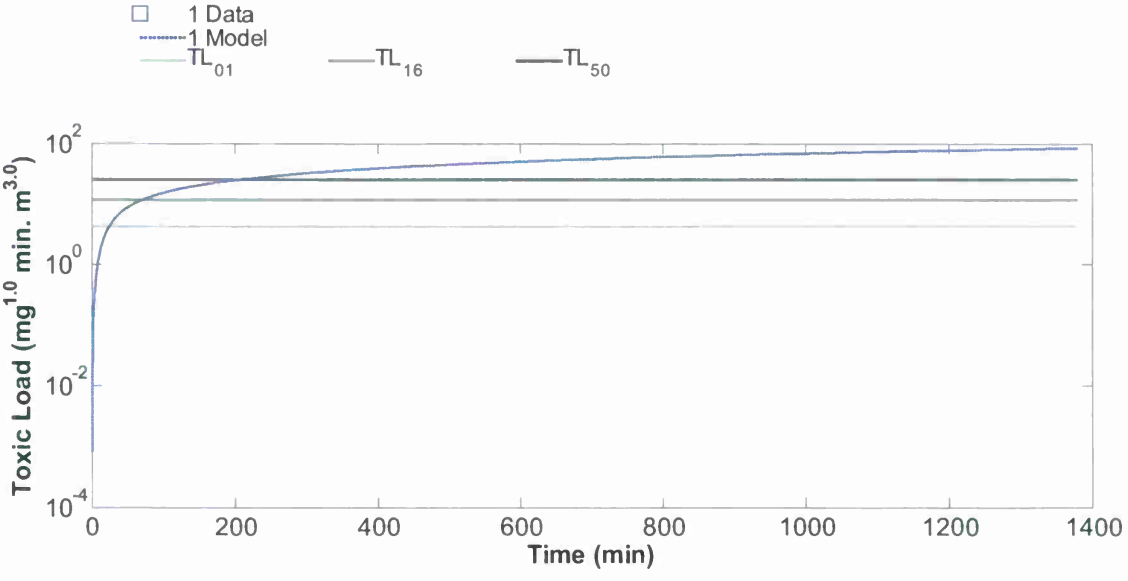

Figure 18. Example toxic-load graph for example data result 2.

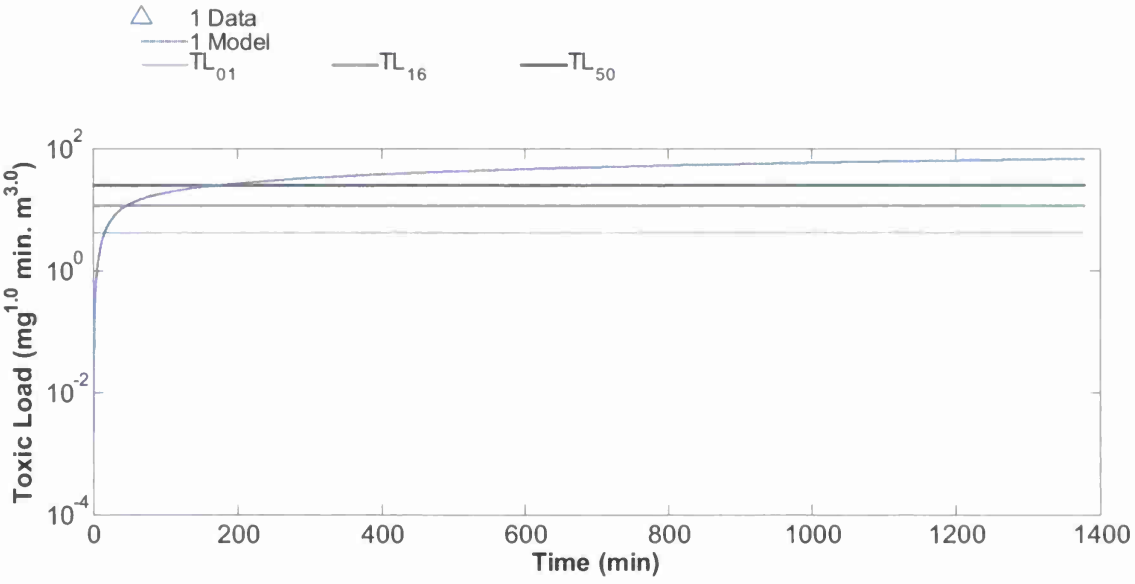

Figure 19. Example toxic-load graph for example data result 3.

PROCEDURE 13: SCENARIO VAPOR CONCENTRATION CALCULATION

Procedure 13 contains the steps used to determine the scenario vapor concentration from the vapor test chamber results. No scenarios were specified during this program to enable example calculations. The figures shown are example figures of how scenario concentration profiles may appear for a set of data in different scenarios.

The scenarios used to evaluate vapor test results should be agreed upon by the test sponsor to address the scenarios of interest to the sponsor, or as specified in the requirement document for a specific acquisition program.

1. Identify the scenario key parameters.

The first step in the process is to identify several key parameters for the scenario of interest. Key parameters include the scenario total volume (V_{S-T}), the airflow rate, and the air-change rate. The scenario concentration calculation uses the free-air volume for the scenario of interest. The free-air volume is the total scenario volume minus the volume of the articles occupying the same space. The 1 m^2 standard panel is the most basic version of this calculation. For this case, the free-air volume is the same as the scenario volume. The scenario concentration calculation for large items (e.g., vehicles in cargo bay) requires the determination of the occupied volume and the calculation of the free-air volume.

