

# **Evaluation of Sample Screening Technologies for the All Hazards Receipt Facility**

## TEST/QA PLAN



**Battelle**

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National Homeland Security  
Research Center

## **Technology Testing and Evaluation Program**

Test/QA Plan for  
Evaluation of Sample Screening  
Technologies for the All  
Hazards Receipt Facility

**TTEP TTEP TTEP TTEP**

A1 TITLE AND APPROVAL PAGE

TTEP 1119  
I.O. 1.21

Test/QA Plan

For

Evaluation of Sample Screening Technologies for the  
All Hazards Receipt Facility

Version 1  
May 26, 2006



EPA Task Order Project Officer

5/24/06

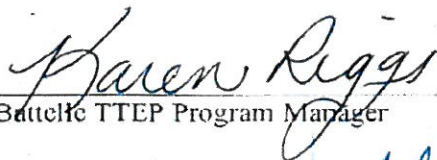
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EPA Quality Assurance Officer

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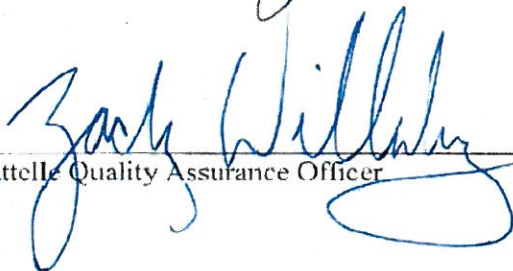
Date



Battelle TTEP Program Manager

5/25/06

Date



Battelle Quality Assurance Officer

5-26-06

Date

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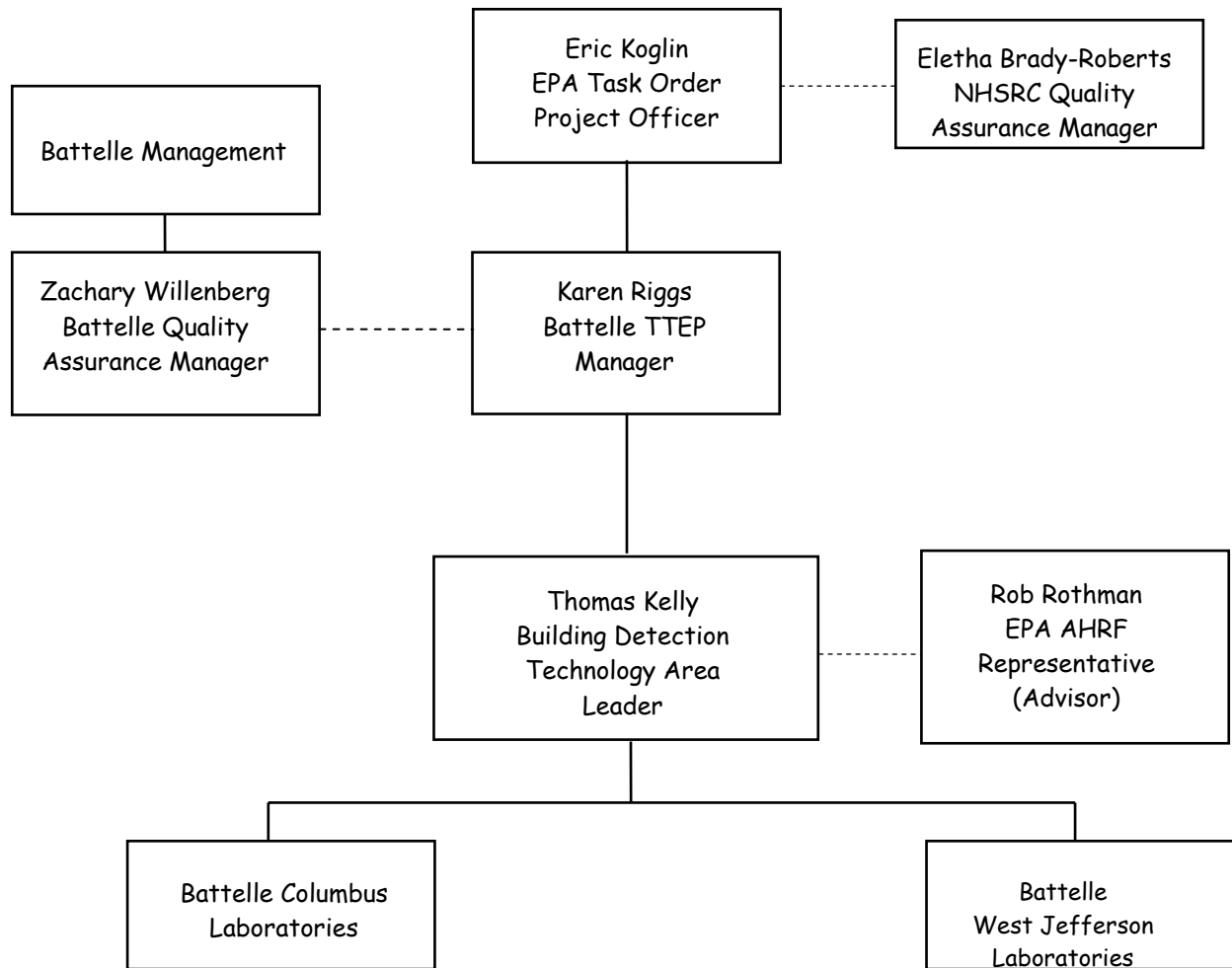
#### **A4 TECHNOLOGY EVALUATION ORGANIZATION**

The technology evaluations described in this test/quality assurance (QA) plan will be performed by Battelle under the direction of the U.S. Environmental Protection Agency's (EPA) National Homeland Security Research Center (NHSRC) through the Technology Testing and Evaluation Program (TTEP). This test/QA plan is for evaluation of technologies selected for use in EPA's All Hazards Receipt Facilities (AHRF), and specifically is designed to be compliant with the draft sample screening protocol developed for the AHRF.<sup>1</sup> This evaluation will be carried out under Task Order 1119 of the TTEP program (Contract GS-23F-0011L-3). The organization chart in Figure 1 shows the individuals from Battelle and EPA who will have responsibilities in the technology evaluation. The specific responsibilities of these individuals are detailed in the following paragraphs.

##### **A4.1 Battelle**

Dr. Thomas Kelly is Battelle's Building Detection Technology Area Leader for TTEP, and the Task Order Leader for this technology evaluation. Dr. Kelly's responsibilities are to:

- Consult with EPA's Task Order Project Officer (TOPO) and AHRF representative in planning for the evaluation.
- Select the appropriate Battelle laboratories to carry out the evaluation.
- Prepare the draft test/QA plan and evaluation reports.
- Arrange for use of the Battelle laboratories and establish a test schedule.
- Arrange for the availability of qualified staff to conduct the evaluation.
- Assure that testing is conducted according to this test/QA plan.
- Revise the test/QA plan and evaluation reports in response to reviewers' comments.
- Keep the Battelle TTEP Manager informed of progress and difficulties in planning and conducting the evaluation.
- Coordinate with the Battelle Quality Assurance Manager for the performance of technical and performance audits as required by Battelle or EPA Quality Management staff.
- Respond to any issues raised in assessment reports and audits, including instituting corrective action as necessary.
- Establish a budget and schedule for the technology evaluation and direct the effort to ensure that budget and schedule are met.
- Coordinate distribution of the final test/QA plan and evaluation reports.



**Figure 1. Organization Chart for the Screening Technology Evaluation**

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

- Maintain communication with EPA's TTEP Program Manager on all aspects of the program.
- Monitor adherence to budgets and schedules in this work.
- Provide the TOPO with monthly technical and financial progress reports.
- Review the draft test/QA plan.
- Review the draft evaluation reports.
- Ensure that necessary Battelle resources, including staff and facilities, are committed to the technology evaluation.
- Support Dr. Kelly in responding to any issues raised in assessment reports and audits.



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

- Review the draft test/QA plan.
- Maintain communication with Battelle's Task Order Leader and TTEP Program Manager.
- Conduct a technical systems audit (TSA) at least once during the technology evaluation.
- Review results of any performance evaluation (PE) audit(s) specified in this test/QA plan.
- Audit at least 10% of the evaluation data.
- Prepare and distribute an assessment report for each audit.
- Verify implementation of any necessary corrective action.
- Notify Battelle's TTEP Manager to issue a stop work order if internal audits indicate that data quality is being compromised. Notify the Task Order Leader if such an order is issued.
- 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 TTEP QMP<sup>2</sup> are followed.

Battelle technical staff will support Dr. Kelly in planning and conducting the technology evaluation. These staff will:

- Assist in planning and scheduling the technology evaluation.
- Become familiar with the use of the technologies to be tested.
- Carry out the test procedures specified in this test/QA plan.
- Assure that test procedures and data acquisition are conducted according to this test/QA plan.

#### **A4.2 EPA**

Mr. Eric Koglin is EPA's TOPO for this program. As such, Mr. Koglin will:

- Have overall responsibility for directing the evaluation process.
- Review the draft test/QA plan.
- Approve the final test/QA plan and any subsequent versions.
- Review the draft evaluation reports.
- Oversee the EPA review process on the draft test/QA plan and reports.
- Coordinate the submission of evaluation reports for final EPA approval.

Ms. Eletha Brady-Roberts, the NSHRC Quality Assurance Manager for this program

will:

- Review the draft test/QA plan and any subsequent versions.
- 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.

Mr. Rob Rothman will serve in an advisory role to Dr. Kelly to convey EPA's needs and expectations for the AHRF screening technology evaluation. In that role he will:

- Provide AHRF planning documents and procedures to assure that this evaluation is consistent with EPA's needs.
- Help identify the commercial screening technologies to be evaluated.
- Help define test procedures to evaluate screening technologies.
- Review the draft test/QA plan and the draft evaluation reports that result from this effort.

#### **A4.3 Test Facility**

The location for the technology evaluation described here will be Battelle's laboratories in Columbus and West Jefferson, Ohio. The Columbus facilities include chemical laboratories equipped for safe handling of volatile toxic industrial chemicals (TICs). The West Jefferson facilities are chemical surety laboratories certified for use of chemical warfare (CW) agents. Other test facilities could be used depending on the availability and capability of the facilities. In general, the responsibilities of the technical staff in these test facilities will be to:

- Ensure that the facility is fully functional prior to the times/dates needed in the technology evaluation.
- Provide requisite technical staff during the technology evaluation.
- Provide any safety training needed by Battelle or EPA staff.
- Review and approve all data and records related to facility operation.
- Review the test/QA plan.
- Adhere to the requirements of this test/QA plan and the program QMP<sup>2</sup> in carrying out the technology evaluation.
- Provide input on facility procedures for the evaluation report.
- Support Dr. Kelly in responding to any issues raised in assessment reports and audits related to facility operation.

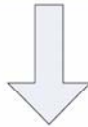
## **A5 PROBLEM DEFINITION/BACKGROUND**

The EPA, U.S. Department of Homeland Security (DHS), and U.S. Department of Defense (DOD) have combined efforts to develop, construct, and implement AHRF capabilities for prescreening unknown and potentially hazardous samples collected during suspected terrorist events. This effort was initiated in response to requests from states and federal agencies, particularly public health laboratories, for standardized guidance on screening samples to protect laboratory staff and ensure sample integrity and the validity of analytical results. The AHRF are intended for in-process screening of unknown samples for chemical (e.g., CW agents), explosive, and radiological hazards to protect laboratory workers and facilities from contamination and injury. The AHRF will serve as a front end assessment and will be used on an “as needed” basis. These facilities will not provide detailed or quantitative analytical results, but instead will provide initial screening of samples prior to full laboratory analysis, for the safety of all laboratory personnel. The screening process is designed to provide an indication of the presence or absence of chemical, radiological, or explosive agents, and is not intended to confirm or quantify specific contaminants. Screening technologies used in the AHRF are intended to be rapid and qualitative, and may be of relatively low cost and “low tech” in design, but must ensure meaningful qualitative results. As directed by EPA, this test/QA plan specifically addresses only technologies for chemical screening in the AHRF, and not radiological or explosives screening.

