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Environmental Technology Verification Program

Building Decontamination Technology Center

Test/QA Plan for Verification of Chlorine Dioxide Gas Technologies for Decontaminating Indoor Surfaces Contaminated with Biological or Chemical Agents

ETV ETV ETV

TEST/QUALITY ASSURANCE PLAN

for

VERIFICATION OF CHLORINE DIOXIDE GAS TECHNOLOGIES FOR DECONTAMINATING INDOOR SURFACES CONTAMINATED WITH BIOLOGICAL OR CHEMICAL AGENTS

Prepared by

Battelle
Columbus, Ohio

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List of Acronyms

AAALAC	American Association for the Accreditation of Laboratory Animal Care
BSCs	biosafety cabinets
BW	biological warfare
CASARM	Chemical Agent Standard Analytical Reference Material
CDC	Centers for Disease Control
CoC	chain-of-custody
CSM	Chemical Surety Material
CV	coefficient of variation
CW	chemical warfare
DEMTMP	diethylmethylthio-methylphosphate
DoD	Department of Defense
EPA	U.S. Environmental Protection Agency
ETV	Environmental Technology Verification
FDA	Food and Drug Administration
FID	flame ionization detectors
FPD	flame photometric detectors
GC	gas chromatograph
GD	soman
HD	sulfur mustard
HMRC	Hazardous Materials Research Center
IS	internal standard
MREF	Medical Research and Evaluation Facility
NHSRC	National Homeland Security Research Center
PA	peak area
PE	performance evaluation
QA	quality assurance
QC	quality control
QMP	Quality Management Plan
RDS	Research Dilute Solutions
RDT&E	research, development, test, and evaluation
SBCCOM	Soldier Biological and Chemical Command
SOP	standard operating procedure
TEP	triethyl phosphate
TGD	thickened GD
TICs	toxic industrial chemicals
TOPO	Task Order Project Officer
TSA	technical systems audit
USDA	U.S. Department of Agriculture
VX	V-agent

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August 2003

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Date 9/17/03

1.0 BACKGROUND

1.1 Technology Verification

Among its responsibilities related to Homeland Security, the U.S. Environmental Protection Agency (EPA) has the goal of identifying methods and equipment that can be used for decontaminating indoor environments, following a terrorist attack on a building using chemical or biological agents. In January 2003, EPA established the National Homeland Security Research Center (NHSRC) to manage, coordinate, and support a wide variety of homeland security research and technical assistance efforts. The Safe Buildings Program, a key research component of the NHSRC, has the aim of verifying the performance of products, methods, and equipment that can decontaminate chemical or biological agents on indoor surfaces or in indoor air.

To accomplish this aim, the EPA has expanded the scope of its Environmental Technology Verification (ETV) program. The ETV process, which has been used since 1997 to verify the performance of over 200 environmental technologies, includes developing a test/quality assurance (QA) plan with input from stakeholders and vendors, applying high-quality test procedures according to that plan, and publicizing separate performance reports for each technology verified. The ETV process does not rank, select, or approve technologies, but instead provides credible performance data to potential users and buyers. Other information about the program is available at the ETV Web site (http://www.epa.gov/etv) and through the NHSRC Web site (www.epa.gov/nhsrc).

In expanding the ETV program to address homeland security needs, the EPA established the ETV Building Decontamination Technology Center, which is managed by Battelle, of Columbus, OH, under contract with EPA. Verification testing of decontamination technologies in the Center generates objective performance data so building and facility managers, first responders, groups responsible for building decontamination, and other technology buyers and users can make informed purchase and application decisions. Verification tests are conducted in the Center in accordance with the ETV process, under the direction of the EPA. All verification

activities are subject to the Quality Management Plan (QMP)[1] and the generic verification protocol[2] for the Center. In performing each verification test, Battelle follows the procedures described in those documents and develops a separate test/QA plan appropriate for the decontamination technology being tested. This document is the test/QA plan for verification testing of decontamination technologies that use chlorine dioxide gas as the decontaminating agent.

1.2 Test Objective

The objective of this test/QA plan is to establish laboratory test procedures to determine the efficacy of chlorine dioxide gas decontamination technologies for removing or inactivating chemical and biological agents and surrogates on a range of representative indoor surfaces.

1.3 Organization and Responsibilities

Verification testing under this test/QA plan will be performed by Battelle under the direction of EPA, with input from expert stakeholders and decontamination technology vendors. The organization chart in Figure 1-1 shows the organizations and individuals who will have responsibilities under this plan. The responsibilities of these organizations and individuals are summarized in the following subsections. Details are provided for the test coordinator, the technology vendor, and the test leaders, who are the most involved in conducting the verification testing.

1.3.1 Battelle

Dr. Michael L. Taylor is the Verification Testing Leader for the ETV Building Decontamination Technology Center. He will have overall responsibility for ensuring that the technical, schedule, and cost goals established for verification testing are met, and that the verification process employed for testing is consistent with Center and ETV program guidelines. For this test, Dr. Taylor will serve as the interface for the Center stakeholder committee.

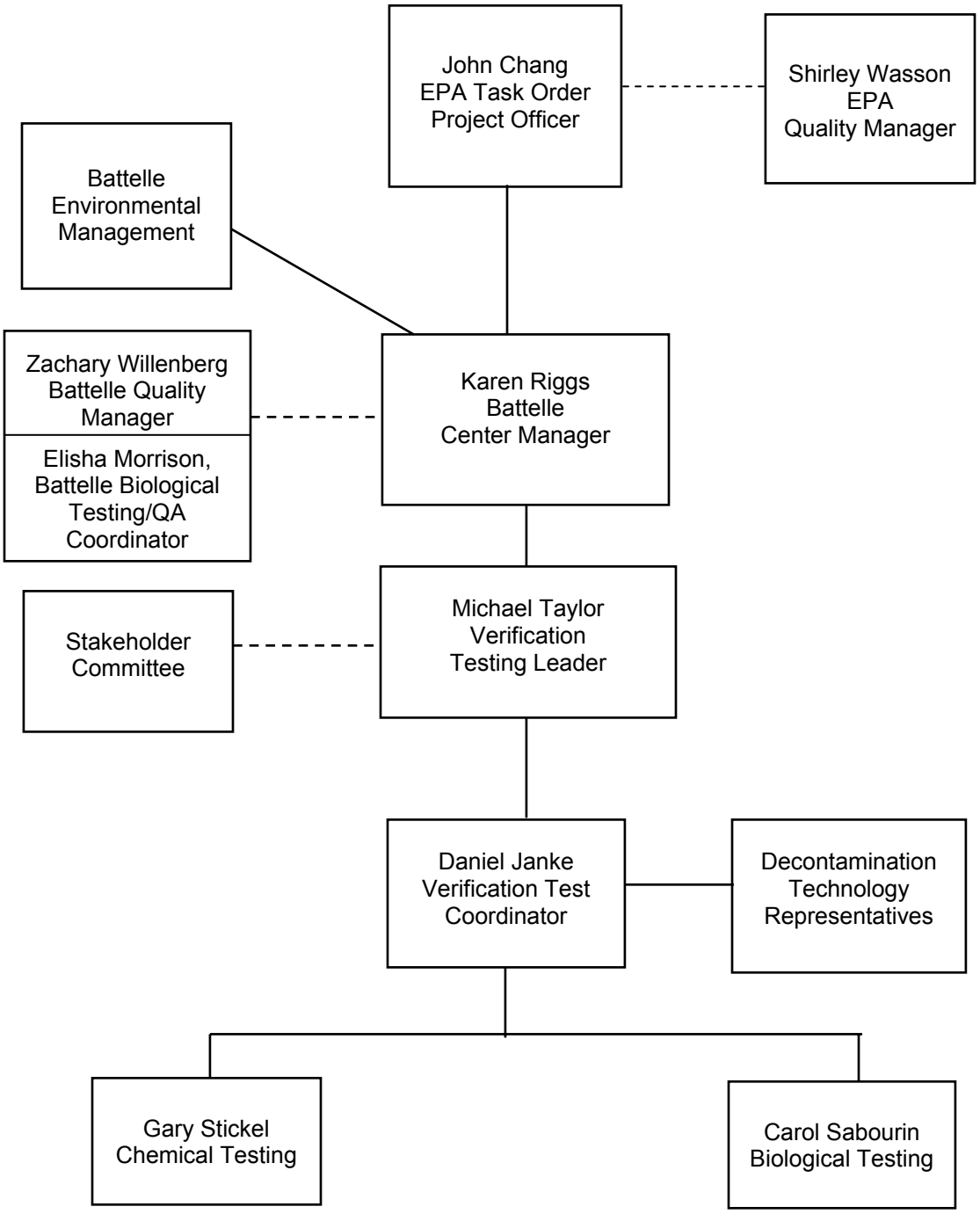

Figure 1-1. Organization Chart for Chlorine Dioxide Gas Decontamination Technology Verification Test

Ms. Karen Riggs is Battelle's Manager for the contract under which the ETV Building Decontamination Technology Center was established. Ms. Riggs will maintain communication with EPA's Task Order Project Officer (TOPO) on all aspects of the program; monitor adherence to budgets and schedules in this work; and ensure that necessary Battelle resources, including staff and facilities, are committed to the verification test.

Mr. Zachary Willenberg is Battelle's Quality Manager for the ETV Building Decontamination Technology Center. He will review the draft test/QA plan, audit at least 10 percent of the verification data, and ensure that all quality procedures specified in this test/QA plan and in the QMP[(1)] are followed. **Ms. Elisha Morrison** will assist Mr. Willenberg and serve as Battelle's Biological Testing/QA Coordinator.

Mr. Daniel Janke is Battelle's Verification Test Coordinator for this test. His responsibilities include

- Selecting the appropriate facility or location for the testing
- Coordinating vendor representatives to facilitate the performance of testing
- Preparing the draft test/QA plan, verification report, and verification statement
- Arranging for use of the test facilities and establishment of test schedules
- Selecting qualified staff to conduct the tests
- Assuring that testing is conducted according to this test/QA plan
- Providing input into revision of the test/QA plan, verification report, and verification statement in response to reviewers' comments
- Updating the Battelle Center Manager and Verification Testing Leader on progress and difficulties in planning and conducting the test
- Coordinating with the Battelle Quality Manager for the performance of technical and performance audits as required by Battelle or EPA Quality Management staff.

