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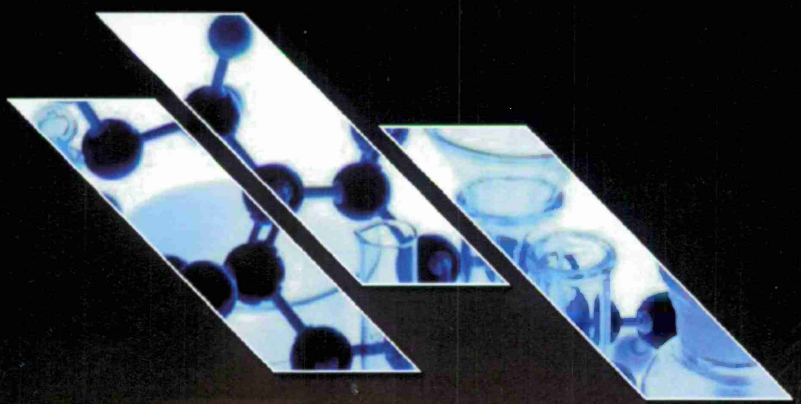
ECBC-TR-934

SMALL-ITEM CONTACT TEST METHOD FY11 RELEASE



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14. ABSTRACT:
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 contact test methods for small items of sensitive equipment. The final contact-sampling 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 Defense Threat Reduction Agency (DTRA) Project No. CA07DEC499. The work was started in October 2007 and completed in March 2009.

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. Manufacturer names and model numbers are provided for completeness. This technical report may not be cited for purposes of advertisement.

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).

This report has been approved for public release. Registered users should request additional copies from the Defense Technical Information Center; unregistered users should direct such requests to the National Technical Information Service.

Acknowledgments

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SMALL-ITEM CONTACT 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 project Nos. BA06DEC414 and CA08DEC420. The SD 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 No. CA08DEC499 extended the Source Document methodology 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 Biological Chemical 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 as 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, and provided 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. These calculations were not part of the original TOP.

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 risk 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 risk. Overestimating the resulting risk 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 then 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. 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 allowed users to compare 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 CONTACT-SAMPLING 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 of 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 contact-sampling method.

The primary objective for the small-item methodology development program was to extend the approaches developed for the robust panel contact test to the contact sampling of small items. The methodology development program was a DTRA-funded program with input from the Product Director, Test and Evaluation Strategy and Support (PD TESS) and the Joint Program Executive Office for Chemical Biological Defense Joint Program Manager (JPEO-CBD JPM) for Decontamination Office. A program expectation was that these new methods would be used to support upcoming testing in the evaluation of the small-item priority list for the Joint Materiel Decontamination Systems (JMDS) acquisition program. A second program expectation was that the

methodology would be universal and would enable the support of programs of records, such as Joint Service Transportable Decontamination System – Large Scale (JSTDS-LS).

The contact test is a measure of the contaminant present after the decontamination process that could pose a risk through transfer to skin or other surfaces. A contact sampler is used to collect contaminant remaining on the item surface that may be bioavailable by touch or available for contact transfer. The contact sampler is extracted in solvent and the extract is analyzed. The contact test value is typically compared to requirements to evaluate decontaminant performance.

The rigorous laboratory-scale contact test method typically uses a standard 2 in. diameter circular panel. A contact sampler is used in this test as a surrogate for dry human skin to collect the contaminant from the panel surface. The contact sampler is extracted, and the contamination collected during a touch is quantified. Figure 1 provides an example of the laboratory-scale contact test sequence.

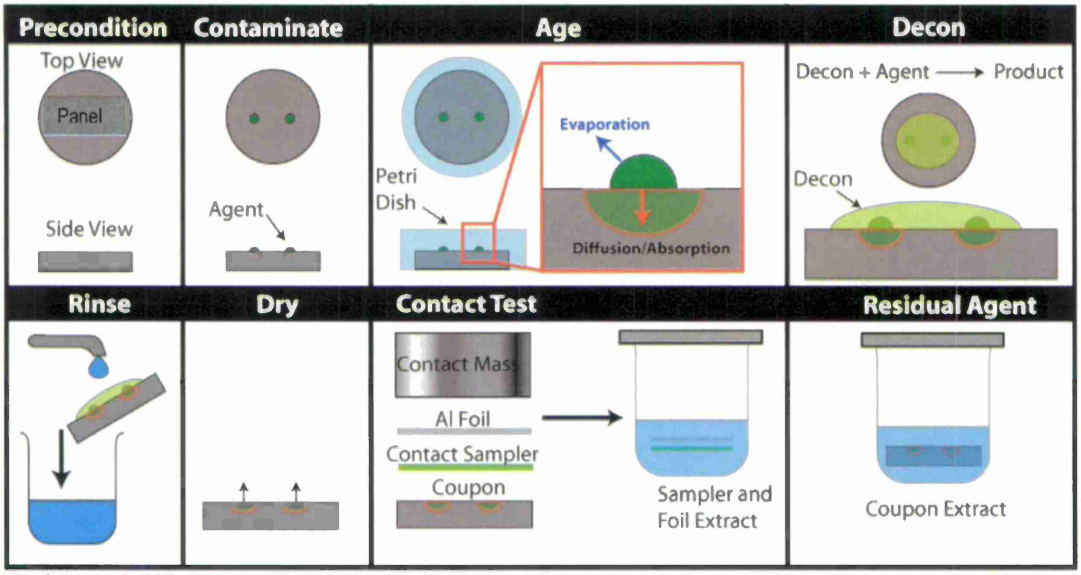

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Figure 1. Laboratory-scale contact test.

At the laboratory scale, a *touch* is defined as each contact test event. In accordance with TOP 8-2-061[1] and the 2007 Source Document[2], the touch is the placement of a contact sampler on the panel for a duration of 15 min. The contact sampler is placed on a flat surface with even contact to the surface area of the panel. Constant pressure is applied to the contact sampler on the panel using a 1 kg weight. The 1 kg weight is called the *contact mass*. Two touches are performed. The first touch is 0–15 min post-decontamination. The second touch is 45–60 min post-decontamination.

The contact test provides a mass of contaminant from the contact sampler in units of nanograms. Through mathematical treatment, the mass of contaminant recovered from the contact sampler is converted to milligrams per square meter, which is typically compared to the requirements.

The amount of contaminant recovered from the sampler extraction alone does not provide a full risk analysis of the future potential contact risks. After contact sampling is complete, the contaminant remaining within the panel may continue to move to the surface of the material, creating a future contact risk. Therefore, full risk analysis requires a characterization of the complete system, including determination of the amount of the contaminant remaining after contact sampling is complete. At the laboratory scale, the use of small panels allows the extraction of the contaminant remaining in or on the material to determine this value, which is called the *residual agent measurement*.

Advancing a decontaminant evaluation to full items adds a level of complexity. Small items are three-dimensional, mixed-material systems with extensive surface features such as curves, junctions, and buttons. The small-item contact test is the measure of the contaminant present after the decontamination process that could pose risk through transfer to skin or other surfaces. This measure of contamination does not represent the full residual contamination, but rather a measure of the contamination that could be available by touch or available for contact transfer. This method can be used to evaluate liquid-phase contaminants such as chemical warfare agents, chemical warfare agent simulants, toxic industrial chemicals, and materials.

The small-item contact test method provides instructions and/or guidance for item pretreatment operations, contamination, decontamination, and contact testing (Figure 2). The contact test is performed using a contact swab to collect the contaminant from the item's surface. The surface of an item in the test configuration is divided into a sampling grid to aid uniform sample collection, and to provide a mechanism for identification of contaminated areas. Each contact swab collected is considered a touch. The contact swab is extracted, and the mass collected during the touch is quantified.

