

# **The Performance of Selected Radiological Decontamination Processes on Urban Substrates**

## TEST/QA PLAN



**Battelle**

*The Business of Innovation*

---

National Homeland Security  
Research Center

## **Technology Testing and Evaluation Program**

Test/QA Plan for  
The Performance of Selected  
Radiological Decontamination  
Processes on Urban Substrates

A1


**TITLE AND APPROVAL PAGE**


**Test/QA Plan**

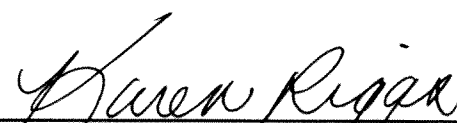
**for**

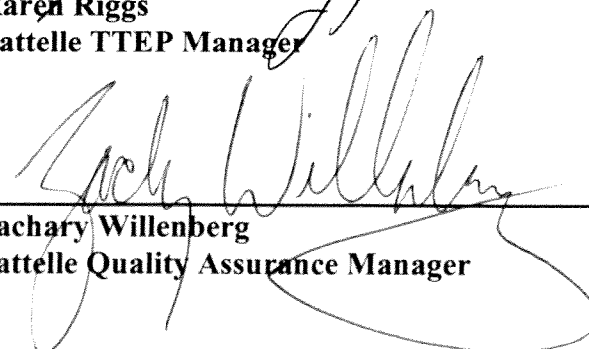
**EVALUATION OF THE PERFORMANCE OF SELECTED RADIOLOGICAL  
DECONTAMINATION PROCESSES ON URBAN SUBSTRATES**

**Version 1.0  
September 10, 2007**

  
\_\_\_\_\_  
**John Drake** **9/18/07**  
**U.S. EPA Task Order Project Officer** **Date**

  
\_\_\_\_\_  
**Eletha Brady-Roberts** **9/18/07**  
**NHSRC Quality Assurance Manager** **Date**

  
\_\_\_\_\_  
**Karen Riggs** **9/10/07**  
**Battelle TTEP Manager** **Date**

  
\_\_\_\_\_  
**Zachary Willenberg** **9-10-07**  
**Battelle Quality Assurance Manager** **Date**

**Battelle  
505 King Avenue  
Columbus, OH 43201-2693**

This page intentionally blank.

**A2 TABLE OF CONTENTS**

	<b>Page</b>
A PROJECT MANAGEMENT .....	2
A1 Title and Approval Page .....	2
A2 Table of Contents .....	4
A3 Distribution List .....	5
A4 Technology Evaluation Organization .....	7
A5 Background .....	12
A6 Technology Evaluation Description and Schedule .....	12
A7 Quality Objectives .....	13
A8 Special Training/Certification .....	14
A9 Documentation and Records .....	14
 B MEASUREMENT AND DATA ACQUISITION .....	 16
B1 Experimental Plan and Test Samples .....	16
B2 Reference Sample Collection .....	24
B3 Sample Handling and Identification .....	24
B4 Analytical Methods .....	25
B5 Quality Control Requirements .....	25
B6 Instrument/Equipment Testing, Inspection, and Maintenance .....	26
B7 Instrument Calibration and Frequency .....	26
B8 Inspection/Acceptance of Supplies and Consumables .....	27
B9 Non-Direct Measurements .....	27
B10 Data Management .....	27
 C ASSESSMENT AND OVERSIGHT .....	 29
C1 Assessments and Response Actions .....	29
C2 Reports to Management .....	30
 D DATA VALIDATION AND USABILITY .....	 31
D1 Data Review, Validation, and Evaluation Requirements .....	31
D2 Validation Methods .....	31
D3 Reconciliation with User Requirements .....	31
 E REFERENCES .....	 33

**Figures and Table**

Figure 1	Organization Chart for Radiological Decontamination Evaluation .....	8
Figure 2	Example Cement Coupon with Identification Marking .....	17
Figure 3	Diagram of the Aerosol Delivery System .....	19
Figure 4	Gamma Counting Equipment At The INL RML .....	20
Figure 5	Schematic Diagram of Front And Side View of Test Stand Face .....	21
Figure 6	Radiological Control Hood .....	23
Figure 7	Glovebox .....	23
 Table 1	 Replicate Concrete Test Coupons for Each Decontamination Technology .....	 22

### **A3     DISTRIBUTION LIST**

Mr. John Drake  
Ms. Eletha Brady-Roberts  
U.S. EPA National Homeland Security  
Research Center  
26 West Martin Luther King Drive  
Cincinnati, Ohio 45268

Dr. Ryan James  
Ms. Karen Riggs  
Mr. Zachary Willenberg  
Battelle  
505 King Avenue  
Columbus, Ohio 43201-2693

Mr. Eric Koglin  
U.S. EPA National Homeland Security  
Research Center  
944 East Harmon Avenue  
Las Vegas, NV 89119

Dr. Robert Fox  
Idaho National Laboratory  
P.O. Box 1625 MS 2208  
Idaho Falls, ID 83415

**List of Abbreviations**

ANSI	American National Standards Institute
ASTM	American Society for Testing and Materials
BG	Background
cm	Centimeters
DARPA	Defense Advanced Research Projects Agency
DF	Decontamination factor
DOD	Department of Defense
EPA	U.S. Environmental Protection Agency
IEEE	Institute of Electrical and Electronics Engineers
INL	Idaho National Laboratory
LRB	Laboratory record book
NHSRC	National Homeland Security Research Center
ORD	Office of Research and Development
QA	Quality assurance
QC	Quality control
QMP	Quality management plan
%R	Percent removal
ppm	Parts per million
RDD	Radiological dispersion device
RH	Relative humidity
RML	Radiological Measurement Laboratory
RTL	Radiochemistry technical lead
TOL	Task Order Leader
TOPO	Task Order Project Officer