The chemical and biological test facilities at Battelle will serve as the location for the testing described in this test/QA plan. These facilities are described in Section 3 of this plan. Biological testing will be led by Dr. Carol Sabourin; chemical testing will be led by Mr. Gary Stickel. In general, the responsibilities of the test leaders will be to

- Assist in planning and scheduling the testing
- Become familiar with the use of the technology to be tested
- Ensure that the facility is fully functional prior to the times/dates needed for verification testing
- Provide requisite technical staff during verification testing
- Provide any safety training needed by Battelle, vendor, or EPA staff
- Review and approve all data and records related to facility operation
- Adhere to the requirements of this test/QA plan and the QMP[1] in carrying out the verification testing
- Provide input on facility procedures for the verification report
- Support Mr. Janke in responding to any issues raised in assessment reports and audits related to facility operation.

1.3.2 Vendor

The decontamination technology vendor will

- Provide input for preparation of the test/QA plan
- Review the draft test/QA plan, and approve the final version
- Sign a vendor agreement specifying the respective responsibilities of the vendor and of Battelle in the verification testing
- Provide the necessary materials and equipment to implement the decontamination technology for testing
- Train Battelle and/or test facility staff in the application of the decontamination technology
- Provide support, if needed, in use of the technology during testing
- Review the draft verification report and verification statement resulting from testing.

1.3.3 EPA

Dr. John Chang is EPA's TOPO for the ETV Building Decontamination Technology Center. As such, Dr. Chang will have overall responsibility for directing the verification process and

Battelle's activities, and will oversee the EPA review process on the draft test/QA plan, verification report, and verification statement.

Ms. Shirley Wasson is EPA's Quality Manager for the ETV Building Decontamination Technology Center. Ms. Wasson will lead EPA's QA oversight on this verification, including, at her option, one external technical systems audit during verification testing.

1.3.4 Stakeholders

Approximately 25 experts from the first responder community, federal and state agencies, military agencies, and academia serve as volunteer advisors to the ETV Building Decontamination Technology Center. Battelle Center staff communicate with these stakeholders regularly by e-mail or telephone and meet periodically with the stakeholder committee and the EPA TOPO. The responsibilities of assigned stakeholders from this committee for testing are to provide input on test procedures for preparation of the test/QA plan, review the draft test/QA plan, and serve as peer reviewers for the verification report.

2.0 APPLICABILITY

2.1 Subject

This test/QA plan is applicable to verification testing of decontamination technologies that generate chlorine dioxide gas to decontaminate indoor surfaces contaminated by chemical or biological agents. This plan is specifically focused on restoring a public building to a usable state after a contamination episode. Decontamination of personnel or equipment is not the subject of this test/QA plan.

The decontamination technologies to be tested under this plan are based on dispersion of chlorine dioxide gas into indoor spaces. Because chlorine dioxide is not stable as a compressed gas, it must be produced on site. Thus, these technologies include the equipment and chemicals for generating and dispersing the chlorine dioxide gas. Chlorine dioxide decontamination technologies may require specific temperatures and humidity levels that enhance the effectiveness of the decontamination process, and may therefore include systems to achieve the optimal temperature and humidity in the space to be decontaminated.

The chemical and biological agents that may pose a threat in the building environment include toxic industrial chemicals (TICs), chemical warfare (CW) agents, and biological warfare (BW) agents (including biotoxins). The chemical and biological agents selected for use in the testing described herein were chosen based on a brief threat summary [(3)] developed from general opinions of Battelle experts, with additional input from Center stakeholders. In the context of decontamination, the contaminants of interest for this plan are those that can persist on indoor surfaces, leading to continuing chance of exposure long after the contamination occurs. Thus, highly volatile TICs and CW agents are not included in testing under this plan because they can be readily removed by ventilation of the building. By the same logic, a highly persistent biological contaminant (anthrax spores) was selected for testing, as opposed to biological agents that cannot survive for long after the contamination event.

The indoor surfaces selected for testing under this plan represent those that must be decontaminated to return a building to use, and do not include those that might simply be removed from the building for disposal. Highly porous, non-structural materials, such as ceiling tiles, cloth-covered furniture and cubicle walls, and draperies, are among those that were deemed likely to be removed from a building for disposal; consequently, those materials are not considered as priority test substrates in this verification plan. Structural materials such as wallboard, painted concrete, metal ductwork, and wood and surfaces of furnishings, such as laminate, are considered essential candidate substrates. Carpeting is also included, as a porous material that could possibly be left in a building for decontamination.

Verification testing requires a basis for establishing the quantitative performance of the tested technology. For the testing conducted under this test/QA plan, quantitative performance is assessed primarily in terms of the efficacy of decontamination. For this assessment, sampling and analysis methods are used to determine the extent of contamination before and after the use of the decontamination technology.

2.2 Scope

The overall objective of the testing called for under this plan is to verify the efficacy of the chlorine dioxide gas decontamination technologies, for removing selected chemical and biological agents from representative indoor surfaces. Testing of each technology is to be conducted at temperatures and relative humidities that would be appropriate for that technology in a building undergoing decontamination.

The performance parameters to be evaluated in verification testing under this test/QA plan include

- Efficacy in destroying chemical agents and surrogates on selected indoor surfaces
- Efficacy in destroying biological agents and surrogates on the same indoor surfaces
- Generation of toxic degradation products from interaction of the decontaminant with the target agents.

Efficacy will be tested by applying chemical and biological agents and surrogates to test surfaces, and comparing the residual contamination after use of the decontamination technology to the contamination originally present. Generation of toxic degradation products will be determined by analysis of the residual contamination for specific degradation products. In addition, any apparent destructiveness of the decontaminant to test surfaces will be assessed by a simple visual inspection before and after use of the decontamination technology.

Under this test/QA plan, verification of chlorine dioxide gas decontamination technologies can include testing with both chemical and biological agents. These components of the complete test are separate, and can be carried out at different times if necessary. Either the chemical or biological agents can be excluded from testing if no efficacy is expected. If these components are conducted separately, they may be the subjects of separate ETV verification reports.

3.0 TEST SITE

Verification testing of chlorine dioxide gas decontamination technologies will be conducted at Battelle's chemical and biological test facilities in West Jefferson, Ohio, near Battelle's headquarters in Columbus, Ohio. The following sections describe the West Jefferson facilities. The testing will be subject to facility-specific methods and standard operating procedures (SOPs) as noted in this test/QA plan, and are required for work at each facility. These documents are cited where appropriate throughout this test/QA plan.

3.1 Site Description

Battelle's chemical and biological test facilities to be used for verification testing are

- The Hazardous Materials Research Center (HMRC), a Department of Defense (DoD) laboratory-scale facility conducting research with CW agents
- Medical Research and Evaluation Facility (MREF), which is a second DoD laboratory-scale facility conducting research with CW and BW agents.

The HMRC is an ISO 9001 certified facility and provides a broad range of materials testing, system and component evaluation, research and development, and analytical chemistry services that require the safe use and storage of highly toxic substances. Since its initial certification by the Chemical Research, Development and Engineering Center in 1981, the facility has functioned as both a research and a technology development laboratory in support of DoD chemical and biological (CB) programs. The HMRC and its personnel have the demonstrated capability for storing and safely handling BZ, tabun (GA), sarin (GB), soman (GD), thickened GD (TGD), sulfur mustard (HD), thickened HD (THD), lewisite (L), mustard-lewisite mixtures (HL), V-agent (VX), and other hazardous materials and toxins, such as arsine (SA), cyanogen chloride (CK), hydrogen cyanide (AC), phosgene (CG), perfluoroisobutylene, as well as agent simulants, Class A poisons, and toxins (e.g., T-2 toxin).

The HMRC complex has approximately 10,000 sq ft of laboratory and support space. It includes the Hazardous Materials Laboratory and the Large Item Test Facility, which provide approximately 2,000 sq ft of laboratory space and 100 linear ft of Chemical Surety Material

(CSM)-approved filtered hoods for working with neat (pure) CW agents; about 630 sq ft of research dilute solution (RDS, diluted chemical agent) laboratory space, including four fume hoods; approximately 2,100 sq ft of laboratory support areas, including wastewater and general laboratory waste disposal, environmental monitoring, emergency power supplies, air filter systems, and general equipment storage room; and about 800 sq ft of staff support areas, including personnel showers, change rooms, laundry facilities, and other common use areas.

The MREF specializes in the research, development, test, and evaluation (RDT&E) of medical countermeasures against highly pathogenic biological and highly toxic chemical materials. This facility is one of a very limited number of U.S. laboratories capable of studying aerosolized etiological agents in animal models under BSL-3 containment. The facility maintains state-of-the-art equipment and professional and technical staffing expertise to safely conduct *in vivo* testing and evaluation of hazardous biological materials under the Food and Drug Administration's (FDA's) GLP Guidelines (21 CFR Part 58).

The MREF facilities are ISO 9001 certified, accredited by the American Association for the Accreditation of Laboratory Animal Care (AAALAC), and inspected and compliant with the U.S. Department of Agriculture (USDA), FDA, Drug Enforcement Agency, Ohio EPA, U.S. Army Safety Team, U.S. Army Inspector General, U.S. Army Medical Research Institute of Chemical Defense Safety and Chemical Operations Branch, U.S. Army Medical Research and Materiel Command Office of Animal Care and Use Review, Madison County Health Department, and Battelle's Institutional Animal Care and Use Committee. The MREF fully complies with all applicable U.S. Army Regulations, and federal government and state of Ohio regulations to conduct and support RDT&E studies using highly toxic chemical and pathogenic biological materials. The MREF is licensed to ship, receive, and handle select agents, as defined by the Centers for Disease Control (CDC).