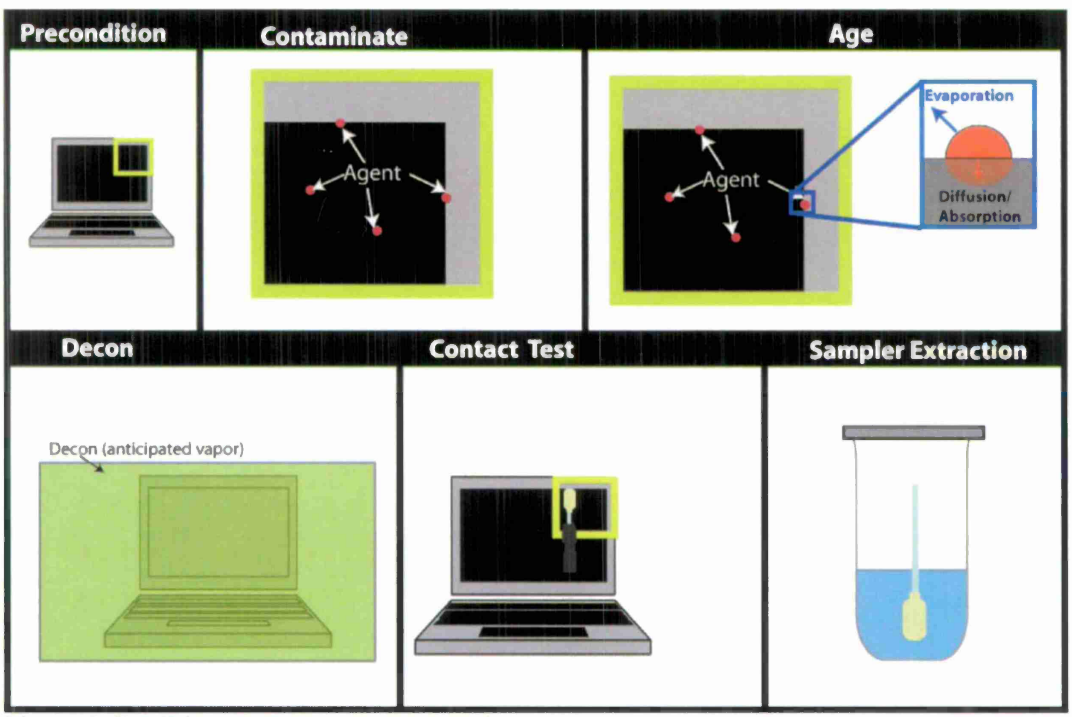

Figure 2. Small-item contact test representation.

This method includes two contact sample types: *gross* and *detail*.

- The larger or *gross* samples are collected over the entire sampling region. The total mass collected is used to determine the *approximated exposure value*. The approximated exposure is the total contaminant mass available in an item that would most likely pose a contact risk.

- The smaller or *detail* samples are used to collect contaminant entrapped in surface features such as crevices and cracks and around buttons. The contaminant in these regions may not be readily accessible but could pose a potential risk to personnel.

Both the gross and detail samples are used to determine the *total potential exposure*. The total potential exposure is the total contaminant mass that could pose a contact risk. At all times, the mass values determined using this method should be presented in the context of the area in which each is collected. This method will provides the mass of contaminant sampled from an item. The final method is documented in the same format as the 2007 SD.

The Small-Item Contact-Sampling Method was developed 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.

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).

Small-Item Contact Test

SUMMARY OF PROCEDURE

The small-item contact test measures the contaminant present after the decontamination process that could pose a risk through transfer to skin or other surfaces. This measure of contamination does not represent the full residual agent, but rather a measure of the contamination that could be bioavailable by touch or available for contact transfer. This method can be used to evaluate decontaminant performance against liquid-phase contaminants such as chemical warfare agents, chemical warfare agent simulants, toxic-industrial-chemicals, and toxic industrial materials. The terms *contaminant* and *agent* are used interchangeably.

This method provides instructions and/or guidance for item pretreatment operations, contamination, decontamination, and contact testing. Analysis and calculations result in the determination of the mass of agent transferred at each contact point, which directly correlates to the approximated exposure and the total potential exposure for a specific item. The contact risk should be assessed with the approximated and total potential exposure values reported.

The contact test uses a contact swab to collect the agent from the item surface. The surface of an item in the test configuration is divided into a sampling grid to aid in collecting uniform samples and to provide a mechanism for the identification of specific contamination locations on an item. At the laboratory scale, a *touch* is defined as each contact test event. Each contact swab collected is considered a *touch*. During small-item contact testing, a touch is characterized by the contact area, contact pressure, contact time duration, and contact swab type. The contact swab is extracted, and the mass collected during a touch is quantified.

This method describes two contact sample types: gross sampling and detail sampling. The gross sample is directly related to the approximated exposure and the total potential exposure. The gross sample provides a mass-per-unit area that could be used alone to provide guidance regarding specific "hot spots" or to aid in full assessment of the total potential exposure of an item. (Hot spots are areas that may not have received satisfactory decontamination.) At all times, these mass values should be presented in the context of the area in which they were collected. This method will provide the mass of agent detected for an item.

COMPARISON TO LABORATORY- SCALE PANEL TESTING

The laboratory-scale decontamination performance evaluation contact test is a rigorous method for execution of decontamination testing using a standard 2 in. disk panel. Tests on this scale result in samples that an operator can easily collect and manage while ensuring that surfaces are sampled uniformly.

Testing decontamination performance on an entire item requires additional considerations. The surface area to be sampled is dependent on the geometry of the item of interest. Surface features such as crevices, folds, and material interfaces are not addressed at the laboratory scale. In addition, the analysis of potential risks resulting from contact transfer requires characterization of the complete system, including the samplers used, mass of contaminant used, contaminant mass removed by touch, contaminant mass remaining after touch, and footprint of the area sampled. Although these parameters can be measured at the laboratory scale, contaminant extraction from a full item may not be practical. These considerations prevent the duplication of the laboratory-scale method when an entire item is under consideration.

The best approach for a full-item assessment is to ensure that all applicable factors are measured. The definition of touch-related variables is critical to experimental execution and data usability. Users should seek to define the "touch" used on an item by defining as many variables that affect that component as possible (i.e., contact time [or number of strokes], sampler extraction efficiency, solvent amount, and contact area [or sample grid size]). Conducting a contact test on an entire item requires additional consideration for the size and amount of surface area to be sampled.

This procedure provides the following information:

- The mass of contaminant, in nanograms, recovered from the contact swab after the decontamination process for each section of the sampling grid.
- The approximate exposure for a specific sampled area and/or an entire item.
- The calculated total potential exposure for an item.

The following prerequisites are required for this test procedure:

- Capability for liquid sample chromatographic analysis.
- Determination of extraction efficiency for contact swab to be used.

Limitations and other test variations:

- Exposure vs. absorbed dose: A contact test is characterized by the contact area, contact pressure, time duration, and sampler type. The value at which personnel may experience toxicological effects is based on the exposure or the dose, which is some fraction of the total amount that may have come into contact with skin. The ability to test every decontaminant-contaminant-material combination on real skin is not realistic. In particular, when sampling a full item with numerous materials, angles, and interfaces, testing every combination, plus duplicating every possible interaction, pressure amount, and time duration is not possible or practical. Therefore, a more prudent, and ultimately more robust, method of determining risk from an operational item should be based on the exposure mass of the agent.

- Comparison of data using different contact swabs should include consideration for the material-uptake characteristics of each swab. These differences should factor into the interpretation of the results.
- The complete small-item contact-testing method provides the contact-test result immediately after decontamination and item drying. In some cases, time extrapolation to determine future potential risk is not recommended, because mass-transport processes may result in the reemergence of entrained agent at later time periods. This potential future risk can only be identified by sampling at later times (e.g., 12–24 h or later).
    - Sorptive/porous materials: Sorptive materials are likely to have significant residual agent post-sampling. Collection of a second sample is recommended at a later time. If additional agent is detected during analysis of the second contact test, then the contact risk at time periods beyond those tested must be reported as uncertain. As a result, the current guidance for many of these contaminated materials is replacement.
    - Surface features: Most actual items will have complex surface features including folds, crevices, cracks, seams, interfaces, etc. within the area to be tested. Although the method does recommend sampling with a detail sampler in these areas, some areas may not be accessible during sampling.
    - Nonsorptive materials: Nonsorptive materials typically yield low-to-no detectable residual agent, which may allow for time extrapolation of the collected value to longer time periods. A reported value that is an estimate or an extrapolation outside the collected data set must be clearly indicated.
- Contaminant Simulant: Substitute 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 a 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 agent data be used to confirm the technology performance and to make risk assessments.
- Residual Agent: Because full-item extraction cannot be performed, it is typically not possible to measure the residual agent present after sampling actual items. A contact sampler may not collect all of the contaminant mass present on the surface of the item. The potential future risk estimation may be limited without residual agent data.