This test/QA plan specifies procedures for the evaluation of commercially available screening devices to rapidly detect toxic chemicals and chemical agents in samples entering an AHRF. The procedures, target chemicals, and sample types called for in this test/QA plan are based on those established in the AHRF draft screening protocol.<sup>1</sup> Figure 2 summarizes the sample screening process to be implemented through the AHRF.<sup>1</sup> As this figure shows, screening of the sample container for chemical contamination occurs in Step 2 of the screening process, and screening of the sample itself for chemical contamination occurs in Step 3. The sample screening in Step 3 may use a wider variety of technologies than that in Step 2, to address the variety of potential sample types. In performing this technology evaluation, Battelle will

### Outside All Hazards Receipt Facility - Sample Receipt

- 1** - Review 1st Responder report  
- Document observations and complete receipt form  
- Establish/continue COC  
- Screen transport container for radioactivity  
- Determine risk for explosive device (if any questions or doubts refer sample to bomb squad)



### Place Transport Container into Fume Hood

- 2** - Unpack samples and note observations  
- Screen sample container: radioactivity (surface scan), explosive screen (colorimetric), CWA screen (colorimetric)



### Transfer Sample into Glove Box

- 3** - Open primary container and expose sample  
- Screen for radioactivity (surface scan), CWA screen (IMS or FSP), organics (PID), hazardous material (colorimetric) and explosives (flame test)  
- Take a swab and/or small aliquot of sample for laboratory biological analysis  
- Take aliquot(s) of sample for lab chemical analysis, if cleared by biological laboratory



### Summarize Sample info for Lab Director

- 4** - Results shared with authority that collected the sample and the FBI WMD Coordinator, chemist or radiological technician consulted when necessary  
- Proper laboratory identified to receive the sample

#### Acronyms:

COC - Chain of Custody

CWA - Chemical Warfare Agent

FSP - Flame Spectrophotometer

PID - Photoionization Detector

IMS - Ion Mobility Spectrometer

WMD - Weapons of Mass Destruction

**Figure 2. Summary of All Hazards Receipt Facility Screening Process**

follow the procedures specified in this test/QA plan and will comply with quality requirements in the Quality Management Plan (QMP) for TTEP.

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

The objective of the technology evaluation is to assess the performance of commercial sample screening technologies in detecting a variety of TICs and CW agents, with a reasonable range of sample types. The evaluation will be conducted in two phases. This test/QA plan addresses primarily Phase 1, which will consist of relatively simple tests to assess whether each technology can detect the target TICs and CW agents when they are present at predetermined hazardous concentrations in simulated samples. Phase 2 will consist of more extensive testing of selected technologies that appear most promising based on their cost, ease of use, and performance in the first phase. Phase 2 of testing will include a broader range of temperature and relative humidity (RH), realistic sample matrices, and assessment of interferent effects. The two phases combined will evaluate the qualitative accuracy of the tested technologies (i.e., the ability to identify hazardous samples), as well as the frequency of false positive and false negative indications, and the effects of expected interferents and normal temperature and RH variations. The ease of using each technology with personal protective equipment (PPE) such as heavy gloves or inside a glove box will also be assessed in Phase 2. These evaluations will not address the detection limit or dynamic range of each technology, but will use concentrations of the TICs and CW agents that a screening technology must be able to detect in the AHRF. This version of the test/QA plan focuses on procedures for the Phase 1 evaluation. Procedures for Phase 2 are described briefly, but will be specified by revision of this plan only after the first phase of testing is complete.

### **A6.1 Applicability**

This test/QA plan focuses on the evaluation of commercially available detection kits and instruments for qualitative screening of samples and sample containers to identify those contaminated with TICs or CW agents. The technologies suitable for this application may range from simple colorimetric test papers and kits, to continuous analyzers based on sophisticated

measurement principles such as ion mobility spectrometry (IMS), photoionization detection (PID), or flame spectrophotometry (FSP). Technologies of various degrees of complexity are required at different steps of the AHRF draft screening protocol,<sup>1</sup> so each technology will be evaluated based on its expected use in that protocol. Appendix A summarizes the technologies to be tested under this test/QA plan.

The toxic chemicals that may pose a threat in a field environment where samples are being screened may include both TICs and CW agents. Chemical agents having relatively low vapor pressures are of particular interest in this test, because of their potential persistence in samples or on sample containers. However, highly volatile TICs and CW agents are also included in testing under this plan, because they may still be present at a contaminated site when sample collection takes place. As described in Section A8.1, different Battelle laboratories will be used for testing with TICs and with CW agents, but essentially the same variety of screening technologies will be tested in both laboratories.

Technology evaluation requires a basis for establishing the performance of the tested technologies. For this evaluation the assessment of technology performance is based on the delivery of TIC or CW agent vapors, or the preparation of samples containing known quantities of the target TICs or CW agents.

## **A6.2 Scope**

The overall objective of this technology evaluation is to evaluate the performance of the screening technologies with selected TICs and CW agents under a realistic range of conditions and procedures of use. The performance parameters on which all screening technologies will be evaluated in each phase of testing under this test/QA plan are listed below:

Phase 1 and Phase 2:

- Analysis time
- Accuracy of identifying hazardous samples
- False positive/false negative rates
- Ease of use
- Data output
- Cost

Phase 2 Only:

- Temperature and humidity effects
- Interference effects

These performance parameters apply to all screening technologies tested, though the nature of the parameter may differ from one technology to another. For example, the analysis time needed to screen a sample is important for any screening technology, because it determines the throughput rate at which samples can be screened. However, analysis time for (e.g.) a colorimetric paper test kit will probably be limited by the physical manipulation of the samples and the kit by the operator, whereas analysis time for a continuous analyzer will be determined by the instrumental response time and recovery time. Operational factors such as ease of use, data output, and cost will be assessed by observations of the test personnel and through inquiries to the technology vendors.

For those screening technologies that provide more than a simple yes/no indication of the presence of a chemical hazard (e.g., IMS, FSP, and PID instruments), one additional performance parameter will be addressed:

- Repeatability

This factor refers to the precision of indications and alarm responses provided by these instruments in the successive challenges with TICs and CWAs. This performance parameter will be evaluated in both Phase 1 and Phase 2 of testing.

The testing to be conducted in Phase 1 is specified in this test/QA plan, and will be based on prepared samples to represent the screening process. Phase 2 evaluation procedures will be defined by revision of this plan after completion of Phase 1. Phase 2 will use realistic samples and sample containers, and will include evaluation of interference effects and a range of temperature and RH representative of field conditions. Testing will be done both with TICs and CW agents, though different laboratory facilities may be used for these two types of chemicals depending on the chemical concentrations and samples used.

Because of the nature of the test activities under this test/QA plan, the screening technologies will be operated by Battelle staff in all testing. Each technology will be used according to the appropriate instructions or operator's manuals for their instrument, and if

needed the technology vendor will be called upon to train Battelle staff in the correct use of the technology. In such a case, Battelle evaluation staff will review all written instructions and manuals before receiving training from the vendor. The Battelle evaluation staff will note the clarity, completeness, and adequacy of the written documentation provided. When the vendor is satisfied that Battelle staff are fully trained in operating their technology, the vendor will be required to attest in writing to that training for the purpose of this technology evaluation.

The primary means of data collection in this screening technology evaluation will be manual recording of readings or indications when the technologies are challenged with test samples. Uniform data recording sheets will be used in all tests for this purpose. In the event that a technology (e.g., a continuous analyzer) allows electronic recording of data, then such recording may be implemented to augment the primary manual data.

### **A6.3 Schedule**

Testing under this test/QA plan is expected to begin in May, 2006. It is anticipated that about three weeks will be required to complete the Phase 1 testing of up to 25 screening technologies. After revision of this test/QA plan to define the Phase 2 test procedures, approximately one month will be needed for the Phase 2 testing.

## **A7 QUALITY OBJECTIVES**

The main objective of the technology evaluation is to assess the performance of commercial screening technologies with realistic screening conditions and samples. This evaluation will rely on the preparation of samples containing CW agents or TICs at hazardous concentrations. Sample quality will rely primarily on the use of high quality source materials for the CW agents and TICs. In addition, reference analyses will be conducted on prepared samples, to confirm that prepared vapor concentrations are within  $\pm 30\%$  for CW agents and  $\pm 20\%$  for TICs, and that concentrations in liquid samples or extracted from surface samples are within  $\pm 15\%$  of the target concentrations. Samples found to be outside this accuracy specification will be remade before being used in testing.



## **A8 SPECIAL TRAINING/CERTIFICATION**

These tests are expected to be conducted at Battelle facilities in Columbus and West Jefferson, Ohio. Those facilities are described below. Alternative facilities could also be used, provided those facilities meet all the requirements for safety, security, and testing capability established by this plan.

### **A8.1 General Site Description**

Battelle has two primary campuses that will be used to conduct the screening technology evaluation. The main chemistry laboratories for non-chemical surety material testing are located at Battelle's King Avenue headquarters in Columbus, Ohio. Testing with the non-surety material – TICs and interferences – will be conducted in those King Avenue laboratories. These facilities have the sample preparation and analysis equipment needed to conduct the tests described in this plan.

Battelle's West Jefferson facility is an 1,800-acre research campus located within a tract of Battelle-owned land in a rural area approximately 17 miles west of downtown Columbus, Ohio. Testing with CW agents under this test/QA plan will use either the Medical Research and Evaluation Facility (MREF) or the Hazardous Materials Research Center (HMRC) at West Jefferson, both of which conduct research with chemical surety material (CSM).

Battelle's Medical Research and Evaluation Facility (MREF) is a Department of Defense laboratory-scale facility conducting research with chemical and biological agents. The MREF is licensed to ship, receive, and handle select agents, as defined by the Centers for Disease Control and Prevention. The facility maintains state-of-the-art equipment and professional and technical staffing expertise to safely conduct testing and evaluation of hazardous chemical and biological materials.

The MREF and its personnel have capability for storing and safely handling CW agents. Handling of CW agents at the MREF is detailed in the following standard operating procedures (SOP): MREF SOP I-002 *Storage, Dilution, and Transfer of GA, GB, GD, GF, TGD, VX, HD, HL, HN and L when CA Concentration/Quantity is Greater than Research Dilute Solution*

(RDS), MREF SOP I-003 *Receipt, Transfer, Storage, and Use of Research Dilute Solution (RDS)*, and MREF SOP I-004 *Disposal of Chemical Agent*.

Battelle's HMRC is an ISO 9001 certified facility that 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. The HMRC can safely store and handle 3-quinuclidinyl benzilate (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 (AsH<sub>3</sub>; SA), cyanogen chloride (CK), hydrogen cyanide (AC), phosgene (CG), perfluoroisobutylene (PFIB), as well as agent simulants, Class A poisons, and toxins (e.g., T-2 toxin). In accordance with SOP HMRC II-001, "*Determination of Delivered CW Agent*," agent concentrations in this work will be determined according to SOP HMRC IV-056 for "*Operation and Maintenance of Gas Chromatographs and for the Analysis of Solutions Containing GA, GB, GD, GF, HD, and VX by Gas Chromatography*."

The HMRC complex consists of approximately 10,000 ft<sup>2</sup> which includes the Hazardous Materials Laboratory (containing 11 chemical hoods certified for CSM) and the Large Item Test Facility (LITF), which together provide approximately 2,000 ft<sup>2</sup> of laboratory space and 100 linear ft of CSM-approved filtered hoods for working with neat (pure) CSM; about 630 ft<sup>2</sup> of research dilute solution (RDS, i.e., diluted chemical agent) laboratory space, including four fume hoods; and approximately 2,100 ft<sup>2</sup> of laboratory support areas, including environmental monitoring, emergency power supplies, and air filter systems.