The air-exchange rate is needed for the scenario concentration calculation. The air-exchange rate is calculated by

$$n_{scenario} = \frac{Q_{scenario}}{V_{scenario}} \qquad \textbf{Equation 28}$$

If the scenario air-change rate is specified, then the airflow rate does not need to be back calculated for the 1 m^2 panel calculation. However, for larger items or composite systems, the airflow rate must be determined in order to calculate the air-change rate for the scenario free volume. Also note what the air-change rate was (in units of min^{-1}), if this was specified by scenario.

2. Calculate the loading factor.

The scenario-loading factor ($l_{scenario}$), is calculated by Equation 29.

$$l_{scenario} = \frac{Z}{V_{scenario}} \qquad \textbf{Equation 29}$$

3. Calculate the scenario vapor concentration.

Calculating the scenario vapor concentration requires scaling the small-scale dynamic vapor chamber data to a "real world" scenario. The mathematics used to perform this operation are well established. However, care must be taken to accurately account for and recognize the assumptions and limitations of the models used to calculate the vapor concentrations. The vapor hazard is calculated by determining the scenario vapor concentration as a function of time, then calculating the toxic-load value associated with the vapor concentration profile. The toxic-load value can be compared to requirements and toxicology data to determine the scenario risk.

The desired accuracy of the vapor concentration in a scenario determines the amount and type of test data to be acquired, in addition to the level of modeling applied in the scale-up calculation. Essentially, higher accuracy scaling requires considerable computational power and complex models such as Computed Flow Dynamics (CFD). It is not reasonable to perform modeling on the CFD level of detail because of the cost of the software, hardware, and expertise required to perform such modeling, in addition to the number of scenarios and the volume of data that could be generated in this type of evaluation. Because this procedure is designed for evaluating decontamination, rather than for providing a detailed simulation, a simple model is provided to give an approximate representation of the vapor concentration that would be encountered in a real world scenario. This calculation will determine the scenario vapor concentration. The following assumptions are made:

- The emission-factor model data was collected for a time period equal to or longer than the scenario duration. Time extrapolation of the emission-factor model is not recommended.
    - Caveat: If the emission factor diminishes to a zero value, zeros can be extrapolated in time, provided that the residual agent measurements indicate that no residual agent is present.
- The initial vapor concentration in a scenario is assigned to C(t=0) = 0 mg/m^3, which indicates that the initial environment is "clean." The initial concentration could be set to any other value if needed.

- If the mass transport mechanism is evaporative, these calculations do not account for the effect of air velocity or concentration gradients that may affect the emission factor. Test conditions (air velocity) should match the scenario to ensure proper scaling of evaporative emission.
- The following calculations apply to enclosed volumes ("indoor" environments), modeling outdoor environments requires dispersion models (e.g., SCIPUFF, VLSTRACK, CFD).
- The enclosed volume is well mixed.
- The presented model does not account for "sinks" that exist in real world scenarios and would absorb the vapor, decreasing the actual vapor concentration.
- The model does not account for changes in emission factors as a function of temperature—the scenario temperature is the same as the test data temperature generated.

This equation is solved numerically by calculating the concentration for discrete time steps (Δt). It is recommended that the time step interval value be set to 0.1 min (if erratic vapor concentrations are observed smaller Δt values should be used). The initial concentration [C(t=0)] should be set to 0 mg/m^3. The calculation should be carried out for the duration of a scenario.

$$C_S(t) = E(t)l_{scenario}\Delta t - C(t-\Delta t)n_{scenario}\Delta t + C(t-\Delta t) \qquad \textbf{Equation 30}$$

where

$C_s(t)$	=	vapor concentration for the scenario at time t (mg/m^3)
$C(t-\Delta t)$	=	vapor concentration for the scenario at the previous time step value $t-\Delta t$ (mg/m^3)
t	=	current time step (min)
Δt	=	time step increment (min)
E(t)	=	emission-factor model for the material (mg m^{-2} min^{-1})
$n_{scenario}$	=	scenario air-change rate (min^{-1})
$l_{scenario}$	=	scenario loading factor (m^2 / m^3)

4. Graph the scenario vapor concentration.

Plot the calculated $C_s(t)$ as a function of time. This corresponds to the vapor concentration to which unprotected personnel in the scenario would be exposed. An example calculation for an item in multiple scenarios may resemble the example provided in Figure 20. The resulting concentration is dependent on the scenario properties.