The MREF BSL-3 facility was completed in 1995, and expanded in 2002 to consist of approximately 31,000 sq. ft. The containment area within the facility is designed to meet or exceed the BSL-3 facility guidelines published by the CDC and National Institutes of Health entitled *Biosafety in Microbiological and Biomedical Laboratories* (4th edition, 1999). The

seven BSL-3 microbiology laboratories contain multiple Class III biosafety cabinets (BSCs), linked together in an H-shaped configuration, and two autoclaves. Additional laboratories within this area include a microbiology laboratory equipped with a Class II BSC connected to a Class III BSC, and a dose configuration room equipped with a Class II BSC.

3.2 Site Operations

Battelle operates its certified chemical and biological test facilities in compliance with all applicable federal, state, and local laws and regulations, including U.S. Army regulations. Battelle's facilities are certified through inspection by personnel from the appropriate government agency. The HMRC has been certified to work with chemical surety material through a Bailment Agreement by the U.S. Army Soldier Biological and Chemical Command (SBCCOM). SBCCOM will terminate its Bailment Agreements on September 1, 2003, and Battelle has begun the process to transition to an AR50-6 surety facility. In this transition, Battelle will demonstrate, via inspections by the appropriate government personnel, that its facilities meet all federal, state, and local laws and regulations, including U.S. Army regulations. Battelle operates the MREF in compliance with requirements contained in 32 CFR 626 and 627, Biological Defense Programs. Our chemical and biological facilities and attendant certifications are listed in Table 3-1.

Test procedures at the HMRC and MREF are governed by established SOPs. Those documents are specified by facility, number, and title. In all cases, the latest version of every such document is used. All relevant documents will be reviewed as part of the Operational Readiness Inspection for verification testing to identify whether any test-specific modifications need to be implemented. The documents that are relevant to testing are indicated where appropriate throughout this test/QA plan.

Table 3-1. Certifications of HMRC and MREF

Facility	Materials	Level	Certification
HMRC	Chemical warfare agents	CSM (Neat) RDT&E (Dilute)	Bailment Agreement No. DAAD13-H-03-0003
Analytical Chemistry Laboratory	Chemical warfare agents	RDT&E (Dilute)	Bailment Agreement No. DAAD13-H-03-0003
MREF	Biological warfare agents	Biosafety Level 3	CDC Select Agents Program (32 CFR 626 and 627) administered through the Biological Defense Research Program
	Chemical warfare agents	CSM (Neat) RDT&E (Dilute)	United States of America Medical Research Materiel Command No. G472501

4.0 EXPERIMENTAL DESIGN

4.1 General Test Design

This test/QA plan specifies procedures for testing chlorine dioxide gas decontamination technologies with chemical and biological agents and surrogates at the laboratory scale using small samples of indoor materials (i.e., coupons). Verification testing will determine efficacy of the technology against agents with representative indoor surface materials. The verification test design will also produce data that will allow correlations to be made between results with actual agents and those with selected surrogates. In all testing, each decontamination technology will be applied in a manner consistent with the manufacturer's recommendations. The technology vendor will provide the equipment for application of their technology and will train Battelle staff in its use. The effect of the decontamination technology on indoor materials will also be assessed by visual inspection of test coupons after they are subjected to decontamination.

The following subsections introduce the primary features of the verification testing approach. Details on the procedures used to conduct testing are presented in Section 6.

4.1.1 Parameters to Be Tested

The following performance parameters of chlorine dioxide gas decontamination technologies will be tested, using coupons of representative indoor materials contaminated with biological and chemical agents in controlled laboratory tests:

- Efficacy in destroying chemical agents and surrogates on selected indoor surfaces
- Efficacy in destroying biological agents and surrogates on the same indoor surfaces
- Generation of toxic degradation products from interaction of the decontaminant with the target chemical agents.

Qualitative biological indicators will also be used in testing to allow correlation of their results with the quantitative efficacy results. Information on surface damage caused by the

decontamination technologies will also be gathered by visual inspection of the test coupons after decontamination.

4.1.2 Scale of Testing

The performance parameters listed above will be evaluated through testing with chemical and biological agents and surrogates at the laboratory scale. These performance tests will use small coupons [approximately ¾ in. x 3 in. (1.9 x 7.5 cm)] of selected indoor materials as test surfaces, and will be carried out in a suitable chemical or biological agent safety hood or cabinet. Multiple coupons of each of several indoor materials will be contaminated with the target agents, and then treated with the decontamination technology. Blank (i.e., uncontaminated) and control (i.e., contaminated but not decontaminated) coupons will also be used for each test material, and will serve as the basis for calculations of decontamination efficacy. This scale of testing will provide a controlled, reproducible approach to assess efficacy with real agents, while also requiring a realistic, though small-scale, application of the decontaminant.

4.1.3 Efficacy

Efficacy (the effectiveness with which the chlorine dioxide gas decontamination technology destroys the agent) will be determined for both chemical and biological agents and surrogates by means of the coupon tests. Efficacy testing will rely on comparing the amount of contaminant on test coupons before decontamination (control coupons) to the amount present after application of the decontamination technology (test coupons). Multiple coupons will be used for both the control and test samples, and the resulting data will be used to calculate efficacy as a percent removal (for chemical agents) or a log reduction (for biological agents).

For building decontamination, the residual amount of agents left after decontamination also needs to be considered, since evaporation of, or physical contact with, any residual could carry a health risk for building occupants. For chemical and biological agents, allowable residual levels have not been determined.[(4)] However, a precedent has been set for the desired end result of decontamination for biological agents: a 6-log kill of biological indicators; and, additionally, no growth is to be detected after decontamination. Though not an official level for building

decontamination, biological efficacy testing under this test/QA plan will follow the 6-log kill objective. For chemical agents, the allowable residual is undefined, but some exposure limits have been set for vapor exposure and for contact hazards.[4, 5] Consequently, the post-decontamination levels of chemical agent will be determined by three methods, including coupon extraction, offgasing, and contact transfer. The extraction method will measure the percent efficacy for destruction of chemical agent on the coupon surfaces. The offgasing method will measure the amount of residual chemical agent vapors offgasing from a test material that could create a vapor hazard. The contact transfer method will measure the amount of residual chemical agent on the coupon surface that could potentially create a contact hazard when transferred to skin or other material during contact.

Efficacy will be evaluated for each chemical and biological agent and surrogate, for each selected indoor surface material. Biological efficacy testing will employ seven coupons of each surface material: three contaminated and subjected to decontamination (test coupons), three contaminated but not subjected to decontamination (control coupons), and one not contaminated (blank coupon). A corresponding set of coupons will be used for chemical efficacy testing; however, chemical testing will also employ additional coupons of each surface material for the vapor offgasing and contact transfer tests.

4.1.4 Temperature and Relative Humidity Conditions

Different chlorine dioxide gas decontamination technologies may require different humidity conditions in the environment to be decontaminated, so that the technology can be most effective. Coupon testing will be carried out at room temperature, and at whatever humidity condition is required and/or maintained by the technology undergoing testing. Temperature and humidity will be monitored during the decontamination process.

4.1.5 Surface Damage

The effect of decontamination on the indoor materials used as test surfaces will be evaluated informally in conjunction with the efficacy testing procedure. After decontamination of the test coupons, the appearance of the decontaminated coupons will be observed, and any obvious

changes in the color, reflectivity, and apparent roughness of the coupon surfaces will be noted. This comparison will be conducted for each of the test materials, before any extraction or sampling of the decontaminated test coupons takes place.

4.2 Agents and Surrogates

The chemical and biological agents to be used in verification testing under this plan were selected based on an evaluation of potential threats to buildings[3] and on subsequent input from stakeholder groups. Note that the threat summary was based on a survey of expert opinions and not on an exhaustive analysis. That evaluation considered the availability of potential contaminants (including chemical and biological agents, biotoxins, and TICs), the lethality of the contaminants, the potential delivery pathways for the contaminants, and the persistence of the contaminants in a building. In addition to chemical and biological agents, surrogates will be used in testing to establish correlations between the decontamination efficacy for surrogates and actual agents.

4.2.1 Chemical Agents and Surrogates

The chemical agents to be used for verification testing, listed in order of priority, are:

Table 4-1. Chemical Agents to Be Used

Agent	Acronym	Type	
V Series	VX	Nerve Agent	> 85%
H Series	HD	Vesicant	> 85%
Thickened Soman	TGD	Nerve Agent	AP

AP: As provided by the U.S Army; see text.

These agents are key representatives of families of similar agents. The agent specified in thickened form (TGD) was chosen because the thickened matrix enhances the persistence of the agent on surfaces. This agent will be obtained in thickened form from the U.S Army, and the Army will provide information on the purity of the thickened agent. However, it will not be possible to confirm the agent's purity by analysis, due to interference from the thickening agent.

For each of the chemical agents listed above, a chemical surrogate will also be used. The selection of chemical surrogates for testing decontaminants is a complex issue. Possible surrogates that have been identified include

- Methyl parathion and malathion for VX
- Methyl phenyl sulfide for HD
- Diisopropyl phosphonofluoridate for GD.

Previous use of these surrogates has been based on the similarity of their physical properties to those of the chemical agents. Alternative choices of surrogates may be used, if evidence is found that the alternative surrogates better mimic the chemical reactivity of the agents with chlorine dioxide gas.

4.2.2 Biological Agents and Surrogates

The primary biological agent used in testing the chlorine dioxide gas decontamination technology will be anthrax spores (*Bacillus anthracis*, Ames strain). To provide correlations with the anthrax results, two biological surrogates will be used:

- *Bacillus stearothermophilus (ATCC 12980)*
- *Bacillus subtilis (ATCC 19659).*

The *B. stearothermophilus* surrogate was chosen because previous tests have indicated that its behavior is similar to anthrax in response to gaseous decontaminants, and it has historically been used as an indicator for chlorine dioxide gas because it is a particularly difficult organism to kill using this technology. The *B. subtilis (ATCC 19659)* surrogate was chosen because it is the same as used in the AOAC Sporicidal Activity Test. Anthrax and the two surrogate organisms will be applied to the test surfaces in the form of spore suspensions.

A commercial spore strip will also be included in testing, of the same spore type [*B. subtilis var niger (B. atrophaeus (ATCC 9372)*], backing (paper), and manufacturer (Raven) as that used during anthrax decontamination in U.S. Postal Service facilities. Furthermore, biological indicators (Apex Labs) containing the surrogates *B. stearothermophilus* and *B. subtilis* will also be included. These biological indicators will contain a spore population of 10^6. The Raven

spore strips and Apex biological indicators will be used for qualitative indication of efficacy to allow correlation with quantitative efficacy results.