## **A8.2 Site Operations**

Battelle operates its certified chemical surety facilities in compliance with all applicable Federal, state, and local laws and regulations, including Army Regulations. Battelle's facilities are certified through inspection by personnel from the appropriate government agency. Battelle is certified to work with CSM through its Bailment Agreement DAAD13-H-03-0003 with the U.S. Army Research, Development & Engineering Command (RDECOM). RDECOM officials and the Army Material Command Inspector General for Chemical Surety Sites regularly inspect

Battelle’s facilities to ensure that Battelle continues to operate its chemical surety laboratories in accordance with all applicable federal regulations. Our chemical agent facilities and attendant certifications are listed in Table 1. Battelle's agent stocks will be analyzed prior to testing to verify the purity of the agent used to make the test samples. Only CW agents with purity greater than 80 percent will be used in this program.

**Table 1. Battelle Facilities for CW Agent Testing**

| Facility                                 | Materials | Level   | Certification  |
|--|-----------|---|--|
| Medical Research and Evaluation Facility | CW Agents | Chemical Surety Materiel (CSM) (Neat)<br>RDT & E (Dilute) | United States of America Medical Research Materiel Command (USAMRMC) Contract No. WB1XWH-05-D-0001 |
| Hazardous Materials Research Center      | CW Agents | Chemical Surety Materiel (CSM) (Neat)<br>RDT & E (Dilute) | Bailment Agreement No. DAAD13-H-03-0003  |
| Analytical Chemistry Laboratory          | CW Agents | RDT & E (Dilute)  | Bailment Agreement No. DAAD13-H-03-0003  |

**A8.3 Training**

Because of the hazardous materials involved in this technology evaluation, documentation of proper training and certification of the test personnel is mandatory before testing takes place. The Battelle Quality Assurance Manager, or a designate, must assure that documentation of such training is in place for all evaluation personnel before allowing evaluation to proceed.

All participants in this evaluation (i.e., Battelle, EPA, and vendor staff) will adhere to the security, health, and safety requirements of the Battelle facility in which testing will be performed. Access to any restricted areas of the test facility will be limited to staff who have met all the necessary training and security requirements. The existing access restrictions of the test facility will be followed, i.e., no departure from standard procedures will be needed for this evaluation.

All visitors to the test facility will be given a site-specific safety briefing prior to the start of any test activities. This briefing will include a description of emergency operating procedures, and the location and operation of safety equipment (e.g., fire alarms, fire extinguishers, eye washes, exits). Evaluation procedures must follow all safety practices of the test facility at all

times. Any report of unsafe practices in this evaluation, by those involved in the evaluation or by other observers, shall be grounds for stopping the evaluation until the Quality Assurance Manager and testing personnel are satisfied that unsafe practices have been corrected.

## **A9 DOCUMENTATION AND RECORDS**

The records for this TTEP evaluation include in this test/QA plan, laboratory record books (LRB), test data collection forms, electronic files (both raw data and spreadsheets), and the final evaluation reports. All of these records will be maintained by the Task Order Leader or his designee during the evaluation and will be transferred to permanent storage at the conclusion of the evaluation. All Battelle LRBs are stored indefinitely, either by the Task Order Leader or Battelle's Records Management Office. EPA will be notified before disposal of any files. Section B10 further details the data recording practices and responsibilities.

All written records must be in ink. Any corrections to notebook or data form 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. All data records will be reviewed prior to use in any calculations, evaluation, or reporting, as described in Section D1 of this test/QA plan.

Documentation of training related to technology testing, field testing, data analysis, and reporting is maintained for all Battelle technical staff in training files at their respective office locations. Any training provided for this evaluation by a vendor of a screening technology will be included in the training record for the relevant staff. The Battelle Quality Assurance Manager may verify the presence of appropriate training records prior to the start of testing. Battelle technical staff will have a minimum of a bachelor's degree in science/engineering or have equivalent work experience.

## **B1 EXPERIMENTAL DESIGN**

### **B1.1 General Test Design**

A description of the performance parameters to be characterized in Phase 1 and the rationale for their inclusion is provided in Section B1.2. The chemicals of interest that will be used in the Phase 1 evaluation are discussed in Section B1.3. The Phase 1 test matrix and schedule are discussed in Sections B1.4 and B1.5, respectively. The technologies to be evaluated are identified in Appendix A.

### **B1.2 Performance Parameters**

The key performance parameters to be evaluated for all selected screening technologies in the Phase 1 technology evaluation are:

- Analysis time
- Accuracy of identifying hazardous samples
- False positive/false negative rates

In addition, technologies providing more than a simple yes/no response will be evaluated for the following performance parameter, using the responses displayed by these devices:

- Repeatability

These performance parameters are defined, and general test procedures are outlined, in Sections B1.2.1 to B1.2.4. Specific test procedures to evaluate these parameters with different sample types are in Sections B2.1.1 to B2.1.3. In addition to these key performance parameters, operational characteristics of the units will be evaluated. These operational characteristics include:

- Ease of use
- Data output
- Cost.

The operational characteristics are summarized in Section B1.2.5, and will be evaluated based on operator observations and available information on the screening technologies.

**B1.2.1 Analysis Time**

Analysis time is defined as the time needed to screen a single sample or group of samples with an individual technology. This parameter is important because it determines the sample throughput that may be achieved in an AHRF. In this evaluation similar screening technologies will be tested with equivalent sets of prepared samples, and the speed with which each sample set can be screened with each technology will be determined based on the recorded start and end times of screening. The relative analysis times of the various technologies will then be compared. For the continuously operating electronic technologies (IMS, FSP, PID) the time to reach a response and the time to return to baseline after a challenge ends will both be recorded, as these will largely determine the overall analysis time of such devices.

**B1.2.2 Accuracy of Hazard Identification**

Accuracy in this context is defined as the ability of a screening technology to identify hazardous samples, so that they can be properly handled to minimize risk to laboratory personnel. Accuracy will be measured in terms of the percentage of prepared hazardous samples that are correctly identified as hazardous by the technology in question. In the Phase 1 evaluation similar screening technologies will be tested with equivalent sets of prepared vapors or simulated samples, and the accuracy of hazard identification will be determined for each technology. The accuracy results for the various technologies will then be compared.

**B1.2.3 False Positive/False Negative Rates**

A false positive screening result occurs when a technology incorrectly identifies a safe sample as being hazardous. Such a result causes unnecessary expense by requiring special sample handling and analysis procedures, but does not jeopardize the health of laboratory staff. A false negative screening result occurs when a technology incorrectly identifies a hazardous sample as being safe. This result can endanger laboratory staff, can result in the contamination and consequent shutdown of laboratory facilities, and can cause extensive expense due to the cleanup of contaminated facilities.

In the Phase 1 evaluation the challenge sample set will include blank samples, i.e., sample matrices not spiked with any TIC or CW agent. Erroneous responses identifying such samples as hazardous will be denoted as false positives. On the other hand, the absence of a hazard indication with a known hazardous vapor or prepared sample will be denoted as a false negative. The false positive and negative rates will be calculated as the percentage of samples producing the respective erroneous result.

#### **B1.2.4 Repeatability**

The responses provided by some technologies undergoing evaluation (e.g., IMS, FSP, PID instruments) are likely to be more complex than the yes/no indications provided by the simple test papers and kits that comprise the majority of technologies evaluated under this plan. Those more complex responses may include intensity readings of a semi-quantitative nature (e.g., High/Medium/Low indications; bar graph indications; approximate concentration values; etc.), visible or audible alarms, or other displays. For any technology providing such indications, all such indications will be recorded and the repeatability or uniformity of such indications will be reported.

#### **B1.2.5 Operational Characteristics**

Key operational characteristics of the screening technologies will be evaluated in both Phase 1 and Phase 2 by means of the observations of test operators, and by inquiry to the respective vendors.

Ease of use will be assessed by operator observations, with particular attention to the conditions of use during screening. In particular, each technology will be used by an operator wearing both nitrile and heavy butyl gloves, and by an operator wearing nitrile gloves alone. This assessment will be done in the course of the evaluation of other performance parameters with TICs or CW agents, i.e., no additional test procedures will be designed specifically to address only the operational characteristics.

For each screening technology, the type of indication or data output will be noted (e.g., color change, intensity of color change, low/med/high indication, audio or visual alarm,

quantitative measure of concentration, etc.). The clarity of the indication will be assessed, as well as the stability of that indication once established, and the availability of multiple forms of data output or display also will be noted, e.g., the availability of both a visual display and an analog voltage output from a continuous analyzer.

Costs for each technology will be assessed based on the purchase and operational costs of the technologies as tested. This technology evaluation will not be of sufficient duration to test long-term maintenance or operational costs of the technologies. Estimates for key maintenance items will be requested from the vendors as necessary.

### **B1.3 Chemical Test Compounds**

Table 2 shows the entire list of target TICs and CW agents identified by EPA as being of potential interest for the AHRF screening technology evaluation. The chemicals that will be used in technology evaluation under this test/QA plan are shown in bold type; footnotes to Table 2 and the following discussion summarize the reasons for selection of those chemicals, and exclusion of the others.

The screening technologies to be evaluated will not be able to distinguish closely similar chemicals (e.g., the G series nerve agents, or H series blister agents) from one another. Consequently, for these two classes of agents, a single representative chemical (GB and HD, respectively) will be used in testing, as shown in Table 2. GB will be used as the representative G series agent because of its relatively high toxicity, water solubility, volatility, and/or volume of production relative to the other G agents.<sup>(3)</sup> HD will be used as the representative H series agent because of its availability in relatively high purity and its large volume of production relative to the other H series agents.<sup>(3)</sup> The pesticides chlorpyrifos and methyl parathion will not be used in this evaluation, because almost none of the screening technologies to be tested claim to be able to detect those compounds. Both the gaseous blood agent hydrogen cyanide (AC) and “cyanide” are on EPA’s list of potential chemicals of interest; AC will be used as a vapor phase target chemical and potassium cyanide will be used in aqueous solution for testing of technologies applicable to water samples. The TIC listed as “arsenic” will be represented by the hazardous gas arsine (AsH<sub>3</sub>, designated SA); however, the CW agent Lewisite also contains arsenic and



will be used in liquid and surface sample screening. A fluoride salt in water will represent the TIC “fluoride”, and the class of “oxidizers” will be represented by hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>).

**Table 2. TICs and CW Agents for Evaluation of Screening Technologies**

| <b>Chemical Category</b>          | <b>Subcategory</b> | <b>Target Chemicals<sup>a</sup></b>   |
|-----------------------------------|--------------------|---|
| Chemical warfare (CW) agents      | Nerve agents       | Tabun (GA), <b>Sarin (GB)</b> , <sup>b</sup> Soman (GD); <b>VX</b>  |
|                                   | Blister agents     | Mustard (H), <b>Distilled mustard (HD)</b> , <sup>c</sup> Nitrogen mustard (HN <sub>x</sub> ), Sulfur mustard (HT); <b>Lewisite (L)</b>   |
|                                   | Blood agents       | <b>Hydrogen cyanide (AC)</b> ; <b>Cyanogen chloride (CK)</b>  |
|                                   | Choking agents     | <b>Phosgene (CG)</b>  |
| Toxic industrial chemicals (TICs) | NA <sup>d</sup>    | <b>Cyanide<sup>e</sup></b><br><b>Arsenic<sup>f</sup></b><br><b>Chlorine</b><br><b>Fluoride<sup>g</sup></b><br><b>Oxidizers<sup>h</sup></b><br><b>Hydrogen sulfide</b><br>Chlorpyrifos<br>Methyl parathion |

a: Chemicals shown in bold type will be used for technology evaluations under this test/QA plan.

b: Representative of G series agents

c: Representative of H series agents

d: Not applicable

e: As potassium cyanide (KCN) in aqueous solution; vapor phase addressed by hydrogen cyanide (AC).

f: As arsine (AsH<sub>3</sub>) gas (designated SA); arsenic also present in Lewisite (L) .

g: As sodium fluoride in water.

h: Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) will be used as a representative oxidizer.