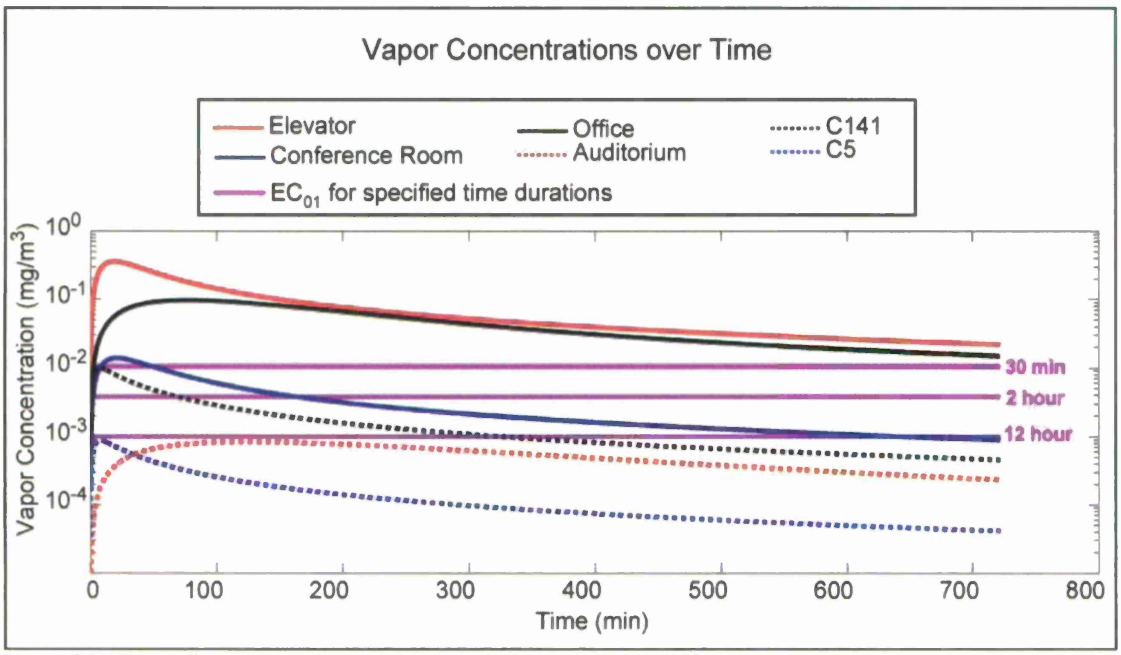

Figure 20. Example vapor concentration graph for an item in different scenarios.

PROCEDURE 14: SCENARIO TOXIC-LOAD CALCULATION

Procedure 14 contains the steps to determine the scenario toxic-load value from the vapor test chamber results. No scenarios were specified during this program to enable example calculations. The figures shown are example figures of what scenario concentration profiles might look like for a set of data in different scenarios.

Toxic-load value is calculated to determine if the scenario vapor concentration poses a risk for unprotected personnel. Historically, time-weighted average vapor concentrations were used to compare to a requirement to determine technology performance. Guidance from toxicology experts and FM 3-11.9 suggest calculating an exposure using a toxic-load (TL) model to provide a more accurate calculation process to determine the scenario risk.

1. **Select the appropriate toxic-load exponent.**

   To calculate a toxic-load exposure, the toxic-load exponent must be selected. The typical variable used to describe the toxic-load exponent is, *n*, not to be confused with the air-change rate using the same variable name. The toxic-load exponent is a function of the agent and can be found in FM 3-11.9. Current values are presented in Table 16. In recent documents, various organizations have used different toxic-load exponents. Decontamination testing should compare the use of toxic-load exponents for mild effects, which are highlighted in yellow in Table 16. For example, if the data corresponds to the agent GD, a toxic-load exponent of $n = 1.4$ is selected.

Table 16. Toxic-load exponents used by FM 3-11.9 and by CHPPM.

Route of Exposure	Effect	GD		VX		HD	
		FM 3-11.9	CHPPM	FM 3-11.9	CHPPM	FM 3-11.9	CHPPM
IH/OC	Lethal	1.25	2	1	2	1.5	1
IH/OC	Severe	1.25	2	1	2	1	1
IH/OC	Mild	1.4	2	1	2	1	1
PC	Lethal	1	N/A	1	N/A	1	N/A
PC	Severe	1	N/A	1	N/A	1	N/A
PC	Mild	1	N/A	1	N/A	1	N/A

IH/OC – inhalation/ocular exposure
PC – percutaneous (through the skin)
Yellow indicates the value that should be used for toxic-load calculations in this report.

2. Calculate the scenario's toxic-load value.

The toxic-load value (*TL*) for a scenario is calculated using the scenario vapor concentration [$C_S(t)$]. This calculation will generate a single number that can be compared to a requirement to determine if a scenario would induce a toxicological response. The toxic-load value is calculated using the ten Berge equation (Equation 31).

$$TL = \int C_S(t)^n \, dt \qquad \textbf{Equation 31}$$

Because the vapor concentration was calculated numerically using discrete time steps, the toxic-load value for any time duration from t_{start} to t_{end} is expressed as the summation shown in Equation 32.

$$TL = \sum_{t_{start}}^{t_{end}} C_S(t)^n \cdot \Delta t \qquad \textbf{Equation 32}$$

Using a notation similar to the numerical method to calculate the vapor concentration, and setting the boundary conditions of $t_{start} = 0$ and $TL(t_{start}) = 0$, the toxic-load value can be calculated as shown in Equation 33.

$$TL(t) = TL(t - \Delta t) + C_S(t)^n \Delta t \qquad \textbf{Equation 33}$$

where

TL(t)	=	toxic-load value at time t (mg^n min/m^{3n}) – note units vary with *n*
TL(t-Δt)	=	toxic-load value at the previous time step
$C_S(t)$	=	scenario vapor concentration at time t (mg/m^3)
n	=	toxic-load exponent (unitless)
t_{start}	=	integration start time (min)
t_{end}	=	integration end time (min)
Δt	=	time step size (min)

The end time should correspond to the scenario duration ($t_{scenario}$). The time step size (Δt) must be the same as that used to calculate the scenario vapor concentration. The quantity $C_S(t)^n\Delta t$ corresponds to the toxic-load exposure for a time step (Δt). The toxic-load value for the scenario duration [$TL(t_{scenario})$], should be compared to a requirement value.

3. Graph the scenario toxic-load value.

Plot the calculated toxic-load value as a function of time. An example calculation for an item in multiple scenarios may resemble the example provided in Figure 21. The resulting concentration is dependent on the scenario properties.