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*.
- **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 sample generated during testing for chromatographic analysis (Gas chromatography and/or Liquid Chromatography) or for other quantitative analytical tools.
- **approximated exposure**: Total amount of agent collected from gross surface samples of an item. Amount detected should correspond to the amount of agent most likely to be available for transfer to skin or other surfaces.
- **bioavailable**: In toxicology, the degree to which a substance becomes available to the target tissue after administration of a defined exposure. In regard to the contact test, bioavailable is defined as the contaminant mass transferred to the contact swab, which could be available for transfer under appropriate conditions.
- **breadboard, brassboard, prototype**: Technology still under development, in differing degrees of configuration, that is not in final form. This can apply to test fixtures, formulations, and/or the decontamination system/applicator.
- **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.
- **confidence interval**: A calculated range for a data set that future results are likely to fall between.
- **contact dose**: The amount of agent that enters a target after crossing an exposure surface.
- **contact risk**: The amount of contaminant remaining on the surface that, based on human toxicological estimates, could pose a threat to unprotected personnel touching the contaminated surface.
- **contact swab**: A tool used in this test to determine the approximate applied exposure and/or potential exposure. The swab sorbs the available surface contamination, which is then extracted to determine the mass of agent available for contact transfer.
- **contact swab transfer efficiency**: The measurement of the contact swab's ability to collect the contaminant from the test material. This measurement is made by using a nonsorptive material as the ideal case to determine the contact swab's ability to sorb the analyte. Transfer efficiency may be different for material-agent combinations.
- **contact transfer**: The capability of a contaminant, present on or in a specific surface, to be moved to another surface through touching the contaminated surface.

- **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 DT.
- **contamination, localized**: Application of the contaminant to selected regions to evaluate specific materials, regions, or surfaces, based on the test objective. This option is best suited for R&D testing to evaluate and optimize decontaminant performance.
- **contamination set**: A specific contamination density, drop volume, and deposition pattern combination used for dose confirmation. Deposition pattern only matters if the contaminant application uses more than one drop.
- **contaminant simulant**: Compounds of lower toxicity that contain at least one property similar to the parent contaminant (e.g., live chemical agent).
- **decontaminant**: For these procedures, a decontaminant is a substance with the ability to remove and/or neutralize chemical agents on/in surfaces of interest. The decontaminant can be liquid, solid (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 *decontamination process* refers to a specific series of treatment tasks performed, which may include contaminating, aging, decontaminating, rinsing, and drying the surface of interest.
- **detail sampler (swab)**: The contact swab used to assess agent that may be in surface features such as cracks, crevices, between buttons, etc. The sum of agent collected in all detail swabs is added to the approximated exposure to determine the total potential exposure.
- **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 to the test item during a test session. Contaminant delivery tools, such as pipettors and syringes, cannot always be assumed to 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.

- **exposure mass**: The amount of agent present in the contact volume. For example, the total mass of agent on the exposed surface of an item, collected with a sampler.
- **extraction**: The separation of a component from a mixture through selective solubility.
- **extraction efficiency**: The quantitative measurement of the solvent's ability to selectively solubilize (i.e., remove) the contaminant from the test material (e.g., panel, contact swab).
- **gross surface sampler (or swab)**: Contact sampler used to collect agent in each section of a sampling grid. The total agent collected for all gross surface samples in an item, which will determine the approximated exposure.
- **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 footprint**: The general area in which an item could be contained. In this method, the item footprint is used to determine the area of contamination.
- **item handling**: The treatment of a 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.*
- **moderate condition**: The test condition in the middle of the testing range, e.g., 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, although 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 to minimize contact and transfer hazards and to sustain operations. This may include decontamination of the individual beyond the scope of immediate decontamination, decontamination of mission-essential spares, and limited-terrain decontamination. (Reference FM 3-11.9.)
- **potential exposure**: A total amount of agent collected from all surface samples. All agent collected could present a hazard via transfer from an item to another surface.

- **quantitation limit**: The lowest amount of analyte in a sample, which 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. Also known as the coefficient of variation (CV).
- **requirement levels**: The documented amount of permissible agent remaining after a decontaminant process, typically expressed as a surface concentration in milligrams per square meter.
- **residual agent**: The amount of contaminant present in or on the material of interest, after the decontaminant process and hazard test have been conducted.
- **rinsate**: The rinse solution collected during 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.
- **sampling grid**: A series of sampling sections on an item. Often designated in a sampling plan and marked on an item to enable quick reference.
- **sessile drop**: A liquid droplet that is firmly attached (not spreading) on a surface. If the droplet significantly spreads across the surface, it is better described as a thin film.
- **sorptive or porous material**: 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, and decontaminant process, and the environmental and test sampling process variables (i.e., contact, remaining, residual).
- **touch**: A contact test event. A touch is characterized by the contact area, contact pressure, contact time duration, and contact swab used. For the small-item test, the contact area is of a size determined before testing. Method development utilized a 3 x 3 in. area for all samples collected, with a 10-stroke sample that was repeated in a lattice pattern. The total contact-sampling time is typically between 10 and 20 s.

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- Zartarian, V.; Bahadori, T.; McKone, T.; Adoption of an Official ISEA Glossary. *Journal of Exposure Analysis and Environmental Epidemiology* **2005**, *15*, 1, 1–5.
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REAGENTS, MATERIALS, AND EQUIPMENT

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 for evaluation are dependent on the test objectives and specific test facility capabilities. Chemical

decontaminants can be liquid, solid, or vapor phase and may contain a reactive functionality for neutralizing chemical contaminants.

- **Extraction Solvents:** The test requires the extraction of sorbed agent from test materials such as the contact swab. Typical solvents 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, distilled or deionized water is recommended 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 has been chosen 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 the interpretation of the starting challenge contamination density. The drop volumes most commonly used will 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 when 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 a survey of commercial items with repeater capability, is about 0.2 µL. Syringes used for the purpose of contaminant delivery should have a maximum inaccuracy of 1%, and a maximum imprecision of 1% of the volume being measured.

- Computerized Dispensing System: 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 a repeatability of <1%. The manufacturer's performance specifications should be reported and evaluated against acceptable measurement uncertainty for the particular test.

- **Decontaminant Delivery Tool:** The tool used to deliver a specific volume of decontaminant to the item's contaminated surface. The specific decontaminant under evaluation will typically determine the delivery tool and decontaminant volume.
  - Pipette: See 'Pipette,' above.
  - Spray Bottle: A spray bottle can sometimes be used to mimic a spray application. The spray bottle should be evaluated to determine the number of pumping actions required to achieve the target decontaminant application. Tools obtained or developed by a testing laboratory, which have no performance specification standard or vendor-provided performance information, should be tested to determine 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 that are under development and are not in final configuration. The decontaminant generation and delivery may not be known. Tools obtained or developed by a testing laboratory, which have no performance specification standard or vendor-provided performance information, should be tested to determine 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 as directed by the vendor. Tools obtained or developed by a 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 able to reproducibly deliver the same amount of decontaminant from test to test, and the exact usage should be documented.

- **Extraction Solvent Delivery Tool**: Tool used for the delivery of specific volumes of solvent to the extraction container. A repeater tool is recommended because multiple samples are collected for each item in a test. The typical delivery volume for extraction of a 3 x 3 in. sampler is 18–20 mL.
  - Bottle-Top Dispenser: These tools are precision liquid dispensers that can be connected to solvent and rinse water bottles. These tools are available in different configurations based on the liquid to be dispensed. Appropriate tools should be used to dispense

organic solvents. Bottle-top dispensers to be used for the purpose of solvent delivery should be compliant with the required performance specifications listed in the most current versions of ISO 8655: Parts 1 and 5 and/or ASTM E 1154 for the volume being measured. (Examples: Dispensette and Brinkman are common brands.)

- **Sample Dilution and Analytical Standard Preparation Tools**: Used to prepare sample dilutions, these tools must be capable of delivering the specified liquid volumes. Single-dispensing tools (as opposed to 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 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 E288 or ISO 1042.
- **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 samples are added or removed. The system must have temperature and relative humidity data logger capability and be able to store and download temperature and humidity data and traces to a computer for analysis. The system must be able to maintain temperature and relative humidity. The system operation and range should be known.
- **Analytical Chromatography Equipment:** The small-item contact test 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, are preferred. Other quantitative detectors may be used.