As feasible, the target chemicals listed in Table 2 will be tested in multiple sample matrices (i.e., vapor, liquid, solid). However, some potential combinations of TIC/CW agent and sample matrix may not be realistic. An example of an unrealistic combination would be VX in the vapor phase; the vapor pressure of this agent is so low that the threat in a sample screening situation would lie in its presence in a liquid sample or on a surface. The actual samples and TIC/CW agent concentrations to be used in Phase 1 testing are specified in Section B1.6, and Appendix A indicates which technologies are planned to be tested with samples of each matrix type. Blank samples of each matrix type will also be used in testing, with blanks comprising at least 10 percent of all samples of each matrix type.

**B1.4 Test Matrix**

Table 3 summarizes the evaluations to be conducted in the Phase 1 screening technology evaluation. Shown are the performance parameters, the objective, and the basis for comparison on which that performance parameter will be addressed.

**Table 3. Summary of Evaluations to be Conducted in Screening Technology Evaluation**

| <b>Performance Parameter</b>      | <b>Objective</b>   | <b>Comparison Based On</b>   |
|-----------------------------------|--|--|
| Analysis Time                     | Determine speed of screening process                                   | Time needed to screen individual samples or sample sets                                  |
| Accuracy                          | Characterize effectiveness of technology to identify hazardous samples | Known composition of prepared samples  |
| False Positive and Negative Rates | Characterize frequency of erroneous screening results                  | Blank samples (for false positives) and prepared hazardous samples (for false negatives) |
| Repeatability                     | Characterize precision of response                                     | Responses observed with replicate challenges   |

**B1.5 Test Schedule**

This evaluation will be organized around the types of samples screened by each screening technology. For example, all technologies capable of screening for TICs and CW agents in the vapor phase will be tested by consistent procedures with all of those chemicals. Each technology will first be tested with a single vapor-phase TIC, before moving on to each of the other TICs in sequence, and ultimately to the CW agents. Similarly all technologies capable of screening liquid or surface samples will be evaluated by consistent procedures with the appropriate target chemicals in water samples, and technologies for surface sampling will be evaluated using material coupons contaminated with appropriate agents. The expected duration of each of these components of the Phase 1 evaluation, once test preparations are complete, is three weeks for vapor-phase evaluation; one week for liquid phase evaluation; and one week for surface evaluation. The duration of the vapor phase evaluation is driven largely by the greater number of TICs and CW agents used in that component of the evaluation, relative to the others, and to the need to modify the vapor delivery system in moving from TICs to the CW agents. However, these three components of the evaluation can be conducted simultaneously, so that the overall

screening process is expected to take less than one month. The evaluation procedures are described in the next section.

## **B2 TEST METHODS**

### **B2.1 Phase 1 Test Procedures**

The screening technologies undergoing evaluation may be applicable to three different scenarios:

- chemical vapors escaping from samples or sample containers
- chemical contamination of liquid samples
- chemical contamination of surfaces or solid samples.

In the Phase 1 evaluation under this test/QA plan, each screening technology will be tested under the scenarios for which it is applicable. The test procedures to be followed in each scenario are presented in the following subsections.

#### **B2.1.1 Vapor Phase Testing**

Screening technologies will be evaluated based on their ability to respond to TICs and CW agents in the vapor phase, using a test apparatus represented schematically in Figure 3. This apparatus has been used in previous evaluations of portable chemical detectors under the TTEP program. The test system consists of a vapor generation system, a Nafion® humidifier, two challenge plenums, a clean air plenum, RH sensors, thermocouples, and mass flow meters. The challenge vapor or gas is generated by the vapor generation system. The appropriate vapor generator, typically a compressed gas cylinder or diffusion cell, will be selected for the TIC or CW agent of interest, respectively. The challenge vapor from the vapor generation system will then mix with the dilution air and flow into the challenge plenum.

As illustrated in Figure 3, the test apparatus allows the temperature and relative humidity (RH) of the challenge gases to be adjusted, and allows multiple challenge concentrations or compositions to be delivered. However, in the Phase 1 testing, these capabilities of the test apparatus will not be exploited: all Phase 1 vapor tests will be conducted with the TIC or CW agent vapor diluted in clean dry air at room temperature, and only a single challenge

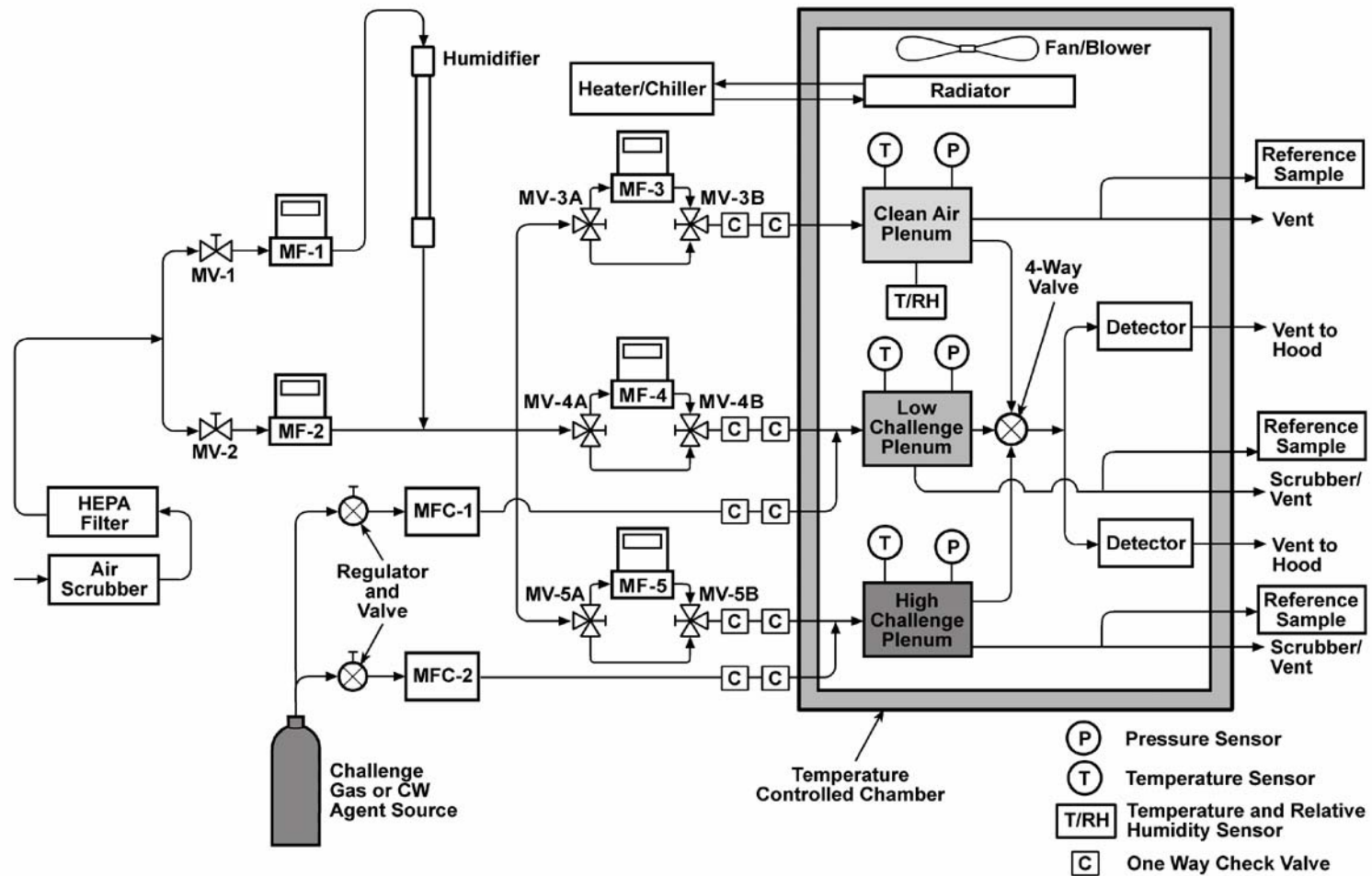


Figure 3. Test System Schematic

concentration will be used. (In the subsequent Phase 2 evaluation, these capabilities will be employed to test over a realistic range of temperature and RH, and to introduce potential interferent vapors along with the target TICs and CW agents.) To conduct Phase 1 evaluation of a screening technology, the test apparatus will be used to establish a flow of clean air through the clean air plenum (Figure 3), and an equal flow of air containing a constant concentration of the target TIC or CW agent through one of the other plenums. Each screening technology will be placed in a bag or other enclosure, which will be connected to the 4-way valve shown in the figure, and through which the clean air or challenge gas will flow before exiting to an appropriate agent trap and/or hood. An exception is that for technologies which draw their own sample flow, such as a PID or IMS instrument, appropriate direct connection will be made to allow the instrument to sample from the air flow. The exact means of connecting such instruments to the test apparatus in Figure 3 will vary depending on the instrument's inlet design, and will be established to prevent over- or under-pressurization, while assuring a sufficient flow of challenge gas to the instrument.

Each screening technology will first sample or be exposed to the clean air flow, and any response or indication from the screening technology will be noted. After this background measurement, the four-way valve will be switched to the challenge plenum to deliver the challenge mixture to the subject technology. Switching between the clean air and challenge gas flows will be rapid, and the residence time of gas in the test system will be short, so that the analysis time determined for each screening technology will not be biased by the limitations of the test apparatus. The reference methods described in Section B4 will be used to quantify the TIC or CW agent concentration in the clean air plenum and the challenge plenum to provide a cross-check of the concentrations measured. The sequence of exposure to clean air followed by exposure to the challenge gas mixture will be carried out three successive times for each screening technology with each gaseous TIC and vapor-phase CW agent. For most of the screening technologies tested, this will require using a new piece of color indicating paper, a new color indicating tube, or a new test kit for each of the three test runs.

Table 4 summarizes the challenge concentrations for each of the target TICs and CW agents to be used in vapor phase testing. Shown in Table 4 are the TIC and CW agent identities,

the challenge concentrations to be used, and the basis for the chosen concentrations. Except in the case of cyanogen chloride (CK) the target concentrations shown are all Acute Exposure Guideline Level (AEGL) values, and specifically AEGL-2 values for a 10-minute exposure.<sup>(4)</sup> The AEGL-2 value is defined as the airborne concentration of a substance above which it is predicted that the general population, including susceptible individuals, could experience irreversible or other serious, long-lasting adverse health effects or an impaired ability to escape. AEGL values are established specifically for the protection of personnel, and thus are appropriate target values for AHRF screening. For CK, no AEGL values have been established, so the target value is based on the Temporary Emergency Exposure Limit (TEEL) for that chemical, and specifically the TEEL-2 value for a 15-minute exposure.<sup>(5)</sup> The TEEL-2 value is defined as the maximum concentration in air below which it is believed nearly all individuals could be exposed without experiencing or developing irreversible or other serious health effects or symptoms that could impair their abilities to take protective action. Delivery of the vapor phase challenges will target the concentrations shown in Table 4, however delivered concentrations will be deemed acceptable if they are within  $\pm 20\%$  of the target value.