4.3 Test Surfaces

The surface materials to be used for testing chlorine dioxide gas decontamination technologies are a subset of the variety of structural, decorative, and functional surfaces that may be found indoors. Excluded from the list of test surfaces are indoor materials that are likely to be removed from a contaminated building for disposal, rather than decontaminated in place. Such materials include draperies, ceiling tiles, and fabric furnishings. However, the surface materials to be used include both smooth and porous surfaces, and a variety of material compositions. The test surfaces that will be used are listed below, with the unique code that will be used for sample identification shown in parentheses:

- Painted (latex, semi-gloss) concrete (cinder block) (PC)
- Painted (latex, flat) wallboard (PW)
- Decorative laminate (DL)
- Galvanized ductwork (GM)
- Glass (GS)
- Bare wood (pine lumber) (BWD)
- Industrial grade carpet (IC).

The test coupons of each surface material will be 1.9 cm x 7.5 cm, with thickness varying from 1/32” to 3/8” as appropriate for the materials. Certain combinations of contaminant and test surface have been avoided in making this selection. For example, hydrolysis of VX has been shown to occur rapidly (half-life = 3 hours) on bare concrete surfaces.[6] Consequently, bare concrete was avoided for testing decontamination efficacy with VX because the substrate efficacy would confound the determination of the decontaminant efficacy.

4.4 Test Sequence

Table 4-2 provides the sequence of testing to be carried out on each technology, listing the names of the test procedures, the performance or operational parameters to be evaluated in each procedure, and a summary of the samples or data comparisons resulting from each procedure. The order of testing will be as shown in Table 4-2, i.e., biological efficacy testing with coupons, followed by corresponding chemical efficacy testing, in each case followed by assessment of surface damage.

Table 4-2. Sequence of Test Procedures in Verification Testing of Chlorine Dioxide Gas Decontamination Technologies

Test Procedure	Parameters Evaluated	Data Produced
Biological efficacy test	Efficacy	Multiple samples, plus controls and blank, for each test surface, for each biological agent and surrogate. Also, multiple spore strip samples, plus controls.
Damage to surfaces	Damage to test coupons	Visual observation of every test coupon in all biological efficacy tests.
Chemical efficacy test	Efficacy	Multiple samples, plus controls and blank, for each test surface, for each chemical agent and surrogate.
Damage to surfaces	Damage to test coupons	Visual observation of every test coupon in all chemical efficacy tests.
Test for known toxic by-products	Analysis of coupon extractions after chemical efficacy tests	Multiple samples, plus blank, for each test coupon/agent combination
Vapor offgas test for chemical agents[a]	Effectiveness at reducing vapor offgasing	Multiple samples, plus controls and blank, for each test surface, for each chemical agent and surrogate.
Contact transfer test for chemical agents[a]	Effectiveness at reducing contact transfer	Multiple samples, plus controls and blank, for each test surface, for each chemical agent and surrogate.

[a] These tests will use separate coupons from those used for other test procedures.

5.0 EQUIPMENT AND MATERIALS

This section provides a description of the key materials and equipment needed to perform verification testing of chlorine dioxide gas technology.

5.1 Materials

5.1.1 Agents

Chemical agent use at the HMRC will be under the terms and conditions of Bailment Agreement DAAD13-H-03-0003. This Bailment Agreement is a contract between Battelle and the U.S. Army that specifies the safety, security, and personnel reliability standards required for storing, handling, and using chemical agents. Battelle's stock of agent will be analyzed prior to testing to verify the purity of the agent used to contaminate the test coupons. An aliquot of diluted agent will be injected into a gas chromatograph (GC) fitted with a flame-ionization detector to determine the purity of the agent. The purity of the agent will be determined through comparison with the analytical standards generated from or based on Chemical Agent Standard Analytical Reference Material (CASARM). Only chemical agents with purity greater than 85 percent will be used in this program. The purity of thickened GD will not be measured due to interference caused by the thickener, but information on the agent purity will be provided by the U.S. Army.

Biological agent use at the MREF will be according to the CDC Select Agents Program (42 CFR Part 73) and the Biological Defense Research Program (32 CFR 626 and 627) in adherence with the Battelle MREF Facility Safety Plan. Anthrax (Ames) spores will be prepared according to *MREF. X-074* (*Production of* Bacillus anthracis *Spores*) or *MREF. X-093* (*Production of* Bacillus anthracis *Spores in a Small Fermentor*). The spores will be characterized according to *MREF. X-075-00* (*Characterization and Qualification of* Bacillus anthracis *Spores*), which requires less than 5 percent debris content for acceptance of spores.

5.1.2 Spore Strips

The Raven commercial spore strips and Apex Labs biological indicators will be purchased for verification testing, in quantities larger than needed for testing.

5.1.3 Surfaces to Be Tested

Section 4.3 lists the materials to be used to simulate indoor surfaces in testing. The representativeness and uniformity of the test materials are important to assure reliable test results. Representativeness means that the materials used are typical of such materials used indoors in buildings. Uniformity means that all test pieces are essentially equivalent for the purposes of testing. Representativeness will be assured by obtaining test materials from appropriate suppliers and by recording the appropriate specifications, manufacturer identification, lot numbers, etc., for each material. Uniformity will be maintained by obtaining a large enough quantity of material that multiple test samples of uniform characteristics can be obtained (e.g., test coupons will all be cut from the interior rather than the edge of a large piece of material). In addition, the uniformity of recovery of biological and chemical agents will be assessed for each test material in method demonstration tests conducted before the start of verification testing (see Section 6.1). The reproducibility of recovery rates will be determined for each material as a measure of the uniformity of the test pieces.

5.2 Delivery and Application Equipment

5.2.1 Agent/Surrogate Surface Application

The equipment needed to apply controlled and reproducible amounts of agents and surrogates to the test surfaces will include the solutions or suspensions to be delivered and the delivery device (a syringe, pipette, or comparable system). These items of equipment are described in Section 6.

5.2.2 Temperature/Humidity Conditions

Commercial decontamination technologies based on chlorine dioxide gas typically include a conditioning system that controls the temperature and/or humidity of the environment to the

optimum conditions for the decontamination. In all verification testing, each technology will be operated according to the vendor's instructions, including the performance of any such conditioning system. The temperature and humidity of the test enclosure will be monitored throughout testing, using vendor provided sensors.

5.3 Test Chamber

A decontaminant exposure chamber will be used to expose the test coupons to the decontaminant. For biological agent testing, a Compact Glove Box Model 830-ABC (Plas Labs, Inc., Lansing, MI; Figure 5-1) will be used. This unit has inner dimensions of 28"w x 23"d x 29"h (71 cm x 59 cm x 74 cm) and outer dimensions of 43"w x 24"d x 31"h (110 cm x 61 cm x 79 cm). The unit also has a top opening of 17" x 23" (43 cm x 58 cm) and a transfer chamber that is 12" (30 cm) long and an inner diameter of 11" (28 cm). The chamber has a total volume of 11.2 cu ft (317 L). A set of glove ports, located on the side, are available for working in the hood. The same type of glove box, but without the transfer chamber, will be used in the chemical agent testing. In both cases, the decontaminant will be directed from the vendor's delivery system through the exposure chamber, at the temperature and humidity conditions established by the delivery system.

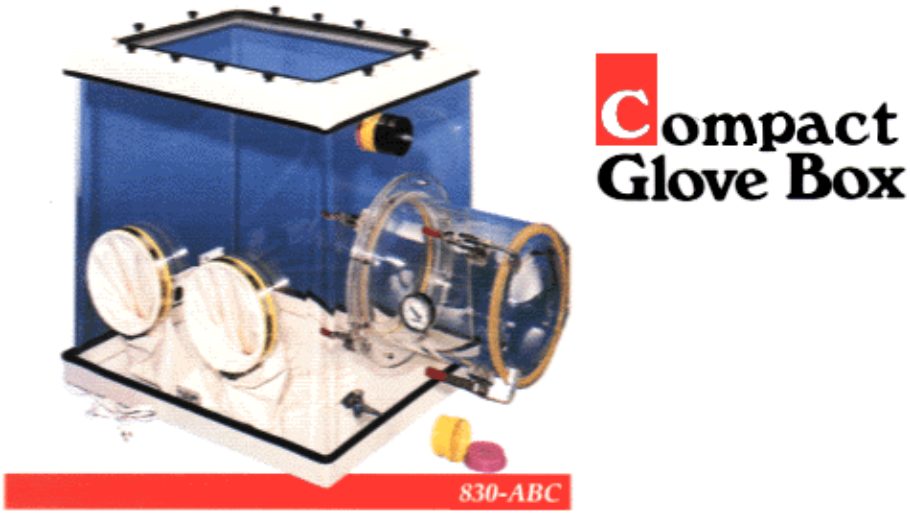

Figure 5-1. Compact Glove Box—BW Agent Tests

5.4 Sampling and Analysis Materials and Equipment

5.4.1 Chemical Agent Testing

5.4.1.1 Contact Transfer Equipment. The contact transfer test equipment will include 2-in diameter pieces of latex dental dam for contact transfer measurements, and 1-in diameter weights (65 g/cm^2) for placing on the latex. Figure 5-2 shows the contact transfer test with the weights applied to the test coupons. The latex will be washed with water and dried at 185°F for 24 hours prior to cutting.

Figure 5-2. Contact Transfer Weights

5.4.1.2 Offgas Sampling Equipment. Offgas sampling will be performed at ambient temperature and relative humidity conditions. The offgas rack (Figure 5-3) will hold up to 25 test cells. Aluminum offgas cells (Figure 5-4) will be used to hold the individual coupons during offgasing, and may include critical orifices to control the flowrate at 0.25 L/minute during offgas collection. Butyl o-rings will be used to seal the cells. Charcoal tubes will be placed on the cell inlet to ensure that clean air is entering the cell. The agent vapors will be collected using solvent-filled impingers or sorbent tubes, depending on the agent and amount of agent offgasing expected from each material type. The agents will be collected in bubblers filled with diethyl phthalate or ethylene glycol diacetate, or with sorbent tubes filled with either Carboxen or Tenax.

