US ERA ARCHIVE DOCUMENT

# **Environmental Technology Verification Report**

BRUKER DALTONICS INC.
RAID-M ION MOBILITY SPECTROMETER

Prepared by Battelle



Under a contract with

**EPA** U.S. Environmental Protection Agency



# Environmental Technology Verification Report

ETV Safe Buildings Monitoring and Detection Technologies Verification Program

# Bruker Daltonics Inc. RAID-M Ion Mobility Spectrometer

by

Kent Hofacre
Tricia Derringer
Dale Folsom
Peter Larkowski
National Security Division

Thomas Kelly
Loraine Sinnott
Cody Hamilton
Zachary Willenberg
Energy and Environment Division

Battelle Columbus, Ohio 43201

### **Notice**

The U.S. Environmental Protection Agency (EPA), through its Office of Research and Development, has financially supported and collaborated in the extramural program described here. This document has been peer reviewed by the Agency. Mention of trade names or commercial products does not constitute endorsement or recommendation by the EPA for use.

#### **Foreword**

The U.S. Environmental Protection Agency (EPA) is charged by Congress with protecting the nation's air, water, and land resources. Under a mandate of national environmental laws, the Agency strives to formulate and implement actions leading to a compatible balance between human activities and the ability of natural systems to support and nurture life. To meet this mandate, the EPA's Office of Research and Development provides data and science support that can be used to solve environmental problems and to build the scientific knowledge base needed to manage our ecological resources wisely, to understand how pollutants affect our health, and to prevent or reduce environmental risks. EPA also addresses responsibilities in homeland security through the National Homeland Security Research Center, by means of research programs in Drinking Water Security, Safe Buildings, and Rapid Risk Assessment.

The Environmental Technology Verification (ETV) Program has been established by the EPA to verify the performance characteristics of innovative environmental technology across all media and to report this objective information to permitters, buyers, and users of the technology, thus substantially accelerating the entrance of new environmental technologies into the marketplace. Verification organizations oversee and report verification activities based on testing and quality assurance protocols developed with input from major stakeholders and customer groups associated with the technology area. ETV consists of seven environmental technology centers. Information about each of these centers can be found on the Internet at http://www.epa.gov/etv.

The ETV approach has also been applied to verification of homeland security technologies. The verification reported herein was conducted by Battelle as part of the Safe Buildings Monitoring and Detection Technologies Verification Program, which is funded by EPA. Information concerning this specific environmental technology area can be found on the Internet at http://www.epa.gov/etv/centers/center11.html.

### Acknowledgments

The authors wish to acknowledge the support of all those who helped plan and conduct the verification test, analyze the data, and prepare this report. We also would like to thank Tom Taylor of the Centers for Disease Control and Prevention and Mark Durno of the U.S. Environmental Protection Agency Region 5 for their reviews of the test/quality assurance plan and this report.

## Contents

		Page	Э
N	otice	i	i
Fo	oreword	ii	i
A	cknowledgments	i	V
Li	ist of Abbreviation	vii	i
1	Background		1
2	Technology Descri	ption	2
3	3.1 Introduct 3.2 Test Des 3.2.1 3.2.2 3.2.3 3.2.4 3.2.5 3.2.6 3.2.7	ocedures ion gn Chemical Test Compounds Test Matrix Test Locations Test Sequence and Schedule Reference Methods Interferents Materials and Equipment edure Response Time  7  7  7  7  7  7  7  7  7  7  7  7  7	4 5 5 6 7 9 1 3 6
	3.3.2 3.3.3 3.3.4 3.3.5 3.3.6 3.3.7 3.3.8 3.3.9	Recovery Time       18         Accuracy       18         Repeatability       18         Response Threshold       18         Temperature and Humidity Effects       19         Interference Effects       19         Cold-/Hot-Start Behavior       20         Battery Life       20         Operational Characteristics       2	8 8 8 9 9 0
4		/Quality Control2/2nt Calibration2/2Reference Methods2/2nstrument Checks2/2	2

	4.2	Audits	24
		4.2.1 Performance Evaluation Audit	24
		4.2.2 Technical Systems Audit	25
		4.2.3 Data Quality Audit	25
	4.3	QA/QC Reporting	25
5	Statistical	Methods	26
	5.1	Statistical Analyses	26
	5.2	Other Analyses	27
6	Test Resu	lts	28
	6.1	Response Time	28
	6.2	Recovery Time	30
	6.3	Accuracy	
	6.4	Repeatability	31
	6.5	Response Threshold	32
	6.6	Temperature and Humidity Effects on Response	32
	6.7	Interference Effects	
	6.8	Cold-/Hot-Start Behavior	36
	6.9	Battery Life	
		Operational Characteristics	
	6.11	Cost	41
7	Performa	nce Summary	42
8	Reference	es	45
A	ppendix A	. Description of Statistical Analysis Methods	
A	ppendix B	. Detailed Statistical Analysis Results	
A	ppendix C	. Tabulation of Data from Verification of the Bruker RAID-M Portable Ion Mobili Spectrometer	ity

## Figures

Figure 2-1.	Bruker Daltonics Inc. RAID-M IMS
Figure 3-1.	Sequence of Possible TIC Verification Tests
Figure 3-2.	Logic Diagram for Determining TIC Test Sequence
Figure 3-3.	Test System Schematic
	Tables
Table 3-1.	Target TIC and CW Agent Challenge Concentrations 6
Table 3-2	Summary of Evaluations Conducted on the RAID-M IMS
Table 3-3.	Test Schedule
Table 3-4.	Summary of Primary Reference Methods
Table 3-5.	Summary of Test Concentrations for Interferents
Table 3-6.	Temperature and Relative Humidity Conditions for RAID-M Testing 16
Table 4-1.	Summary of Performance Evaluation Audit Results
Table 6-1.	Summary Illustrative Data from RAID-M Verification Test
Table 6-2.	Response Threshold Data for the TIC and CW Agent Testing
Table 6-3.	Summary Data Illustrating Interference Effects
Table 6-4.	Summary of Cold-/Hot-Start Test Data
Table 6-5.	Responses Recorded from the RAID-Ms in Battery Life Testing

#### **List of Abbreviations**

AC hydrogen cyanide ANOVA analysis of variance

Cl<sub>2</sub> chlorine CG phosgene

CK cyanogen chloride CW chemical warfare

DEAE N,N-diethylaminoethanol

EPA U.S. Environmental Protection Agency
ETV Environmental Technology Verification

FID flame ionization detection FPD flame photometric detection

GB sarin

GC gas chromatography

HD sulfur mustard

HMRC Hazardous Materials Research Center

ID identification

IDLH immediately dangerous to life and health

IMS ion mobility spectrometer(ry)

L liter

LCD liquid crystal display

μg/m<sup>3</sup> microgram per cubic meter

μl microliter
mL milliliter
mm millimeter

MSD mass selective detection PE performance evaluation

ppb part per billion ppm parts per million

ppmC parts per million of carbon

psig pounds per square inch gauge

QA quality assurance QC quality control

QMP quality management plan

RH relative humidity

RSD relative standard deviation

SA arsine

THC total hydrocarbon

TIC toxic industrial chemical
TSA technical systems audit
VOC volatile organic compound

### Chapter 1 Background

The U.S. Environmental Protection Agency (EPA) supports the Environmental Technology Verification (ETV) Program to facilitate the deployment of innovative environmental technologies through performance verification and dissemination of information. The goal of the ETV Program is to further environmental protection by accelerating the acceptance and use of improved and cost-effective technologies. ETV seeks to achieve this goal by providing high-quality, peer-reviewed data on technology performance to those involved in the design, distribution, financing, permitting, purchase, and use of environmental technologies.

ETV works in partnership with recognized testing organizations; with stakeholder groups consisting of buyers, vendor organizations, and permitters; and with the full participation of individual technology developers. The program evaluates the performance of innovative technologies by developing test plans that are responsive to the needs of stakeholders, conducting field or laboratory tests (as appropriate), collecting and analyzing data, and preparing peer-reviewed reports. All evaluations are conducted in accordance with rigorous quality assurance (QA) protocols to ensure that data of known and adequate quality are generated and that the results are defensible.

Subsequent to the terrorist attacks of September 11, 2001, this ETV approach has been applied to verify the performance of homeland security technologies. Monitoring and detection technologies for the protection of public buildings and other public spaces fall within the Safe Buildings Monitoring and Detection Technologies Verification Program, which is funded by EPA and conducted by Battelle. In this program, Battelle recently evaluated the performance of the Bruker Daltonics Inc. RAID-M portable ion mobility spectrometer (IMS).

## **Chapter 2 Technology Description**

The objective of the ETV Safe Buildings Monitoring and Detection Technologies Verification Program is to verify the performance characteristics of monitoring technologies for chemical and/or biological contaminants that might be introduced into the building environment. This verification report provides results for the verification testing of the RAID-M portable IMS made by Bruker Daltonics Inc. Following is a description of the RAID-M, based on information provided by the vendor. The information provided below was not subjected to verification in this test.

The RAID-M is a chemical detector that uses the principle of IMS to detect, classify, quantify, and continuously monitor concentrations of chemical warfare (CW) agents and toxic industrial chemicals (TICs). The identity of substances detected is displayed both by class (e.g., "G," "H," or "T" for G series agents, H series agents, and TICs, respectively) and by specific agent, simulant, or TIC (e.g., "GB," "HD," or "TDI"). All classes can be displayed independently. Relative concentrations are indicated by a bar display with eight increments. In addition to use in the field, the RAID-M is designed to be capable of operating within collective protection facilities.

The RAID-M can be operated while being held in one hand. It has no protruding parts and weighs less than 2.80 kilograms (6.4 pounds), excluding battery. The RAID-M contains a small radioactive sealed source that is completely housed and is such that RAID-M can be stored in bulk. The RAID-M is 400 millimeters (mm) (15.7 inches) long, 115 mm (4.5 inches) wide, and 165 mm (6.5 inches) high. The RAID-M is of a one-tube design, with automatic polarity



Figure 2-1. Bruker Daltonics Inc. RAID-M IMS

switching (i.e., both positive and negative ions are automatically monitored, in alternate intervals of 2 to 3 seconds), and is fully microprocessor-controlled. It has a remote display and control option. The display shows agent identity and a relative indication of hazard level. The RAID-M incorporates a built-in audible alarm to indicate agent detection, and visual alarms to warn of a low battery and other faults.