**Table 4. Challenge Concentrations for Vapor Phase Testing**

| TIC/CW Agent                        | Concentration <sup>a</sup>           | Basis for Concentration <sup>b</sup> |
|-------------------------------------|--------------------------------------|--------------------------------------|
| Hydrogen cyanide (AC)               | 17 ppm (18.7 mg/m <sup>3</sup> )     | AEGL-2 value                         |
| Cyanogen chloride (CK)              | 0.4 ppm (1 mg/m <sup>3</sup> )       | TEEL-2 value                         |
| Phosgene (CG)                       | 0.6 ppm (2.4 mg/m <sup>3</sup> )     | AEGL- 2 value                        |
| Chlorine (Cl <sub>2</sub> )         | 2.8 ppm (8.4 mg/m <sup>3</sup> )     | AEGL- 2 value                        |
| Arsine (SA)                         | 0.3 ppm (1 mg/m <sup>3</sup> )       | AEGL- 2 value                        |
| Hydrogen sulfide (H <sub>2</sub> S) | 41 ppm (57.4 mg/m <sup>3</sup> )     | AEGL- 2 value                        |
| Sarin (GB)                          | 0.015 ppm (0.087 mg/m <sup>3</sup> ) | AEGL- 2 value                        |
| Sulfur mustard (HD)                 | 0.09 ppm (0.6 mg/m <sup>3</sup> )    | AEGL- 2 value                        |

a: At normal temperature and pressure, 1 ppm = (MW)(0.0409) milligrams per cubic meter (mg/m<sup>3</sup>), where MW is the molecular weight of the compound.

b: AEGL = Acute Exposure Guideline Level; TEEL = Temporary Emergency Exposure Limit.

In addition to the TIC and CW agent challenges listed in Table 4, each screening technology will be tested in three successive trials with blank (i.e., clean) air as the challenge. Thus, for a technology that is applicable to all eight vapor phase TICs and CW agents in Table 4,

the total vapor phase test regimen in Phase 1 will consist of 24 test runs and 3 blank runs (i.e., blanks will be at least 11% of all trials in vapor phase testing).

### **B2.1.2 Liquid Sample Testing**

Screening technologies that are applicable to liquid samples will be tested with prepared samples of selected CW agents (GB, HD, VX, and L) and TICs (cyanide, fluoride, and hydrogen peroxide) at known concentrations. Each selected TIC or CW agent will be prepared at a single concentration, in one or more appropriate solvents that assure stability of the challenge samples, and that mimic sample matrices that might be encountered in the field. Each liquid challenge sample will contain a single TIC or CW agent, i.e., no mixed samples will be prepared. Each screening technology capable of screening liquid samples will then be tested three times with each combination of target chemical and appropriate solvent(s) for that chemical. This evaluation will typically involve applying a drop of the liquid sample to the test paper, test kit, or analyzer, or immersing a portion of the kit in the sample, and observing the response. Three corresponding analyses with the pure solvent will also be conducted with each screening technology, as a baseline test. Table 5 lists the target chemicals, the appropriate solvents to be used, and the planned concentrations to be used in the evaluation of liquid screening technologies.

Because the purpose of the AHRF screening protocol is to protect analytical personnel from toxic exposures in handling and analyzing samples, the use of challenge concentrations taken from drinking water standards is not appropriate. It is unrealistic to assume that an analyst would ever ingest a sample provided for analysis. Furthermore, drinking water standards assume the ingestion of several liters of water per day, and lead to allowable concentrations that are too low to be detected by sample screening technologies (e.g., concentrations in the low  $\mu\text{g/L}$ , or part per billion (ppb) range for the CW agents). As a result, for this evaluation, the levels set by the U.S. Government for samples in Research, Development, Test, and Evaluation (RDT&E) laboratories will be used as a starting point for the CW agents. Allowable RDT&E levels are set specifically to protect laboratory staff from hazards associated with spillage or inadvertent contact with hazardous samples, and thus fit the intent of the AHRF screening protocol. For this

**Table 5. Challenge Concentrations for Liquid Phase Testing**

| Chemical            | Concentration          | Solvent <sup>e</sup> | Basis for Concentration     |
|---------------------|------------------------|----------------------|-----------------------------|
| Sarin (GB)          | 1 mg/ml <sup>a</sup>   | IPA; hexane          | 0.5 x RDT&E limit           |
| Sulfur Mustard (HD) | 1.5 mg/ml <sup>b</sup> | IPA; hexane          | 0.15 x RDT&E limit          |
| VX                  | 0.1 mg/ml <sup>c</sup> | Water; hexane        | 0.1 x RDT&E limit           |
| Lewisite (L)        | 2.5 mg/ml <sup>d</sup> | IPA; hexane          | 0.5 x RDT&E limit           |
| Cyanide             | 0.7 mg/ml              | Water                | 0.1 x Oral LD <sub>50</sub> |
| Fluoride            | 0.7 mg/ml              | Water                | 0.1 x Acute Toxic Dose      |
| Hydrogen peroxide   | 10% (100 mg/ml)        | Water                | ATSDR Guidelines            |

a: Total quantity of agent present at any time will be 20 mg or less.

b: Total quantity of agent present at any time will be 100 mg or less.

c: Total quantity of agent present at any time will be 10 mg or less.

d: Total quantity of agent present at any time will be 50 mg or less.

e: IPA = isopropyl alcohol; water used for VX and cyanide solutions will be properly pH buffered.

test, consistent with the usual practice in Battelle’s laboratories, liquid concentrations of the CW agents will be kept at a fraction of their respective RDT&E limits. As the footnotes to Table 5 indicate, there are also RDT&E limits on the total amount of CW agents that can be present in the test laboratory; those limits will be adhered to in all evaluations.

The solvents listed in Table 5 were chosen to represent both polar and non-polar potential sample matrices, including a pure hydrocarbon solvent (hexane) that represents potential “oily” sample matrices such as fuels. Although water is likely to be the most common type of liquid sample encountered in the field, most of the target CW agents are not stable enough in water to assure reliable testing. Isopropyl alcohol (IPA) will be used as an alternate polar solvent. Water will be used as a solvent only for VX in this testing, because of the stability of that agent in water when properly pH buffered.

For the TICs, concentrations suitable for testing of liquid sample screening technologies are not as well defined. Primarily this is because dermal exposure is not as well studied or understood as inhalation or ingestion pathways. For example, although the U.S. Agency for Toxic Substances and Disease Registry (ATSDR) sets Minimum Risk Levels (MRLs) for many chemicals for the inhalation and oral ingestion pathways, no MRLs have been set for dermal exposures. As a result, the aqueous challenge concentrations for cyanide, fluoride, and hydrogen peroxide in Table 5 are based on reasonable assumptions and/or the interpretation of information on toxic effects. The concentration shown for cyanide is based on the assumption



that a water sample of 50 ml volume, containing an amount of the target chemical equal to one-tenth of the oral dose that would be lethal to half the population ( $LD_{50}$ ), is spilled on the skin and that all of the chemical is then absorbed into the body through the skin. For cyanide, with an  $LD_{50}$  of 5 mg/kg of body weight, and an assumed body weight of 70 kg, the total mass of cyanide would be 35 mg, and the concentration in a 50 ml sample would be 0.7 mg/ml, as shown in Table 5. The cyanide solutions will be buffered at a slightly basic pH to assure stability of the cyanide in solution. Similarly, the acute toxic dose of fluoride is generally reported as 3 to 5 mg/kg. Taking the higher number, and making the same one-tenth adjustment and assumptions as above for cyanide, results in the 0.7 mg/ml concentration shown in Table 5. For hydrogen peroxide, the concentration of 10% (by weight) in Table 5 is identified by ATSDR as being strongly irritating and potentially corrosive to skin.

In addition to the chemical and CW agent challenges listed in Table 5, each screening technology will be tested with three blank samples of each solvent used to prepare any challenge solution. Thus, for a technology that is applicable to all seven chemicals in Table 4, the total liquid sample test regimen in Phase 1 will consist of 33 test samples (6 for each of the four CW agents plus 3 for each of the three other chemicals) and 9 blank samples (3 for each of the three solvents used). Thus, blank samples will be at least 21% of all trials in liquid testing.

### **B2.1.3 Solid Sample Testing**

Solid materials to be screened under the AHRF field protocol could potentially include sample containers, soil, or solid debris. However, the screening of primary sample containers and individual sample containers is the focus of the current draft AHRF protocol.<sup>1</sup> Consequently, in Phase 1 evaluation of screening technologies under this test/QA plan, the solid samples used will consist of glass slides contaminated with selected target chemicals. The use of this simple sample matrix will provide uniformity and efficiency in Phase 1 evaluation, and will avoid the complexities of selecting, preparing, and homogenizing samples in a soil or other complex matrix.

The target chemicals to be used in evaluation of screening technologies for solid samples will be the CW agents VX and Lewisite (L). These two chemicals were chosen for this

component of the evaluation because their low volatility assures that, if present, they will exist on material surfaces for extended periods of time. These chemicals will be placed in small but potentially hazardous amounts on glass slides or test coupons approximately 1 x 3 inches in size, by applying a solution of the agent dropwise onto the slide and allowing the solvent to evaporate away. The amount of VX and L applied to the test coupons for this evaluation will be based on the hazardous surface concentrations determined for these agents. Table 6 summarizes the surface loadings that will be used in this component of the evaluation.

**Table 6. Surface Loadings to be Used in Solid Sample Screening Evaluation**

| <b>Chemical</b> | <b>Surface Loading</b> | <b>Basis for Loading</b>    |
|-----------------|------------------------|-----------------------------|
| VX              | 0.2 mg/cm <sup>2</sup> | 0.1 x Skin LD <sub>50</sub> |
| Lewisite (L)    | 40 mg/cm <sup>2</sup>  | 0.1 x Skin LD <sub>50</sub> |

The surface loadings in Table 6 will be applied over a total area of approximately 5 cm<sup>2</sup>, and are based on one-tenth the LD<sub>50</sub> values for the respective agents. For example, for VX the skin LD<sub>50</sub> is 0.142 mg/kg, or 10 mg for a person weighing 70 kg, therefore the surface loading of VX to be used in testing is calculated as  $0.1 \times 10 \text{ mg} / 5 \text{ cm}^2 = 0.2 \text{ mg/cm}^2$ . For L, the corresponding skin LD<sub>50</sub> is approximately 30 mg/kg, or about 2 g for a person weighing 70 kg, and thus the resulting surface loading to be use in testing is  $0.1 \times 2,000 \text{ mg} / 5 \text{ cm}^2 = 40 \text{ mg/cm}^2$ . The actual application of agent to each coupon will be done by applying drops or spots of agent solution from a pipette in a regular pattern across a 2.2 cm x 2.2 cm area of the coupon.

The evaluation will be conducted by contacting (i.e., touching, wiping, pressing) the test coupon with the screening technology as required for use of the technology. Each screening technology applicable to solid samples will be tested three times with each of the two CW agents. Three test runs will also be conducted with each technology using blank glass coupons, as a baseline test. Thus a technology applicable to both VX and L on surface samples will be tested with three blank coupons and six loaded test coupons, i.e., blank samples will comprise at least one-third of all samples in surface sample testing.

### **B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS**

Samples for reference analyses will be analyzed in the same form as they are prepared, in most cases, as described in Section B4. In all cases the analyst will document the date and time when the sample was taken or the *in-situ* analysis was conducted, the identity of the TIC or CW agent being used, and the screening technology that was undergoing testing at the time the reference sample was taken. Written records of the analysis must be retained in the analyst's laboratory notebook, and in the form of any printouts, chromatograms, or summaries provided by the reference analytical instrumentation.

### **B4 REFERENCE METHODS**

#### **B4.1 Reference Sample Collection**

The vapor, liquid, and solid surface samples used in this technology evaluation will be generated as described in Section B2. Selected samples will be taken from these evaluation samples and analyzed by reference methods to confirm that the prepared samples are in fact close to the target challenge concentrations. The following sections describe how these reference determinations will be made.