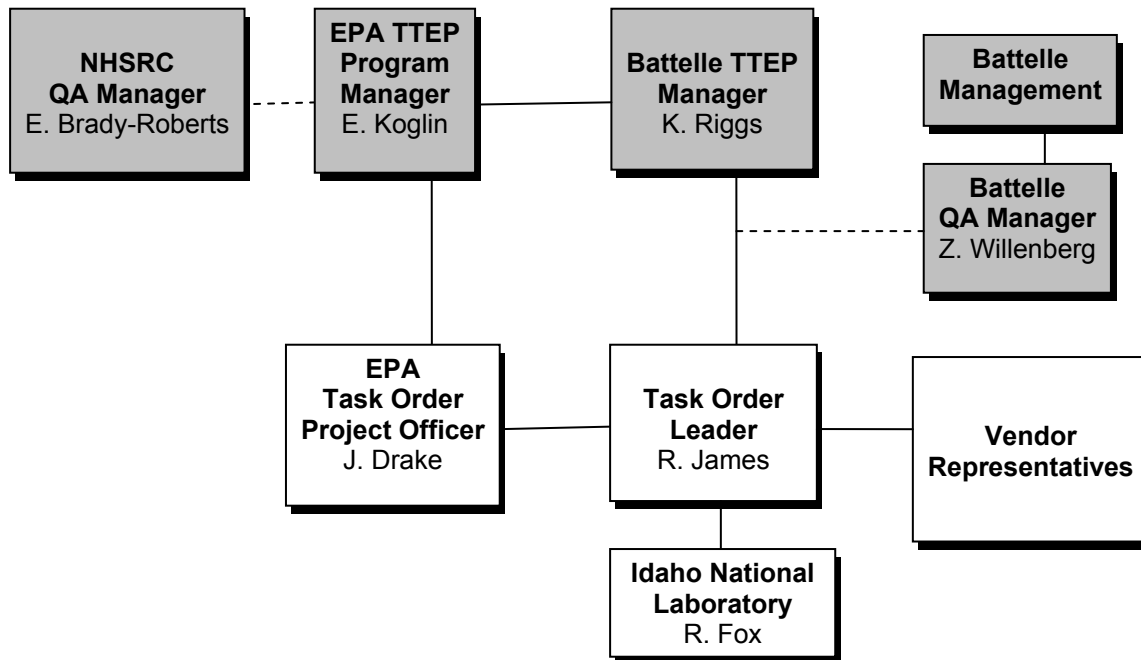
## **A4 TECHNOLOGY EVALUATION ORGANIZATION**

The technology evaluation will be performed by Battelle with assistance from Idaho National Laboratory (INL) under the direction of U.S. Environmental Protection Agency's (EPA) National Homeland Security Research Center (NHSRC) through the Technology Testing and Evaluation Program (TTEP), with input from the vendors whose decontamination technologies will be evaluated. The organization chart in Figure 1 shows the individuals from Battelle, INL, the vendor companies, and EPA who will have responsibilities in the technology evaluation. The specific responsibilities of these individuals are detailed below.

### **A4.1 Battelle**

Dr. Ryan James is Battelle's Task Order Leader (TOL) for this technology evaluation. In that role, Dr. James will oversee the evaluation of the radiological decontamination technologies. Dr. James's responsibilities are to:

- Maintain communication with EPA's Task Order Project Officer (TOPO) on all aspects of the program
- Select the appropriate laboratory for the evaluation
- Coordinate with vendor representatives to facilitate receipt and operation of the radiological decontamination technologies as well as the review of the draft test/QA plan and report
- Prepare and revise the draft test/QA plan
- Review and revise the draft evaluation reports
- Establish a budget and schedule for the technology evaluation and direct the effort to ensure that budget and schedule are met
- Assure that the evaluation is conducted according to this test/QA plan
- Keep the Battelle TTEP Manager informed of progress and/or difficulties in planning and conducting the evaluation
- Coordinate with the Battelle Quality Assurance (QA) Manager for the performance of technical and performance audits as required by Battelle or EPA Quality Management staff
- Respond to any issues raised in QA assessment reports and audits, including instituting corrective action as necessary
- Coordinate distribution of final test/QA plan and evaluation reports.



**Figure 1. Organization Chart for Radiological Decontamination Evaluation**

Ms. Karen Riggs is Battelle's TTEP Manager. As such, Ms. Riggs will:

- Monitor adherence to budgets and schedules in this work
- Maintain regular communication with the EPA TTEP Program Manager on program level issues and provide oversight of Dr. James and the rest of the evaluation staff
- Provide the TOPO with monthly technical and financial progress reports
- Review the draft test/QA plan and approve the final version
- Review the draft evaluation reports
- Ensure that necessary Battelle resources, including staff and facilities, are committed to the technology evaluation
- Ensure that vendor data is not shared with other participating vendors prior to publication of results
- Issue stop work order if health and safety of workers or quality of work is compromised
- Support Dr. James in responding to any issues raised in QA assessment reports and audits.

Mr. Zachary Willenberg is Battelle's QA Manager for TTEP. As such, Mr. Willenberg will:

- Review the draft test/QA plan and approve the final version
- Conduct himself, or appoint a designee, to perform a technical systems audit (TSA) at least once during the technology evaluation
- Audit at least 10% of the evaluation data

- Prepare and distribute an assessment report for each audit
- Verify implementation of any necessary corrective actions
- Notify Battelle's TTEP Manager to issue a stop work order if internal audits indicate that data quality is being compromised
- Provide a summary of the QA/quality control (QC) activities and results for the evaluation reports
- Review the draft evaluation reports
- Ensure that all quality procedures specified in this test/QA plan and in the QMP<sup>1</sup> are followed.

Dr. Robert Fox is the Idaho National Laboratory (INL) radiochemistry technical lead (RTL) who will coordinate the experimental work that is conducted at INL during this evaluation. In that role, he will:

- Assist in the development of the test/QA plan for the evaluation
- Determine the schedule for the experimental work
- Direct the INL staff in carrying out the evaluation procedures specified in this test/QA plan
- Provide the Battelle TOL a weekly status report via phone and a written monthly report describing the progress of the experimental work
- Ensure that all quality assurance measures dealing specifically with measuring radionuclides are followed
- Assist in drafting the evaluation reports.

Evaluation staff will include INL staff that will perform the experimental work in the laboratory. In that role, they will:

- Review the final version of the test/QA plan
- Perform the evaluation of the radiological decontamination technologies as described in the test/QA plan and as directed by Dr. Fox.

#### **A4.2 Vendors**

Vendors that volunteer radiological decontamination technologies for evaluation will:

- Review the draft test/QA plan and final version prior to the evaluation of their radiological decontamination technologies
- Sign a vendor agreement specifying the respective responsibilities of the vendor and of Battelle in the evaluation
- Provide radiological decontamination technologies for use during the evaluation
- Review the draft evaluation report specific to their technology.