The RAID-M is powered by an integral, primary battery and can accept power input from a

variety of sources including vehicles (12- to 24-volt direct current nominal) or from a 240-volt, 50-Hertz, alternating current power supply. A diagnostic input/output socket provides data output, power input, personal computer connectivity, and built-in test information. The carrying case is designed to protect the RAID-M from exposure to air blasts, thermal radiation, neutron radiation, gamma radiation, and electromagnetic pulse.

Consumables do not need to be changed when the RAID-M detects a challenge, and consumables are designed to have a maximum life of not less than 500 hours. There are no scheduled preventive maintenance tasks. Daily checks are designed to not require dismantling the equipment and to not typically exceed an average of 10 minutes per day.

# **Chapter 3 Test Design and Procedures**

#### 3.1 Introduction

When first responders arrive at a potentially contaminated site, they need to immediately and accurately identify chemicals that may be present. Chemicals and chemical agents that may pose a threat in a building could include both TICs and CW agents.

The objective of this verification test of the RAID-M, a commercially available, portable IMS, was to evaluate its ability to detect toxic chemicals and chemical agents in indoor air. This verification focused on the scenario of a portable IMS used by first responders to identify contaminants and guide emergency response activities after chemical contamination of a building. This verification was conducted according to a peer-reviewed test/quality assurance (test/QA) plan<sup>(1)</sup> that was developed according to the requirements of the Quality Management Plan for the ETV program.<sup>(2)</sup> The following performance characteristics of the RAID-M were evaluated:

- Response time
- Recovery time
- Accuracy
- Repeatability
- Response threshold
- Temperature and humidity effects
- Interference effects
- Cold-/hot-start behavior
- Battery life
- Operational characteristics.

Response time, recovery time, accuracy, and repeatability were evaluated by challenging the RAID-Ms with known vapor concentrations of target TICs and CW agents. RAID-M performance at low target analyte concentrations was evaluated to assess the response threshold. Similar tests conducted over a range of temperatures and relative humidities (RH) were used to establish the effects of these factors on detection capabilities. The effects of potential interferences in an emergency situation were assessed by sampling those interferences both with and without the target TICs and CW agents present. The RAID-Ms were tested after a cold start (i.e., without the usual warm-up period) both from room temperature and from cold storage conditions, and after hot storage, to evaluate the delay time before readings could be obtained

and the response speed and accuracy of the RAID-Ms once readings were obtained. Readings of a target TIC were obtained with the RAID-Ms operated on alternating current power, and subsequently on battery power, to assess any differences. Battery life was determined as the time until RAID-M performance degraded as battery power was exhausted in continuous operation. Operational factors such as ease of use, data output, and cost were assessed by observations of the test personnel and through inquiries to the vendor.

It was intended to conduct all tests simultaneously with two RAID-Ms. However, due to occasional RAID-M problems, testing continued in some cases with only one RAID-M. A total of three RAID-Ms were used during the four-month test period.

Testing was limited to detecting chemicals in the vapor phase because that mode of application is most relevant to use by first responders. Testing was conducted in two phases: detection of TICs (conducted in a non-surety laboratory at Battelle) and detection of CW agents (conducted in a certified surety laboratory at Battelle's Hazardous Materials Research Center [HMRC]).

#### 3.2 Test Design

#### 3.2.1 Chemical Test Compounds

The TICs used in testing were

- Hydrogen cyanide (HCN, North Atlantic Treaty Organization designation AC)
- Cyanogen chloride (ClCN, designated CK)
- Phosgene (COCl<sub>2</sub>, designated CG)
- Chlorine (C1<sub>2</sub>)
- Arsine (AsH<sub>3</sub>, designated SA).

The CW agents were sarin (GB) and sulfur mustard (HD).

It should be noted that the RAID-Ms tested were programmed to detect all of these compounds except SA; neither of the software libraries in the RAID-Ms at the time of testing included SA. Also, it should be noted that the chemical identification that the RAID-Ms displayed upon detection of a chemical varied slightly among the target compounds. For Cl<sub>2</sub>, GB, and HD, the RAID-M identifiers were "CL2," "GB," and "HD," i.e., the same as the chemical designations themselves. For AC, the RAID-M display identifier was "CY," indicating a cyanide compound. For CG, the RAID-M identifier was "CIX," indicating a chlorine-containing compound. For CK, the RAID-M identifier was either "CIX" or "CY," indicating either a chlorine-containing or cyanide-containing compound (CK contains both).

Table 3-1 summarizes the concentrations of each TIC and CW agent used in this verification test. For the TICs AC, CK, CG, and Cl<sub>2</sub>, tests were conducted at both 0.1 and 1 times the respective immediately dangerous to life and health (IDLH) level. The 0.1 IDLH level was added to the test procedure because full-scale readings were often obtained with the TICs at the IDLH concentrations. For SA, the RAID-Ms did not respond at the IDLH level, so the 0.1 IDLH level

was not needed. For the CW agents GB and HD, testing was conducted at a single concentration level that produced less than full-scale readings on the RAID-Ms under normal temperature and humidity conditions. The concentration used for GB was 0.014 ppm (0.080 mg/m³), which is 0.4 times the IDLH concentration of 0.035 ppm (0.2 mg/m³). No IDLH level has been set for HD, so the concentration used was based on an alternative toxic effects guideline, as noted in the footnote to Table 3-1.

Table 3-1. Target TIC and CW Agent Challenge Concentrations

Chemical	Concentrations	Type of Level	
Hydrogen cyanide (AC)	50 ppm (50 mg/m <sup>3</sup> ) and 5 ppm (5 mg/m <sup>3</sup> )	1 and 0.1 x IDLH <sup>a</sup>	
Cyanogen chloride (CK)	20 ppm (50 mg/m <sup>3</sup> ) and 2 ppm (5 mg/m <sup>3</sup> )	1 and 0.1 x IDLH	
Phosgene (CG)	2 ppm (8 mg/m <sup>3</sup> ) and 0.2 ppm (0.8 mg/m <sup>3</sup> )	1 and 0.1 x IDLH	
Chlorine (Cl <sub>2</sub> )	10 ppm (30 mg/m <sup>3</sup> ) and 1 ppm (3 mg/m <sup>3</sup> )	1 and 0.1 x IDLH	
Arsine (SA)	3 ppm (10 mg/m <sup>3</sup> )	1 x IDLH	
Sarin (GB)	0.014 ppm (0.080 mg/m <sup>3</sup> )	0.4 x IDLH	
Sulfur mustard (HD)	0.063 ppm (0.42 mg/m <sup>3</sup> )	$0.7 \text{ x AEGL-}2^{\text{b}}$	

<sup>(</sup>a) IDLH = Immediately Dangerous to Life and Health; IDLH value for CK estimated from value for AC.

#### 3.2.2 Test Matrix

Table 3-2 summarizes the evaluations that were conducted in the verification test. As Table 3-2 indicates, except for cold-/hot-start behavior, battery life, and assessment of false positive interference effects (i.e., the interferent alone), all performance parameters were evaluated during both the TIC and CW agent testing.

#### 3.2.3 Test Locations

Two laboratories were used to conduct the verification tests. Testing with the non-chemical surety materiel—TICs and interferents—was conducted in a new laboratory at Battelle's Columbus, Ohio, campus, which has the needed vapor generation, collection, and analysis equipment. This laboratory has been used previously to conduct IMS instrument and filter tests using CG, AC, CK, and Cl<sub>2</sub> under controlled environmental conditions. Testing with CW agents was conducted at the HMRC at Battelle's West Jefferson, Ohio, campus. 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 requiring the safe use and storage of highly toxic substances. Battelle operates the HMRC in compliance with all applicable federal, state, and local laws and regulations, including Army regulations.

<sup>(</sup>b) AEGL = Acute Exposure Guideline Level; AEGL-2 levels are those expected to produce a serious hindrance to efforts to escape in the general population. The AEGL-2 value of 0.09 ppm (0.6 mg/m³) for HD is based on a 10-minute exposure.

Table 3-2. Summary of Evaluations Conducted on the RAID-M IMS

Performance Parameter	Objective	Comparison Based On
Response Time	Determine rise time of RAID-M response	RAID-M readings with step rise in analyte concentration
Recovery Time	Determine fall time of RAID-M response	RAID-M readings with step decrease in analyte concentration
Accuracy	Characterize agreement of RAID-M with reference results	Reference method results
Repeatability	Characterize consistency of RAID-M readings with constant analyte concentration	RAID-M readings with constant input
Response Threshold	Estimate minimum concentration that produces RAID-M response	Reference method results
Temperature and RH Effects	Evaluate effect of temperature and RH on RAID-M performance	Repeat above evaluations with different temperature and RH
Interference Effects	Evaluate effect of building contaminants that may interfere on with RAID-M performance	Sample interferents and TICs/CW agents together (and interferents alone <sup>(a)</sup> )
Cold Start	Characterize startup performance after cold storage	Repeat tests with no warm-up <sup>(a)</sup>
Hot Start	Characterize startup performance after hot storage	Repeat tests with no warm-up <sup>(a)</sup>
Battery Operation	Characterize battery life and performance	Compare RAID-M results on battery vs. alternating current power and duration of operation on batteries <sup>(a)</sup>

<sup>(</sup>a) Indicates this part of the test performed only during TIC testing.

#### 3.2.4 Test Sequence and Schedule

The sequence of tests performed with the TICs in this study is outlined in Figure 3-1. Since analyzer performance was not known *a priori*, the concentrations used in testing depended on the results of the first few tests performed. The decision logic used to determine the actual TIC concentrations and the test sequence is shown in Figure 3-2. After completion of TIC testing, a similar, but slightly reduced, set of tests was performed with CW agents. Table 3-3 summarizes the actual schedule of testing for the TICs and CW agents. As Table 3-3 indicates, testing with AC was interrupted as a result of depletion of the AC source gas. Testing was completed on CK, and then AC testing resumed. Testing with SA took only one day because this TIC is not detected by the RAID-M.

#### **Test 1: Vapor Challenge with TIC**

Alternating clean air with IDLH level concentration of TIC five times with IMS operating on alternating current power, fully warmed up per manufacturer's instructions prior to testing, and room temperature  $(22 \pm 3^{\circ}\text{C})$  and  $50 \pm 5\%$  RH.

#### Test 2: Vapor Challenge with TIC at reduced concentration

Test 1 is repeated at 0.1 times the IDLH concentration.