MATERIALS AND SMALL EQUIPMENT

- **Aluminum Foil:** Aluminum foil is typically used to line a workspace.
- **Analytical Vials and Caps**: Appropriate analytical vials for use on the chromatographic equipment. The vial cap should be lined with an inert material. polytetrafluoroethylene (PTFE)/Teflon coating is the preferred material to prevent the extraction of plasticizers or other impurities into the sample.
- **Contact Swab(s)**: Adsorptive material used to collect available contamination from surface of interest. The use of polyurethane foam swabs, wetted with isopropyl alcohol (IPA), is suggested. Testing will typically involve a sampling technique using two sets of sampling strokes for a defined surface area.

- **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 excess volume of household bleach to allow submersion of the items.
- **Extraction Containers**: The small-item contact test procedure involves the extraction of the contact swab and/or panel. A glass container, such as a vial or jar of sufficient size to hold the contact swab, panel (if required), and extraction solvent volume, is required. The container cap should be lined with an inert material. PTFE/Teflon is the preferred material to prevent the extraction of plasticizers or other impurities into the sample. Use of plastic containers is not recommended for chemical agent testing.
- **General Laboratory Items**: Items may include glassware (vials, flasks, cylinders, bottles, beakers, jars, etc.), paper towels, beakers, vials, spatulas, paraffin-coated film, etc.
- **Items**: The test sample used for evaluation.
- **Sample-Handling Tools**: The tools used to handle items during testing. These may include forceps, tweezers tongs, aluminum foil, or wax paper.
- **Sample Tray:** Optional item for the handling and movement of items during testing.
- **Sample Area Marker:** Used to create a sampling grid for each contact swab used. Items may include wax pencil or markers.
- **Standard Laboratory Record-Keeping Items**: Record-keeping items may include a computer, data test forms, laboratory notebooks, and writing utensils.
- **Timing Device(s):** Test method requires accurately timing key steps. Digital timers reporting in minutes and seconds are preferred.

ADDITIONAL MATERIALS AND EQUIPMENT: The test facility and decontaminant preparation and application processes may require additional materials and equipment. Additional materials and equipment may include, but are not limited to, analytical balances, stir plates, stir bars, vortexers, pH meters, 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 method user to establish the appropriate environmental, health, and safety practices for using this method and handling the waste generated while complying 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.

PROCEDURE

This method contains several procedures to execute a small-item contact test. Each procedure provides steps to define several variables. 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.

PROCEDURE 1: CONTACT-SAMPLING PLAN DEVELOPMENT

1. Identify the Sampling Grid

1.1 Place the sample item on a flat surface with the surface to be contaminated facing up. Orient the item in the configuration that it will most likely assume in an actual contamination scenario.

1.2 Divide the selected surface into regions of equivalent size (3 x 3 in. squares are depicted and recommended). The number of regions selected will determine the number of gross surface swabs collected (or "touches" performed). A laptop illustration is provided in Figure 3 (depicted in yellow).

1.3 Determine the number of interfaces, crevices, and corners to be sampled with detail samplers to aid in determining potential total exposure. A laptop illustration is provided in Figure 3 (depicted in green).

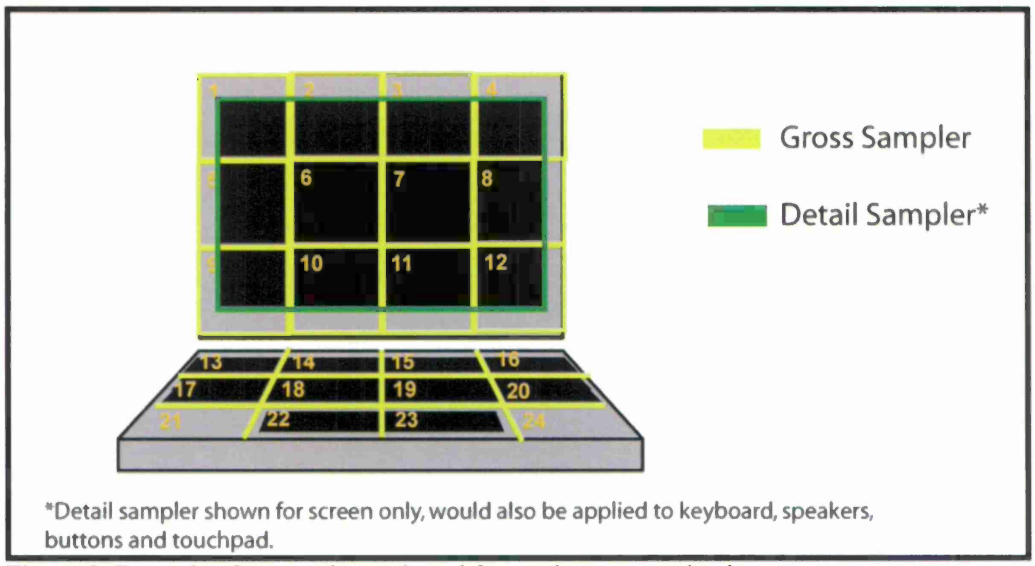


Figure 3. Example of area regions selected for test item contamination.

1.4 Determine total number of surface swabs to be collected from an item.

A sampling size of 3 x 3 in. will require the use of a a swab with a 0.5 in. head and 500 µL of solvent. Any difference in sampling area should result in a proportional change to the size of the swab and the amount of solvent used.

Regions of the sampling grid could align with the contamination regions.

For both types of contamination (localized and full item), sampling of all exposed surfaces in the test configuration is recommended. During the aging process, mass transfer due to dripping, smearing, etc. may occur, and surfaces that were not originally contaminated may actually contain agent at the time of sampling.

2. Create Guide Lines on the Sample Item

2.1 Divide the selected surface into regions of equivalent size (3 x 3 in. squares are depicted and recommended). Use a sample area marker to mark the edges of an item to denote where the sampling grid lines for gross samples will begin.

Wax pencil is recommended because the marks are easy to identify on an item and can be removed from the surface fairly easily after testing.

2.2 Mark the anticipated line intersections with a small "+" to help guide sample collection during Procedure 2, Step 10: Sample Item with Gross Surface Sampler. An illustration of a laptop divided into regions is provided in Figure 4.

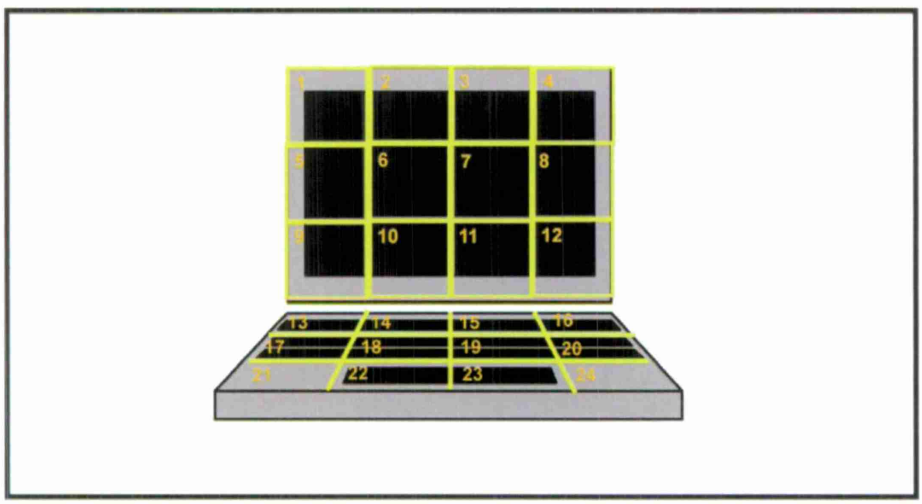


Figure 4. Example of area regions selected for test item contamination.

Drawing complete lines on an item is not recommended because the marks may create a barrier that impedes agent transfer between areas, resulting in an inaccurate assessment of the item.

PROCEDURE 2: ITEM TREATMENT AND CONTACT TEST

Procedure 2 specifies the sample treatment and contact-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. Figure 5 provides a representation of the test steps.