Figure 5-3. Offgas Rack

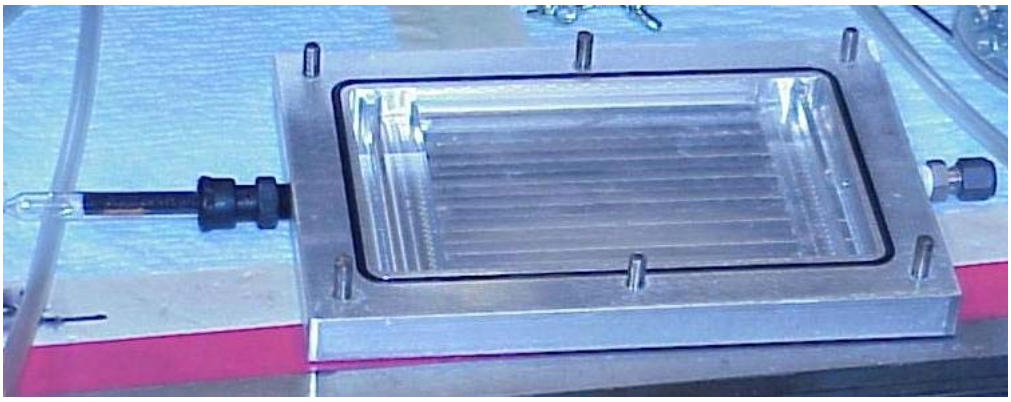

Figure 5-4. Offgas Cell

The Carboxen tubes will be solvent extracted for analysis. The Tenax sorbent tubes will be thermally desorbed to analyze for the chemical agents.

5.4.1.3 Analytical Equipment. Chemical agent analyses will be performed using Hewlett Packard 5890 or 6890 GCs equipped with flame photometric detectors (FPD) or flame ionization detectors (FID). Internal standard (IS) will be added to the chloroform to produce a concentration of 1.7 µg/mL using triethyl phosphate (TEP) and diethylmethylthio-methylphosphate (DEMTMP). The TEP is used as the IS for GD analysis, and DEMTMP is used as the IS for HD and VX analysis. The internal standard for the surrogate analysis will be either TEP or DEMTMP, based on the relative retention times of the surrogates and the IS.

5.4.2 Biological Agent Testing

5.4.2.1 Sampling Media. The procedures and media used to extract the biological agent or surrogates from the test surfaces are described in Section 6.2.5.2.

5.4.2.2 Sample Analysis. Section 6.2.5.2 describes the culturing and enumerating procedures for biological samples.

5.5 Performance Evaluation Audit Materials

The performance evaluation (PE) audit (Section 7.2) will use independent standards to check the analysis methods for chemical agents and chemical surrogates. These independent standards will be RDS, prepared in Battelle's Columbus facilities, and analyzed with the GC equipment used for sample analysis at the West Jefferson facilities. At least one such RDS solution will be prepared for each of the chemical agents and surrogates identified in Section 4.2.1.

No PE audit will be done for biological agents, due to the lack of suitable audit standards. The application confirmation procedure (Section 6.2.3.2), controls, blanks, and method validation procedures will be used to document the biological test results.

6.0 TEST PROCEDURES

This section provides a discussion of procedures for method validation, and for chemical and biological coupon testing of chlorine dioxide gas decontamination technologies.

6.1 Method Demonstration

Many of the test coupon materials to be used are likely to be new to decontamination testing. Consequently, method trials will be performed as necessary to demonstrate the methods included in this test/QA plan.

6.1.1 Chemical Agent Method Demonstration

Method demonstration will be performed as necessary to determine the optimum methods for extracting chemical agents and surrogates from the various test coupons, and for quenching the chlorine dioxide gas reactions to analyze for chemical agent, surrogates, and toxic degradation products after decontamination. Demonstration trials will be conducted with each chemical agent/surrogate/coupon combination, using 5 percent bleach as the positive control, and distilled water as the negative control. Extraction efficiencies for various solvents (e.g., chloroform, hexane) from these combinations will be determined. Furthermore, prior to testing, one coupon of each material type and the samplers used as the contact transfer material (see Section 6.2.5.1.3) will be extracted in solvent to ensure that no analytical interferences would inhibit agent analysis. Up to three solvents will be tested.

The objective of the quenching demonstration study is to establish a quenching method that will stop the reaction between the chlorine dioxide gas and the chemical agent. Typically samples are quenched with an organic compound containing sulfur that is also soluble in the extraction solvent to stop oxidation of the agent. Sodium sulfite has often been used for this purpose, as it reacts very rapidly with chlorine dioxide gas. In addition, dilution or extraction may also be effective at quenching the reaction. Up to four methods will be tested. Based on the results of method trials, an appropriate extraction solvent will be selected. The solvent selection process

will consider extraction efficiencies, analytical interferences, material compatibility, and other observations made during the trials.

In addition, trials of the offgasing method will be performed to determine the appropriate vapor collection system design based on the amount of agent vapor leaving the various coupons. Trials will be performed on each type of test coupon using each type of agent. Results will be used to determine how many coupons should be in the offgasing cell, and what is most the most appropriate system for collecting vapors (e.g., solvent-filled impingers, sorbent-filled tubes, or both, which solvent, which sorbent, etc.).

6.1.2 Biological Agent Method Demonstration

Method demonstration trials will be performed as necessary to determine the optimum methods for extraction of biological agents from the test coupons and for quenching the decontamination reaction. The objective of the quenching demonstration study is to determine a quenching method that will stop the reaction between the decontaminant and the biological agent so that decontamination does not continue after sampling. For example, a solution of sodium metabisulfite can be used to quench the reactivity of some decontaminants that act by oxidation. Once a method has been established, the method demonstration trials will determine the average spore recovery efficiency from each type of surface material and the reproducibility of that efficiency. The reproducibility will be determined as a percent coefficient of variation (%CV) of repeated trials with each surface material. The %CV values will indicate the uniformity of the test coupons for each material. The average recovery values will determine what log kill can be determined based on an initial spore loading of 10^8.

6.2 Coupon-Scale Testing

Decontamination efficacy testing with coupons will be conducted based on procedures described in *TOP 8-2-061* (*Decontamination Systems Laboratory/Field Testing*). The testing will evaluate decontamination efficacy for chemical and biological agents by extracting and measuring the initial and residual agent on test coupons. Chemical analysis or biological enumeration of the

resulting extracts will allow efficacy to be calculated as the percent removal for chemical agents, or the log reduction for biological agents.

For chemical agents, as discussed in Section 4, measurements will also be made of vapor offgasing and contact transfer to help evaluate efficacy from the perspective of residual chemical agent allowed to remain in a building after decontamination. The offgasing tests will measure the amount of chemical agent vapors evaporating from the coupon, potentially creating a vapor hazard. Contact transfer tests will measure the amount of chemical agent transferred to a simulated skin material touching the coupon surface, simulating a contact hazard when transferred to skin or other material. Detailed descriptions of these tests are presented in Section 6.2.5.1.

6.2.1 Preparation of Test Materials

For testing chemical agent decontamination, no special preparation of test surfaces is required. To ensure normal cleanliness and prevent contamination of test surfaces, care will be exercised and the test coupons will be packaged in individual sample bags. At most, surface preparation will involve washing with a solvent or water. The test coupons will be cut to 1.9 cm x 7.5 cm size from the interior of a large piece of test material. Edges and damaged areas will be avoided in cutting test coupons. The test coupons will be visually inspected upon receipt and any evidence of damage will be recorded. The length, width, and thickness of the test coupons will be measured and recorded.

Chain-of-Custody (CoC) forms will be used to ensure that the test coupons are traceable throughout all phases of testing. Each coupon will be assigned a unique identifier code that matches it with the sample, test parameters, and sampling scheme. The testing staff receiving the test coupon will be responsible for comparing the identifier code with the test matrix.

6.2.2 Application of Agents to Test Coupons

6.2.2.1 Application of Chemical Agents and Surrogates to Test Coupons

To assess decontamination efficacy, the conditions specified in *TOP 8-2-061* will be used, i.e., a contamination density of 10 g/m^2 and a droplet size of 1-µL. For the three chemical agents, the number of agent drops to be administered per coupon will be determined prior to testing based on the contamination density, agent density, and the measured agent purity.

The test coupons will be removed from their individual packages and allowed to equilibrate to the laboratory temperature and relative humidity for a minimum of one hour prior to agent application. A 1-inch diameter circle will be drawn on the test coupons with a non-interfering grease pencil to provide a known area for agent application.

The test coupons will be laid flat in the chemical agent fume hood. The chemical agents will be applied to the test coupons using microliter-sized drops to achieve the target contamination density (10 g/m^2). A Hamilton gastight syringe with a Hamilton repeatable stepper will be used to produce the drops. Separate syringes will be used for each chemical agent to prevent cross-contamination. After agent application, the coupons will be covered with a Petri dish to minimize agent evaporation. Coupons will be allowed to weather overnight (i.e., approximately 16 to 18 hours) after application of chemical agent. SOP HMRC-II-001 (*General Provisions for Handling Chemical Agent in the Hazardous Materials Research Center*) will be used for agent operations. The same procedures used for application of the chemical agents will also be used for application of the surrogates.

6.2.2.2 Application of Biological Agents and Surrogates to Test Coupons

Testing will be performed in a Compact Glove Box (Plas Labs, Inc.) (see Section 5.3). Test coupons will be laid flat in the cabinet and contaminated at challenge levels of 10^8 spores per coupon. Stock suspensions of the agent at the required concentration will be prepared, transferred to the coupon using a micropipette, and spread over the sample surface (e.g., by smearing the suspension over the coupon with the tip of the pipette or placing the suspension over the surface as small droplets similar to the chemical agent approach). After contamination

with biological agent or surrogate suspension, the test coupons will be allowed to dry undisturbed to completion.