#### Test 3: Vapor Challenge with TIC at increased concentration

Test 1 is repeated at roughly 10 times the IDLH concentration.

#### Test 4: Response Threshold of TIC

Test 1 is repeated at a concentration below IDLH. If a response is recorded, the concentration is cut in half until no response is recorded. If no initial response is recorded, the concentration is increased by a factor of 2 until a response is recorded.

#### Test 5: IDLH/0.1 IDLH/Clean Air Challenge

Test 1 is repeated by alternating IDLH, low concentration (either 0.1 IDLH or response threshold concentration. See logic chart, Figure 3-2), and clean air three times, and alternating low concentration, IDLH, and clean air three times.

#### Test 6: Vapor Challenge with TIC at room temperature, low humidity

Test 1 is repeated at room temperature  $(22 \pm 3^{\circ}\text{C})$  and less than 20% RH. The test is performed at the concentration(s) determined via the logic in Figure 3-2.

#### Test 7: Vapor Challenge with TIC at room temperature, high humidity

Test 1 is repeated at room temperature  $(22 \pm 3^{\circ}\text{C})$  and  $80 \pm 5\%$  RH. The test is performed at the concentration(s) determined via the logic in Figure 3-2.

#### Test 8: Vapor Challenge with TIC at high temperature, medium humidity

Test 1 is repeated at high temperature ( $35 \pm 3^{\circ}$ C) and  $50 \pm 5\%$  RH. The test is performed at the concentration(s) determined via the logic in Figure 3-2.

#### Test 9: Vapor Challenge with TIC at high temperature, high humidity

Test 1 is repeated at high temperature  $(35 \pm 3^{\circ}\text{C})$  and  $80 \pm 5\%$  RH. The test is performed at the concentration(s) determined via the logic in Figure 3-2.

#### Test 10: Vapor Challenge with TIC at low temperature, medium humidity

Test 1 is repeated at low temperature ( $5 \pm 3^{\circ}$ C) and  $50 \pm 5\%$  RH. The test is performed at the concentration(s) determined via the logic in Figure 3-2.

#### Test 11: Interferent false positive tests

Test 1 is repeated alternating interferent only with clean air. The test is repeated for all interferents in both libraries.

#### **Test 12: Interferent false negative tests**

Test 1 is repeated alternating TIC and interferent with clean air. The test is repeated for all interferents.

#### **Test 13: Opposite Library test**

Test 1 is repeated for the library opposite of the one recommended by the manufacturer for TICs.

#### Test 14: Room Temperature, cold start behavior

Repeat Test 1 with the IMS at room temperature for a minimum of 12 hours and no warm-up.

#### Test 15: Cold-/Cold-start behavior

Repeat Test 1 after the IMS has been kept refrigerated (5-8°C) overnight for a minimum of 12 hours, with no warm-up.

#### Test 16: Hot-/Cold-start behavior

Repeat Test 1 after the IMS has been kept heated (40°C) overnight for a minimum of 12 hours, with no cool down or warm up.

#### **Test 17: Battery test**

Repeat Test 1 with the IMS operating on battery power. The TIC at IDLH concentration is alternated with clean air once every half hour until the unit stops responding or shuts down due to loss of power.

#### Figure 3-1. Sequence of Possible TIC Verification Tests

**Step 1:** Perform Test 1. Depending on the results of this test, go to Step 2a, 2b, or 2c as appropriate.

**Step 2a:** If there is no response in Test 1, then perform Test 3, then go to Step 4.

**Step 2b:** If the response in Test 1 is on scale, then skip to Step 3 and perform all subsequent tests at the IDLH concentration.

**Step 2c-1:** If the response in Test 1 is full- or off-scale, then perform Test 2. Depending on the results of Test 2, go to Step 2c-2a or 2c-2b as appropriate.

**Step 2c-2a:** If there is no response in Test 2, then perform Test 4. Perform all subsequent tests at IDLH and response threshold concentrations.

**Step 2c-2b:** If there is a response in Test 2, then perform all subsequent tests at IDLH and 0.1 times the IDLH concentrations.

**Step 3:** Perform Test 4 (if not already done), Tests 5-10, Tests 12-13 at the concentration(s) determined above. For the first TIC, also perform Test 11 and Tests 14-17.

**Step 4:** Return to Step 1 and repeat for all other TICs.

Figure 3-2. Logic Diagram for Determining TIC Test Sequence

Table 3-3. Test Schedule

	Chemical	Test Dates (2003)
AC		August 6–September 3 September 15–25
CK		September 4–15
CG		September 25–October 3
SA		October 6
$Cl_2$		October 13–21
GB		November 19–December 8
HD		December 9–18

#### 3.2.5 Reference Methods

Table 3-4 summarizes the primary reference methods used to determine the challenge concentrations of the target TICs and CW agents. 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 concentration range of each method. For CK and AC, low concentration samples were injected directly for determination by gas chromatography (GC) with flame ionization detection (FID). A gas chromatographic method for CG proved to have inadequate sensitivity, so an impinger-based

**Table 3-4. Summary of Primary Reference Methods** 

Analyte	Concentration Range (ppm)	Sampling Method	Analysis Method
AC	0.05 to 100	Air sample injected directly	GC/FID
CK	2 to 100	Air sample injected directly	GC/FID
CG	1 to 100	Collection in impingers with nitrobenzyl pyridine	Visible absorption at 475 nm
$Cl_2$	0.1 to 100	Continuous electrochemical detector with chlorine-specific sensor	Continuous detection
SA	0.05 to 100	Capillary gas chromatography with syringe injection from bag sample	MSD
GB	0.01 to 100	Air sample collected with solid sorbent tube	GC/FPD
HD	0.01 to 100	Air sample collected with solid sorbent tube	GC/FPD

visible absorption measurement was implemented.<sup>(3)</sup> This method was satisfactory for confirming the levels of CG provided to the RAID-Ms with sampling intervals of 20 minutes and a sampling flow rate of one liter (L) per minute. Cl<sub>2</sub> was determined by a continuous electrochemical analyzer with a Cl<sub>2</sub>-specific sensor to allow rapid determination of Cl<sub>2</sub> levels delivered to the RAID-M during testing. SA was determined by a GC with a capillary column and mass selective detection (MSD), using samples collected by syringe from the test apparatus. The CW agents GB and HD were collected in solid sorbent cartridges, and determined by GC with flame photometric detection (FPD), according to existing HMRC test procedures.

Summaries of these primary methods, and of supplemental methods also used, are as follows.

Hydrogen cyanide (AC) and cyanogen chloride (CK)—The reference method for AC and CK was an Agilent 6890 GC with a capillary column and FID. This GC was positioned next to the laboratory hood containing the test system during the TIC testing and sampled automatically from the flow line delivering the challenge gases to the RAID-Ms.

Phosgene (CG)—To quantify CG, a sample of air was drawn at a known, constant flow rate through a midget impinger containing 10 milliliter (mL) of an indicating solution, consisting of a mixture of 4-(4'-nitrobenzyl)pyridine (0.25% w/w) and n-phenylaniline (0.5% w/w) in diethyl phthalate. In this solution, CG reacts to produce a product having a brilliant red color. The amount of CG reacted was determined by measuring the absorbance of the indicating solution at 475 nm using a spectrophotometer. Due to the complexity of the impinger method for CG, a Draeger Pac III Single Gas Monitor also was used in some tests to provide rapid response. This device was new, and its factory calibration was used in all monitoring.

Chlorine  $(Cl_2)$ —  $Cl_2$  was monitored with a new Draeger MiniWarn Multi-Gas Monitor, which was factory calibrated.

Arsine (SA)—SA was determined using an Agilent 6890 GC coupled to a 5970 mass selective detector. Air samples ( $100 \,\mu\text{L}$ ) were injected onto a GS-Q capillary column held at - $30^{\circ}\text{C}$ . SA was separated from xenon at a constant flow of 3 mL/minute helium using the following temperature program: hold at - $30^{\circ}\text{C}$  for 1 minute; ramp at  $50^{\circ}\text{C}$ /minute to  $150^{\circ}\text{C}$ ; hold at  $150^{\circ}\text{C}$  for 5 minutes (total time 9.6 minutes). The injector temperature was maintained at  $100^{\circ}\text{C}$ , operated in splitless mode, and was purged after 0.5 minute at 5 mL/minute. Single ion monitoring at m/z = 129 and 76 was used to record signals for xenon and SA, respectively. SA was quantified using its relative response compared with xenon as the internal standard. In addition, to provide rapid response in SA determination, a new Draeger Miniwarn Multi-Gas Monitor also was used. This monitor was factory calibrated.

Sarin (GB) and sulfur mustard (HD)—The analytical method for these CW agents involved collecting the agents by sampling air through sorbent sampling tubes. The tubes were then thermally desorbed, and the agents were determined using a capillary GC with FPD. Concentrations were determined based on a linear regression of peak area with the amount of agent, and accounting for the volume of air sampled.

Total hydrocarbons—A continuous FID was used for the determination of the total hydrocarbon (THC) content of interferent mixtures provided to the RAID-Ms during testing. The THC concentrations characteristic of realistic interferent levels in buildings were determined, either by direct measurement or by interpretation of published data. The interferent delivery systems were then adjusted to achieve the desired THC indication in parts per million of carbon (ppmC) for each interferent during testing.

#### 3.2.6 Interferents

Interferents were selected for testing based upon their prevalence in a building. The interferents selected were the volatile chemicals in latex paint, air freshener, and ammonia-based floor cleaner, as well as gasoline engine exhaust and N,N-diethylaminoethanol (DEAE). DEAE is a common additive to boiler systems and is released into the heating, ventilating, and air conditioning system when boiler steam is used to humidify the air. These selected interferents were tested for false positives by exposing the detectors to selected levels of the interferents in clean air, to see whether the interferents generated a positive response from the instruments when no TICs or CW agents were present. Each interferent also was introduced to the instruments along with each TIC and CW agent, to determine false negatives, i.e., whether the interferent prevents the RAID-M from indicating that the TIC or CW agent is present. The following sections describe the materials and concentrations used for testing.

The interferents are mixtures of chemicals and determining the interferent concentration requires the quantification of all the chemicals present. However, monitoring each component would be time and cost prohibitive. For this reason, interferent concentrations were monitored using a THC analyzer. THC analysis is appropriate because all the interferents consist of a significant amount of carbon-containing compounds. Because quantification is based on carbon content, the test concentrations are reported on a per carbon basis such as ppmC. The use of the

hydrocarbon analyzer also provided real-time continuous monitoring of the interferent concentration during testing.