Sample collection for liquid samples will involve direct analysis of the prepared liquid sample itself. Sample collection for vapor phase challenges may involve direct analysis of the challenge vapor, or collection of the target chemical from the challenge air stream using liquid reagent impingers, sampling bags, or sorbent traps. Sample collections for surface samples will involve extracting the target agent from the coupon surface with an appropriate solvent for analysis. The relevant approaches are noted for each TIC and CW agent in Section B3.2.

#### **B4.2 Laboratory Reference Methods**

Table 7 summarizes the reference methods to be used for determining the challenge concentrations of the target TICs and CW agents in the test. Listed in the table are the target TICs and CW agents, the sampling and analysis methods to be used for each compound, and the applicable sample matrix types of each method. References to the methods used are footnoted in Table 7. For the TICs cyanogen chloride and hydrogen cyanide, air samples will be injected

**Table 7. Planned Reference Methods for Target TICs and CW Agents**

| Analyte                     | Sample Matrix | Sampling Method  | Analysis Method                                    |
|-----------------------------|---------------|--|--|
| Hydrogen cyanide (AC)       | Vapor         | Air sample injected directly   | GC/FID <sup>a</sup>                                |
| Cyanogen chloride (CK)      | Vapor         | Air sample injected directly   | GC/FID <sup>b</sup>                                |
| Phosgene (CG)               | Vapor         | Capillary gas chromatography with direct injection   | GC with mass selective detector (MSD) <sup>c</sup> |
| Chlorine (Cl <sub>2</sub> ) | Vapor         | Continuous electrochemical detector with chlorine-specific sensor                              | Continuous detection <sup>d</sup>                  |
| Arsine (SA)                 | Vapor         | Capillary gas chromatography with direct injection   | GC/MSD <sup>e</sup>                                |
| Hydrogen sulfide            | Vapor         | Continuous electrochemical detector  | Continuous detection <sup>d</sup>                  |
| Sarin (GB)                  | Vapor         | Whole air sample collected in gas sampling bag, or agent selectively collected on sorbent trap | GC/FPD <sup>f</sup>                                |
| Sulfur mustard (HD)         | Vapor         | Whole air sample collected in gas sampling bag, or agent selectively collected on sorbent trap | GC/FPD <sup>f</sup>                                |
| VX                          | Surface       | Extract from surface into DI water   | GC/FPD <sup>f</sup>                                |
| Lewisite (L)                | Surface       | Extract from surface into DI water   | HPLC <sup>g</sup>                                  |
| Sarin (GB)                  | Liquid        | Direct analysis of liquid sample   | GC/FPD <sup>f</sup>                                |
| Sulfur Mustard (HD)         | Liquid        | Direct analysis of liquid sample   | GC/FPD <sup>f</sup>                                |
| VX                          | Liquid        | Direct analysis of liquid sample   | GC/FPD <sup>f</sup>                                |
| Lewisite (L)                | Liquid        | Direct analysis of liquid sample   | HPLC <sup>g</sup>                                  |
| Cyanide                     | Liquid        | Direct analysis of liquid sample   | Ion chromatography                                 |
| Hydrogen peroxide           | Liquid        | Direct analysis of liquid sample   | Test kit <sup>h</sup>                              |
| Fluoride                    | Liquid        | Direct analysis of liquid sample   | Ion chromatography                                 |

- a: Reference 6.
- b: Reference 7.
- c: By adaptation of method in reference 8.
- d: Commercially available detector, e.g., Draeger MiniWarn, Jerome 631-X, or Model 860 electrochemical sensor.
- e: Reference 8.
- f: These measurements governed by HMRC SOPs HMRC-IV-056 and -IV-067.
- g: This measurement governed by HMRC SOP HMRC-IV-057.
- h: Hach HYP-1 H<sub>2</sub>O<sub>2</sub> Test Kit, or similar.

directly for determination by gas chromatography (GC) with flame ionization detection (FID).<sup>(6,7)</sup> Arsine will be determined by a gas chromatographic method with a capillary column and mass selective detection (MSD), using samples collected in gas sampling bags from the test apparatus.<sup>(8)</sup> A retention time of about seven minutes is expected for arsine, allowing repeated analysis within each test procedure. The method for phosgene will be based on GC with mass selective detection (MSD), similar to that for arsine.<sup>(8)</sup> Chlorine will be determined by a

commercially available continuous electrochemical analyzer with a chlorine-specific sensor (Draeger Mini Warn, or similar). Hydrogen sulfide will be determined with a commercial continuous monitor based on electrochemical detection of sulfide ion (e.g. Jerome 631-X, or similar). The CW agents GB, HD, and VX will be determined in the various sample matrices by GC with flame photometric detection (FPD). Determination of these CW agents will be conducted according to the procedures and quality requirements of HMRC Standard Operating Procedure (SOP) “*HMRC-IV-056, for Operation and Maintenance of Gas Chromatographs and for the Analysis of Solutions Containing GA, GB, GD, GF, HD, and VX by Gas Chromatography.*” Lewisite will be determined by high performance liquid chromatography (HPLC) according to the HMRC SOP for that analysis (*HMRC-IV-057*). Cyanide and fluoride will be determined as anions by ion chromatography, and hydrogen peroxide in aqueous samples will be determined by a commercially available colorimetric test kit. In all cases the QA/QC procedures defined in the appropriate SOP, reference, or manufacturer’s instructions will be followed and documented.

## **B5 QUALITY CONTROL REQUIREMENTS**

### **B5.1 Sample Acceptance**

Acceptance ranges for the vapor, liquid, and surface concentrations used to challenge the screening technologies were stated in Section A7, i.e.,  $\pm 30\%$  for CW agent vapors,  $\pm 20\%$  for TIC vapors, and  $\pm 15\%$  for all chemicals in liquid and on surface samples, based on reference analyses. Blank samples will not be subject to any reference analysis or acceptance criteria, as these samples (i.e., a clean air stream, pure solvents, or undosed clean coupons, for vapor, liquid, and surface samples, respectively) are deliberately prepared to contain no target chemicals.

### **B5.2 Performance Evaluation Audit**

The equipment needed for conducting the performance evaluation audit will consist of independent standards used to check the reference measurements that confirm challenge sample concentrations. The PE audit will be conducted only for five of the six vapor-phase TICs, and for the aqueous phase TICs cyanide, fluoride, and hydrogen peroxide. No PE audit will be

conducted for the vapor phase TIC cyanogen chloride (CK), since the standard for this TIC is made by Battelle from pure starting materials, and no independent standard is available. For the other five TICs (AC, CG, Cl<sub>2</sub>, SA, and H<sub>2</sub>S), the independent standards will be gaseous standards of the target TICs, obtained from different commercial suppliers than those providing the standards used for reference method calibrations. Also, no independent PE standards are available for the CW agents, i.e., all CW agents used in testing are obtained from the U.S. Army. In lieu of a true PE audit, one or more QC check samples will be prepared for each CW agent used in testing, by spiking blank sample matrices and analyzing them by the same method used to analyze test samples. Description of the criteria for the PE audit is provided in Section C1.2.

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

### **B6.1 Vapor Delivery Equipment**

Different vapor delivery equipment will be used depending on the TIC or CW agents to be tested. Compressed gas cylinders will be used as the vapor delivery source for all the vapor phase TICs. Vendor certificates of analysis will be required for all such TIC gas standards. For the CW agents GB and HD, a diffusion cell will be used. A calibrated temperature controlled water bath will be installed to control the temperature of the diffusion cell, to maintain a stable and controllable vapor generation rate. Suitable valving will be included in the flow path downstream of the vapor generation source, so that the dilution and test equipment can be totally isolated from the source if necessary. Gas mass flow controllers for use in the vapor delivery system will be obtained from Battelle's Instrument Laboratory, and must be accompanied by currently valid documentation of calibration by that laboratory. Similarly temperature measurement and recording instruments will be obtained for control of the CW agent source water bath; current calibration records are also required for that equipment. A schematic of the entire vapor generation, dilution and delivery system is shown in Section B2.1, Figure 3.

### **B6.2 Phase 2 Temperature/Humidity Control**

When temperature and RH effects are assessed in Phase 2, all delivered challenge samples (whether vapor in air, liquid, or solid surface) and the screening technologies will be

equilibrated within the specified temperature and RH range before testing. For testing above ambient temperature, the test enclosure will be warmed, using an electronic temperature controller. For testing below ambient temperature, the test enclosure will be cooled, using a chilled radiator and fan in the enclosure. For all tests, thermocouples installed in the test enclosure will provide real-time temperature monitoring. All temperature and RH measurement devices must have currently applicable calibration records from Battelle's Instrument Laboratory.

A commercial Nafion® humidifier (Perma Pure, Inc.) will be used to generate controlled high humidity air (50 to 100% RH), which will then be mixed with dry dilution air and the target vapor stream to obtain the target RH ( $\leq 20\%$  to 80%) in the challenge air.

### **B6.3 Screening Technology Checks**

All screening technologies will be operated and maintained according to the vendors' instructions throughout the technology evaluation. Vendors will be required to provide such instructions before testing. Maintenance of any tested technologies will be performed only according to a preset schedule, or in response to predefined instrument diagnostics. Any vendor-specified confidence checks intended to assure proper operation of a technology will be carried out each day before evaluation procedures begin. No evaluation of a technology will be conducted unless proper response is observed with such checks. The vendor will be required to repair or replace any technology that repeatedly fails such confidence checks.

## **B7 INSTRUMENT CALIBRATION AND FREQUENCY**

### **B7.1 Reference Methods**

The reference methods to be used for the determination of TICs and CW agents are listed in Section B3. The analytical equipment needed for these methods will be calibrated, maintained and operated according to the quality requirements of the respective methods or SOPs indicated in Section B3, and the normal operational procedures of the test facility. Continuous monitoring equipment, such as an electrochemical monitor used to confirm Cl<sub>2</sub> concentrations, will be

operated, calibrated, and maintained according to the pertinent operations manual for the equipment.

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

### **B8.1 TICs, CW Agents, and Other Chemicals**

As stated above, the vapor phase TICs to be used in this technology evaluation will include: hydrogen cyanide (AC), cyanogen chloride (CK), phosgene (CG), chlorine (Cl<sub>2</sub>), arsine (SA), and hydrogen sulfide (H<sub>2</sub>S). All these TICs will be purchased as dilute compressed gas mixtures from commercial vendors, in nitrogen. The concentrations of those purchased mixtures will be specified based on the required final challenge concentrations and reasonable dilution ratios achievable with a mass flow control system. Acceptance of these gas mixtures will require that they be accompanied by a certificate of analysis (COA) indicating concentration and traceability to NIST standards (if applicable).

The CW agents planned for use in the technology evaluation (GB, HD, VX, and L) will be obtained from the U.S. Army, under the bailment agreement noted in Section A8. Acceptance for use will be based on a check of purity; as noted in Section A8.2 a minimum purity of 80 percent will be required.

Other chemicals used in testing (e.g., cyanide and fluoride salts; hydrogen peroxide) will be obtained from commercial suppliers at a purity of American Chemical Society (ACS) reagent grade or higher. Confirmation that the chemical is of at least ACS reagent grade, based on vendor-supplied specifications, will be grounds for acceptance.

### **B8.2 Technologies To Be Tested**

The screening technologies to be tested will be purchased from the respective manufacturers or their distributors, in the quantities needed for testing. No additional acceptance testing will be performed, i.e., delivery of the purchased technologies in the manufacturer's packaging will be taken as proof of the acceptability of the technologies for testing.



### **B8.3 Chain of Custody**

Chain of custody procedures are not required due to the nature of the testing to be conducted under this plan.

### **B9 NON-DIRECT MEASUREMENTS**

No non-direct measurements will be used for this technology evaluation.