#### **A4.3 EPA**

Mr. John Drake is the EPA's TOPO for this program. As such, Mr. Drake will:

- Have overall responsibility for directing the evaluation process under the oversight of the TTEP Program Manager
- Review the draft test/QA plan and approve the final version
- Coordinate involvement of the NHSRC QA Manager
- Review the draft evaluation reports
- Oversee the EPA review process on the draft test/QA plan and reports
- Notify the Battelle TTEP Manager to issue a stop work order if an external audit indicates that data quality is being compromised
- Coordinate the submission of evaluation reports for final EPA approval.

Ms. Eletha Brady-Roberts, the NHSRC QA Manager for this program will:

- Review the draft test/QA plan and approve the final version
- Perform, at her option, one external TSA during the technology evaluation
- Notify the EPA TOPO to issue a stop work order if an external audit indicates that data quality is being compromised
- Prepare and distribute an assessment report summarizing the results of the external audit, if one is performed
- Review the draft evaluation reports.

#### **A4.4 Test Facility**

The experimental work involved in this evaluation will be performed by Idaho National Laboratory. Over the past several years the U.S. Department of Defense (DOD) Defense Advanced Research Projects Agency (DARPA) and the U.S. Department of Homeland Security have been engaged in a Radionuclide Detection and Decontamination program which included a Radionuclide Capture and Decontamination component and Wide-Area Radionuclide Detection component<sup>1</sup>. Under that program, INL developed and implemented an effective method by which five decontamination technologies for concrete surfaces were evaluated. Because of their experience with that previous work, INL was selected to perform the upcoming TTEP evaluation of concrete decontamination technologies by employing similar methodology. In performing this evaluation, Idaho National Laboratory will follow the procedures specified in this test/QA plan and will comply with quality requirements in the TTEP Quality Management Plan (QMP)<sup>2</sup>. In general, the responsibilities of the technical staff of INL will be to:

- Ensure that any necessary laboratory equipment and hood(s) are fully functional and available prior to the times/dates needed in the technology evaluation.
- Have the appropriate training and experience to adequately complete the evaluation according to this test/QA plan.

- Review, approve, and maintain all data and records related to facility operation.
- Adhere to the requirements of the test/QA plan and the program QMP<sup>1</sup> in carrying out the technology evaluation.
- Provide input on facility procedures for the evaluation report.
- Support Dr. James in responding to any issues related to facility operations raised in QA assessment reports and audits.

## **A5 BACKGROUND**

The National Response Plan, Nuclear/Radiological Annex, published in December of 2004, designates EPA as the lead agency for long term recovery following terrorist incidents involving radioactive materials. Consistent with EPA's legislated mission, this directive gives the EPA the primary governmental responsibility for protecting human health and the environment releases of radiological materials. To meet the expected technology needs associated with acts of radiological terrorism, the EPA's Office of Research and Development (ORD), NHSRC, is conducting decontamination technology evaluations through the TTEP. These technology evaluations will provide data to be used in support of decisions concerning the selection and use of decontamination technologies for large outdoor environments contaminated with radiological threat agents. The technology evaluation data may also be used in clean-up guidance pertaining to specific threat agents and release scenarios. This test/QA plan details the experimental plan for the evaluation of selected commercially available radiological decontamination technologies and/or processes that are applicable to urban building materials, specifically, concrete, contaminated as would be the case following terrorist use of a radiological dispersion device (RDD), sometimes known as a "dirty bomb".

## **A6 TECHNOLOGY EVALUATION DESCRIPTION AND SCHEDULE**

### **A6.1 Decontamination Technologies**

The experimental approach and evaluation matrix described within this test/QA plan is not exclusive to one particular type of radiological decontamination technology, but one that would be appropriate for a variety of decontamination technologies. The exact technologies to be evaluated during this test have not yet been determined. However, EPA intends to test both "chemical" (e.g., wet application, radionuclide dissolution, and removal) and "physical" (e.g., physical removal of contamination from the concrete surface with minimal damage) technologies.

### **A6.2 Scope of Evaluation**

This technology evaluation will include concrete coupon selection, contaminant application, radiation measurement of contamination present on coupons (by laboratory gamma

counting), application of the decontamination technologies, and then subsequent measurement of residual contamination in order to determine the decontamination factor (i.e., efficiency of radionuclide removal; defined in Section B1.6) attained by each technology. Following the technology evaluation, reports describing the quantitative (i.e., decontamination factor) and qualitative performance of each technology will be generated. The qualitative aspects, while subjective, are a very important part of the evaluation and will include: 1) a full description of the technology including its mechanism of decontamination, how much ancillary equipment was required, its applicability to other contaminants and substrates; 2) an itemization of the capital and operating costs incurred during use of these technology including, reagents or media, waste disposal, decontamination of equipment; 3) deployment and operational data including rate of surface area decontamination, applicability to irregular surfaces, skilled labor requirement, utilities requirements, extent of portability, set-up/tear-down time, shelf life of media, the reliability of equipment; 4) secondary waste management including the estimated amount and characteristics of the spent media; and 5) any health, safety, or legal concerns over any aspect of the use of a technology.

### **A6.3 Schedule**

The initial TTEP evaluation of the decontamination technologies will be conducted during the fall of 2007. The bulk of this experimental work will likely be completed from October through December with the evaluation reports being drafted and peer reviewed from approximately December through February.

## **A7 QUALITY OBJECTIVES**

The outcome of this evaluation is based largely on the measurement of gamma radiation from the surface of concrete coupons. Accuracy of the detectors will be ensured through routine calibration and duplicate sample analyses. In addition, for each sample, gamma ray counting will be continued until the activity level of Cs-137 on the surface has a relative standard deviation of less than 2% based on nuclear counting statistics. The final activity assigned to that coupon is a compilation of information obtained from all components of the electronic assemblage which comprises the "gamma counter", including the raw data, and the spectral

analysis conducted by the spectroscopist using an INL data analysis program. Final spectra and all data which comprise the spectra are sent to a data analyst who independently confirms the "activity" number arrived at by the spectroscopist. When both the spectroscopist and an expert data analyst independently arrive at the same number in agreement then the data are certified. This would be described as the full gamma counting QA process for certified results.

In addition, the same coupons will be counted before and after application of the decontamination technology in order to determine the decontamination factors of the technologies. The concrete (prepared in a single batch) will have been verified that it falls within the American Society for Testing and Materials (ASTM C150) standards for Type II Portland cement<sup>3</sup>.