Test concentrations for the interferents were based on direct measurements or published data. Concentrations found in published data were converted to a per carbon basis as described below. Table 3-5 is a summary of the test concentrations. The following sections contain a detailed description of how the test concentrations were determined.

Table 3-5. Summary of Test Concentrations for Interferents

Interferent	Test Concentration (ppmC)
Latex Paint Fumes	5-10
Floor Cleaner Vapors	10
Air Freshener Vapors	1
Gasoline Exhaust Hydrocarbons	2.5
DEAE	0.02

#### 3.2.6.1 Latex Paint Fumes

Concentrations of latex paint fumes were evaluated directly in and around a freshly painted office. Samples were obtained using a 25 L Teflon bag and analyzed for THC content. Each wall in the office was painted, and the room dimensions are 11 feet by 11 feet with an alcove that is 4 feet by 10 feet and ceiling that is 12 feet high. Immediately after painting, the hydrocarbon concentration was 170 ppmC. After 2.5 hours, the hydrocarbon concentration in the office fell to 38 ppmC. At this time, the hydrocarbon content was determined just outside the entrance to the office and in the hallway 80 feet away from the office. Hydrocarbon content just outside the office was 20 ppmC; in the hallway 80 feet away from the office, it was 3 ppmC. Based on these measurements, the test concentration was maintained at 5 to 10 ppmC.

#### 3.2.6.2 Floor Cleaner Vapors

The test concentration for the ammonia-based floor cleaner was inferred from studies on latex paint fumes. Similar to paint, floor cleaner is applied to a surface and allowed to dry. Floor cleaner vapors containing both ammonia and fragrances will disperse into the hallway. Because of the similarity, a test concentration of 10 ppmC was used for the floor cleaner.

#### 3.2.6.3 Air Freshener Vapors

Concentration levels of air freshener for interferent testing were based upon values reported at recent indoor air quality conferences. Volatile organic compound (VOC) emission for a plug-in air freshener was reported to be 30 to 80 milligrams per hour, resulting in a concentration of 300 to 500 micrograms per cubic meter ( $\mu g/m^3$ ) for the average room. Assuming the VOC emitted consists of hydrocarbons similar to limonene, a common fragrance component, the concentration on a per carbon basis can be calculated. Limonene contains 10 carbons and has a molecular weight of 136. A concentration of 5.56  $\mu g/m^3$  of limonene is the same as 1 part per

billion (ppb). With a room concentration of  $500 \,\mu\text{g/m}^3$  and limonene as a representative molecule, the fragrance concentration on a per carbon basis is estimated to be 1 ppmC. This THC level was maintained for all tests with the air freshener.

#### 3.2.6.4 Gasoline Engine Exhaust

Of the constituents in gasoline engine exhaust fumes, the aromatic components were considered most likely to interfere with the performance of the RAID-M. A recent study reported that urban areas have benzene concentrations of 7 to 9 ppb with comparable concentrations of other aromatics. (4) The test mixture used to simulate exhaust contains 61 compounds ranging in size from 2 to 10 carbons, with an average concentration of 200 ppb for each component. To obtain a concentration of 7 to 9 ppb for the aromatic compounds, the test mixture was diluted 30:1. Assuming an average size of six carbons, the THC of the mixture was approximated to be 73 ppmC. After dilution, the THC content was 2.5 ppmC, and this target concentration was maintained for all the experiments.

#### 3.2.6.5 DEAE

DEAE is a common additive to boiler systems to prevent corrosion. When boiler steam is used to humidify the air in a building, DEAE is released into the building as well. Generally, the DEAE concentration is kept below 40 ppb, the threshold for odor detection. One study has shown DEAE concentrations of 1 ppb in a building that uses direct steam injection for humidification. For testing purposes, the concentration was set at 20 ppbC, which correlates to 3.3 ppb DEAE given that DEAE contains six carbons. This concentration was not detectable by THC analysis, so the interferent concentration was set by dilution of a concentrated standard.

#### 3.2.7 Materials and Equipment

#### 3.2.7.1 TICs and CW Agents

The commercial gas standards used as sources of the TICs for testing included standards of 10,000 parts per million (ppm) AC (Cylinders 151531 and 7035, Scott Specialty Gases); and 991 ppm CG (Cylinder RR37345), 997 ppm SA (Cylinder KE-50368), and 5,811 ppm Cl<sub>2</sub> (Cylinder RA64239), all from Matheson Gas Products. The source of CK was a 10,000 ppm compressed gas standard made at Battelle starting with neat CK gas. The neat CK gas was from Atomergic Chemetals Corp. (Lot No. L6196). To prepare the CK standard, 210 mL of the neat gas was diluted by pressurizing to 1,000 pounds per square inch gauge (psig) with hydrocarbonfree air in a 5.9-L cylinder. The CW agents GB and HD were obtained as neat materials from the U.S. Army under Bailment Agreement No. DAAD13-H-00-0002.

#### 3.2.7.2 Vapor Delivery Equipment

The compressed gas mixtures noted in Section 3.2.7.1 were diluted as the vapor sources for CK, AC, CG, Cl<sub>2</sub>, and SA. For the CW agents GB and HD, a diffusion cell containing the pure agent was used. A temperature-controlled water bath was installed to control the temperature of the diffusion cell to maintain a stable and controllable vapor generation rate. A two-way valve was

included in the flow path downstream of the vapor generation source, so that the dilution and test equipment could be totally isolated from the source. A schematic of the entire vapor generation, dilution, and delivery system is shown in Figure 3-3.

#### 3.2.7.3 Temperature/Humidity Control

The RAID-Ms were evaluated at the temperature and humidity conditions indicated by an "X" in Table 3-6. Both the delivered air temperature and the RAID-M units were maintained within the specified temperature range. For testing at 35°C, the vapor delivery system was warmed with a heat-traced line, using an electronic temperature controller. For testing at 5°C, the dilution and delivery system was enclosed in a cooled chamber to provide approximate temperature control. For all tests, thermocouples were installed in both the clean air plenum and the challenge plenum to provide real-time temperature monitoring.

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

#### 3.2.7.4 Interferent Sources

Interference test concentrations were obtained by diluting a concentrated feed with air. For latex paint, floor cleaner, and air freshener, the concentrated feeds were made by purging the head space of a large boiling flask containing about 100 mL of the bulk liquid of each interferent. THC analysis of the head space samples found that the concentrated feeds contained 394, 886, and 233 ppmC for latex paint, floor cleaner, and air freshener, respectively. Gasoline engine exhaust was simulated using a mixture of 61 organic compounds ranging from 2 to 10 carbon atoms ( $C_2$  to  $C_{10}$ ). This mixture was prepared by adding 1 microliter ( $\mu$ L) of 51 neat liquid components and 250  $\mu$ L of 10 gaseous components into a 15.7-L cylinder and diluting to a final pressure of 1,200 psig with nitrogen. A concentrated standard of 1 ppm for DEAE was made by adding zero nitrogen to 6  $\mu$ L of liquid neat DEAE to a final pressure of 1,200 psig.

#### 3.2.7.5 Performance Evaluation Audit Materials

As part of the quality assurance effort in this verification, a performance evaluation (PE) audit was performed on reference methods used to confirm the TIC and CW agent concentrations provided to the RAID-Ms. This audit involved conducting analyses on independent standards, obtained from different sources than those used for the calibration standards. The results from the independent standards were then compared with those from the calibration standards, to assess the degree of agreement. The target agreement in the PE audit was within 20% for TICs and within 30% for the CW agents.

For the TICs, the PE audit standards were 967 ppm AC (Cylinder SD-10271, Matheson Gas Products); and 997 ppm CG (Cylinder NA021189), and 5,830 ppm Cl<sub>2</sub> (Cylinder 1C1857), both from Scott Specialty Gases. A PE audit gas for SA (nominal 1,000 ppm, Scott Specialty Gases) was obtained, but contained less than 1 ppm of SA, so no PE audit was done for arsine. Also, no PE audit could be done for CK because of the unavailability of commercial standards for that gas.

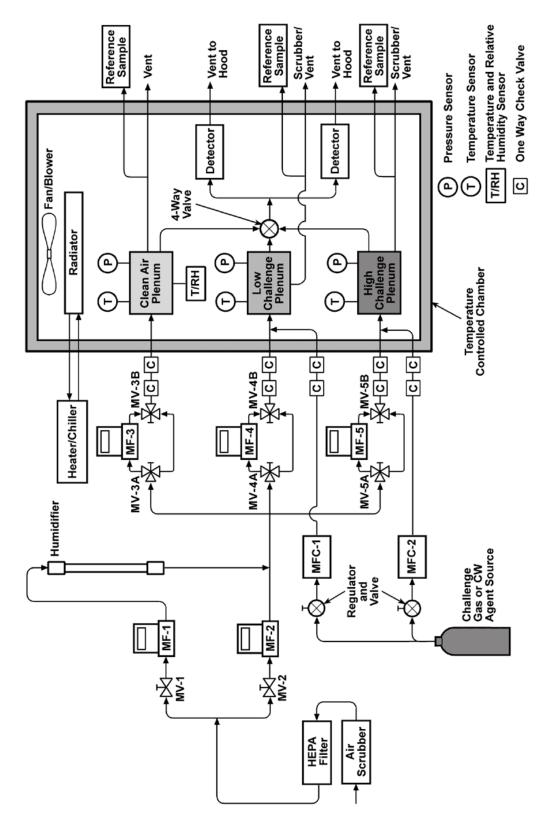


Figure 3-3. Test System Schematic

15

Table 3-6. Temperature and Relative Humidity Conditions for RAID-M Testing

	Temperature (°C)		
<b>RH</b> (%)	5 ± 3	22 ± 3	$35 \pm 3$
≤ 20		X	
$50 \pm 5$	X	X	X
$80 \pm 5$		X	X

For the CW agents, PE audit samples were prepared at the HMRC by an analyst other than the staff who conducted routine calibration of the reference method. The PE audit samples were sorbent tubes, spiked with known quantities of GB or HD, starting from a different batch of agent than that used to prepare the calibration samples. These tubes were analyzed by the same approach used for analysis of reference samples from GB and HD testing, and the results were compared with the spike amounts.