6.2.3 Confirmation of Surface Applications

6.2.3.1 Confirmation of Surface Application Density of Chemical Agents

Each chemical agent will be applied to three Teflon control coupons at the desired density using the procedure described in Section 6.2.2.1. These coupons will be extracted using the same procedure used for the decontaminated coupons (see Section 6.2.5.1.1) immediately after agent application. They will be analyzed by the same procedure used for decontaminated coupons (see Section 6.2.5.1.4), to verify the initial application density.

6.2.3.2 Confirmation of Surface Application Density of Biological Agents

To confirm the application density of biological agents and surrogates, the anthrax and surrogate spore suspensions used to contaminate the coupons will be reenumerated on each day of use. This enumeration will be carried out as described in Section 6.2.5.2.

6.2.4 Application of Chlorine Dioxide Gas Decontamination Technology

After application of agents and surrogates to the test coupons and completion of the drying or weathering time, the test coupons will be decontaminated. Each decontamination technology undergoing testing will be used in accordance with the vendor's instructions, to supply the test enclosure with the required levels of chlorine dioxide gas for decontamination. The duration and chlorine dioxide gas level used for decontamination will be as recommended by the vendor.

If feasible (and if resources are available), monitoring of the concentration of chlorine dioxide gas may be conducted during the technology verification testing in accordance with vendor's instructions.

6.2.5 Determination of Decontamination Efficacy

The primary test of decontamination efficacy will determine the fraction of agent destroyed by the chlorine dioxide gas treatment, through extraction of residual agent from the coupons after decontamination. In addition, analysis of end point conditions will be made (i.e., by performing vapor offgasing and contact transfer testing for chemical agents and verifying no growth for biological agents). The vapor offgasing and contact transfer tests will provide alternative measures of efficacy for the chemical agents and surrogates.

6.2.5.1 Decontamination Efficacy for Chemical Agent on Coupons

6.2.5.1.1 Extraction of Residual Chemical Agent from Coupons

After application of the decontaminant, extraction of the residual chemical agent will be performed. Decontaminated test coupons and the control coupons will be placed directly into jars containing the extraction solvent. After a 1-hour extraction, an aliquot of the solvent will be transferred to a GC vial for analysis. Depending on the outcome of the method validation effort, a phase separation may be performed to minimize analytical interferences by separating coupon debris from the extraction solution. The sample will be analyzed for chemical agent using a GC with an appropriate detector as discussed in Section 6.2.5.1.4.

For chemical agents, decontamination efficacy will be calculated based on the amount of agent applied to the test coupon and the amount of residual agent measured after decontamination, as described in Section 8.2.2. Decontamination efficacy results will be presented as percent agent neutralized/removed. The upper limit for calculated efficacy values is based on the detection limit of the GC and the amount of solvent used for extraction; typically these limitations do not come into play unless efficacy exceeds 99.9 percent.

6.2.5.1.2 Offgasing Measurements

The offgasing test will be performed using different coupons than are used for extracting the residual agent for the primary determination of efficacy (Section 6.2.5.1.1). Larger coupons may be required for the offgasing test to produce sufficient agent vapor for analysis.

The vapor offgasing test will be performed on coupons that have been contaminated with chemical agent and subsequently decontaminated with the chlorine dioxide gas, as well as on contaminated coupons that have not been decontaminated (i.e., control coupons). Each coupon will be sealed in an aluminum offgas cell. A charcoal filter will be placed on the cell inlet to provide clean airflow into the cell. A sorbent tube or impinger will be attached to the cell exhaust. Critical orifices or mass-flow controllers will be used to control the flow through the sorbent tubes or impingers at 0.25 liters per minute. The offgas will be sampled over specific time intervals of 0 to 2, 2 to 4, and 4 to 12 hours. The sorbent tubes will be extracted with 3 mL of solvent and analyzed for chemical agent by GC. The impinger solutions will analyzed directly by GC, or extracted prior to analysis by GC. *SOP HMRC-X-049* (*Offgas Testing of Materials*) will be followed for this test. The efficacy of reducing the vapor offgasing will be calculated by comparing the offgasing rates for the decontaminated coupons to those from the control coupons, as described in Section 8.2.4.

6.2.5.1.3 Contact Transfer

The contact transfer test will be performed using different coupons than used for the vapor offgasing test (Section 6.2.5.1.2) or for extracting the residual agent for the primary determination of efficacy (Section 6.2.5.1.1). The contact transfer test will be performed after the vapor offgasing test for both the decontaminated and the control coupons.

The amount of agent transferred by contact will be measured using a piece of latex dental dam (dental dam is made from natural rubber latex and other ingredients and is used as a barrier during endodontic and other restorative procedures). A 2-in diameter piece of latex will be placed on the test coupon as a sampler. A 2-in piece of aluminum foil will be placed on top of the latex, and a 2-inch weight (65 g/cm^2) will be applied to simulate the force of a hand touching the surface. After 15 minutes of contact, the weight will be removed, and the latex sample will be placed in a jar containing 20 mL of solvent. After a 60-minute extraction, an aliquot of the solvent will be transferred to a GC vial for GC-FPD analysis. If the agent concentration is below the GC-FPD detection limit, a 10-mL aliquot of the solvent extract will be evaporated in a concentration of 1 mL and reanalyzed. If the agent concentration is still below the GC-FPD

detection limit, it will be reported as a non-detect. *Contact Transfer and Offgas Testing Following Chemical Agent Contamination and Decontamination (SOP HMRC-X-070)* will be followed for this test. The efficacy of reducing the contact transfer of agent will be calculated by comparing the offgasing rates for the decontaminated coupons to those from the control coupons, as described in Section 8.2.3.

6.2.5.1.4 Sample Analysis

A quantitative analysis of chemical agent in coupon extracts, latex extracts, and vapor offgas samples will be conducted using GC/FID, FPD, or mass spectrometry detectors. Analysis will be performed and standards for reference analysis will be prepared in solvent using neat agent in accordance with *HMRC Standard Operating Procedures IV-056-06* (*Standard Operating Procedure for Operation and Maintenance of Gas Chromatographs and for the Analysis of Solutions Containing GA, GB, GD, GF, HD, VX by Gas Chromatography.* Analytical standards will be generated from or based on the CASARM standard (see Section 5.1).

A detector is selected based on the chemical agent being analyzed, the expected concentration range, any interferences identified during the method validation process, and the time required for analysis. The FPD will be used for the chemical agents and surrogates because it has the highest sensitivity for measurement and can also be used to analyze more samples per day. The FID will be used if the agent concentration in the samples is high.

Extracts from the test coupons will be analyzed for specific degradation products. A possible degradation product of VX, EA 2192, will be determined by liquid chromatography/mass spectrometry. *SOP HMRC-III-001* (*General Provisions for RDTE Dilute Solutions Utilized in JN-4*) will be used for handling laboratory samples.

The analytical results for each extract will be fitted to the calibration curve for the specific GC used to analyze the extract. The agent concentrations for each extract will be determined by Equation 1:

$$C = \frac{A}{M} - w \tag{1}$$

where

C = agent concentration (μg/mL)
M = slope of the calibration line
A = the peak area (PA) for the agent, normalized to the internal standard PA
w = Y intercept of the calibration line.

As given in Equation 1, the agent concentration for each sample is determined from the ratio of the IS concentration to that of the agent. Analytical results in excess of the daily method detection limit for the instrument will be recorded in μg/mL. The agent (density) on the coupons will be determined by Equation 2.

$$R = \frac{C\,E_v}{S_a} \tag{2}$$

where

R = residual agent density (μg/cm^2)
C = GC concentration (μg/mL) from Equation 1
E_v = extract volume (mL)
S_a = contaminated surface area (cm^2).

6.2.5.2 Decontamination Efficacy for Biological Agent on Coupons

6.2.5.2.1 Extraction of Spores from Test Coupons

The testing will quantify decontamination efficacy by measuring the anthrax or surrogate spores on surface material coupons, both those exposed (test coupons) and unexposed (control coupons) to the decontaminant. Following the decontamination process, each surface material coupon will be placed in a 50-mL test tube containing 10 mL of sterile phosphate-buffered solution with 0.1 percent Triton X-100 and catalase. The purpose of the Triton X-100 is to minimize clumping of spores, and the purpose of the catalase is to neutralize any residual chlorine dioxide. For spore extraction, the tubes will be agitated on an orbital shaker for 15 min at room temperature. Samples will then be heat shocked at 60 ºC for 1 hr to kill any vegetative bacteria. Following heat shock, 1.0 mL of each extract will be removed, and a series of dilutions through 10^{-7} will be prepared in sterile water.

An additional qualitative assessment of chlorine dioxide gas efficacy will be conducted following spore extraction. After the extraction process described above, each coupon will be transferred to a clean 50-mL tube containing 20 mL of liquid nutrient broth. The vials will be sealed and incubated overnight at 37ºC on an orbital shaker. The next day, the tubes will be assessed qualitatively for viability as "growth" or "no growth."

6.2.5.2.2 Enumeration of Spore Samples

The number of viable spores present on the surface materials will be determined using the coupon extracts produced by the procedure in Section 6.2.5.2.1. Spore viability will be determined by dilution plating, using both the undiluted extracts, and the successive dilutions of each extract, to assure that accurate spore counts are achieved. One hundred microliters of the undiluted extract and of each serial dilution will be plated onto Trypticase Soy Agar plates in triplicate, allowed to dry, and incubated overnight at 35 to 37 ºC for *B. anthracis* and *B. subtilis* and at 55 to 60 ºC for *B. stearothermophilus*. Plates will be enumerated the next day, and the colony-forming units/mL will be determined by multiplying the average number of colonies per plate by the reciprocal of the dilution, as described in *MREF SOP X-054* (*Enumeration of BL-2 and BL-3 Bacterial Samples Via the Spread Plate Technique*). Data will be expressed as mean $\pm$ standard deviation of the numbers of colony-forming units observed. To calculate the efficacy of the decontamination treatment, the number of spores remaining on the decontaminated test coupons will be compared to the number of spores on the control coupons. Efficacy for biological agents will be calculated in terms of a log reduction, as described in Section 8.2.2.