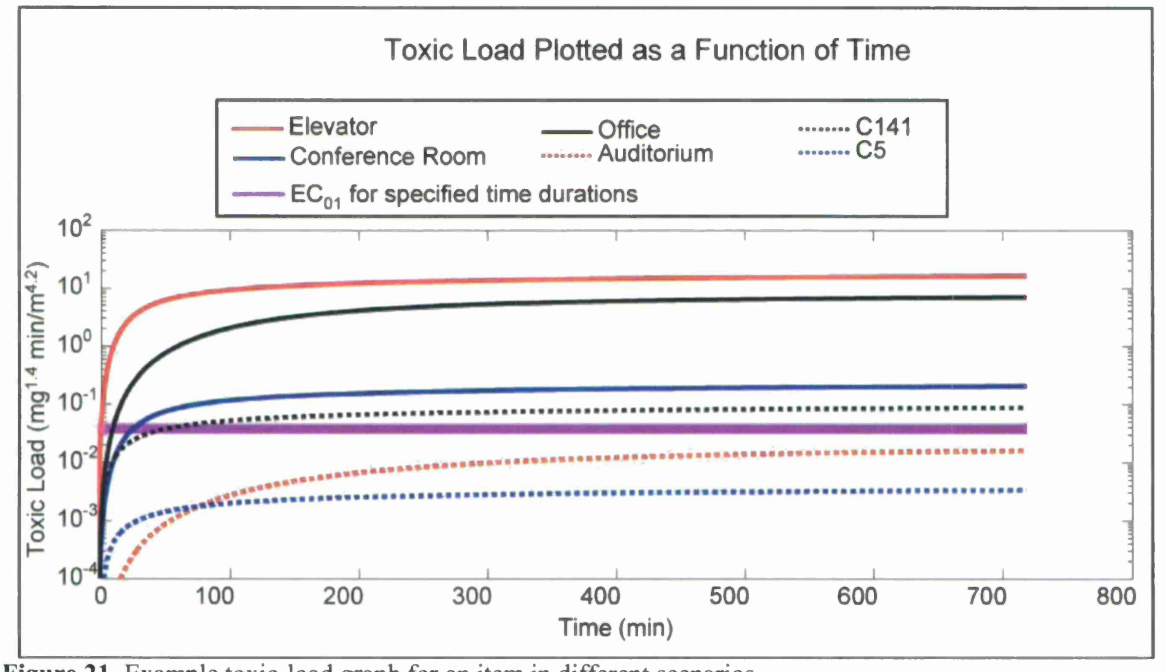

Figure 21. Example toxic-load graph for an item in different scenarios.

4. Perform scenario and toxicological toxic-load comparison.

The toxic-load value can be compared to a requirement to determine if a toxicological response will be observed. FM 3-11.9 lists toxic-load exposures that would induce a response in 50% of the population. Toxic-load values for 16% (ETL_{16}) or 1% (ETL_{01}) of the military population can also be acquired. The toxic-load values, using the field manual exponents for 1, 16, and 50% of the military population, are provided in Table 17. If the calculated TL value is greater than the value listed in Table 17, then a toxicological response will be observed for the corresponding population percentage.

Table 17. Toxic-load values for common test agents using field manual values.

Agent	**HD & L**			**VX**		
Toxic-Load Exponent	n = 1			n = 1		
Effects	Mild (eye irritation)			Mild (miosis, rhinorrhea)		
Units	mg min/m^3			mg min/m^3		
Level	ETL_{50}	ETL_{16}	ETL_{01}	ETL_{50}	ETL_{16}	ETL_{01}
Toxic-load value	25.0	11.6	4.2	0.1	0.056	0.026

Agent	**GD & GF**			**GA & GB**		
Toxic-Load Exponent	n = 1.4			n = 1.5		
Effects	Mild (miosis, rhinorrhea)			Mild (miosis, rhinorrhea)		
Units	$mg^{1.4}$ $min/m^{4.2}$			$mg^{1.5}$ $min/m^{4.5}$		
Level	ETL_{50}	ETL_{16}	ETL_{01}	ETL_{50}	ETL_{16}	ETL_{01}
Toxic-Load Value	0.0796	0.0572	0.0371	0.179	0.128	0.0831

PROCEDURE 15: SCIENTIFIC DISCUSSION AND REPORTING GUIDANCE

Prior comparisons of vapor data to requirements were often binary approaches to evaluate whether a decontaminant met a requirement value. In many cases this binary comparison was made using different data treatment approaches. In addition, a vapor hazard is scenario-dependent. The lack of sufficient information from requirement documents limited the ability to correctly calculate for the scenario. The binary data evaluation approach did not provide proper guidance for decontaminant development. The data analysis method did not provide the context (e.g., scenario) or proper utilization (e.g., toxicological effects).

The approach of this method update has been to build a foundation to compare test results to toxicological values, and account for how the scenario affects the hazard. This section uses scenario vapor concentration profiles to determine the scenarios for when a hazard is present, when a hazard is not present, and when trade space considerations may exist. These curves can also identify when detectors may alarm. The toxic-load values and curves determine if the material or item in the scenario poses a vapor hazard to unprotected personnel by comparison to a toxicological value. The examples in this section compare results to the toxic-load value for which 1% of the population would experience a toxicological response. All of the scenarios shown in this section are based on a 12 h mission length.

This type of data analysis is best suited for later R&D and field testing to determine potential applicability to an acquisition program. Early decontaminant evaluations should focus on

removing as much contaminant as possible. Later R&D activities should assess the hazard that the remaining agent creates. This type of testing and data analysis does not replace the need for later DT/OT on actual items, using the final applicator system and decontaminant process. This analysis is meant to enhance the overall research, development, and testing process.

1. **Document the vapor concentration profiles.**

   The vapor concentration profiles provide guidance regarding the decontaminant treatment performance for the reduction of agent contamination on and in the material of interest. The 12 h EC_{01} for GD is 0.00087 mg/m^3. This value is shown in Figure 22 using a thick, pink dividing line labeled 12 h. The scenarios fall into three groups for concentration profiles that are (a) above, (b) cross, or (c) below the 12 h dividing line. The results are discussed further in the following list.