#### 3.3 Test Procedure

The test system (Figure 3-3) consisted of a vapor generation system, a Nafion® humidifier, two challenge plenums, a clean air plenum, an RH sensor, thermocouples, and mass flow meters. The challenge vapor or gas was generated by the vapor generation system. The challenge vapor was then mixed with the humid dilution air and flowed into the challenge plenum. Interference vapors were added to the challenge mixtures as needed for testing.

The RH and target concentration of the challenge vapor were obtained by adjusting the mixing ratio of the humid air (from the Nafion® humidifier) to the dry dilution air, and the mixing ratio of the vapor generation stream to the humid dilution air, respectively. To avoid potential corrosion or malfunction of the RH sensor from exposure to the challenge vapor, the RH meter was installed upstream of the inlet of the vapor stream. The RH of the challenge vapor stream was calculated based on the measured RH of the humid dilution air and the mixing ratio of the vapor generation stream to the humid dilution air.

To establish the background readings of the two RAID-Ms being tested, a clean air plenum was installed. Part of the humid dilution air was introduced directly into the clean air plenum. When establishing the RAID-M background, the four-way valves connected to the two RAID-Ms were switched to the clean air plenum to collect baseline data.

After the background measurement, the four-way valves connected to the two RAID-Ms were switched to one of the challenge plenums to allow the RAID-Ms to sample the challenge mixture. Switching between the challenge and clean air plenums was rapid, and the residence time of gas in the test system was short to allow determination of the response and recovery times of the RAID-Ms. The reference methods described in Section 3.2.5 were used to confirm that the gas concentrations in the challenge plenums were within  $\pm 20\%$  of the target level.

Concentrations outside that tolerance range triggered a repeat of any test procedures conducted since the last analysis.

#### 3.3.1 Response Time

To evaluate IMS response time, the environmental target conditions were established at  $22 \pm 3^{\circ}$ C and  $50 \pm 5\%$  RH. Initially 10 L/min of clean humidified air were passed through the clean air plenum. The RAID-M sampled the clean air for a minimum of 5 minutes or until a stable reading was indicated, but not exceeding 10 minutes, to obtain a baseline. At the same time the clean air was sampled by the RAID-M, the clean air plenum also was sampled with the appropriate reference method. This sampling took place after the RAID-M reading was stabilized.

Concurrent with the background measurements, the target challenge concentration in the high challenge plenum was established. The high challenge concentration was generated at the target environmental conditions. Adjustments were made to the generator operating conditions and the dilution flow as needed to establish a challenge concentration within ±20% of the IDLH or other target concentration, with a stability characterized by a relative standard deviation (RSD) of 10% or less in successive reference measurements. Reference samples were collected and analyzed immediately to establish the challenge concentration and demonstrate stability prior to testing. A challenge concentration was considered stable if it could be maintained within the target challenge bounds on three consecutive reference sample measurements for AC and CK over a minimum of 5 minutes of continuous operation prior to the test. A challenge concentration was considered stable for CG and SA if one sample taken prior to testing was consistent with the method calibration curve. A challenge concentration was considered stable for Cl<sub>2</sub> when the continuously monitoring reference method reached a stable reading.

After a stable reading was obtained from the RAID-M on background air, and the challenge mixture was stable and at the target concentration, the four-way valve at the RAID-M's inlet was switched to sample from the high challenge plenum. The response of the RAID-M was recorded, and the time to reach a stable response was determined. The RAID-M sampled from the challenge plenum for a minimum of 5 minutes, up to a maximum of 10 minutes, unless the RAID-M responded with an alarm of 8 bars (i.e., a full-scale response). In that case, the RAID-M sampled from the challenge plenum for only 30 seconds. The high challenge vapor concentration was determined by the reference method as frequently as possible during the procedure. For AC, CK, and Cl<sub>2</sub>, a reference sample was taken prior to every challenge with the RAID-M. For CG and SA, a reference sample was taken prior to and at the conclusion of every set of tests.

After the challenge sampling was concluded, the sample inlet four-way valve was switched to again sample from the clean air plenum. The time required for the RAID-M to clear (i.e., the time to return to its starting baseline or non-alarm reading) was recorded as the recovery time. A minimum of 5 minutes was permitted to allow the RAID-M response to return to baseline. After a maximum of 10 minutes, regardless of whether the RAID-M returned to baseline, subsequent cycles of alternating challenge/clean air sampling were carried out, controlled by the four-way valve. Five such challenge/clean air cycles were completed.

The same sampling procedure (i.e., challenge/clean air cycles) was carried out at different temperatures and RHs or challenge concentrations to evaluate temperature and RH effects and response thresholds. For the TIC testing, the initial test was conducted at a concentration equal to the target chemical's IDLH level. If the RAID-M alarmed at 8 bars, the five challenge/clean air cycles also were conducted at a concentration of 0.1 times the original IDLH (see Table 3-1). In the CW agent testing, only a single target concentration was used that produced responses below 8 bars (Table 3-1).

Following the five challenge/clean air cycles, six cycles also were conducted in which the RAID-M alternated sampling from the high and low challenge plenums. For the TICs, the high challenge plenum provided the IDLH or target concentration, and the low challenge plenum provided a concentration of 0.1 times that level. For the CW agents, two different levels below about 0.7 IDLH were used. This procedure simulated using the RAID-M in locations having different degrees of contamination. In three of the six cycles the high plenum was sampled first, then the low plenum; in the other three the order was reversed (this change in order was not carried out in testing with HD). Clean air was sampled before the first cycle, and again after every high/low cycle. This test with alternating concentrations was conducted only at the normal temperature and RH conditions (i.e., 22°C and 50% RH).

#### 3.3.2 Recovery Time

The time for the RAID-M to return to its baseline reading or non-alarm state after removing a challenge concentration was measured as described in Section 3.3.1.

#### 3.3.3 Accuracy

In all of the response threshold and response time tests, the challenge concentration was measured using a reference method or monitor. Reference samples were collected prior to, during, and after RAID-M testing to ensure that a stable concentration was maintained for the AC, CK, and Cl<sub>2</sub> testing. Reference samples were collected prior to and after RAID-M testing to ensure a stable concentration was maintained for the CG and SA testing and the CW agent tests. The reference samples were the ground truth samples used to assess accuracy.

#### 3.3.4. Repeatability

Repeatability was assessed using data obtained from the five repeated challenge/clean air cycles or the six repeated high challenge/low challenge/clean air cycles. The repeated test results at the same environmental and concentration conditions were used to quantify the repeatability of the measurements and the effects of test conditions on repeatability.

#### 3.3.5. Response Threshold

The response threshold of each RAID-M was evaluated by repeating the procedure in Section 3.3.1 at successively lower concentrations. The response threshold was determined at the baseline environmental condition of 22°C and 50% RH, in the absence of any interfering chemicals. The manufacturer's reported detection limit (±50%) was used as the starting

concentration. If the manufacturer did not provide a detection limit, a concentration below the IDLH or target concentration was chosen. Three cycles of challenge/clean air were conducted at each concentration. If the RAID-M showed a response at that challenge concentration, the challenge concentration was decreased. The decrease in concentration was continued until the response threshold had been bracketed. The minimum challenge concentration producing a response was denoted as the response threshold. In some cases, the RAID-Ms continued to alarm even at the lowest challenge concentration obtainable from the testing system. In such cases, that minimum challenge concentration was denoted as an upper limit for the response threshold.

#### 3.3.6 Temperature and Humidity Effects

The tests described in Section 3.3.1 were repeated at the IDLH or other selected target concentration of significant health concern, over the range of environmental conditions shown in Table 3-3. Five repeat runs were performed at each set of test conditions for each target TIC or CW agent. The data at different temperature and RH conditions were used to infer whether these conditions affected the detection (i.e., accuracy, repeatability, response threshold) of the RAID-M for the target chemical. The effect on response time and recovery time also was assessed.

#### 3.3.7 Interference Effects

To evaluate the effects of the interferents described in Section 3.2.6, the test system shown in Figure 3-3 was modified by adding an interferent vapor generator. The output from this source was directed as needed to mix with the humidified air flowing to the challenge plenum. Interference effects were evaluated with the interferent alone and with the interferent and TIC or CW agent together. Testing with the interferent alone allowed evaluation of false positive responses, and testing with the interferent and target chemical together allowed evaluation of false negatives. Interference effects on response time and recovery time also were observed.

Interferent testing involved only one interferent vapor at a time. False positive testing was done by alternately sampling clean air and an interferent vapor, for a total of up to five times each, in a procedure analogous to that described in Section 3.3.1. However, if no interferent effect was observed after three such test cycles, the test was truncated. The same process was used for testing for false negatives with interferents and TICs (or interferents and CW agents) together, with the two compounds diluted together in humidified air delivered to the challenge plenum. In the interference tests, all TICs were tested at their IDLH levels, and the two CW agents were tested at the concentrations shown in Table 3-1. The interferent concentrations used were those shown in Table 3-5. A response from the RAID-M with the interferent alone was recorded as a false positive. The absence of a response, or a reduced response, to the TIC or CW agent in the presence of the interferent was recorded as a false negative.

If the RAID-M alarmed at 8 bars during the false negative tests, the five challenge and interferent/clean air cycles also were conducted at a concentration 0.1 times the original target concentration for the TIC testing.

The replicate test runs conducted with the interferent plus TIC or CW agent allowed the response time and recovery time of the RAID-M to be assessed with interferents present.

Differences in response and recovery times, relative to those in previous tests with only the TIC or CW agent present, were attributed to the effect of the interferent vapor.

#### 3.3.8 Cold-/Hot-Start Behavior

The cold-/hot-start tests were conducted in a manner similar to the response time test in Section 3.3.1. Prior to these tests, however, the RAID-M was not allowed to warm up per the manufacturer's recommendation.

The cold-start test was conducted both with the RAID-M at room temperature and subsequently at reduced temperature for a minimum of 12 hours prior to start-up. In the former test, the RAID-M was stored at  $22 \pm 3$  °C for at least 12 hours prior to testing. The cold-start effect was assessed using an IDLH challenge concentration of AC, at the baseline conditions of 22°C and 50% RH. The time from powering up the RAID-M to its first readiness to provide readings was considered the start-up delay or standby time. After challenge with AC, the response time was measured, followed by the recovery time. Repeatability and accuracy in five replicate challenge/clean air cycles also were noted.