6.2.5.2.3 Qualitative Indicators

The spore strips and biological indicators will be exposed to the decontamination treatment along with the surface material coupons, but will be used to determine only qualitative (i.e., growth/no growth) efficacy, as described in Section 6.2.5.2.1 for the material coupons. Following the decontamination process, the spore strips and biological indicators will be placed in liquid nutrient broth, and the presence of any viable spores will be determined. No enumeration of the spores will be attempted.

6.2.6 Observation of Surface Damage

Following application of the decontamination technology, each test surface will be examined visually to establish whether use of the decontamination approach caused any obvious damage to the surface. Surface damage will be observed immediately after completing the decontamination process but before post-decontamination sampling to assess efficacy. If wetted by the decontamination process, the test surface will be allowed to dry before any inspection for damage. The surface will then be inspected visually through side-by-side comparison of the decontaminated test surface and the control coupons of the same test material. Differences in color, reflectivity, contrast, and roughness will be assessed in this way. These observations will be made by the testing staff and recorded.

7.0 QUALITY ASSURANCE/QUALITY CONTROL

7.1 Equipment Calibrations

The methods to be used to determine chemical and biological agents and surrogates are described in Section 6. The analytical equipment needed for these methods will be maintained and operated according to the quality requirements and documentation of the test facility. All equipment will be calibrated with appropriate standards on a pre-set schedule, and calibration results will be clearly and consistently recorded.

Hewlett Packard GCs will be used for analysis of the extract, offgas, contact transfer, and residual rinse samples. For GC analysis, five calibration standards will be analyzed at the beginning of each sample analysis. The GC will be recalibrated if the correlation coefficient (R^2) from the regression analysis of these standards is less than 0.99. In addition, the percent bias for the low standard must be less than 25 percent, and the percent bias for the remaining standards must be less than 15 percent. One or two calibration check standards will be run for every five samples. The criteria for evaluation of the GC performance is listed below:

- R^2 should be greater than 0.99
- The bias for the lowest standard should be less than 25 percent
- The bias for the remaining standards should be less than 15 percent
- For duplicate samples, the difference between should be less than 20 percent
- The areas of the internal standards should be within 40 percent of the average of the standards
- The difference between the shortest retention time and the longest retention time for the target agent should be less than 0.5 minutes.

The calibration range and associated example detection limits for each agent are listed in Table 7-1. In the event that agent concentrations are above the highest calibration level for the GC-FPD analysis, the samples will be analyzed using a GC equipped with an FID, or diluted and reanalyzed by GC-FPD. The GC-FID will be calibrated in a range of 10 to 250 µg/mL.

Table 7-1. Calibration Ranges

	Calibration Range	Extraction ($\mu g/cm^2$)	Offgas ($\mu g/cm^2$)	
TGD	0.1 to 10 µg/mL	0.5 to 50	0.03 to 3	0.1 to 10
HD	0.5 to 10 µg/mL	2.5 to 50	0.15 to 3	0.5 to 10
VX	0.25 to 10 µg/mL	1.25 to 50	N/A	0.25 to 10

* Actual detection limits depend on contamination area and extract volume.

The critical orifices and mass-flow controllers used for flow control in the offgas test will be calibrated using a Buck Calilogger. The flowrate of 0.25 L/minute will be used for the offgas testing.

7.2 Assessment and Audits

7.2.1 Technical Systems Audits

Battelle's Quality Manager will perform a technical systems audit (TSA) once during the performance of this verification test. The purpose of a TSA is to ensure that verification testing is being performed in accordance with the test/QA plan and that all QA/quality control (QC) procedures are being implemented. In this audit, the Quality Manager may review the sampling and analysis methods used, compare actual test procedures to those specified in this test/QA plan, and review data acquisition and handling procedures. The Quality Manager will prepare a TSA report, the findings of which must be addressed either by modifications of test procedures or by documentation in the test records and verification report.

At EPA's discretion, EPA QA staff may also conduct an independent on-site TSA during verification testing. The EPA TSA findings will be communicated to testing staff at the time of the audit and documented in a TSA report.

7.2.2 Performance Evaluation Audit

A PE audit will be conducted to assess the quality of the chemical agent and surrogate analyses made during verification testing. This audit addresses only those measurements that factor directly into the data used for verification, i.e., the decontamination technology is not the subject

of the PE audit. Similarly, auxiliary measurement systems used to establish test conditions (e.g., temperature, relative humidity, and flow measurement devices) are subject to their own usual calibration requirements, but are not subject to the PE audit.

The PE audit of chemical measurements will be made by independently preparing RDSs of the agents and surrogates, in the same solvent and with the same nominal concentrations as the calibration solutions used for the GC analysis. Successive analysis of these independent solutions will then be conducted as a check on the calibration solutions. An acceptable tolerance of ±25 percent will apply to this comparison. Failure to meet this criterion will require repreparing the independent test solutions; a subsequent failure will trigger an investigation of the calibration process and flagging of test data for the agent or surrogate. This audit will be the responsibility of Battelle and will be carried out once during verification testing. Battelle's Quality Manager will assess PE audit results.

No PE audit will be done for biological agents and surrogates because quantitative standards for these materials do not exist. The confirmation procedure, controls, blanks, and method validation efforts will be the basis of support for biological test results.

7.2.3 Data Quality Audit

Battelle's Quality Manager will audit at least 10 percent of the verification data acquired during verification testing. The Quality Manager will trace the data from initial acquisition, through reduction and statistical comparisons and to final reporting. All calculations performed on the data undergoing audit will be checked.

7.2.4 Assessment Reports

Each assessment and audit will be documented in accordance with the QMP for the ETV Building Decontamination Technology Center.[1] Assessment reports will include the following:

- Identification of any adverse findings or potential problems
- Space for response to adverse findings or potential problems
- Possible recommendations for resolving problems

- Citation of any noteworthy practices that may be of use to others
- Confirmation that solutions have been implemented and are effective.

7.2.5 Corrective Action

The Quality Manager, during the course of any assessment or audit, will identify to the technical staff performing experimental activities any immediate corrective action that should be taken. If serious quality problems exist, the Quality Manager is authorized to stop work. Once the assessment report has been prepared, the Verification Test Coordinator will ensure that a response is provided for each adverse finding or potential problem and will implement any necessary follow-up corrective action. The Quality Manager will ensure that follow-up corrective action has been taken.

8.0 DATA ANALYSIS AND REPORTING

8.1 Data Acquisition

Data acquisition during verification testing includes proper recording of the procedures used in testing to assure consistency in testing and adherence to this test/QA plan, documentation of sampling conditions and analytical results for the reference methods, determination of damage to surfaces from the decontamination process, and recording of efficacy results and test conditions. Data acquisition will be carried out by the Battelle testing staff, in the form of test notebooks, analytical data records, and data recording forms. Appendix A shows examples of Test Performance Control Sheets and a Test Coupon Sample Form that will be used during testing.

Laboratory analytical data (e.g., method results quantifying the chemical or biological contaminants used) may be produced electronically. Other test data will be recorded manually in laboratory notebooks or on data forms prepared prior to the test. These records will be reviewed to identify and resolve any inconsistencies. All written records must be in ink. Any corrections to notebook entries, or changes in recorded data, must be made with a single line through the original entry. The correction is then to be entered, initialed, and dated by the person making the correction. A brief explanation of the basis for the correction will also be recorded.

Strict confidentiality of test data will be maintained. At no time will Battelle staff engage in any comparison of the technology undergoing testing with any other decontamination technologies.

Table 8-1 summarizes the types of data to be recorded; how, how often, and by whom the recording is made; and the disposition or subsequent processing of the data. The general approach is to record all test information immediately and in a consistent format throughout all tests. This process of data recording and compiling will be overseen by the Battelle Verification Test Coordinator and Quality Manager.

Table 8-1. Summary of Data Recording Process for Verification Testing

Data to Be Recorded	Where Recorded	How Often Recorded	Disposition of Data
Dates, times of test events	Laboratory record books, data forms	Start/end of test, and at each change of a test parameter	Used to organize/ check test results; manually incorporated in data spreadsheets as necessary
Test parameters (agent/surrogate identities and concentrations, test surfaces, temperature and relative humidity, gas flows, etc.)	Laboratory record books, data forms	When set or changed, or as needed to document the sequence of test.	Used to organize/ check test results, manually incorporated in data spreadsheets as necessary
Sampling data (identification of sampling media, sampling flows, etc.)	Laboratory record books, data forms	At least at start/end of reference sample, and at each change of a test parameter	Used to organize/ check test results; manually incorporated in data spreadsheets as necessary
Chemical analysis or biological enumeration analysis, chain of custody, and results	Laboratory record books, data sheets, or data acquisition system, as appropriate.	Throughout sample handling and analysis process	Transferred to spreadsheets
Records and observations on decon use	Laboratory record books	Throughout implementation of decon technology; during discussions with decon vendor	Reviewed and summarized to support data interpretation

8.2 Calculation Procedures

8.2.1 Data Screening

ANOVA models will be fitted to the residual extraction, contact transfer, and offgas results for each agent (TGD, HD, and VX). Factors to be included in the models will be material, agent, chlorine dioxide gas concentration, and time, as appropriate. Data will be checked for normality and equal variance between groups and appropriate transformations (log) taken if necessary. Outliers with normalized residuals greater than three standard deviations will be considered for removal from the data.

8.2.2 Efficacy

The primary assessment of efficacy will rely upon comparing the concentration of the target agent or surrogate on the test coupons, before and after the application of the decontamination technology. For chemical agents and surrogates, efficacy (E) in percent will be calculated as

$$E = (C_o - C_f)/C_o \bullet 100\% \qquad (3)$$

where C_o is the concentration of agent or surrogate before decontamination (determined from the control coupons of each surface material) and C_f is the concentration on the test coupons after decontamination.

For biological agents and surrogates, decontamination efficacy will be calculated as the log reduction in viable organisms achieved by the decontamination technology. That is, efficacy (E) for biological agents or surrogates will be calculated as

$$E = \log (N°/N) \qquad (4)$$

where N° is the number of viable organisms present on the control coupons (i.e., those not subjected to decontamination), and N is the number of viable organisms present on the test coupons after decontamination.