   - Group (a) above: The elevator and office scenarios have vapor concentrations *above the dividing line* for the entire 12 h time period. These scenarios are expected to yield high toxic-load values and result in a hazard to unprotected personnel. The decontaminant treatment performance was not sufficient for the material placed in these scenarios.
   - Group (b) cross: The conference room and C141 cargo bay scenarios have vapor concentrations that *cross the dividing line* over the course of the 12 h period. It is not possible to determine the hazard to unprotected personnel solely from the concentration chart. The toxic-load values must be considered. The conference room and C141 cargo bay scenarios identify a potential trade space to determine the acceptable risk level for unprotected personnel.
   - Group (c) below: The auditorium and C5 cargo bay have vapor concentrations *below the dividing line* for the entire 12 h period. These scenarios are expected to yield lower toxic-load values and result in a scenario that poses less risk for unprotected personnel. The decontaminant treatment performance was sufficient for the material placed in these scenarios.

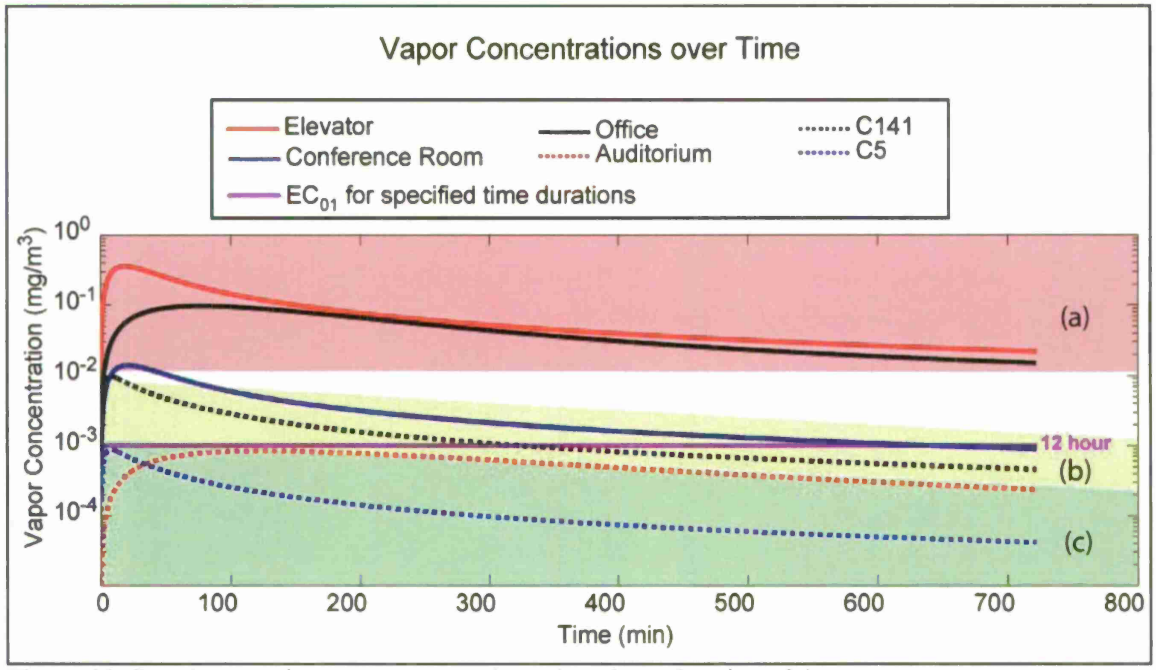

Figure 22. Sample scenario vapor concentrations plotted as a function of time.

2. Evaluate the toxic-load value.

The toxic-load value should be used to determine the hazard to unprotected personnel. The ETL_{01} for GD is 0.0371 $mg^{1.4}$ $min/m^{4.2}$ and is denoted in Figure 23 using pink colored text. The calculated toxic-load values for the decontaminated material in the scenarios are shown in Figure 23. The auditorium and C5 cargo bay scenarios toxic-load values are well below the ETL_{01} for GD. The conclusion for these scenarios, based on the experimental test data, is that the decontaminant treatment was effective for this 1 m^2 decontaminated panel and that a vapor hazard was not present. The same decontaminated material, placed in the elevator and office, however, poses a significant vapor hazard to unprotected personnel. The calculated toxic-load values are significantly greater than the ETL_{01} for GD. The calculated toxic-load values for the C141 cargo bay and conference room scenario are greater than the ETL_{01} for GD. The decontaminant treatment was not effective for these scenarios. A vapor hazard to unprotected personnel is present, but not as severe as the elevator and office scenarios.