For the reduced temperature cold-start test, the RAID-M was placed in a refrigerated enclosure (5 to 8°C) for at least 12 hours overnight. At the start of the next day, the cold-start test with AC was repeated, under the same baseline conditions (22°C and 50% RH) and again recording the start-up delay or standby time and other performance parameters.

For the hot-start test, the RAID-M was placed in a heated enclosure at  $40 \pm 3^{\circ}$ C for at least 12 hours overnight. At the start of the next test day, the hot-start test was conducted using AC in the same fashion as in the cold-start tests, at the baseline test conditions (22°C and 50% RH).

For the cold-/hot-start tests, the RAID-M was connected to the clean air manifold and switched on. The time between switching the RAID-M on and when it indicated it was ready to begin providing readings was recorded as the delay or standby time. Then the RAID-M was connected (by the four-way valve shown in Figure 3-3) to the challenge plenum, which was supplied with the IDLH level of AC. The response time, stable reading, and recovery time of each RAID-M were recorded for each of five successive periods of alternating challenge mixture and clean air. The recorded data were used to evaluate whether response and recovery times, repeatability, and accuracy were affected by a cold or hot start relative to normal (i.e., fully warmed up) operation. Only one cold-/hot-start test was performed per day so that the RAID-M could equilibrate to storage conditions prior to the test.

#### 3.3.9 Battery Life

Battery life was evaluated by assessing the degradation of performance with extended continuous operation. Fully charged batteries were installed, and the RAID-Ms were turned on and allowed to warm up, and an initial response time test was performed (see Section 3.3.1). An IDLH concentration of AC was used in this evaluation. The indicated concentration signal from the RAID-M was recorded. The RAID-M then sampled clean air for 30 minutes, and then the AC mixture was sampled again. This procedure was repeated with the RAID-M operating

continuously until the RAID-M no longer responded to the AC challenge. The total time of operation was recorded as the measure of battery life.

#### 3.3.10 Operational Characteristics

Key operational characteristics of the RAID-M were evaluated by means of the observations of test operators and by inquiry to the RAID-M vendor. Ease of use was assessed by operator observations, with particular attention to the conditions of use by emergency first responders.

Signal or data output capabilities were assessed by observations of the personnel who operated the RAID-M during testing. The type of data that was output was noted on the data sheets (e.g., audio and/or visual alarm, bar graph, low/med/high indication, and/or quantitative measure of concentration). In addition, the clarity and readability of the output were noted, especially in low light conditions or when holding the RAID-M while walking, as in use by a first responder. The availability of multiple forms of data output or display also was assessed (e.g., the availability of both a visual display and an analog voltage output for recording purposes).

The vendor was asked for the purchase and operational costs of the RAID-M as tested. Estimates for key maintenance items also were requested from the vendor.

# Chapter 4 Quality Assurance/Quality Control

QA/quality control (QC) procedures were performed in accordance with the ETV quality management plan (QMP)<sup>(2)</sup> and the test/QA plan for this verification test.<sup>(1)</sup>

#### 4.1 Equipment Calibration

#### 4.1.1 Reference Methods

The reference methods used for determining TICs and chemical agents are summarized in Section 3.2.5. The analytical equipment needed for these methods was calibrated, maintained, and operated according to the quality requirements of the reference methods and Battelle's normal documentation. Procedures for blank sampling during testing and for calibration of reference methods are described below.

For AC, CK, and Cl<sub>2</sub> testing, blank reference samples were run between each challenge concentration. The sequence of reference sampling thus included establishing the concentration prior to testing the RAID-Ms, running a blank on clean air, switching to challenge gas and taking a reference sample immediately prior to challenging the RAID-Ms with the challenge gas, and again running a blank when the RAID-Ms were once more sampling clean air. Blank reference samples were taken before and after, but not during, SA and CG testing, because the blank methods used were not conducive to running the large number of reference samples analyzed during the other TIC tests. In testing with GB and HD, blank sorbent tubes were run at the start of each test day.

Calibration procedures for the reference and other analyses were as follows:

Hydrogen cyanide (AC) and cyanogen chloride (CK)—The GC reference method for AC and CK was calibrated by preparing gas mixtures in 1-L gas sampling bags. For AC, calibration standards were prepared by diluting 0.5 to 4 mL of commercial concentrated AC gas standards (e.g., 10,000 ppm AC in  $N_2$ ) in 800 mL of clean air in a bag. Three samples from each bag were injected by syringe into the GC, and the peak area was recorded. Several such calibration standards ranging from 1.25 to 50 ppm HCN were prepared and analyzed over a three-day period. The regression of peak area versus AC standard concentration had the form Peak Area =  $3.0671 \times (AC, ppm)$ , with an  $r^2$  value of 0.9984. For CK, 800-mL bag standards were prepared in a similar manner; but, since CK was available as a neat gas, a two-stage dilution was needed.

An initial bag dilution of the neat material was made, and then a small volume of that mixture was diluted further in a second bag, producing CK standards of about 6 to 25 ppm. Again, three samples from each final bag were analyzed, and the peak areas were used to generate a regression line, which had the form Peak Area =  $1.8374 \times (CK, ppm)$ , with an  $r^2$  value of 0.9987.

Phosgene (CG)—For calibrating the impinger-based method for CG, 25-L Tedlar bags were filled with a known amount (~24 L) of zero air spiked with known amounts of CG from a high concentration commercial standard. Final concentrations in the bags ranged from 0 (method blank) to 2 ppm. Approximately 23 L of the phosgene/air mixture in each bag was then drawn through a midget impinger containing the indicating solution. A multi-point calibration at the beginning of the study showed linear response and an r² value of 0.98. Before proceeding with the analysis of samples on each test day, a one-point calibration check was conducted, typically at a phosgene concentration of 1 ppm, by the procedures outlined above. Inclusion of each day's calibration check data in the calibration curve showed correlation coefficients of 0.96 to 0.98, which were deemed acceptable.

Due to the complexity of the impinger method for CG, a Draeger Pac III Single Gas Monitor also was used for the monitoring of phosgene. This device was new, and its factory calibration was used in all monitoring.

Chlorine (Cl<sub>2</sub>)— Cl<sub>2</sub> was monitored with a new Draeger MiniWarn Multi-Gas Monitor, which was factory calibrated. The Cl<sub>2</sub> calibration was checked by injecting a known amount of high concentration gas into a known volume of clean air in a Tedlar gas sampling bag, to give a concentration of a few ppm. The MiniWarn readings in this check were within 5% of the expected standard concentrations.

Arsine (SA)—SA was quantified by GC using its relative response compared with xenon as the internal standard. Xenon is a ubiquitous component of ambient air with a concentration of 90 ppb. Triplicate injections of SA standards at 0.3, 3, and 50 ppm and one method blank yielded a linear calibration plot having an r<sup>2</sup> value of 0.996.

A new Draeger MiniWarn Multi-Gas Monitor also was used for monitoring SA. This monitor was factory calibrated, but the SA calibration was checked by injecting a known amount of high concentration gas into a known volume of clean air in a Tedlar gas sampling bag, to give a concentration of a few ppm. The MiniWarn readings in this check were within 5% of the expected standard concentrations.

Sarin (GB) and sulfur mustard (HD)—Calibration standards for these CW agents were prepared by diluting stock agent to 50 nanograms/ $\mu$ L, and then injecting  $\mu$ L volumes of that standard onto sorbent tubes used for the agent sampling. These tubes were thermally desorbed in the same manner as for all sample tubes, and a regression of peak area versus amount of agent was prepared.

Total hydrocarbons—The THC analyzer used to document the interferent levels provided in testing was calibrated by filling a 25-L Tedlar bag with 33 ppm of propane in air from a commercial gas standard. Since propane is a three-carbon molecule, this standard constitutes a THC

concentration of 99 ppmC. This standard was used for calibrating the THC analyzer throughout the verification. Clean air from the room was used for zeroing.

#### 4.1.2 Instrument Checks

The RAID-Ms were operated and maintained according to the vendor's instructions throughout the verification test. Maintenance was performed according to predefined RAID-M diagnostics. Daily operational check procedures for the RAID-Ms were performed with a vendor-supplied simulant tube. Proper response of the RAID-Ms to the simulant was required before testing could proceed.

#### 4.2 Audits

#### 4.2.1 Performance Evaluation Audit

As described in Section 3.2.7.5, a PE audit was conducted to assess the quality of reference measurements made in the verification test. For TICs, the PE audit was performed once during the verification test by analyzing gaseous standards that were obtained from different suppliers than those providing the standards used during the testing. The acceptable tolerance for each target TIC was ±20%. For the two CW agents, the PE audit was conducted by an analyst other than the person who prepared the normal calibration standards, using a sorbent trap spiked with one of the target agents. This spiked trap was prepared using a different batch of agent than that used to prepare calibration standards. The expected tolerance on the CW agent PE audit was ±30%. Table 4-1 shows that the results of the PE audits were all within the target tolerances.

Table 4-1. Summary of Performance Evaluation Audit Results(a)

TIC or				Agraement
CW Agent	Sample	Concentration	Result	Agreement (%)
CW Agent	Sample	Concentration	Kesuit	(70)
AC	Standard (Cylinder 7035)	10,000 ppm	42.2 ppm	
	PE Audit Std (Cyl. SD-10271)	967 ppm	4.1 ppm	0.5
CG	Standard (Cylinder RR37345)	990.7 ppm	1.92 ppm	
	PE Audit Std (Cyl. NA021189)	997 ppm	1.93 ppm	0.1
$\mathrm{Cl}_2$	Standard (Cylinder RA64239)	5,811 ppm	9.90 ppm	
	PE Audit Std (Cyl. 1C1857)	5,830 ppm	8.95 ppm	9.9
GB	Standard (GB50 4-16-04 PR)	50 ng/tube	$49.9 \; ng^{(b)}$	
	PE Audit Std (GB50 4-18-04	50 ng/tube	43.2 ng	13.4
	FM)			
HD	Standard (1000 4-16-11 PR)	1,000 ng/tube	966 ppm	
	PE Audit Std (1000 4-19-04 FM)	1,000 ng/tube	1,034 ppm	7.0

a) As noted in Section 3.2.7.5, PE audits were not performed for SA (audit standard had incorrect concentration) or CK (no independent standard available).

24

<sup>(</sup>b) ng = nanogram.