#### **A8 SPECIAL TRAINING/CERTIFICATION**

Documentation on training related to standard radiochemistry laboratory techniques and methods is maintained for INL technical staff in training files. The RTL from INL will verify the presence of appropriate training records prior to the start of testing. All technical staff will have a minimum of a bachelor's degree in science/engineering or have equivalent work experience. Prior to the formal evaluation of radiological decontamination technologies, any vendors that volunteer their technologies will have the option of training INL technicians on the operation of their technologies. This can be done during a teleconference or during an in person training session. Following the training, the vendors will sign a consent form that states that they have trained the technicians and will accept the data generated by these trained operators or designee.

#### **A9 DOCUMENTATION AND RECORDS**

The records for this evaluation include the test/QA plan, laboratory record books (LRB), electronic files (both raw data and spreadsheets), audit reports, and evaluation reports. The LRBs will serve as the primary repository of information during the experimental work conducted during this evaluation. The LRBs will be stored in the laboratory and periodically reviewed by the RTL or the TOL. Following the evaluation, all evaluation records will be transferred to permanent storage at Battelle's or INL's Records Management Office except for



quality assurance records which will be maintained by the Battelle Quality Assurance Manager.

All LRBs will be stored indefinitely, either by the RTL, TOL, or INL's Records Management Office and final location will be noted in the evaluation records. EPA will be notified before disposal of any files. Section B10 further details the data recording practices and responsibilities.

## **SECTION B**

### **MEASUREMENT AND DATA ACQUISITION**

#### **B1 EXPERIMENTAL PLAN AND TEST SAMPLES**

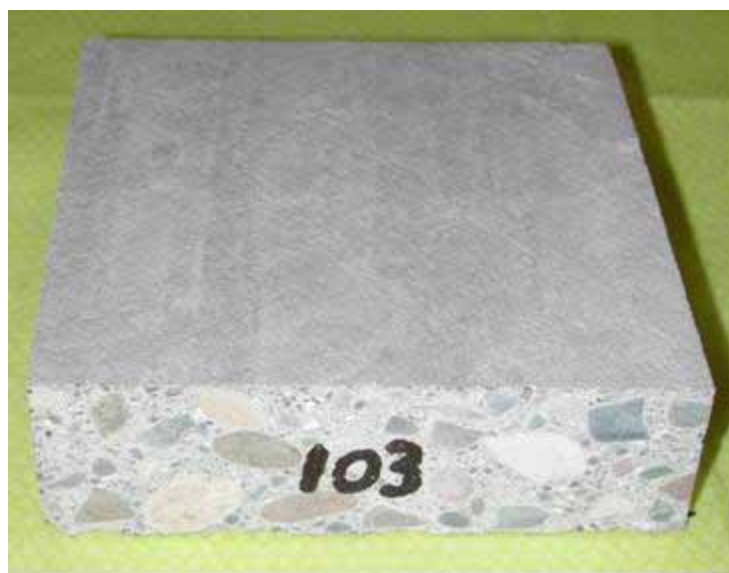
##### **B1.1 Concrete Test Coupons**

INL will supply up to 200 coupons composed of structural concrete. The coupons will be approximately 4 centimeters (cm) thick, 15 cm square, and have a surface finish that is consistent across all the coupons and, most importantly, that is representative of that which would be typically found on the exterior of an urban structure (Type II Portland cement). These coupons (2,500 of them are available) were prepared for the DARPA/DHS study<sup>1</sup> in a single batch of concrete provided by a redi-mix concrete supplier according to Uniform Building Code requirements for structural concrete<sup>4</sup>. Specifically, the redi-mix was poured into 0.9 m square plywood forms with the surface exposed, the surface “floated” to get the smaller aggregate and cement paste to float to the top, and then cured for 21 days. Following curing, the squares were cut to the desired size with a laser guided rock saw. For this evaluation, the “floated” surface will be used because of the possibility of interferences due to release agents, in the case of metal forms, and cellulose residual, in the case of wood forms.

The concrete has certification papers that details cement:water ratio, percent air entrainment, admixtures, the ratio of tricalcium silicate and dicalcium aluminate, etc. Because all of the coupons will have been made from the same batch, tensile strength testing to confirm uniformity within batches will not be performed. However, for the DARPA/DHS work<sup>1</sup> mentioned previously, INL completed a comparison of the radionuclide absorption properties of this concrete with concrete samples collected from several locations around the United States<sup>5</sup>. This comparison showed that the concrete coupons to be used for this evaluation are indeed “typical” in terms of how radionuclides interact with the surfaces.

Prior to contaminant application, the surface of the coupons will be examined for obvious cracks or abnormalities and, if none are found, they will be cleaned with a soft nylon brush and nanopure water and allowed to air dry for at least five days. Each coupon will be marked with an identifying number using a permanent marker, as shown in Figure 2. In addition, in order to protect against the possibility of any contamination seeping into the coupons through the edges, and ensuring that contaminant is applied only via the surface of the coupons, the edges will be

sealed with epoxy. In addition, EPA has recommended that the concrete coupons need to be preconditioned at  $23.9\text{ }^{\circ}\text{C} \pm 1.7^{\circ}\text{C}$  and between 40%–60% relative humidity (RH). However, during the DARPA/DHS work<sup>1</sup>, INL completed a comparison of the radionuclide absorption properties as a function of preconditioning moisture level on the surface of the coupons. They showed that the difference in cesium absorption depth between coupons preconditioned in an environment of 12% RH and those saturated with water was on the order of nanometers. This difference corresponded to a very small difference in decontamination efficiency. INL determined that uncontrollable differences in surface characteristics of the coupons proved to have a more significant impact. In addition, ambient temperatures within the INL laboratories are typically within the above range. Therefore, while the temperature and RH will not be controlled, those parameters will be measured in the coupon storage location. However, if new findings show that RH plays a significant role in cesium absorption, we will obtain an environmental chamber for controlled pre-conditioning.