### **B10 DATA MANAGEMENT**

#### **B10.1 Data Acquisition**

Data to be recorded include the times and conditions of steps in testing; sampling conditions and analytical results for the reference methods; the responses (or lack thereof) of the screening technologies in each portion of the test; and observations about ease of use, cost, etc. These data will be recorded by the testing staff in laboratory record books, analytical data records, and data recording forms. An example data sheet is shown in Appendix B.

Table 8 summarizes the types of data to be recorded, how the data will be recorded, and how often the data will be recorded. All data will be recorded by Battelle staff. 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 Task Order Leader and Quality Assurance Manager.

#### **B10.1.1 Instrumental Data Acquisition**

For sophisticated screening devices such as IMS, FSP, or PID instruments, the acquisition of data will be tailored to the data output capabilities of those instruments. It is expected that a visual display of readings, coupled with an audible or visual alarm, will be the primary data output of most portable IMS instruments. For those IMS instruments, data will be recorded manually by the evaluation staff, on data forms prepared before the technology evaluation. Some IMS, FSP, or PID instruments may have on-board data logging capabilities, or may provide an electronic output signal. In such cases, data acquisition will be conducted

electronically, using the instrument’s own software or a personal computer-based data acquisition system in the test facility.

**Table 8. Summary of Data Recording Process for the Technology Evaluation**

| <b>Data to be Recorded</b>  | <b>Where Recorded</b>   | <b>How Often Recorded</b>  | <b>Disposition of Data<sup>(a)</sup></b>  |
|---|---|--|---|
| 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/TIC identities and concentrations, gas flows, etc.)              | Laboratory record books, data forms   | When set or changed, or as needed to document the sequence of tests.               | Used to organize/check test results, manually incorporated in data spreadsheets as necessary. |
| Reference method 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. |
| Reference method sample 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   |
| Screening technology responses, readings, and diagnostic displays                       | Electronically if possible; prepared data forms otherwise                         | Upon challenge with each TIC/CW agent sample or blank                              | Transferred to spreadsheets   |

a: All activities subsequent to data recording are carried out by Battelle.

Whether collected manually or electronically, all such data will be entered into electronic spreadsheets, set up to organize the screening response, reference method, and test data for each screening procedure. Organization of the data in this way will allow evaluation of the performance parameters clearly and consistently. The accuracy of entering manually-recorded data into the spreadsheets will be checked at the time the data are entered, and a portion of the data will also be checked by the Battelle Quality Assurance Manager as part of the Data Quality Audit (Section C1.3). A separate spreadsheet will be set up for each screening technology tested, and no intermingling or intercomparison of data from different instruments will take place until the draft evaluation reports are prepared.

### **B10.1.2 Laboratory Data Acquisition**

Laboratory analytical data (e.g., reference method results quantifying the TICs or CW agents used) may be produced electronically, from (e.g.) gas chromatographic or electrochemical instruments. These records will be reviewed at least on a weekly basis to identify and resolve any inconsistencies. All written records must be in ink, and signed (or initialed) and dated by the person recording the information. All written records must be entered promptly, legibly, and accurately. 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.

### **B10.2 Confidentiality**

In all cases, strict confidentiality of test data will be maintained until the draft evaluation reports are ready for internal review. Separate files (including manual records, printouts, and electronic data files) will be kept for each technology.

### **B10.3 Statistical Calculations**

The screening technologies to be tested under this test/QA plan will provide primarily qualitative responses. That is, they will indicate the presence or absence, and in some cases the relative concentration, of a target TIC or CW agent, rather than a quantitative concentration. Consistent with the qualitative nature of the responses, the data produced in the Phase 1 test will be subjected to relatively simple analyses to assess the effectiveness of the screening technologies on the three performance parameters of analysis time, accuracy, and false positive/false negative responses.

#### **B10.3.1 Analysis Time**

The data collected in Phase 1 to evaluate analysis time will be the measured time periods required to screen each set of challenge samples. These data will be determined based on the recorded start and end time of each screening test with a given technology. Three replicate analysis time measurements will be recorded in all tests, whether the challenge samples are

vapor in air, liquid, or surface coupon samples. Thus, for a technology that is applicable to all the target TICs and CW agents in all the Phase 1 target sample matrices, a total of 24 analysis time values will be recorded with vapor phase samples, 21 with liquid samples, and 6 with surface samples (based on the number of target chemicals in Tables 4, 5, and 6, respectively).

The recorded analysis time data will be tabulated in the evaluation report, and will be summarized in terms of the mean, variance, and range of response times observed for each type of sample matrix (vapor, liquid, surface). Data analysis will include comparison of the observed analysis times for different screening technologies for each TIC or CW agent, and for each type of sample matrix. Such comparisons will be based on appropriate small-sample statistical tests such as the t test, comparison of ranges, or F test.

### **B10.3.2 Accuracy**

Accuracy will be assessed in Phase 1 in terms of the percentage of prepared samples that each screening technology properly identifies as being hazardous. Accuracy (A) will be calculated as follows:

$$A = (PR/HS) \times 100$$

where PR is the number of positive screening responses observed from a technology, and HS is the number of prepared hazardous challenge samples that were screened. Accuracy will be calculated in this way for each screening technology for each matrix type (vapor, liquid, surface), including all the target chemicals that the screening technology in question is intended to respond to. Individual TICs or CW agents for which accuracy is markedly higher or lower than for others, within each sample matrix type, will also be identified.

### **B10.3.3 False Positives and False Negatives**

The rate of false positives and false negative responses from each screening technology will be calculated from Phase 1 data as a percentage of the corresponding samples used to assess these performance factors.

False positive rates will be determined based on the response of screening technologies to blank sample matrices of each type (clean air, clean water, and clean glass coupons, for vapor,

liquid, and surface samples, respectively). For each screening technology, the false positive rate (FP) will be calculated as:

$$FP = (PRB/B) \times 100$$

where PRB is the number of positive responses observed when screening blank samples, and B is the number of blank samples that were screened. This calculation will be done for each technology, for each of the three sample matrix types.

False negative rates will be determined based on the absence of response of screening technologies to known prepared hazardous samples of each matrix type. For each screening technology, the false negative rate (FN) will be calculated as:

$$FN = (NR/HS) \times 100$$

where NR is the number of negative responses (i.e., failures to identify a hazardous sample), and HS is the number of samples screened, as defined above in Section B10.2. This calculation will be done for each technology, for each of the three sample matrix types.

Individual TICs or CW agents for which false positive or negative rates are markedly higher or lower than for others, within each sample matrix type, will also be identified.

#### **B10.3.4 Repeatability**

The repeatability of screening technology responses will be evaluated for those responses that are other than simple yes/no indications. Repeatability of yes/no indications will be covered by evaluation of accuracy and false positive/negative rates. However, a technology that provides (e.g.) a High/Medium/Low response may correctly indicate the presence of a TIC or CW agent with any of those responses.

At a minimum, repeatability will be evaluated by recording and reporting the responses observed in each of three successive challenges with each TIC or CW agent. Those results will be tabulated to compare the uniformity of responses in each case. For a technology that provides a numerical response (e.g., a bar graph indication or approximate concentration) the mean, range, and relative standard deviation of each set of replicate results will be tabulated for comparison of results.

## **B11 PHASE 2 EVALUATION PROCEDURES**

The test procedures to be used in the Phase 2 testing will be fully specified once the Phase 1 test results are complete. This approach will be taken so that the Phase 2 results are targeted toward those technologies that performed best in the Phase 1 evaluation, or are most appropriate for the AHRF screening protocol. This test/QA plan will be revised following completion of the Phase 1 evaluation, and as a result only a general description of the planned Phase 2 procedures is provided below.

### **B11.1 Temperature and Humidity Effects**

During Phase 2 testing, both temperature and RH will be varied to assess the impact of these conditions on the performance of selected screening technologies, which will be chosen based on their performance in Phase 1. The planned temperature and RH ranges for such testing are approximately 10 to 30° C and 20 to 80% RH, respectively. The Phase 2 tests will involve a subset of the same target TICs and CW agents and the same test matrices as used in Phase 1. In the Phase 2 temperature/RH testing, the test procedures will be identical to those conducted in Phase 1, but the screening technologies and test samples will be equilibrated at the target temperature and RH conditions (e.g., 10° C, 80% RH) before the test procedure is conducted. The data from these tests will be evaluated to assess whether temperature or RH have an effect on the screening performance of each technology.

### **B11.2 Interference Effects**

The effect of potentially interfering compounds will be assessed in Phase 2 testing because such compounds can potentially produce either false positive or false negative screening responses. The interferents of interest will be materials that might occur in the vapor phase, or in solid or liquid samples collected in the field. Interferent testing will involve only one interferent at a time.

To evaluate vapor phase interference effects, the test system shown in Figure 3 (Section B2.1) will be modified with the addition of an interferent vapor generator. The output from this source will be directed as needed to mix with the humidified air flowing to the challenge plenum.

The target TIC or CW agent source will be independently controlled such that the interferent can be generated either in the absence or the presence of the target chemical. This will allow interference effects to be evaluated with the interferent alone, and with an interferent and TIC or CW agent together. Testing with the interferent alone will allow evaluation of false positive responses, and testing with the interferent and chemical together will allow evaluation of false negatives. Testing will be done by alternately conducting screening of clean air and the interferent mixture, for a total of up to three times each, in a procedure analogous to that described in Section B2.1. The same TIC and CW agent concentrations used in the initial testing under Section B2.1 will be used in this test, i.e., one-half the IDLH level or other target level. The test procedures will also allow observation of interferent effects on the analysis time of each screening technology. Potential vapor-phase interferents include (e.g.) fuel vapors, diesel engine exhaust, and vapors from common cleaning supplies such as ammonia-based cleaner.

Evaluation of interference effects in liquid and solid surface sample screening will involve potential interferents such as soil minerals and powders, liquid fuels (e.g., diesel fuel), salt water, commercial cleaning products, or explosives residues (e.g., ammonium nitrate). False positive interferences will be assessed by screening samples containing an individual interferent in a simple sample matrix (e.g., water), in the absence of any TIC or CW agent. False negative interferences will be assessed by screening similar samples containing both an interferent and a representative TIC or CW agent. To the extent possible, allowance will be made in these tests for potential interactions between the interferent and the TIC or CW agent. Evaluation of interferences in screening for surface contamination will be conducted by applying the potential interferents to the same type of material coupons used for the solid surface evaluation in Phase 1.

## **C1 ASSESSMENTS AND RESPONSE ACTIONS**

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

Battelle's Quality Assurance Manager will perform a TSA at least once during the performance of this technology evaluation. The purpose of this TSA is to ensure that the technology evaluation is being performed in accordance with this test/QA plan and that all QA/QC procedures are being implemented. In this audit, the Quality Assurance Manager may review the reference analysis methods used, compare actual test procedures to those specified in this plan, and review data acquisition and handling procedures. The Quality Assurance 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 report.

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

### **C1.2 Performance Evaluation Audit**

A PE audit will be conducted to assess the quality of the measurements made in this technology evaluation. This audit addresses only those reference measurements that factor into the data used for evaluation, i.e., the screening technologies are not the subject of the PE audit. This audit will be performed once during the technology evaluation, and will be performed by analyzing a standard that is independent of standards used during the testing. Table 9 summarizes the PE audits that will be done and indicates the acceptance criteria for the PE audit. This audit will be the responsibility of Battelle evaluation staff.