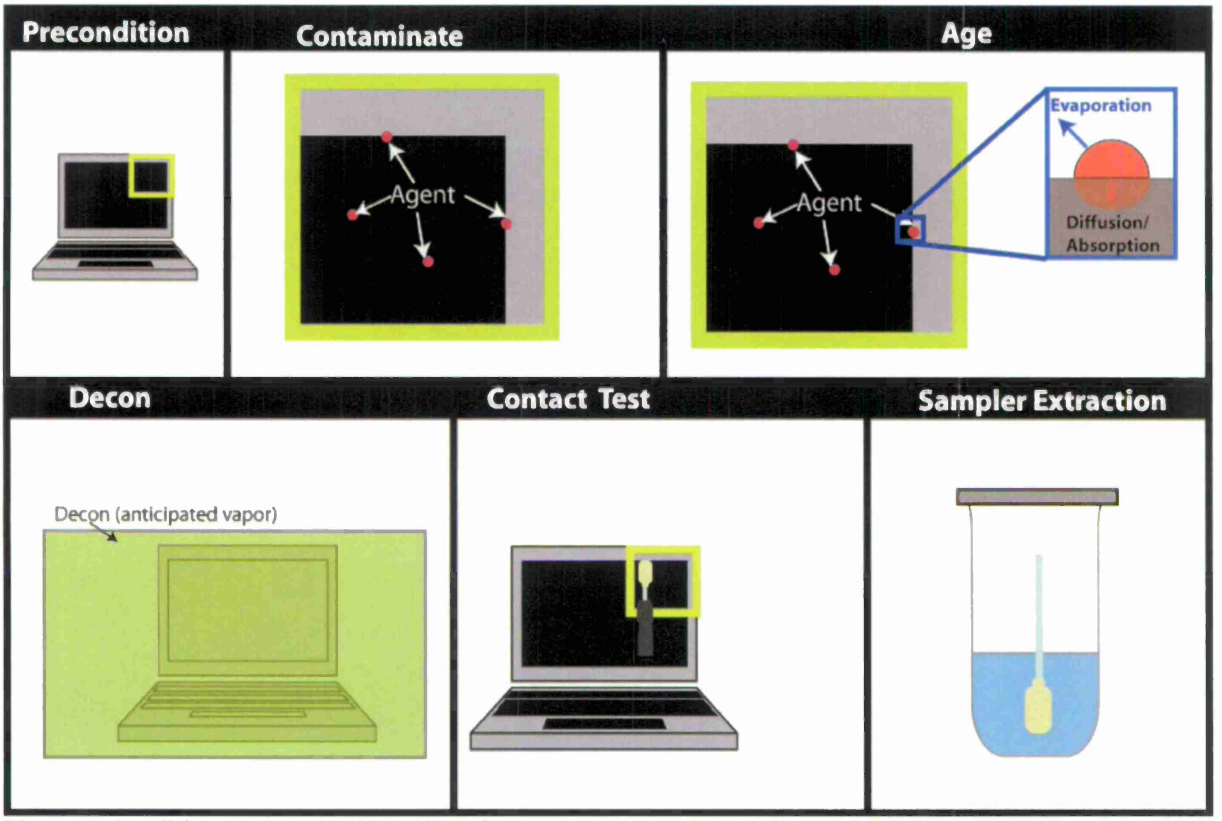


Figure 5. Small-item contact test representation.

1. Test Preparation Tasks

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

- Turning on equipment that will need to thermally equilibrate (i.e., environmental chamber).
- Selecting the contamination method and completing preparatory calculations.
- Obtaining the documented decontaminant application method.
- Completing the test area setup tasks, including any labeling (i.e., vials, trays) and other associated pretasks that can be performed before the start of testing.
- Preparing and inspecting items. All items should be cleaned (if required) and inspected to remove any unacceptable specimens before the start of testing.
- Preparing the decontaminant.
- Checking that the necessary equipment is operational and that the calibration is current (if applicable).
- Obtaining the contaminant. Contaminant may require thermal equilibration to room temperature (or other sponsor specified temperature) before 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. Using timing charts to stagger

major steps is strongly encouraged because subtle differences in item treatment may contribute to data scatter.

The number of replicate items depends on the test objectives and item availability. Reusing items is not recommended because items may contain entrapped agent, which could resurface over time, creating a false-positive test interference. Five dose-confirmation samples per contamination set are recommended.

2. Precondition 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 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 moderate condition case temperature is 21 ± 3 °C (70 ± 5 °F) preferred, 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 specifications. 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 upward.

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

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 provided in ASTM D4442.

2.5 Minimize temperature fluctuations by removing samples from the environmental chamber immediately before executing Step 5: Contaminate Items.

3. Contaminate Items

3.1 Select the contamination option from the following choices:

- Full contamination.
- Localized contamination.
- No contamination: This is a negative control test to evaluate whether an item or decontaminant-item pair contains compounds that may interfere with the chromatographic analysis of the analyte of interest. Proceed to Step 8: Decontaminate the Items.

3.2 Determine the contaminant volume and drop size as follows:

3.2.1 Select the target starting challenge (i.e., contamination density), in grams per square meter.

3.2.2 Determine the item footprint in square meters.

Item footprint and sampling area are independent values. The footprint is defined as the area in which an item can be contained. Sampling area includes all of the regions that have been identified as contamination surfaces.

3.2.3 Determine the mass of contaminant.

3.2.4 Determine the volume of 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 as follows:

3.3.1 Identify the test item configuration. This parameter determines how the item will be placed for contamination and potential decontamination. The item configuration may also determine which surfaces are sampled in Step 10: Sample Item with Gross Surface Sampler, because it is likely that some surfaces will not be exposed to contaminant. For example, a laptop may have several configurations, including closed and opened as shown in Figure 6. In the closed position, exposure to the inside of the item may not be likely, and sampling may not be necessary. In the opened position, exposure to the bottom, and possibly other surfaces, may not be necessary, as determined by actions in Step 5.3.2.

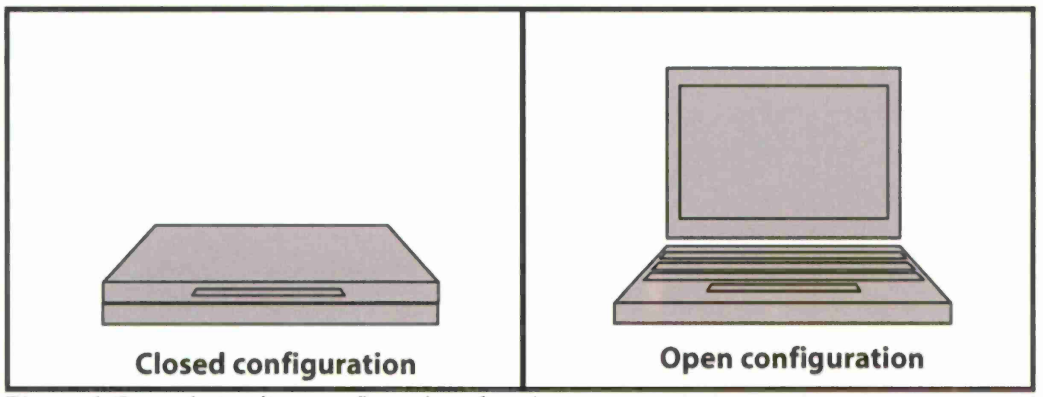


Figure 6. Example test item configurations for a laptop.

3.3.2 Select the contamination areas. A test item may have several different contamination options as determined by the test objective and procedures for handling the contaminated item safely. Three potential areas for a laptop, with contaminated surfaces highlighted in red, are shown as an example in Figure 7.

3.3.3 In all cases, despite the changes in the sampling area, the footprint is constant and is determined by the item, not the test (blue highlight).

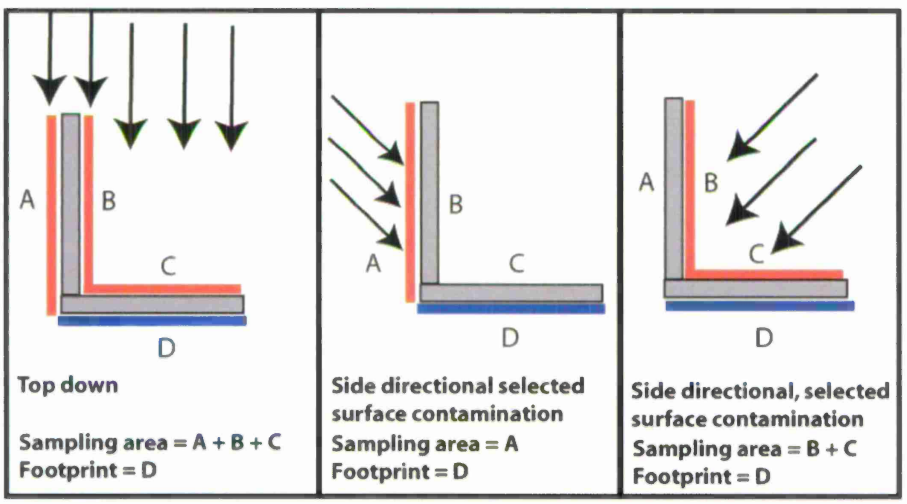


Figure 7. Example of test item contamination areas for a laptop in the open configuration.