**Figure 2. Example Cement Coupon with Identification Marking**

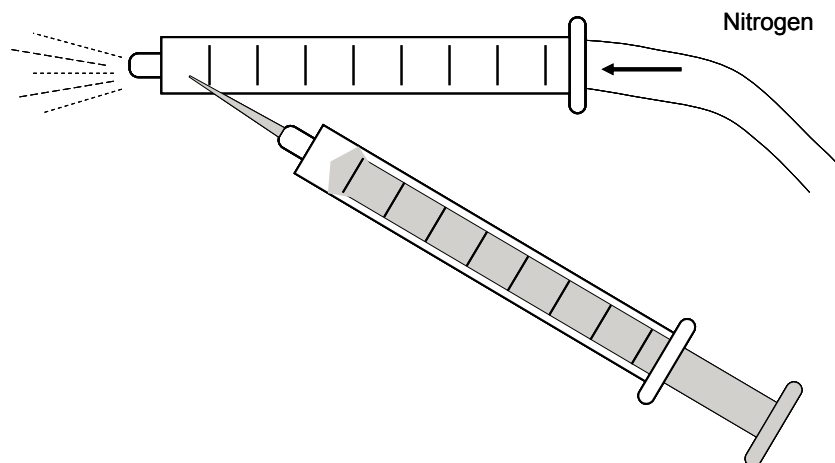
## **B1.2 Contaminant Application**

Each contaminated coupon will be spiked with 250 microliters of unbuffered, slightly acidic aqueous solution containing 137 ppm Cs-137 which corresponds to an activity level of approximately 53 microCuries. The liquid spike will be delivered to each coupon using an aerosolization technique used for the DARPA/DHS RDD program<sup>1</sup>. The aerosol delivery device

is constructed of two syringes and is shown in Figure 3. The first syringe has had the plunger removed from it and a nitrogen gas line has been attached to the rear of the syringe. The second syringe contains the contaminant spiking solution and is equipped with a 27 gauge needle which penetrates through the plastic housing near the tip of the first syringe. Nitrogen gas is turned on at a flow of approximately 1 - 2 liters per minute creating a turbulent flow through the first syringe. The liquid spike in the second syringe is introduced and becomes nebulized by the turbulent gas flow. A very fine aerosol is ejected from the tip of the first syringe creating a controlled and uniform spray of fine liquid droplets onto the coupon surface. The contaminant spray is applied all the way to the edges of the coupon which are taped (after having previously been sealed with epoxy) to insure that the contaminant is only applied to the surfaces of the coupons.

Due to the heterogenous nature of the texture of concrete coupons, perfect homogeneous distribution of contaminant will likely never be attained. However, a reasonably uniform coverage is expected because as the contaminant is being applied, the technician can observe whether or not the mist has been applied uniformly across the entire surface of the coupon. In addition, the intrinsic germanium detector that is used to measure the gamma rays from the surface of these coupons has been set up specifically to measure the activity from the entire surface of the concrete coupons. Therefore, small differences in coverage across the coupons, while not quantified, will not impact the results. To protect against an extremely rare instance occurred in which the contaminant was applied in a very heterogeneous fashion (i.e., most or all of the contaminant deposited onto one quadrant or one half of the coupon) a semi-quantitative gamma counter will be used to confirm a reasonably uniform distribution of activity across the surface of the coupons. If this measurement suggests an uneven distribution of contaminant, that coupon will not be used. Because this is a semi-quantitative measurement tool, this will rely on the technician's experience of evaluating if there are areas of the coupons exhibiting abnormally low or high levels of activity compared with the rest of the coupon. In summary, the contaminant application step requires a degree of skill that only comes with much experience in performing this task. Therefore, while difficult to measure quantitatively, the technician that has this skill is in the position to determine if a non-homogeneous application has occurred.

Because the activity from each full coupon will be measured before and after application of the decontamination technology, differences in contaminant load between coupons is also not critical. However, the acceptable amount of variation in activity on each coupon is 10%. If a coupon varies from the target activity level by more than 10%, it will not be used. Before coupons are spiked, the coupon edges will be covered with masking tape to prevent accidental overspray from contaminating the epoxy coating on the edges. The target activity level on each coupon will be 53 microCuries Cs-137.



**Figure 3. Diagram of the Aerosol Delivery System**

### **B1.3 Contaminant Measurement**

Each spiked coupon will be allowed to air dry for at least 2 hours, then will be removed from the radiological buffer area and gamma counted. Gamma ray counting will be continued until the average activity level of Cs-137 from the surface stabilizes to a relative standard deviation of less than 2%. Gamma-ray spectra acquired from Cs-137 spiked coupons will be analyzed using the INL Radiological Measurement Laboratory (RML) data acquisition and spectral analysis programs. Radionuclide activities on coupons are calculated based on the efficiency, emission probability, and half-life values. Decay corrections are made based on reference time and date and the duration of the counting period. Full RML gamma counting

QA/QC (as described in Section A7) will be employed and certified results will be sent via inter-department letter to the RTL. Counting equipment is shown in Figure 2 below. This is not a destructive measurement technique so the coupons that have been spiked with contaminant and measured will be aged for either 7 or 28 days before they will be positioned in a radioactive hood for application of a decontamination technology. Following application of the decontamination technology, the residual radioactive contamination on the same coupons will be measured again in order to calculate the decontamination factor.