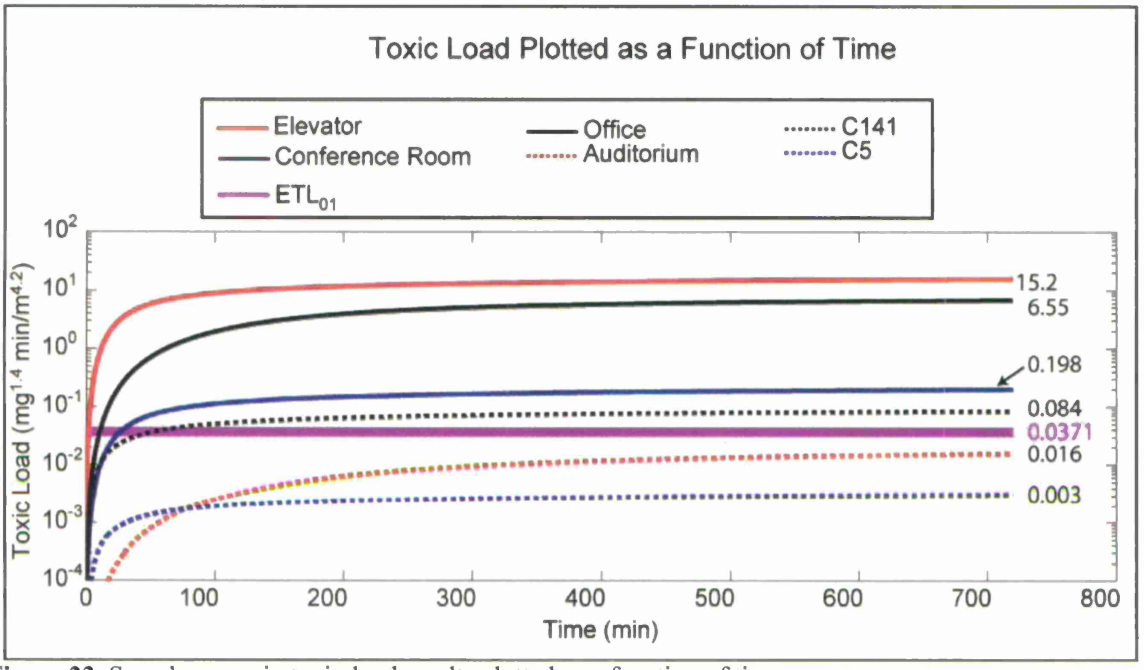

Figure 23. Sample scenario toxic-load results plotted as a function of time.

3. Evaluate the trade space scenario.

The scenario-based evaluation offers greater context for the evaluation of decontaminant performance. The decontaminant treatment can be analyzed to determine the trade space for the decontaminant tested. This ability for trade space is evident for the C141 cargo bay and conference room example scenarios. The C141 cargo bay demonstrated a toxic-load value of 0.056 $mg^{1.4}$ $min/m^{4.2}$, which is above 0.0371 $mg^{1.4}$ $min/m^{4.2}$, the ETL_{01} for GD. However, this value is slightly below the ETL_{16} for GD listed in Table 18. If it is acceptable for an operation to have up to 16% of the unprotected personnel affected in order to complete the mission, then this decontaminant performance may be reasonable. A 1 m^2 panel was decontaminated in this scenario.

The majority of the toxic-load value for the conference room and C141 scenarios occurred during the first 3 h post-decontamination treatment. If the material could be placed elsewhere to offgas for 3 h before being brought into the C141 scenario, then the resulting toxic-load value would be below the ETL_{01} for GD (Figure 24). If the material or contamination source could not be placed elsewhere, then the personnel would require protection until a satisfactory level was achieved.

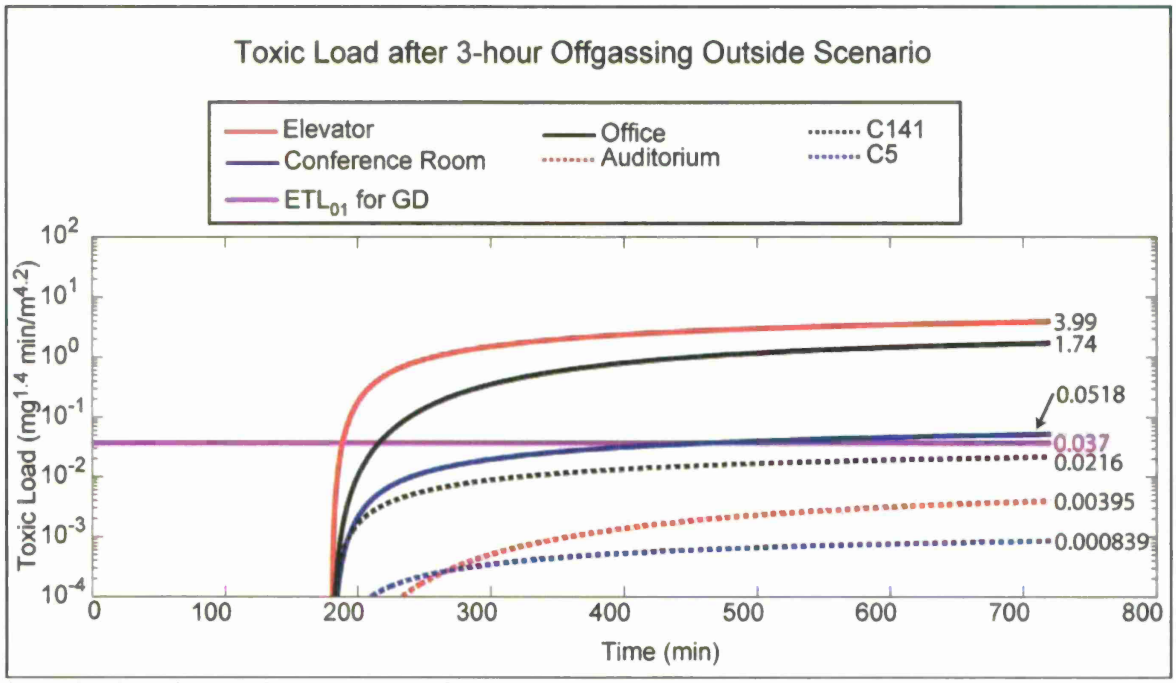

Figure 24. Sample toxic-load results as a function of time after a 3 h offgassing period.

Table 18. Sample toxic-load values for scenarios after a 3 h offgassing period.