As indicated by Table 9, the PE audit will be conducted for TICs, in both vapor and liquid samples, but not for the CW agents. The reason for this is that there is no independent source of the CW agents, i.e., all agents used in testing are obtained from the U.S. Army. In lieu of a PE audit for the CW agents, sorbent traps or sampling bags will be spiked with known quantities of the agents, and subjected to analysis as a QC check. This check will be conducted once in each technology evaluation, with at least one spiked sample prepared for each of the CW agents used in testing. The target agreement for this QC check will be  $\pm 30\%$ . Also, no PE audit



**Table 9. Summary of PE Audit**

| <b>Parameter</b>                        | <b>Audit Procedure</b>        | <b>Expected Tolerance</b> |
|---|-------------------------------|---------------------------|
| TIC vapor Concentrations <sup>a</sup>   | Analyze independent standards | ± 20%                     |
| TIC aqueous concentrations <sup>b</sup> | Analyze independent standards | ± 15%                     |

a: AC, CG, SA, Cl<sub>2</sub>, and H<sub>2</sub>S only

b: Cyanide, fluoride, and hydrogen peroxide only.

will be done for the TIC cyanogen chloride (CK) because the source gas for that TIC is prepared by Battelle, and no independent standard is available.

In the event that results of analysis of the PE audit standard do not meet the acceptance criteria, then the reference analysis method will be recalibrated with the laboratory standards, and then the PE audit standard will be reanalyzed. Continued failure to meet the PE audit criteria will result in the pertinent data being flagged, and potentially the purchase of new standards for repetition of the PE audit. Battelle’s Quality Assurance Manager will assess PE audit results.

**C1.3 Data Quality Audit**

Battelle’s Quality Assurance Manager will audit at least 10 % of the evaluation data acquired in the technology evaluation. The Quality Assurance 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.

**C1.4 Corrective Action**

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

## **C2 REPORTS TO MANAGEMENT**

Each assessment and audit will be documented in accordance with Sections 3.3.4 (Internal Assessment Reporting) and 3.3.5 (Response) of the program QMP.<sup>(2)</sup> 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.

Copies of the TSA assessment report will be provided to EPA.

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

Records generated in the technology evaluation will receive a one-over-one review within two weeks after generation, before these records are used to calculate, evaluate, or report results. These records will include laboratory record books, completed data forms, electronic spreadsheets or data files, and reference method analytical results. Appendix B shows an example of a data recording sheet that would be used during testing. The data review will be performed by the Battelle Task Order Leader or his designate, but in any case someone 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.

## **D2 VALIDATION AND VERIFICATION METHODS**

Section C of this test/QA plan provides a description of the validation safeguards employed for this technology evaluation. Data validation and verification efforts include the performance of TSA, PE, and data quality audits as described in Section C.

## **D3 RECONCILIATION WITH DATA QUALITY OBJECTIVES**

The data comparisons described in Section B will be conducted separately for each screening technology undergoing evaluation. Two evaluation reports will then be prepared, one presenting the results of testing with TICs, and the other presenting the results of testing with CW agents. Each evaluation report will summarize the respective test data, as well as the results of the evaluation of those data. Each evaluation report will briefly describe the TTEP program, and will refer to this test/QA plan and any amendments of this plan for the procedures used in the technology evaluation. The results of the technology evaluation will then be stated for each technology tested, so that the relative performance of the screening technologies can be assessed. The preparation of the two draft evaluation reports, the review and revision of those reports, final approval, and the distribution of the reports, will be conducted as stated in the program QMP.<sup>(2)</sup>

Each report will also discuss the limitations of the test data, emphasizing that the Phase 1 evaluation is a screening approach preliminary to Phase 2.

## E REFERENCES

1. Draft Interim All Hazards Receipt Facility Protocol Standard Operating Procedures (Guidance), prepared by Computer Sciences Corporation, Alexandria, VA, for U.S. EPA National Homeland Security Research Center, Cincinnati, OH, July 13, 2005.
2. Quality Management Plan (QMP) for the Technology Testing and Evaluation Program, Version 1, Battelle, Columbus, Ohio, January 21, 2005.
3. Chemical warfare agent information gathered from Jane's Chem-Bio Web ([www4.janes.com](http://www4.janes.com)); Canadian Centre for Occupational Safety and Health ([www.ccohs.ca](http://www.ccohs.ca)); U.S. Agency for Toxic Substances and Disease Registry; U.S. Army Center for Health Promotion and Preventive Medicine; National Library of Medicine ChemID Plus ([chem.sis.nlm.nih.gov/chemidplus](http://chem.sis.nlm.nih.gov/chemidplus)); and additional internet sources (e.g., [cbwinfo.com](http://cbwinfo.com)).
4. Acute Exposure Guideline Levels published by the National Research Council, National Academy of Sciences, and available from the U.S. Environmental Protection Agency at <http://www.epa.gov/oppt/aegl/sitemap.htm>.
5. Temporary Emergency Exposure Limits established for the U.S. Department of Energy by the Subcommittee on Consequence Assessment and Protective Actions (SCAPA), information and TEEL values available at <http://www.orau.gov/emi/scapa/teels.htm>.
6. Battelle Chromatography Method for Hydrogen Cyanide (HCN), Method Designation C:\HPCHEM\1\METHODS\ETV\_HCN.M, May 2003.
7. Battelle Gas Chromatography Method for Cyanogen Chloride (CK), Method Designation C:\HPCHEM\1\METHODS\ETV\_CK.M, May 2003.
8. Battelle Gas Chromatography Method for Arsine, Method Designation C:\MSDCHEM\1\METHODS\OLD\ARSINE5.M, May 2004.

**Appendix A**  
**Tabulation of Screening Technologies to be Evaluated for Use in the**  
**All Hazards Receipt Facility**

The tables in this Appendix identify the screening technologies that will be evaluated for detection of each target TIC or CW agent, in each of the three sample matrices (vapor, liquid, and surface samples). Table A-1 lists the technologies to be evaluated with vapor samples, Table A-2 those to be evaluated with liquid samples, and Table A-3 those to be evaluated with surface samples. The left column in each table indicates the TIC or CW agent, and the right column shows the vendor and name of each technology to be evaluated with samples of that type with that chemical.

**Table A-1: Technologies to be Evaluated with Vapor Samples**

| VAPOR SAMPLE                        | TECHNOLOGY   |
|-------------------------------------|--|
| Hydrogen cyanide (AC)               | Draeger - Civil Defense Kit; Nextteq - Civil Defense Kit; ; Safety Solutions – HazMat Smart Strip; Agentase – CAD Kit; Sensidyne – Gas Detection Tubes; MSA – Single CWA Sampler Kit; Truetech – M18A3; Anachemia – M256A1; Anachemia – Chemical Agent Detector Kit; Draeger – CMS Analyzer  |
| Cyanogen chloride (CK)              | Draeger - Civil Defense Kit; Nextteq - Civil Defense Kit; Agentase – CAD Kit; MSA – Single CWA Sampler Kit; Truetech – M18A3; Anachemia – M256A1; Anachemia – Chemical Agent Detector Kit  |
| Phosgene (CG)                       | Draeger - Civil Defense Kit; Draeger - CMS Analyzer; Nextteq - Civil Defense Kit; Truetech – M18A3; MSA – Gas Detection Tubes  |
| Arsine (SA)                         | Draeger - Civil Defense Kit; RAE Systems - MultiRae Plus; Sensidyne – Gas Detection Tubes  |
| Chlorine (Cl <sub>2</sub> )         | Draeger – Civil Defense Kit; Draeger - CMS Analyzer; Safety Solutions – HazMat Smart Strip; Sensidyne – Gas Detection Tubes; Proengin – AP4C   |
| Hydrogen sulfide (H <sub>2</sub> S) | Draeger - CMS Analyzer; Safety Solutions – HazMat Smart Strip; Sensidyne – Gas Detection Tubes; Proengin – AP4C  |
| Sarin (GB)                          | Nextteq - Civil Defense Kit; RAE Systems - MultiRae Plus; Anachemia – Chemical Agent Vapor Detector; Agentase – CAD Kit; Safety Solutions – HazMat Smart Strip; Safety Solutions – M8 Nerve Agent Badge; Truetech – M18A3; Anachemia – M256A1; Anachemia – Chemical Agent Detector Kit; Proengin – AP4C; Smiths Detection - APD 2000; Draeger – Civil Defense Kit; MSA – Gas Detection Tubes |
| Sulfur mustard (HD)                 | Draeger - Civil Defense Kit; Nextteq - Civil Defense Kit; RAE Systems - MultiRae Plus; Agentase – CAD Kit; Safety Solutions – HazMat Smart Strip; Safety Solutions – M8 Nerve Agent Badge; MSA – Single CWA Sampler Kit; Truetech – M18A3; Anachemia – M256A1; Anachemia – Chemical Agent Detector Kit; Proengin – AP4C; Smiths Detection - APD 2000   |

**Table A-2: Technologies to be Evaluated with Liquid Samples**

| <b>WATER SAMPLE</b>                                | <b>TECHNOLOGY</b>  |
|--|--|
| Sarin (GB)   | Agentase – CAD Kit; Truetech M272 Water Kit; Anachemia – M256A1; Anachemia – 3-way Paper; Anachemia – M-8 Paper; Anachemia M-9 Paper; Anachemia – Chemical Agent Detector Kit; Safety Solutions – HazMat Smart Strip; Proengin – AP4C;   |
| Sulfur mustard (HD)                                | Agentase – CAD Kit; Truetech M272 Water Kit; Anachemia – M256A1; Anachemia – 3-way Paper; Anachemia – M-8 Paper; Anachemia M-9 Paper; Anachemia – Chemical Agent Detector Kit; Safety Solutions – HazMat Smart Strip; Proengin – AP4C;   |
| VX   | Agentase – CAD Kit; Truetech M272 Water Kit; Anachemia – M256A1; ; Anachemia – 3-way Paper; Anachemia – M-8 Paper; Anachemia M-9 Paper; Anachemia – Chemical Agent Detector Kit; Safety Solutions – HazMat Smart Strip; Proengin – AP4C; |
| Lewisite (L)                                       | Truetech M272 Water Kit; Anachemia – M256A1;   |
| Cyanide (in the form of potassium cyanide, KCN)    | Agentase – CAD Kit; Truetech M272 Water Kit; Anachemia – M256A1;   |
| Hydrogen peroxide (H <sub>2</sub> O <sub>2</sub> ) | Safety Solutions – HazMat Smart Strip  |
| Fluoride (as sodium fluoride, NaF)                 | Safety Solutions – HazMat Smart Strip; Proengin – AP4C   |



**Table A-3: Technologies to be Evaluated with Surface Samples**

| <b>SURFACE SAMPLE</b> | <b>TECHNOLOGY</b>  |
|-----------------------|--|
| VX                    | Agentase – CAD Kit; Truetech M272 Water Kit; Anachemia – M256A1; ; Anachemia – 3-way Paper; Anachemia – M-8 Paper; Anachemia M-9 Paper; Anachemia – Chemical Agent Detector Kit; Safety Solutions – HazMat Smart Strip; Proengin – AP4C; |
| Lewisite (L)          | Truetech M272 Water Kit; Anachemia – M256A1;   |

## **APPENDIX B**

### **Example Data Sheet for Evaluation of AHRF Screening Technologies**

**AHRF Screening Technologies Evaluation  
Data Recording Sheet**

Date \_\_\_\_\_ Technology \_\_\_\_\_

Sample Matrix (V/L/S) \_\_\_\_\_ Testing Staff \_\_\_\_\_

| <b>Sample ID</b> | <b>Target Chemical</b> | <b>Nominal Conc.</b> | <b>Response</b> | <b>Notes</b> |
|------------------|------------------------|----------------------|-----------------|--------------|
|                  |                        |                      |                 |              |
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|                  |                        |                      |                 |              |
|                  |                        |                      |                 |              |

Start Time \_\_\_\_\_ End Time \_\_\_\_\_

Entered By \_\_\_\_\_ Date \_\_\_\_\_

Reviewed By \_\_\_\_\_ Date \_\_\_\_\_





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