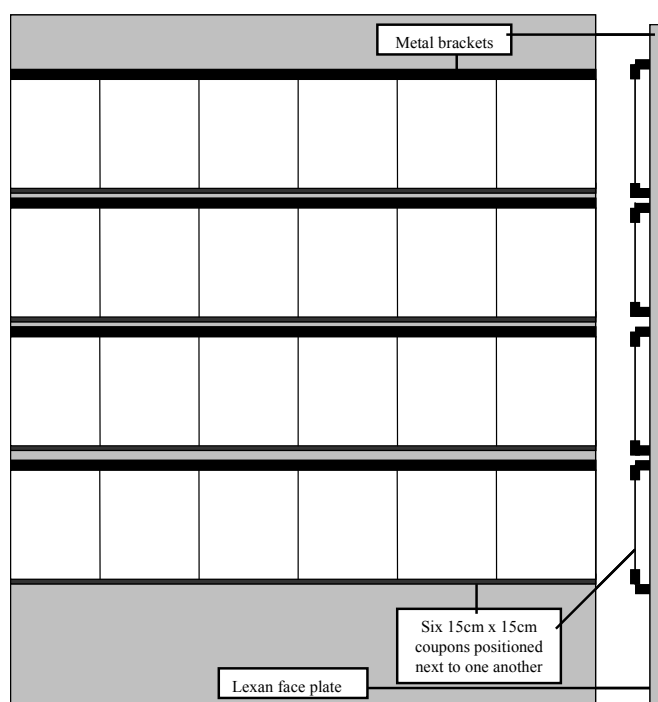


**Figure 4. Gamma Counting Equipment at the INL RML**

#### **B1.4 Construction of the Test Stand**

In order to test these technologies in a way that simulates how they would be used in a real-world setting, a test stand will be constructed to hold the concrete coupons in a vertical orientation. This stand, depicted as a schematic in Figure 5, will be constructed from a face plate made from a non-reactive material such as Lexan. Non-reactive metal brackets extending across the width of the face plate will be spaced adequately apart so that the concrete coupons can be slid into these brackets from both sides and held firmly. The stand itself will be wide enough so that up to six concrete coupons will fit into each level of the stand at once. In all, this stand will provide an area of up to six square feet of concrete surface (24 concrete coupons) to apply the decontamination technologies. This surface area of 24 coupons will be constructed four times

(using different coupons each time) in a vertical orientation, once after 7 days and once after 28 days for both technology types. The stand will be constructed in such a way to fit within the radiological buffer area hood. Throughout the construction of the test stand, INL, EPA, and Battelle will consider the effectiveness of the stand for attaining the objective of this evaluation. It is possible that another design will be implemented. Changes in stand design will be documented by amendment of the test/QA plan once the final design is determined.



**Figure 5. Schematic Diagram of Front and Side View of Test Stand Face**

### **B1.5 Application of Decontamination Technologies**

Throughout the course of this evaluation, two decontamination technologies will be applied to 24 concrete coupons arranged in 2' x 3' rectangular surfaces (referred to as the test surface). Each test surface that is constructed will only be used for evaluation of one decontamination technology. For each technology, separate test surfaces will be evaluated in a vertical orientation and a horizontal orientation. In addition, separate vertical surfaces will be evaluated  $7 \pm 2$  days after the contaminant was applied to the coupons and  $28 \pm 2$  days after it was applied. The same goes for the horizontal surfaces. Of the 24 coupons that are used for

each test surface, six coupons, placed at edge and middle locations across the test surfaces, will have been contaminated with radionuclide at the contaminant application step (it would be impractical to apply contaminant to and measure all 24 coupons for each testing scenario). Therefore, 18 coupons in each of those configurations will not have been contaminated at all, their presence is to provide a scenario (of a wall or floor) that is similar to what might be the case in an actual decontamination event. Therefore, the decontamination efficiency of each technology under each set of conditions will be measured at least six times. Simply stated, each test surface will be built in part with six coupons of a known activity, and following the application of a decontamination technology, the remaining activity on those six coupons will be measured, from which the decontamination factor can be calculated. Table 1 summarizes the matrix of replicate test coupons in the various orientations and time delays.

The decontamination technologies will be applied to the coupons as per manufacturer's specifications. All tests will either be conducted in a radiological fume hood or in a glove box similar to those shown in Figures 6 and 7. One coupon that has not been contaminated will be measured for background (BG) activity with each set of conditions. The background coupons will not be exposed to the decontamination technologies being evaluated.

**Table 1. Replicate Concrete Test Coupons for Each Decontamination Technology**

	<b>Horizontal Contamination Measurement</b>	<b>Horizontal Decontamination Measurement</b>	<b>Vertical Contamination Measurement</b>	<b>Vertical Decontamination Measurement</b>
<b>7 Day</b>	6	6	6	6
<b>7 Day BG</b>	1	1	1	1
<b>28 Day</b>	6	6	6	6
<b>28 Day BG</b>	1	1	1	1

BG=Background activity measurement



**Figure 6. Radiological Control Hood****Figure 7. Glovebox**

### **B1.6 Calculation of Decontamination Efficacy**

The efficacy of decontamination will be calculated for each contaminated coupon in order to evaluate the performance of each decontamination technology. The decontamination efficacy will be represented using the following equations for decontamination factor (DF) and percent removal (%R):

$$DF = A_o/A_f \text{ and } \%R = (1-A_f/A_o) \times 100\%$$

where  $A_o$  is the radiological activity from the surface of the coupon before application of a decontamination procedure and  $A_f$  is radiological activity from the surface of the coupon after application of the decontamination technology. Each of these equations represents the same data in a slightly different way. The DF is the factor by which the contamination was removed and the %R is the percent of contamination that was removed by the decontamination technology.

### **B1.7 Documentation of Operational Factors**

An important part of this evaluation will include the collection of qualitative data pertaining to the operational aspects of each technology that is evaluated. This information will include deployment and operational data including rate of surface area decontamination, which will be measured simply by recording the time it takes to apply the decontamination technology. It will also include applicability to irregular surfaces, skilled labor requirement, power and water requirements, extent of portability, set-up/tear-down time, shelf life of media, and the reliability

of equipment. The information will be obtained by having the evaluation staff responsible for operating the technology during the evaluation test fill out a questionnaire that includes questions about these issues.. These staff will be INL technicians that have been trained by the vendor in the operation of the technology. Some of the qualitative information will be easily gleaned through their operational experience during the evaluation, but other information, such as the applicability to irregular surfaces and the extent of portability, will require them to think about how effective these technologies would be if deployed for a field decontamination scenario.

The approximate volume and weight of any secondary waste generated per unit surface area decontaminated will be reported. This might be reported in units of number of containers that had to be disposed of as well as the approximate weight of those containers. The physical characteristics of the waste will be described as well (e.g., if the waste is a solid, liquid, slurry, etc.). The ease of clean-up and of secondary waste containment will also be described by the operators. Lastly, the evaluation staff questionnaire will also include be questions about any real or perceived health or safety concerns over any aspect of the use of the technology and documented and reported.

## **B2     REFERENCE SAMPLE COLLECTION**

The radiological activity of the contaminated coupons will be measured after contamination and then again after application of the decontamination technologies. Because of the direct nature of radionuclide measurement from the surface of the coupons, no reference samples will be collected during this evaluation.

## **B3     SAMPLE HANDLING AND IDENTIFICATION**

The key concern with sample handling and custody during this evaluation will be the systematic organization of sample labeling so the concrete coupons that are contaminated and then decontaminated remain properly identified from coupon contamination through final gamma counting. This will be done by providing an identification number that is marked on the edge of each concrete coupon in permanent marker and then covered with clear epoxy to ensure its integrity. This number will be recorded in a laboratory record book and used throughout the measurement and decontamination components of the evaluation. After contaminant application,

each coupon will be stored in its own transparent plastic bag to allow visibility of the identification number and protect against cross-contamination between coupons.