3.4 Identify the specific locations for contaminant placement (sampling grid) as follows:

3.4.1 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 2 µL in volume, is shown in Figure 8.

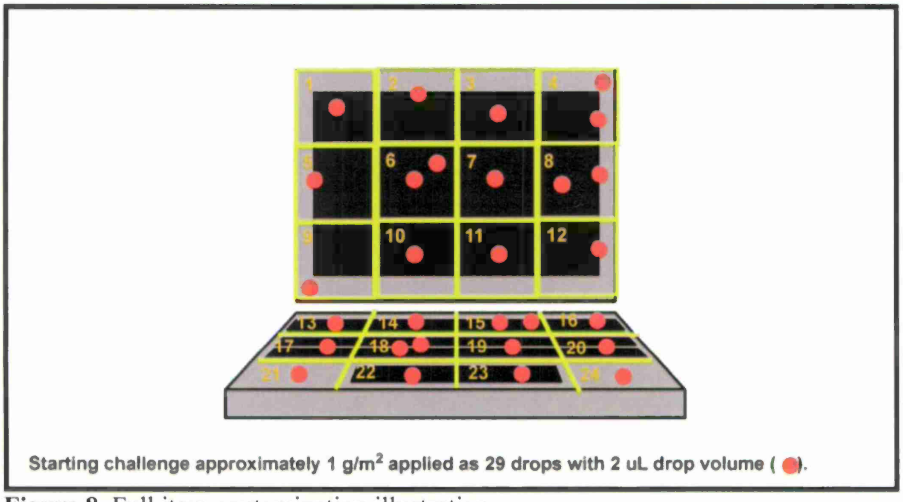


Figure 8. Full item contamination illustration.

- Localized contamination: The contaminant should be placed on specified regions, as determined by 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 2 µL in volume, is shown in Figure 9. This example meets a test objective to evaluate the ability to decontaminate the screen-case interface.

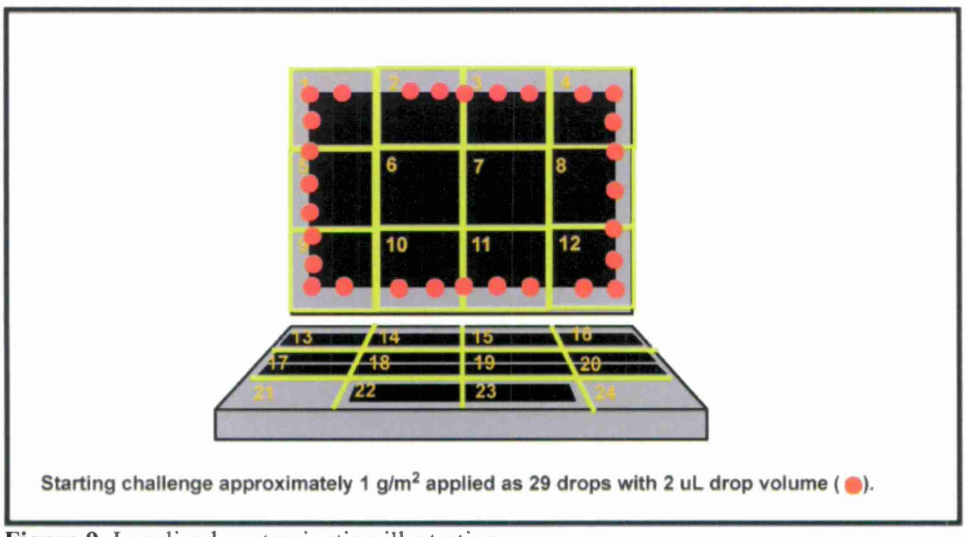


Figure 9. Localized contamination illustration.

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. Any alternate methods should be documented in the test report.

3.5.1 Set the tool to the appropriate drop volume.

- The pipette volume should not be changed within a set of procedures. Tests have shown that changing the tool's dispensing volumes 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 the 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.

Steps 5.5.4.5, 5.5.4.6, and 5.5.4.7 may be performed later in the test when samples are diluted and prepared for analysis. This delay typically occurs in tests that include a large number of panels to facilitate completion of the staggered timing chart. 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 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.

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.

Steps 5.5.6.5, 5.5.6.6, and 5.5.6.7 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. Item Aging

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

4.2 Select and execute the item-aging procedure.

Option A: At 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 the 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. A generalized procedure for item preconditioning using an environmental chamber could include the following steps:

4.2.1 Set the environmental chamber to the specified test condition.

4.2.2 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. Maintaining temperature and humidity at the setpoint, for at least 30 min prior to the start of aging, is recommended.

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

4.2.4 Allow items to age for the desired aging time.

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

5. Prerinse or Clean 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 rinsing. Rinsing is not performed. Please continue to Step 8: Decontaminate the Items.

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 that are based on the liquid to be dispensed. Appropriate tools should be used to dispense organic solvents. Examples are the Dispensette and Brinkman brands. Bottle-top dispensers 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 rinsate 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 rinsing procedure may call for the use of hot soapy water.

OPTION C: Preclean 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.

6. Decontaminate the Items

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

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

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

6.3 Select and execute the decontamination procedure.

Option A, Vaporous decontaminants. An item is placed in a decontamination chamber. 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 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 an application method and 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 cover the entire contaminated surface adequately, which may yield 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: Achieving the desired environmental conditions may require the use of an environmental chamber. The moderate condition case temperature preferred 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. A generalized procedure for item decontamination using an environmental chamber could include:
    - Set the environmental chamber to the specified test condition.
    - 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. Maintaining temperature and humidity at the setpoint, for at least 30 min prior to the start of aging, is recommended.
    - Apply the decontaminant.
    - Place the items in the chamber. Items should be placed with the contaminated test surface facing upward. Items should be appropriately spaced to minimize contact between items.

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 in accordance with 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 an item baseline for the test treatment process and environmental conditions under investigation.

6.4 Wait the desired residence time.

6.5 Remove the items from the environmental chamber, if necessary.

7. Post-Rinse and Dry

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

Option A: No rinsing. Rinsing is not performed. Please continue to Step 10: Sample Item with Gross Sampler.

Option B: Rinsing is performed. 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 approximately 100 linear feet per minute (lfm) airflow. Controlled air dryers can be used. Blotting, wiping, or other direct-surface contact methods are not recommended because the contaminant may be removed as part of the process.
- Item placement: The items should be positioned to increase 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 since contaminant evaporation could occur, resulting in potential differences in test results using different drying time periods.
- Inspection of the surface after dry time is complete: The appearance of the surface should be inspected for changes or residual water and documented.

7.2 Record the date and time then note when the end of the item-treatment process timeline is complete.

8. Sample Item with Gross Surface Sampler

8.1 Conduct the first touch with the gross sampler as follows:

8.1.1 Wet the gross sampler with solvent:

Option A: Wet a polyurethane foam swab with IPA. A sampling size of 3 x 3 in. will require a swab with a 0.5 in. head and 500 µL of solvent. Any difference in the sampling area should result in a proportional change to the size of the swab and the amount of solvent used.

Option B: Wet the swab with type and amount of solvent. The extraction efficiency of any swab-solvent combination should be determined before testing begins to discover the likelihood of contaminant interactions with the swab, which could prevent an accurate assessment of "available agent".

8.1.2 Sample the item surface within a specific grid region using a gross sampler as follows:

Option A: 3 x 3 in. touch area. Stroke the surface with the swab 20 times, with each stroke lasting approximately 1 s.

- Place the swab head in one corner, parallel to bottom of the touch area.
- Stroke from front to back (away from operator) toward the opposite corner of touch area (Figure 10, Step 1).
- Repeat for strokes 2–10, with each stroke overlapping slightly, until the entire touch area has been covered.
- Rotate the swab so that the next stroke uses a fresh surface of the swab.
- Stroke from left edge to right edge of touch area (Figure 10, Step 3).
- Repeat for strokes 12–20, with each stroke overlapping slightly until the entire touch area has been covered. The entire technique is depicted in Figure 10.