Scenario	Volume (m^3)	Air Change (min^{-1})	Loading Factor (m^2/m^3)	TL[a] ($mg^{1.4}$ min $m^{4.2}$)
Elevator	6.75	0.102	0.0222	3.99
Conference Room	175	0.0871	0.000857	0.0518
Office	87.5	0.0129	0.00171	1.74
Auditorium	11960	0.006370	0.000013	0.00395
C141	86.5	0.333	0.00174	0.0216
C5	880	0.333	0.000170	0.000839

[a]3 to 12 h calculation after allowing item to aerate "outside" of scenario for 180 min

4. Impact of reporting Time-Weighted Average (TWA) instead of toxic-load value.

In some cases, reporting the TWA instead of the toxic-load value can give a false impression that a vapor hazard does not exist. The TWA calculation method should not be used to determine if a vapor hazard exists for agents with a toxic-load exponent not equal to one.

5. Impact of reporting vapor test chamber as scenario.

Common practice was to determine vapor hazard based on the vapor emission from a test material in a small vapor chamber. This practice resulted in the inability to compare lab-to-lab data and raised questions regarding lab data utility when compared to field data. The vapor test chamber is a scenario. If the same calculation used to generate the vapor concentration and toxic-load profiles was performed for the vapor chamber, the result, when compared to the six scenarios, is a significantly higher vapor concentration (Figure 25) and toxic-load value (Figure 26). Requiring R&D to develop decontamination technologies that result in no vapor hazard for the vapor chamber scenario could result in more time, logistical burden, and potential material

incompatibility than should be required for adequate decontamination. The use of scenario-based evaluations can better guide R&D efforts.

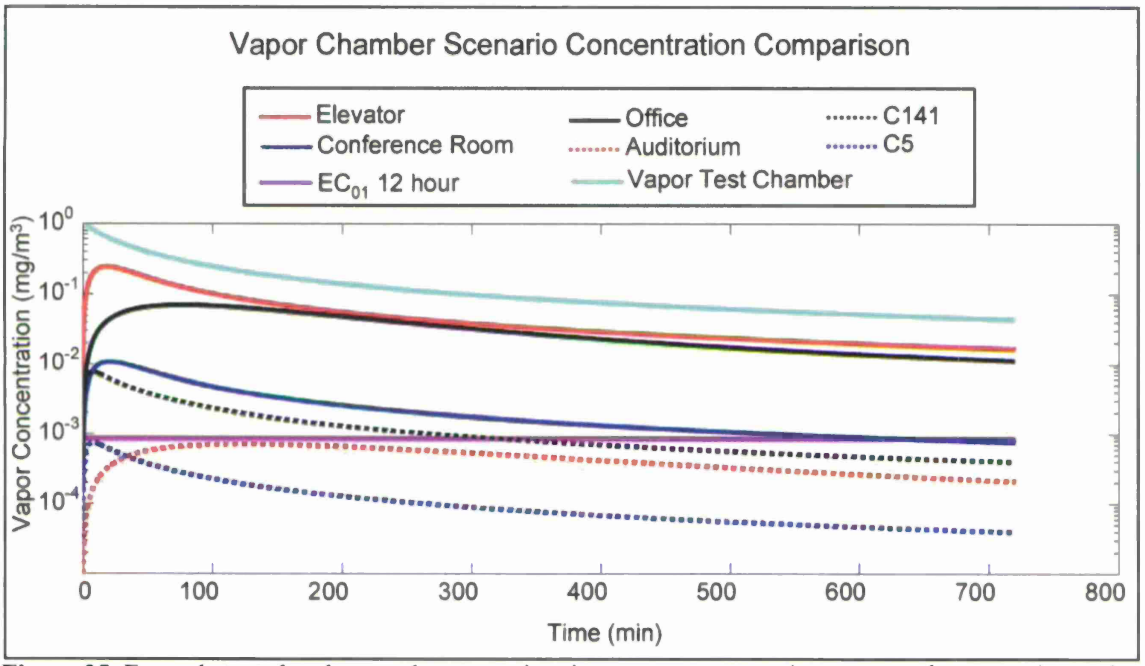

Figure 25. Example test chamber result, over estimating vapor concentration compared to scenario results.

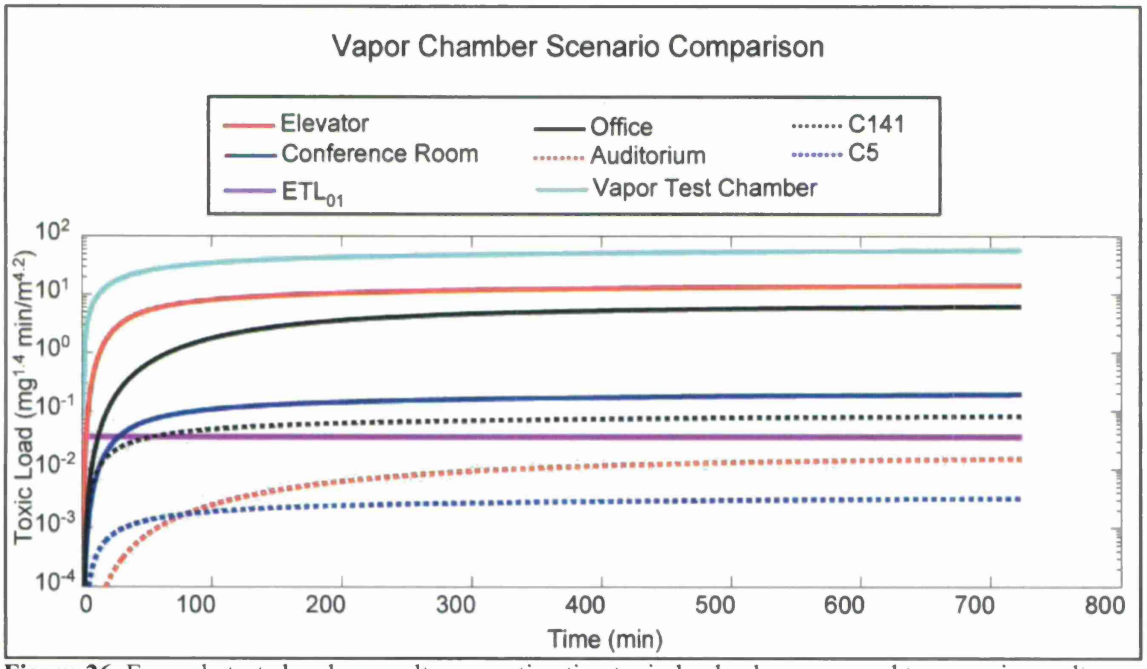

Figure 26. Example test chamber result, over estimating toxic-load value compared to scenario results.