#### 4.2.2 Technical Systems Audit

The Battelle Quality Manager also conducted a technical systems audit (TSA) to ensure that the verification test was performed in accordance with the test/QA plan<sup>(1)</sup> and the ETV QMP.<sup>(2)</sup> As part of the audit, the Battelle Quality Manager reviewed the reference sampling and analysis methods used, compared actual test procedures with those specified in the test/QA plan,<sup>(1)</sup> and reviewed data acquisition and handling procedures. Observations and findings from this audit were documented and submitted to the Battelle Verification Test Coordinator for response. The only technical finding of the TSA concerned carrying out test procedures for CK while reference analyses for that compound were still in progress. A deviation form was prepared and approved, describing that departure from the test/QA plan.<sup>(1)</sup> This deviation had no impact on data quality, as all CK concentrations were confirmed to be within the target specification. The records concerning the TSA are permanently stored with the Battelle Quality Manager.

#### 4.2.3 Data Quality Audit

All of the data acquired during the verification test were audited, not merely the 10% required by the test/QA plan. Battelle's Quality Manager traced the data from the initial acquisition, through reduction and statistical analysis, to final reporting, to ensure the integrity of the reported results. All calculations performed on the data undergoing the audit were checked.

The data quality audit disclosed three items requiring the preparation and approval of deviation forms:

- The use of the impinger/visible absorption method for CG, rather than the planned GC/FID method, which proved ineffective
- The failure to reverse the order of low and high concentrations of HD in the oscillating concentration test with that CW agent (low/high was always done, and not high/low)
- The lack of completion of one-over-one review of all test data sheets within two weeks after data collection.

The first two of these had a negligible impact on the test data. The impact of the third deviation was minimized by the fact that all data sheets were scrutinized in compiling electronic data files for statistical evaluation. In the end, only a handful of small corrections to the test data were needed as a result of this deviation, with minimal impact on the verification results.

#### 4.3 QA/QC Reporting

Each assessment and audit was documented in accordance with the test/QA plan. (1) Once the assessment report was prepared, the Battelle Verification Test Coordinator ensured that a response was provided for each adverse finding or potential problem and implemented any necessary follow-up corrective action. The Battelle Quality Manager ensured that follow-up corrective action was taken. The results of the TSA were sent to the EPA.

## Chapter 5 Statistical Methods

To extract the most information about RAID-M performance from the test procedures, a statistical analysis of the test results was performed whenever appropriate. Such an analysis used all available data to explore the impact of test parameters on RAID-M performance. Section 5.1 summarizes the statistical approaches and the parameters tested. The performance parameters of response threshold and battery life were assessed with simple comparisons that did not require statistical analysis. Section 5.2 describes the analyses used for these performance parameters.

## **5.1 Statistical Analyses**

Several successive readings of the RAID-M responses, alternating with RAID-M baseline readings, were recorded in each step of the test procedure. That is, for each RAID-M and each TIC or CW agent, such readings were recorded with each concentration, at a range of temperature and RH conditions (Table 3-3), both with and without each interferent. In addition, readings were obtained at the normal temperature and RH conditions at different RAID-M starting conditions. These data were the basis for the statistical analysis of RAID-M performance.

The statistical analyses focused on the following performance parameters:

- Response time
- Recovery time
- Accuracy
- Repeatability
- False positives/false negatives

by considering the following explanatory variables:

- Identity of the target TIC or CW agent
- Concentration of the target TIC or CW agent
- Temperature
- Humidity
- Start state (i.e., warmed up, cold start, etc.)
- Identity and presence/absence of interferent.

The RAID-M does not provide quantitative concentration readings, but instead provides various qualitative outputs, including an intensity of response (0 to 8 bar scale), alarm/no alarm state, and identification of the TIC or CW agent that is detected. As a result, the statistical analysis often relied on methods for categorical rather than continuously variable data.

Appendix A of this report provides a detailed description of the statistical procedures used for the verification of the performance parameters listed above. In brief, a cumulative logit model was used for analysis of the effects of TIC or CW agent identity and concentration, temperature, RH, and RAID-M start state on RAID-M response. Response time and recovery time were evaluated using an analysis of variance (ANOVA) model. The treatment of recovery times took into account the presence of censored data for recovery time (i.e., RAID-M response that did not return to baseline within 600 seconds and was therefore truncated at a recovery time of 600 seconds). Accuracy and repeatability were evaluated using a binomial logit model, and false negatives and false positive were evaluated using a cumulative logit model. The mathematical forms of the models used are given in Appendix A.

## 5.2 Other Analyses

The data used to evaluate the response threshold were the five replicate RAID-M readings obtained at each succeeding TIC or CW agent concentration. These data were tabulated, along with the corresponding reference method data that established the challenge concentration. The response threshold was determined by inspection as the lowest reference method concentration that produced a positive RAID-M response in all replicate runs. In this evaluation, any positive RAID-M response was taken as detection of the target TIC or CW agent, i.e., RAID-M response of 1 bar was sufficient in terms of the response threshold evaluation.

Both battery life and the effectiveness of battery operation were assessed. Battery life is reported as the total time from start-up to battery exhaustion when a RAID-M is warmed up and operated continuously solely on battery power at room temperature and 50% RH. This time was measured from initial start-up to the point in time when the RAID-M no longer responded to a challenge mixture of AC in air. Battery effectiveness was evaluated by comparing responses obtained while operating on battery power to those obtained under identical sampling conditions immediately before switching from alternating current power to battery power.

## Chapter 6 Test Results

As discussed in Chapter 5 and detailed in Appendix A, cumulative logit models and other approaches were used to test for the effects of different conditions on RAID-M performance. The following sections present summaries of the statistical model results from these tests. An extensive and detailed presentation of the modeled statistical results is included as Appendix B of this report. In addition, a tabulation of all test data is included as Appendix C to this report. Each section of Appendix B indicates which test identifications (IDs) in Appendix C were used for the evaluation. Also note that the statistical evaluations reported in this chapter were not applied to SA, since the RAID-Ms did not respond to that TIC.

Two CW agents were tested, GB and HD. In contrast to the TIC testing, in most testing each CW agent was tested at only one concentration level, and the levels were different for each agent: GB was tested at about 0.4 IDLH and HD at about 0.7 of its AEGL-2 level. Because of the concentration difference across agents, the effects of concentration and agent on the behavior of the RAID-M could not be separated. For this reason, the CW agent statistical analyses described below and in Appendix B do not include agent or concentration effects. In descriptive comments about figures that summarize results, differences may be noted that appear across the agents. It should be kept in mind that such differences may be the result of a difference in agents, a difference in concentration, or a mixture of both effects.

## **6.1 Response Time**

Results of the response time analysis are summarized here and detailed in Appendix B, Section B.1. Table 6-1 summarizes data used for the analysis of response time and other performance parameters. This table is for illustration purposes, and for brevity the TIC results shown are drawn only from data obtained at the IDLH concentration.

The RAID-M units produced an alarm and a visual display of response typically within a few seconds after the initiation of sampling of one of the TICs. For CG, CK, and AC, the modeled geometric mean response times were all between 3 and 5 seconds. Response for Cl<sub>2</sub> was slightly slower, with a modeled geometric mean response time of about 9 seconds. Neither temperature nor relative humidity had any significant effect on the TIC response times. The effect of start state (cold start, hot start) was tested with AC only, and no effect was found of start state on the response time for that TIC.

Table 6-1. Summary Illustrative Data from RAID-M Verification Test

sponse lange	Response Time Range (s)	Recovery Time Range (s)	Alarms (Indicated
bars)	2-4	185-299	Chemical) 10/10 (CY)
8	2-4 2-5		
		112-323	10/10 (CY)
8	3-5	70-201	10/10 (CY)
8	2-6	51-82	10/10 (CY)
8	1-4	25-36	10/10 (CY)
7-8	3-6	>600	10/10 (CY)
5-8	2-8	14-24	6/10 (CY)
			4/10 (CL2)
8	2-4	17-39	9/10 (CY)
			1/10 (CL2)
7-8	1-4	13-36	5/10 (CY)
			5/10 (CL2)
4-8	3-10	1-32	9/10 (CY)
			1/10 (CL2)
4-8	3-4	8-29	6/10 (CY)
			4/10 (CL2)
8	2-4	15-18	5/10 (CY)
			5/10 (CL2)
8	2-5	7-9	10/10 (CLX)
8	2-5	6-9	10/10 (CLX)
8	2-5	7-9	10/10 (CLX)
8	2-4	6-9	10/10 (CLX)
6-8	2-4	6-9	10/10 (CLX)
8	2-4	6-8	10/10 (CLX)
8	3-15	24-35	10/10 (CL2)
8	2-23	21-34	10/10 (CL2)
6-8	3-15	0-39	10/10 (CL2)
7-8	5-16	21-58	10/10 (CL2)
5-6	4-31	0-35	5/6 (CL2)
ın - NR)			,
7-8	10-17	12-35	10/10 (CL2)
3	7-14	42-55	5/5 (GB)
3	10-13	66-73	5/5 (GB)
3	7-12	36-54	5/5 (GB)
1-2	8-13	20-23	5/7 (GB)
ns - NR)	0 13	20 23	3// (GB)
2	8-14	17-26	5/5 (GB)
2	19-26	47-67	5/5 (GB)
3-8	5-9	24-44	10/10 (HD)
3-8 4-8	4-8	29-73	10/10 (HD) 10/10 (HD)
	3-7		10/10 (HD) 10/10 (HD)
3-8		34-105	, ,
			10/10 (HD)
			5/5 (HD)
	7-9	34-41	4/5 (HD)
ı	2-7 7 4-8 <u>In - NR)</u>	2-7 3-10 7 5-7 4-8 7-9	2-7 3-10 11-22 7 5-7 19-22 4-8 7-9 34-41 m - NR)

<sup>(</sup>a) Data shown are for illustration, TIC results shown are from data at IDLH level only. NR - indicates "No Response"

For the CW agents, response times were about 5 to 8 seconds for HD under most conditions, and about 10 seconds for GB under most conditions. The only exception was that at the lowest temperature (approximately 5°C) the modeled response time for GB was approximately 20 seconds. Relative humidity had no effect on the response times for either GB or HD.