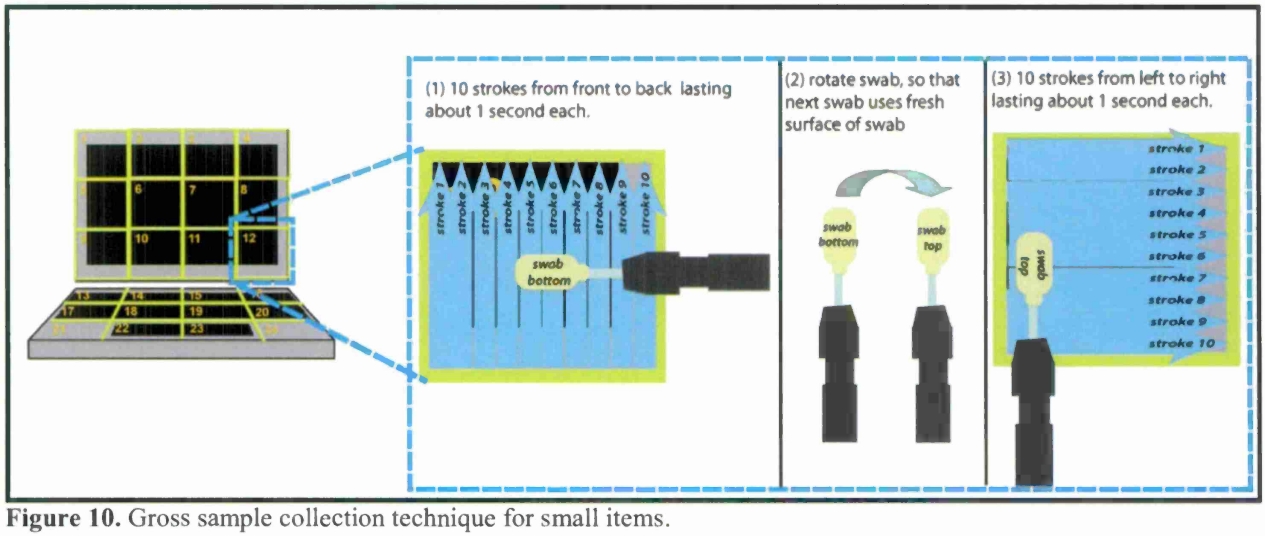


Figure 10. Gross sample collection technique for small items.

Option B: Any size touch area. The number of strokes should be proportional to the 20 strokes per 3 x 3 in. area as explained in Option

A. The actual wiping pattern (perform half the number of strokes, rotate the swab, and repeat) should be executed regardless of the touch area chosen.

8.2 Extract the contact swab as follows:

8.2.1 Place the contact swab into a scintillation vial (or equivalent glass container).

8.2.2 Add 20.0 mL of extraction solvent.

8.2.3 Place a PTFE/Teflon-lined lid on extraction scintillation vial.

8.2.4 Thoroughly mix the contents by inverting the vial three times.

8.2.5 Leave the contact swab in the extraction solvent for 60 min. Note: Other extraction times can be used, but the extraction efficiency measured must incorporate the same extraction time.

8.3 During extraction time, repeat Steps 10.1 and 10.2 for each block in the touch grid until the entire item has been sampled.

8.4 At the end of the extraction period, thoroughly mix the contents by inverting the vial three times.

8.5 Using a clean pipette, transfer a sample into an analytical vial for analysis.

9. Conduct First Touch with Detail Sampler

9.1 Dip a smaller detail swab, with a 0.188 in. head, into vial of IPA for 5 s.

9.2 Trace around area of interest for this particular swab with foam head.

9.3 Follow Steps 10.2 through 10.5 for extraction.

10. Chromatographic Analysis for Agent

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

- Dose-confirmation liquid sample
- Contact swab extract

10.2 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.

10.3 Obtain the list of analytical results for extracts in units of nanograms per milliliter.

10.4 Correct the results for dilutions.

11. Complete the Required Reporting for This Procedure

The test report should contain the following items:

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, 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**
  - **Pipettes, Syringes, and Commercial Applicators:** Provide the tool identification including 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:** For systems without a documented performance specification standard, provide the following information: 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 should be included in the final report.
- **Decontaminant Delivery Tool**
  - **Pipettes and Syringes:** See the 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**
  - **Pipettes and Syringes:** See the contaminant delivery tool reporting requirements for pipettes and syringes.
  - **Volumetric Glassware**: Provide the glassware description including 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.
- **Defined Area Marker:** Provide a description of the tool used including the source and part number.
- **Analytical Chromatography:** Provide a description of the entire unit configuration. For major components, include the component description (e.g., detector, injection system, etc.), manufacturer, and model number.

MATERIALS

- **Items:** Provide information on the item including description, manufacturer, and model number.
- **Contact Swab:** Provide information on the swab including description, manufacturer, and model number.

CONTACT TEST

- **Precondition Items:** Description of how the conditioning was performed, including the following:
    - Location.
    - Preconditioning length of time in hours and minutes.
    - Temperature average with the high and low standard deviation for the conditioning period.
    - Relative humidity average with the high and low standard deviation for the conditioning period.
    - Identification and discussion of any temperature or humidity excursions including the excursion value, duration, and suspected cause.
- **Contamination:** Include the following information:
    - Description of how the contamination was performed.
    - Target contamination density in grams per square meter.
    - Total agent volume in microliters applied per item.
    - Agent drop volume size(s) in microliters per item.
    - Test location temperature and relative humidity during contamination.
    - Contaminant temperature at the time of contamination.
    - Description of contaminant handling. For example, if the contaminant is applied "cold" or "warm," provide a description of how the contaminant was chilled or warmed. If the contaminant was warmed to room temperature before application, note this or any other handling information.
- **Dose-Confirmation Sample Preparation:** Include the contamination density in grams per square meter, the total agent volume in microliters per vial, the agent drop volume size(s) in microliters per vial, the solvent identification, and the solvent volume.
- **Aging:** Description of how the aging was performed, including the following:
    - Aging length of time with applicable units.
    - Temperature average with the high and low standard deviation for the aging period.
    - Relative humidity average with standard deviation, high, and low for aging period.
    - Identification and discussion of any temperature or humidity excursions, including the 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:

- Description of the rinse solution, including water quality, temperature, and, if used, the 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, relative humidity, and the end of aging surface visual inspection, including any residual water on the surface.

- **Decontamination:** Include the following information:
  - Decontaminant information including the vendor and part number for commercial items and the configuration for developmental items.
  - Documented decontamination method including the following:
    - Decontaminant residence time.
    - Temperature average with high and low standard deviation for the decontamination period.
    - Relative humidity average with high and low standard deviation for the decontamination period.
    - Decontaminant temperature at time of application (e.g., room temperature, chilled, warmed, etc.). If decontaminant is applied "cold" or "warm," provide a description of how the decontaminant was chilled or warmed.
    - Decontaminant amount delivered to item.
    - For vaporous decontaminants: injection rate, flow rate, fumigant concentration, temperature, and relative humidity.
    - For liquid decontaminants: volume delivered.
    - For solid decontaminants: mass delivered.
    - For other decontaminants: amount delivered.
- **Contact Test:** For each touch provide the following information:
  - Temperature of test.
  - Contact sampling period (in number of strokes).
  - Volume of solvent used per touch.
  - Extraction solvent used.
  - Extraction time.
- **Chromatographic Analysis:** Describe the chromatographic analysis, including the queue design, the analytical method, continuing calibration verification (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:** These results should be maintained on file and reported to capture the amount delivered per test.
- **Reporting Statement (Recommended):** Small-item contact 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 3: DOSE-CONFIRMATION SAMPLE CALCULATION

1. Calculate the Contaminant Mass Delivered

1.1 Obtain the raw chromatography results, in nanograms per milliliter, for the dose-confirmation samples (DC_E).

1.2 Correct the raw result for any dilutions performed between the sample collection and analysis. Report the corrected value (DC_{E-C}) in nanograms per milliliter.

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

$$Del = DC_E \times SV \qquad \text{Equation 1}$$

1.4 The *Del* value may be better represented in grams 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, the calculated average, and standard deviation including units.

PROCEDURE 4: CONTACT TEST MASS CALCULATION

Procedure 4 contains the contact test mass data calculation to be used in the determination of approximated exposure (AE) and the total potential exposure (TPE) of an item.