#### **B4 ANALYTICAL METHODS**

Direct gamma counting is the only measurement method that will be used during this evaluation. Coupons with an unknown Cs-137 activity will be placed into gamma ray counters and nuclear counting will continue until the average activity level of Cs-137 measured on the surface stabilizes to a relative standard deviation of less than 2% based on nuclear counting statistics. The sensitivity of these counters is 10 picoCuries of activity. Gamma ray counters employ pulsed, intrinsic, High Purity germanium (HPGe) detectors which are either Low Energy (Canberra LEGe Model GL 2825R/S) or Broad Energy (Canberra BEGe Model 2025S) detectors cooled with liquid nitrogen in a shielded, integral cryostat. Detectors are equipped with an integral pre-amplifier which utilizes dynamic charge restoration methods to discharge the integrator. High voltage is supplied to the detectors using a Canberra Model 3125 power supply. Signal from the detector is processed through a Canberra Model 2026 spectroscopy amplifier to achieve correct shaping and amplitude. Shaped signal then goes to a Canberra Model 8713 analog to digital converter (ADC) where the signal is digitized into a pulse height spectrum. A Canberra Model 556A acquisition interface module (AIM) is used to control the ADC, and to collect and transfer the data over an ethernet connection to a computer. Spectral analysis is conducted using the a software program developed at the INL. Radionuclide activities on the coupons are calculated based on individual detector efficiency, emission probability, and half-life values. Decay corrections are made based on reference time and date and the duration of the counting period. If specified, interference corrections between the desired photopeak and background interference are made. Data are also manually analyzed by an experienced gamma ray spectroscopist to confirm the value which the computer has determined.

#### **B5 QUALITY CONTROL REQUIREMENTS**

Approximately 10% of the total coupons to be gamma counted will be counted two times (with a requirement of agreement within 5%) as a quality check on the reproducibility of the counting method. In addition, the background activity of at least one coupon will be measured

for each set of test conditions (technology, orientation, aging time, etc.). See Section A7 for a description of the quality assurance of the certified activity levels.

## **B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE**

The key piece of equipment is the intrinsic high purity germanium detector. It and the accompanying electronics which comprise the “gamma counter” will be maintained according to INL standard procedures. Other equipment and supplies may include adjustable volume pipettes, beakers, etc. In general, all equipment will be operated and maintained per manufacturer recommendations, or existing facility requirements.

## **B7 INSTRUMENT CALIBRATION AND FREQUENCY**

The intrinsic, high purity germanium detector energy calibrations will be established using standardized techniques called out in American National Standards Institute(ANSI)/Institute of Electrical and Electronics Engineers (IEEE) 325-1996, 1996. IEEE Standard Test Procedures for Germanium Gamma-ray Detectors<sup>6</sup>, and ANSI N42.14-1999, 1999. Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-ray Emission Rates of Radionuclides, ANSI<sup>7</sup>. In brief, detector energy calibrations will be established using thorium-228 daughter gamma rays at 238.6, 583.2, 860.5, 1620.7, and 2614.5 kilo electron volts. A quadratic fit of the energy of each photopeak versus detector channel will be done with an estimated uncertainty of  $\pm 0.03$  keV over the given range. Detector efficiency calibrations will be accomplished using the Cs-137 photopeak at 661.6 keV. Detector efficiency at 661.6 keV will be determined both as a function of detector height from the surface of the coupon and as a function of known Cs-137 activity on the surface. Efficiency calibration curves will be established to an estimated uncertainty of  $\pm 2.0\%$  at the 68.3% confidence level (1 sigma). This rigorous calibration is performed weekly and documented by the RML and in addition, weekly calibration checks are performed using a europium-152 standard. These calibration checks provide assurance that the detectors are functioning properly. All calibrations or calibration checks will be documented in the project laboratory record book. The instrument records will be reviewed by the Battelle QA Manager or designee prior to the evaluation.

## **B8 INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES**

All materials, supplies, and consumables to be used during the evaluation will be ordered by the RTL or designee. Battelle and INL will rely on previous experience or recommendations to guide selection of manufacturers and materials. The ingredients of the concrete used to prepare the coupons will be reviewed to confirm that it meets the criterion for structural concrete. The Ready-mix supplier will provide documentation to INL that the concrete used in the coupons meets the ASTM standards for Type II Portland cement.

## **B9 NON-DIRECT MEASUREMENTS**

Data published previously in the scientific literature will not be used during this evaluation.

## **B10 DATA MANAGEMENT**

Data will be acquired and recorded electronically or manually by Battelle or INL technical staff during this evaluation test. All data and observations for the operation of the radiological decontamination technologies will be documented by Battelle technical staff in laboratory record books. All hand written entries will be recorded in ink and corrections to the entry will be made with a single line so as to not obliterate the original entry. The correction will be initialed and dated. An explanation will accompany all non-obvious corrections. Records received by or generated by any of the technical staff during the evaluation will be reviewed by a Battelle or INL staff member within two weeks of receipt or generation, respectively, before the records are used to calculate, evaluate, or report evaluation results. If a staff member generated the record, this review will be performed by a technical staff member involved in the evaluation test, but not the staff member that originally received or generated the record. The review will be documented by the person performing the review by adding his/her initials and date to the hard copy of the record being reviewed. In addition, data calculations performed by technical staff will be spot-checked by other staff to ensure that calculations are performed correctly. Calculations to be checked include any statistical calculations described in this test/QA plan. The data obtained from this evaluation will be compiled for each radiological decontamination

technology. During the course of any assessment or audit, the Battelle QA Manager or designee will inform the technical staff of any immediate corrective action that should be taken. If serious quality problems exist, the Battelle QA Manager, or designee, will contact the TTEP Manager, who is authorized to stop work. Once the assessment report has been prepared, the TOL will ensure that a response is provided for each adverse finding or potential problem, and will implement any necessary follow-up corrective action. The Battelle QA Manager will ensure that follow-up corrective action has been taken.