DATA ACCEPTANCE CRITERIA AND CORRECTIVE ACTION

This section contains some guidance for establishing data acceptance criteria and corrective actions for small-item vapor testing. Test items can vary greatly in construction materials, which can result in test variations. The end use of the data may determine many of the test parameters, and should be established between the testing facility and the test sponsor. Small-item test data can be greatly affected by the environmental parameters (temperature, and relative humidity), event timing (aging duration, decontaminant residence time, item transfer to vapor test chamber, etc.), contaminant application, and decontaminant treatment process. Because there are many potential test designs, this section contains data acceptance guidance specific to the test-timing events, contaminant application, vapor test chamber, and the solid-sorbent tubes. Additional guidance may be added, as appropriate, by the performing laboratory for the specific test under investigation. Corrective actions should be added by the method user.

- Amount of Contaminant Delivered: Precision-dispensing tool (e.g., pipette) calibration should be current and compliant with the required performance specifications listed in the most current versions of the ISO 8655, Parts 1 and 2, or ASTM E 1154 for the volumes being delivered.
    - Rationale: The percent neutralization, percent efficacy, and reduction in starting challenge calculations require measurement of the contaminant delivered to determine the difference. The amount of contaminant delivered is confirmed through the analysis of the tool-characterization samples. The tool-characterization samples provide the actual contamination density, compensating for agent temperature (altering the density of the agent) and purity differences.

- Aging Time: Standard test aging time is 60 ± 3 min. For other aging times, the acceptance criterion is target time ±5%.
    - Rationale: The amount of time a contaminated item is aged influences the amount of contaminant absorbed into the item materials. For example, mass adsorbed for sorptive non-porous materials (based on Fick's first law) is proportional to square root of the aging time. A 5% time deviation could result in a 2.5% variation in mass absorbed into the item materials.

- Test-Event Timing: The time between treatment tasks should not exceed 3 min. Transfer to the vapor test chamber is facility-dependent and should be reproducible from test to test. For other event times, the acceptance criterion is target time ±5%.
    - Rationale: Once a test has begun, event timing is crucial. The time between events should be minimized. Event times that are outside the acceptance criteria will induce error and/or bias into the final test results, making the test results potentially unusable, especially for regulatory requirement test-to-test and lab-to-lab comparisons. For example, a contaminated item that was allowed to age longer may induce a negative bias.
    - Note: For tests executed at temperatures other than the room condition, the amount of time spent outside of a temperature-

controlled region may alter the temperature of the test materials, and should be minimized.

- Decontaminant Residence Time: The total decontaminant residence time should be within ±5% of the target time.
    - Rationale: The main objective of the test is to measure the effectiveness of a decontaminant in reducing the contaminant. The decontaminant–contaminant interaction time will be proportional to the amount of agent removed and/or neutralized, most likely in a nonlinear manner.

- Vapor Sampling: The tube-sampling time should be <±2% of target tube sampling time, with ±5% maximum.
    - Rationale: Tube-sampling time should be as accurate as possible since this time is directly proportional to agent mass on tube and the sampled air volume. Any inaccuracy in actual tube-sampling time will result in an under/over estimation of chamber vapor concentration and emission factors.

  The tube-sampling flow rate should be within ±5% of the target flow rate.
    - Rationale: The tube-sampling flow rate should be as accurate as possible. Tube-sampling flow rate directly contributes to the sampled air volume. Any inaccuracy in the tube flow rate will result in under/over estimation of the chamber vapor concentration and emission factors.

  The acceptance of a reported "below detection" vapor concentration should be carefully evaluated. For situations where low concentrations are expected, the sampling time should be as long as reasonable.
    - Rationale: A below-detection vapor concentration is dependent on the analytical detection limits and how the sample was collected. Low vapor concentrations, sampled for short periods of time, may mislead the analyst to conclude that the vapor concentration is zero. It is less than the Limit of Detection (LOD) divided by sampled volume, but may not be zero. This may lead to underestimating the emission factor and ultimately the hazard.

  Prior to test, the SSV specific for the solid-sorbent type and agent tested should be determined.
    - Rationale: Samples collected in excess of the SSV are likely to exhibit breakthrough, resulting in an underestimate of the vapor concentration and ultimately in an underestimation of the hazard.

  The free-air volume of the chamber should be determined as accurately as possible. The volume should be known within ±10%.

- Rationale: The chamber free-air volume is used to calculate the loading factor and air-change rates. Error in the chamber volume will induce scatter in the emission-factor calculations. This may be difficult to physically measure or calculate.

- Vapor-Emission Model: The vapor-emission model should provide a best fit for the data. Though some materials may provide a significant distribution of results, an average RPD value for a model of a single item should be <20%. However, it is recognized that some materials may never meet this criterion. In all cases, the average RPD value should be reported.
    - Note: It is not safe to time-extrapolate vapor emission models beyond the last sampled time.

REVISION HISTORY

December 2008:	Original vapor method.
April 2009:	Final small-item vapor method, original release.
July 2011:	Small-item vapor method, reissued.

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