A separate efficacy calculation will be made for each of the surface materials, with each chemical agent/biological agent/surrogate. In addition, since each surface material will be represented by multiple sample coupons of that material in the efficacy tests, each combination of a material and an agent/surrogate will result in multiple values of percent efficacy or log reduction. For each material and agent/surrogate combination, a mean and range of the efficacy values will be reported. Thus, the primary efficacy results from the coupon testing will be a matrix table in which each entry shows the mean and range of efficacy results for one of the agents/surrogates on one of the surface materials.

8.2.3 Contact Transfer

The contact transfer of chemical agent is calculated based on the amount of agent transferred to the sampler and the surface area sampled. Contact transfer (CT) is calculated according to Equation 5:

$$CT = \frac{M}{A} \tag{5}$$

where M is the mass of agent (in mg) collected on the latex contact surface of area A (in cm^2). The units of CT thus are mg/cm^2.

The effectiveness with which the decontamination technology reduces the chemical contact transfer will be calculated in a manner analogous to Equation 3, i.e.:

$$E_{CT} = (CT_o - CT_f)/CT_o \bullet 100 \tag{6}$$

where E_{CT} is the percent efficacy for reducing contact transfer, and CT_o and CT_f are the contact transfer rates determined from the control and test coupons, respectively.

The residual contact hazard is estimated based on the contact transfer, the surface area contacted, and the estimated hazard level for the percutaneous exposure to chemical agents. Criteria for contact hazard estimation are defined in the NBC Contamination Survivability Criteria for Military Equipment.[5] This document defines negligible risk percutaneous contact transfer values for chemical agents, with negligible risk defined as mild incapacitation for 5 percent of the military personnel. These values are listed below. Much lower levels would have to be established for the general public since these numbers reflect battleground risks.

- GD: 30 mg/70-kg man
- VX: 1.4 mg/70-kg man
- HD: 180 mg/70-kg man.

8.2.4 Offgas Flux

The offgas flux is calculated based on the amount of agent transferred to the sampler and the surface area sampled. Offgas flux (OF) is calculated according to Equation 7:

$$OF = \frac{M}{A \bullet T} \tag{7}$$

where M is the mass of agent (in mg) collected on the sorbent tube or in the impinger over the sampling interval T (in minutes), due to offgasing from the contaminated surface area A (in cm^2). Thus the units of OF are $mg/cm^2/min$.

The effectiveness with which the decontamination technology reduces the chemical vapor offgasing will be calculated in a manner analogous to Equation 3, i.e.:

$$E_{OF} = (OF_o - OF_f)/OF_o \bullet 100 \tag{8}$$

where E_{OF} is the percent efficacy for reducing the vapor offgas flux, and OF_o and OF_f are the contact transfer rates determined from the control and test coupons, respectively.

The agent vapor hazard is estimated based on the offgas flux and assumed exposure time, room ventilation, material surface area, and the hazard level for the vapor exposure to chemical agents. Possible criteria for vapor exposure are listed below:[4]

- GD: 0.000001 mg/m^3
- HD: 0.003 mg/m^3
- VX: 0.000003 mg/m^3.

8.3 Data Review

Records generated during verification testing will receive a one-over-one review before these records are used to calculate, evaluate, or report verification results. These records may include laboratory record books, completed data sheets, or reference method analytical results. This review will be performed by a Battelle technical staff member other than the person who originally generated the record. Testing staff will be consulted as needed to clarify any issues

about the data records. The review will be documented by the person performing the review by adding his/her initials and date to a hard copy of the record being reviewed. This hard copy will then be returned to the Battelle staff member who generated or who will be storing the record.

8.4 Reporting

The efficacy calculations described in Section 8.2, the assessment of material damage, and other observations during verification testing will be compiled in a verification report. The verification report will present all the test data, supporting information on the measurement methods, as well as the quantitative evaluation of the test results. The verification report will briefly describe the ETV Building Decontamination Technology Center and will describe the procedures used in verification testing. The results of verification testing will then be stated quantitatively, without comparison to any other technology, or any comment on the acceptability of the technology's performance. The preparation of the draft verification report, the review of the report by vendors and others, the revision of the report, the final approval, and the distribution of the report will be conducted as stated in the QMP[(1)] for this Center. Preparation, approval, and use of the verification statement summarizing the results of the testing will also be subject to the requirements of that same QMP. For a technology undergoing testing with both biological and chemical contaminants, separate verification reports will be prepared on those two tests.

9.0 HEALTH AND SAFETY

All participants in verification testing (i.e., Battelle, EPA, and vendor staff) will adhere to the security, health, and safety requirements of HMRC and MREF. Vendor staff will train test personnel in the use of their decontamination technology, but will not be the technology users during the testing. For reasons of safety and controlled access at the West Jefferson facilities, vendor staff may be able to observe some test procedures, but will not conduct any of the testing activities.

9.1 Access

Access to restricted areas of the West Jefferson facilities will be limited to staff who have met all the necessary training and security requirements. The existing access restrictions of the facilities will be followed, i.e., no departure from standard procedures will be requested for verification testing.

9.2 Potential Hazards

Verification testing conducted under this plan will involve the use of extremely hazardous chemical and biological materials. Use of those materials must only be implemented in properly certified surety facilities, capable of handling such materials safely.

In addition, surrogate materials used in such verification testing may also be toxic and must be used with appropriate attention to good laboratory safety practices.

9.3 Training

Because of the hazardous materials that will be involved in testing conducted under this plan, documentation of proper training and certification of the test personnel is mandatory before any testing takes place. The Battelle Quality Manager or counterpart at the West Jefferson facilities will assure that such training is documented for all test personnel before allowing testing to proceed.

9.4 Safe Work Practices

All visiting staff at the test facilities will be given a site-specific safety briefing prior to the start of any test activities conducted under this plan. This briefing will include a description of emergency procedures. Testing procedures must follow all specified safety practices at all times. Any report of unsafe practices, by those involved in testing or by other observers, shall be grounds for stopping testing until the appropriate facility safety officer and testing personnel are satisfied that unsafe practices have been corrected.

10.0 REFERENCES

1. Quality Management Plan (QMP) for the Technology Verification of Commercially Available Methods for Decontamination of Indoor Surfaces Contaminated with Biological or Chemical Agents, Version 1, prepared by Battelle, Columbus, Ohio, November 22, 2002.

2. Generic Verification Protocol for Technology Verification of Commercially Available Methods for Decontaminating Indoor Surfaces Contaminated with Biological or Chemical Agents, Version 1, prepared by Battelle, Columbus, Ohio, March 11, 2003.

3. Priority Threat Summary, prepared for EPA's Safe Buildings Program by Battelle, Columbus, Ohio (draft), March 7, 2003.

4. Raber, E., Jin, A., Noonan, K., McGuire, R., and Kirvel, R.D. Decontamination Issues for Chemical and Biological Warfare Agents: How Clean is Clean Enough? *Int. J. Environ. Health Res.,* 11, 128-148 (2001).

5. NBC Contamination Survivability Criteria for Military Equipment, Quadripartite Standardization Agreement 747, Edition 1. 12 August, 1991.

6. Groenewold, G.S., Williams, J.M., Appellans, A.D., Gresham, G.L., Olson, J.E., Jeffrey, M.T., and Rowland, B. Hydrolysis of VX on Concrete: Rate of Degradation by Direct Surface Interrogation Using an Ion Trap Secondary Ion Mass Spectrometer, *Environ. Sci. Technol.*, 36, 4790-4794 (2002).

APPENDIX A

TEST PERFORMANCE CONTROL SHEETS

TEST COUPON SAMPLE FORM

TEST PERFORMANCE CONTROL SHEETS

CONTACT/EXTRACT TEST

DATE		OPERATOR	
TRIAL #		ASSISTANCE	
AGENT		RECORDER	

SPIKE CONTROLS

MATERIAL	AGENT DROPS	EXTRACT VOLUME	SAMPLE ID
GLASS			
GLASS			
GLASS			

POSITIVE CONTROLS (NOT DECONTAMINATED)

MATERIAL TYPE	REP	AGENT APPLIED		COUPON EXTRACTED		ID
		DROPS	TIME	TIME	VOLUME	
A	1					
A	2					
A	3					
B	1					
B	2					
B	3					
C	1					
C	2					
C	3					
D	1					
D	2					
D	3					

TEST SAMPLES

MATERIAL TYPE	REP	AGENT APPLIED		CHLORINE DIOXIDE GAS GENERATION		CONTACT TRANSFER		EXTRACT	
		DROPS	TIME	START	END	TIME	VOLUME	TIME	VOLUME
A	1								
A	2								
A	3								
B	1								
B	2								
B	3								
C	1								
C	2								
C	3								
D	1								
D	2								
D	3								
A		NONE							
B		NONE							
C		NONE							
D		NONE							

TEST PERFORMANCE CONTROL SHEETS

OFFGAS TEST

DATE		OPERATOR	
TRIAL #		ASSISTANCE	
AGENT		RECORDER	

SPIKE CONTROLS

MATERIAL	AGENT DROPS	EXTRACT VOLUME	SAMPLE ID
GLASS			
GLASS			
GLASS			

TEST SAMPLES

MATERIAL TYPE	REP	AGENT APPLIED		CHLORINE DIOXIDE GAS GENERATION		OFFGAS SAMPLE			
		DROPS	TIME	START	END	INTERVAL (TBD)	INTERVAL (TBD)	INTERVAL (TBD)	VOLUME
A	1								
A	2								
A	3								
B	1								
B	2								
B	3								
C	1								
C	2								
C	3								
D	1								
D	2								
D	3								
A		NONE							
B		NONE							
C		NONE							
D		NONE							

TEST COUPON SAMPLE FORM

Study No. G604302
Method No. 80/Microbiology
QC Review By/Date: _____
Tech Review By/Date: _____

Bacillus _____
Spore Lot No. _____
Spore Source: _____

STERIS Parameters:
Air Flow Rate = _____ SCFM
Injection Rate = _____ g/min
Exposure Time = _____ min
Enclosure Concentration = _____ mg/L
Percent Saturation = _____ %

Sample ID	Test Coupon Code	Test Coupon Description	Tier No.	Time Spores Added	Heat-Shock Extract from…	Comments/Observations (e.g., Color, Reflectivity, Apparent Roughness)	Initials & Date