The contact test on a small item includes two types of sampling: the gross sample and the detail sample. The gross sample is directly related to both the AE and TPE. The gross sample provides a mass-per-unit area that could be used alone for guidance regarding specific "hot spots," or to aid in full assessment of bioavailable agent on an item. (Hot spots are areas that may not have received satisfactory decontamination.) At all times, these mass values should be presented in the context of the area in which they were collected. For example, in the instance of hot-spot identification, users should report the mass of agent detected in the specific grid square. For full-item assessment (i.e., AE, TPE), the sum of all agent detected should be reported as mass per item.

Contact risk should be assessed with the AE and the TPE values reported.

1. Convert the Results From Nanograms per Milliliter to Nanograms for Each Sample Collected

1.1 Obtain the chromatography data in nanograms per milliliter for the contact swab extract (CS_E), and the dose-confirmation samples (DC_E) that have been corrected for any dilutions performed between sample collection and analysis.

1.2 Convert the contact test result from mass in solution (CS_E) to mass (CS_M).

For each contact swab extract, convert the analytical results in nanograms per milliliter to mass results in nanograms (ng) by multiplying the extraction solvent volume (EV) in milliliters. For the method as written, the extraction solvent volume is 20 mL.

$$CS_M = CS_E \times EV \qquad \text{Equation 2}$$

1.3 Match each mass found to the corresponding location in the touch grid from which it was obtained.

2. Calculate the Contact Test Results Corrected for Extraction Efficiency

2.1 Obtain the calibration curve developed during the extraction efficiency determination.

2.2 Calculate the extraction efficiency corrected contact test result (CS_C) in nanograms.

3. Calculate the AE

3.1 Obtain all gross sampler results from Step 2.

3.2 Calculate the full item approximated exposure in nanograms per item:

$$AE = \Sigma\, CS_{C\,(gross)} \qquad \text{Equation 3}$$

4. Calculate the TPE

4.1 Obtain the result from Step 3.

4.2 Obtain all detail swab results from Step 2. $[\Sigma\, CS_{C\,(detail)}]$

4.3 Calculate the full item total potential exposure in nanograms per item:

$$TPE = \Sigma\, CS_{C\,(gross)} + \Sigma\, CS_{C\,(detail)} \qquad \text{Equation 4}$$

DATA-ACCEPTANCE CRITERIA AND CORRECTIVE ACTIONS

This section contains some guidance for establishing data-acceptance criteria and corrective actions for small-item contact 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 test 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, etc.), contaminant application, decontaminant treatment process, and contact swab selected for use. Because there are many potential test designs, this section contains data-acceptance guidance specific to the test-timing events and contaminant application. 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 the knowledge of the amount of contaminant delivered to determine the difference. The amount of contaminant delivered is confirmed through 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, the mass adsorbed for sorptive nonporous materials (based on Fick's first law) is proportional to the 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 from the decontamination area to the contact test area is facility-dependent and should be reproducible from test to test. For other tasks, the acceptance criterion should be ±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, the longer a contaminated item is allowed to age, the more likely a negative bias may be induced. A contact touch that has a longer duration than specified will most likely induce a positive bias.
- For tests executed at temperatures different from 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 object of the test is to measure the effectiveness of the 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.

Contact Test Touch Duration: Using the recommendations throughout this method, the contact test is a series of 20 swipes over a 3 x 3 in. area for a duration of 20 s. If other touch

durations are used, operators should conduct preliminary studies to determine the target time needed to sample the area size reproducibly. The contact test touch time should be within ±20% of the target.

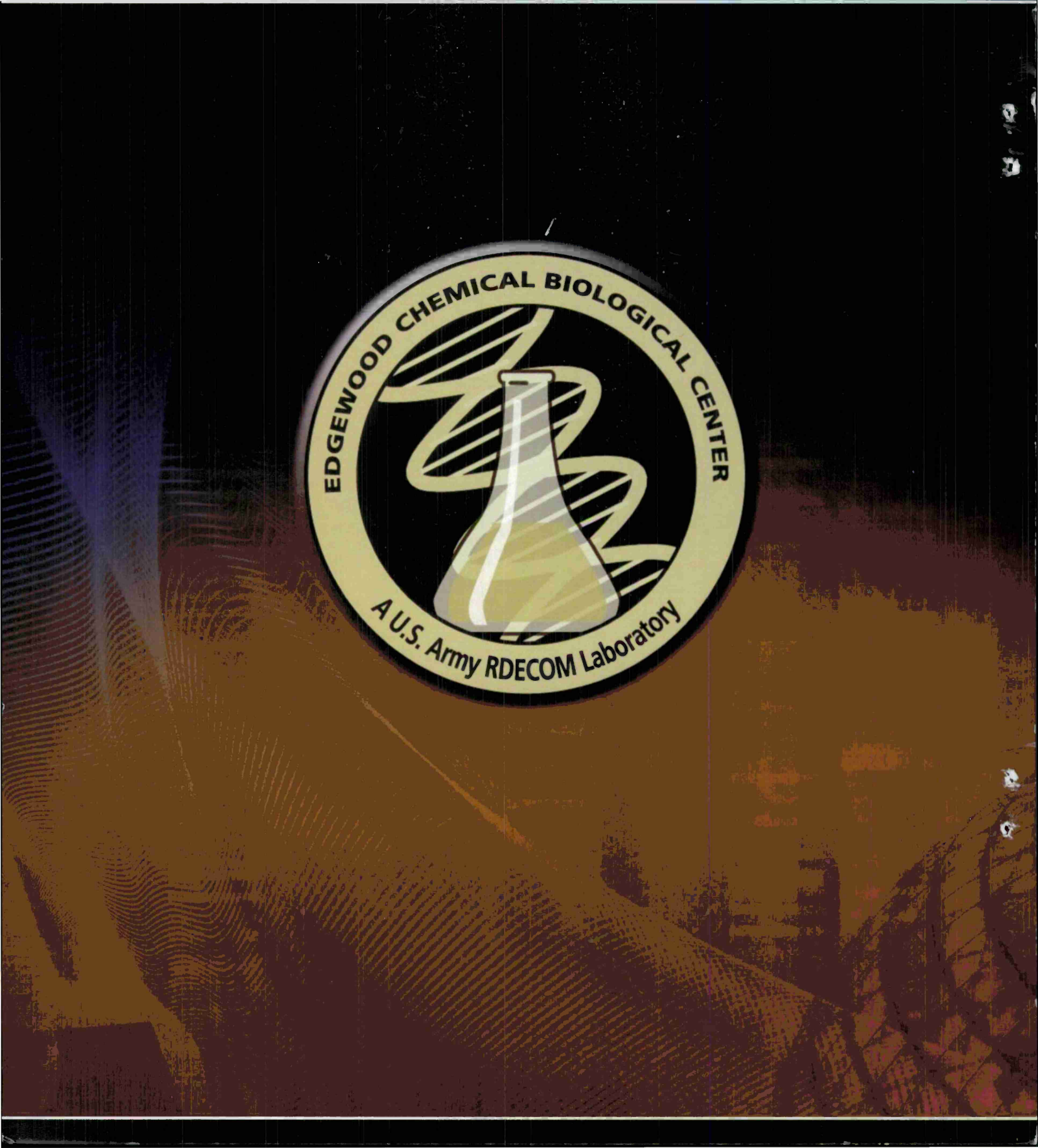
- Rationale: The contact test result will vary with the size of the contaminated surface area and the length of touch time. For nonsorptive surfaces, the majority of mass adsorbed by the contact swab is likely to occur within the first pass-through of a wet swab, and is less affected by time. The contact touch time duration is proportional to the contact test result for sorptive surfaces.

Contact Swab: The contact swab used during method development was a polyurethane foam swab. Because there is no guidance for the measurement of skin uptake on an item, the small-item contact test measures the removal of bioavailable contaminant and places emphasis on repeatability of that removal. The comparison of data using different contact swab material may not produce similar results, which limits the direct comparison of test data.

- Rationale: The contact test is supposed to measure the mass of agent that would be transferred to skin. The mass of agent transferred to the contact swab is a function of the mass transport phenomenon, which is material-dependent (see EPA/600/8-91/011B 1992, *Dermal Exposure Assessment: Principles and Applications*). Materials with different mass transport properties will yield different mass absorption results. Comparison of contact results using different contact swab materials is not advised.

REVISION HISTORY

December 2008:	Original method.
July 2011:	Reissued method.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