## **SECTION C ASSESSMENT AND OVERSIGHT**

### **C1 ASSESSMENTS AND RESPONSE ACTIONS**

Every effort will be made in this evaluation test to anticipate and resolve potential problems before the quality of performance is compromised. One of the major objectives of the test/QA plan is to establish mechanisms necessary to ensure this. Internal quality control measures described in this test/QA plan, implemented by the technical staff and monitored by the TOL, will give information on data quality on a day-to-day basis. The responsibility for interpreting the results of these checks and resolving any potential problems resides with the TOL. The technical staff has the responsibility to identify problems that could affect data quality or the ability to use the data. Any problems that are identified will be reported to the TOL, who will work with the Battelle QA Manager to resolve any issues. Action will be taken to control the problem, identify a solution to the problem, and minimize losses and correct data, where possible. Independent of EPA QA activities, Battelle will be responsible for ensuring that the following audits are conducted as part of this evaluation test.

#### **C1.1 Technical Systems Audits**

The Battelle QA Manager, or designee at INL, will perform a technical systems audit (TSA) at least once during this evaluation test. The purpose of this audit is to ensure that the evaluation is being performed in accordance with the QMP<sup>1</sup> and this test/QA plan. In this audit, the Battelle QA Manager, or designee, may review the gamma counting methods used, compare actual test procedures to those specified or referenced in this plan, and review data acquisition and handling procedures. A TSA report will be prepared, including a statement of findings and the actions taken to address any adverse findings. The NHSRC QA Manager will receive a copy of Battelle's TSA report. At EPA's discretion, EPA QA staff may also conduct an independent on-site TSA during the evaluation. The TSA findings will be communicated to technical staff at the time of the audit and documented in a TSA report.

## **C1.2 Data Quality Audits**

The Battelle QA Manager will be responsible for auditing at least 10% of the evaluation data acquired in the evaluation test. The Battelle QA Manager will trace the data from initial acquisition, through reduction and statistical comparisons, to final reporting. All calculations performed on the data undergoing the audit will be checked.

## **C1.3 QA/QC Reporting**

Each assessment and audit will be documented in accordance with Section 3.3.4 and 3.3.5 of the QMP<sup>1</sup>. The results of the assessment reports will be submitted to EPA. Assessment reports will include the following:

- Identification of any adverse findings or potential problems
- Response to adverse findings or potential problems
- Recommendations for resolving problem
- Confirmation that solutions have been implemented and are effective
- Citation of any noteworthy practices that may be of use to others

## **C2 REPORTS TO MANAGEMENT**

The Battelle QA Manager, or INL designee, 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 Battelle QA Manager, or INL designee, will contact the Battelle TTEP Manager who is authorized to stop work. In such an event, the RTL and TOL would work together to quickly resolve whatever issues exist and document any corrective actions. Once the assessment report has been prepared, the TOL will ensure that a response is provided for each adverse finding or potential problem and will implement any necessary follow-up corrective action. The Battelle QA Manager will ensure that follow-up corrective action has been taken. The test/QA plan and final report are reviewed by EPA QA staff and EPA program management staff.



## **SECTION D**

### **DATA VALIDATION AND USABILITY**

#### **D1 DATA REVIEW, VALIDATION, AND EVALUATION REQUIREMENTS**

The key data review requirements include a comparison of laboratory record book data and comments against final data to flag any suspect data and a review of final data to resolve any questions about apparent outliers. The quality assessments, as described within Section C of this document are designed to assure the quality of this data.

#### **D2 VALIDATION METHODS**

Data validation is conducted as part of the data review in Section D1 of this test/QA plan. This validation will include a visual inspection of hand written data to ensure that all entries were properly recorded and that any erroneous entries were properly noted. Calculations used to determine Cs-137 concentrations will be spot checked to ensure accuracy and the appropriateness of the calculations. Data validation efforts include the completion of QC activities and the performance of a TSA as described in Section C.1.2. An audit of data quality will be conducted by the Battelle QA Manager to ensure that data review and validation procedures were completed, and to assure the overall quality of the data.

#### **D3 RECONCILIATION WITH USER REQUIREMENTS**

The purpose of this test is to evaluate the performance of radiological decontamination technologies. The data obtained shall include thorough documentation of the performance of each technology. The data review and validation procedures described in the previous sections will assure that data meet these requirements and are accurately presented in the evaluation reports generated from this test. Any limitation to the data will be discussed in the report.

The data generated in this evaluation will be compiled into one TTEP evaluation report for each technology evaluated. The report will be submitted to EPA in Word and Adobe pdf format and subsequently posted on the TTEP website. This test/QA plan and the resulting TTEP evaluation report(s) will be subjected to review by the radiological decontamination technology vendors, Battelle and INL staff, EPA, and expert peer reviewers. The reviews of this test/QA

plan will assure that this evaluation and the resulting reports meet the needs of potential users of the evaluated technologies.

## **SECTION E**

### **REFERENCES**

1. Radionuclide Detection and Decontamination Program, Broad Agency Announcement 03-013, U.S. Department of Defense (DOD) Defense Advanced Research Projects Agency (DARPA) and the U.S. Department of Homeland Security, classified program.
2. Quality Management Plan for the Technology Testing and Evaluation Program, Version 2.0, Battelle, Columbus, Ohio, January 2006.
3. ASTM Standard C 150-07, 2007, "Standard Specification for Portland Cement," ASTM International, West Conshohocken, PA, [www.astm.org](http://www.astm.org).
4. ACI Manual of Concrete Practice, American Concrete Institute, October 1978.
5. Adsorption of cesium and cobalt onto cement, granite, and marble, Fox, R. V.; Ball, R. D.; Houghton, T.P.; and Fox S. L., poster -62<sup>nd</sup> Annual Meeting of the Northwest Regional American Chemical Society, June 2007.
6. Standard Test Procedures for Germanium Gamma-ray Detectors ANSI/IEEE Std. 325-1996, Issue 29, May 1997.1996.
7. Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-ray Emission Rates of Radionuclides, American National Standards Institute ANSI N42.14-1999. New York, IEEE 1999 (Rev. 2004).

# SCIENCE CENTER



PRESORTED STANDARD  
POSTAGE & FEES PAID  
EPA  
PERMIT NO. G-35

Office of Research and Development  
National Homeland Security Research Center  
Cincinnati, OH 45268

Official Business  
Penalty for Private Use  
\$300



**Recycled/Recyclable**  
Printed with vegetable-based ink on  
paper that contains a minimum of  
50% post-consumer fiber content  
processed chlorine free