## **6.2 Recovery Time**

Results of the recovery time analysis are summarized here and detailed in Appendix B, Section B.2. Recovery time results also are illustrated in Table 6-1. In general, recovery times differed considerably from one TIC to another. For CG, recovery times were always within 10 seconds, whereas those for CK and Cl<sub>2</sub> ranged from about 10 to 40 seconds under nearly all conditions. Recovery times for AC were longer and more variable, ranging from about 15 seconds to 600 seconds (the maximum value recorded) over all tests. AC recovery times were shorter at higher temperatures, with model-estimated mean values of about 280 seconds at the lowest temperature (5°C), about 100 seconds at room temperature, and about 35 seconds at the highest temperature (35°C). Recovery times for AC also were shorter at lower concentrations, with model-estimated mean values of less than 50 seconds at 0.1 IDLH and about 210 seconds at IDLH concentrations. Relative humidity did not have a consistent effect on TIC recovery times.

The effect of the start state of the RAID-Ms was tested with AC at the IDLH level and was found to have a strong effect on the recovery times. Under fully warmed-up conditions, the model-estimated geometric mean recovery time for AC was about 240 seconds; under cold start conditions after an overnight hot soak (at 40°C), the recovery time was only slightly longer, with a geometric mean of 286 seconds. However, after a cold soak (5 to 8°C) or with cold start-up from room temperature, modeled geometric mean recovery times for AC were at least 600 seconds (recovery times were not recorded beyond that time).

Model-estimated geometric mean recovery times for the CW agents under normal conditions were about 50 seconds for GB and about 34 seconds for HD. For both agents, higher temperatures led to shorter recovery times, with estimated recovery times of about 24 seconds for GB and 16 seconds for HD at the highest temperature (35°C). Relative humidity did not have a consistent effect on recovery time for HD, but for GB recovery time increased as relative humidity decreased. GB model-estimated recovery time ranged from 69 seconds at the lowest humidity to 46 seconds at the highest humidity.

## 6.3 Accuracy

Results of the accuracy analysis are summarized here and detailed in Appendix B, Section B.3. Results of tests that involved alternating different challenge concentrations, as opposed to alternating clean air and a single challenge concentration, are detailed in Appendix B, Section B.8. Accuracy results also are illustrated in Table 6-1. The RAID-M units were 100% accurate in identifying the target TICs and CW agents under the majority of test conditions. Accuracy below 100% was observed primarily for CK. Estimated mean accuracy for CK ranged

from about 50% to nearly 100% at both 0.1 and 1 IDLH, with the lowest accuracy occurring at the lowest temperature and at the highest relative humidity. As Table 6-1 illustrates, the inaccurate responses for CK occurred when the RAID-Ms identified CK as chlorine gas ( $\text{Cl}_2$ ). Accuracy was less than 100% for CG only at 0.1 IDLH and the highest temperature tested, and for  $\text{Cl}_2$  only at IDLH and the highest relative humidity. TIC accuracy was 100% in all other tests and conditions.

Neither temperature nor relative humidity had a significant effect on accuracy for the CW agents. Excluding those interferences that suppressed GB response, overall accuracy for GB was 97.5%, based on 81 total readings. Overall accuracy for HD was 99.4%, based on 161 readings in 17 total tests, including all interferent tests. In both cases, inaccuracies were in the form of absence of response to the agent. It should be noted that, in addition to correctly identifying GB or HD, in most tests with the CW agents, the RAID-Ms also gave an indication of "HN."

The accuracy of RAID-M response when alternating between different TIC or CW agent challenge concentrations also was essentially the same as that when alternating between clean air and a challenge concentration.

## 6.4 Repeatability

Results of the repeatability analysis are summarized here and detailed in Appendix B, Section B.4. Repeatability of response was always perfect for AC, as the RAID-M units always gave a full-scale reading of 8 bars at both 0.1 and 1 IDLH for this TIC. Concentration had a marked effect on response repeatability for CG, with a model-estimated mean repeatability of 50% at 0.1 IDLH, but with perfect repeatability at 1 IDLH. Concentration had minimal effect on response repeatability for CK and Cl<sub>2</sub>, with modeled repeatability values near or above 90% for both these TICs. On the other hand, temperature had no effect on repeatability for CG, but strongly affected repeatability for CK and Cl<sub>2</sub>. Repeatability was 100% for these TICs at the lowest temperature (5°C), but the model-estimated mean dropped to about 60% at the highest temperature (35°C). Humidity had a strong effect on repeatability of response for Cl<sub>2</sub>, with better repeatability at lower humidity. Repeatability of response also was reduced to a model-estimated mean of about 50% for CK at medium and high humidities.

The modeled repeatability of response times varied among the TICs, from 24 to 28 %RSD for AC to 70 to 75 %RSD for CK, but neither temperature nor relative humidity affected the repeatability of response times.

The repeatability of TIC recovery times also varied from 11 %RSD for AC to 47 %RSD for CK and was unaffected by temperature. However, for CG and Cl<sub>2</sub>, repeatability of recovery times was better at 1 IDLH than at 0.1 IDLH; for Cl<sub>2</sub>, repeatability of recovery times was very sensitive to humidity. Model-estimated mean %RSD for Cl<sub>2</sub> was 20 to 30% at low and medium humidities, but at high humidity was 110%.

The RAID-M response data for the CW agents showed no significant effect of relative humidity on the repeatability of response for either GB or HD. However, the repeatability of RAID-M

response for HD was highly dependent on temperature. For that agent, the model-estimated mean response repeatability was very low at the lowest temperature (5°C), but increased to about 70% at the highest temperature (35°C). In contrast, GB mean response repeatability was 100% at low and medium temperatures, but fell below 60% at the highest temperature.

The analysis of response time repeatability for GB and HD suggested that HD response times became less repeatable (more variable) with increasing temperature (observed means of 12, 21, and 36 %RSD at low, medium, and high temperatures, respectively), whereas for GB response time variability peaked at 27 %RSD at the medium temperature (22°C). HD response time repeatability showed little dependence on relative humidity, and for GB the response time was least repeatable (most variable) at medium humidity (27 %RSD).

The analysis of recovery time repeatability for the CW agents showed similar results to that of response time repeatability. HD recovery times became less repeatable as temperature increased (observed means of 9, 19, and 27 %RSD at low, medium, and high temperatures, respectively), whereas temperature had little effect on the repeatability of recovery times for GB. For both GB and HD, the repeatability of recovery times was lowest at the highest humidity (means of 14 and 44 %RSD for GB and HD, respectively), though only GB showed a consistent trend of recovery time repeatability with humidity.

## 6.5 Response Threshold

The response threshold for each TIC was determined by challenging the RAID-Ms with successively lower concentrations until both RAID-Ms no longer responded. Table 6-2 provides the results for each TIC for the response threshold test. The concentrations used in each of these tests are given in the table and are well below the IDLH or other target concentration used in the other challenge/clean air tests. The responses listed in the table give the results for three successive challenge/clean air cycles.

For AC and CK, the response threshold could be determined only as an upper limit of < 0.06 ppm and < 0.6 ppm, respectively. Both RAID-Ms were responding with readings of 4 to 6 bars at the lowest concentration obtainable. Thus, the actual threshold response levels of the RAID-M for AC and CK are well below these upper limits. For CG, the response threshold was different for the two RAID-Ms tested. RAID-M BW000701 had a response threshold between 0.08 and 0.15 ppm. RAID-M BW01335 had a response threshold between 0.15 and 0.33 ppm. For Cl<sub>2</sub>, both RAID-Ms had a response threshold between 0.25 and 0.50 ppm. No response threshold test was conducted for SA because the RAID-Ms did not respond to SA at the IDLH concentration level. The response threshold for GB was 0.0035 to 0.007 ppm (0.02 to 0.04 mg/m³) and for HD was 0.01 to 0.02 ppm (0.07 to 0.13 mg/m³).

## 6.6 Temperature and Humidity Effects on Response

The results of investigating temperature and relative humidity effects on RAID-M response are summarized here and are detailed in Appendix B, Section B.5. Table 6-1 also illustrates T/RH

effect data. In general, the effects of temperature and relative humidity on RAID-M response were small. Considering the TICs, full-scale (i.e., 8 bars) responses to AC, CK, and Cl<sub>2</sub> were observed in most tests at both 0.1 and 1 IDLH. Responses to CG varied more widely than those for the other TICs, and responses to 0.1 IDLH of CG were clearly lower than corresponding responses to 1 IDLH levels.

Table 6-2. Response Threshold Data for the TIC and CW Agent Testing

	RAID-M Identification Number					
TIC (Concentration)	BW000701	BW001001	BW01335			
AC (0.50 ppm)	8 bars	8 bars	NA			
AC (0.25 ppm)	8 bars	8 bars	NA			
AC (0.13 ppm)	7 bars	8 bars	NA			
AC (0.06 ppm)	4-5 bars	6 bars	NA			
CK (2.50 ppm)	8 bars	NA	7-8 bars			
CK (1.25 ppm)	8 bars	NA	5-7 bars			
CK (0.63 ppm)	5-6 bars	NA	4 bars			
CG (0.33 ppm)	2 bars	NA	1 bar			
CG (0.15 ppm)	1 bar	NA	No Response			
CG (0.08 ppm)	No Response	NA	No Response			
Cl <sub>2</sub> (0.50 ppm)	7-8 bars	NA	6 bars			
Cl <sub>2</sub> (0.25 ppm)	No Response	NA	No Response			
GB (0.007 ppm) (0.04 mg/m <sup>3</sup> )	1 bar (8 - 16 s)	NA	1 bar (17 - 28 s)			
GB (0.0035 ppm) (0.02 mg/m <sup>3</sup> )	No Response	NA	No Response			
HD (0.02 ppm) (0.13 mg/m <sup>3</sup> )	NA	NA	1 bar (8 s)			
HD (0.01 ppm) (0.07 mg/m <sup>3</sup> )	NA	NA	No Response			

The effect of temperature on RAID-M response was most noticeable for CK, with higher predicted RAID-M readings at low temperature. A significant effect of relative humidity also was found. For CK and Cl<sub>2</sub>, higher predicted RAID-M readings were seen at lower humidity.

Considering the CW agents, relative humidity did not have a significant effect on RAID-M response to either GB or HD. A significant effect of temperature on response was found, with the highest temperature (35°C) producing slightly lower readings for both GB and HD.

### **6.7 Interference Effects**

The results of investigating interference effects on RAID-M response are summarized here and are detailed in Appendix B, Section B.6. Table 6-3 summarizes data used for the analysis of

33

interference effects. This table is for illustration purposes, and for brevity the TIC results shown are drawn only from data obtained at the IDLH concentration.

Table 6-3. Summary Data Illustrating Interference Effects

TIC/CN/A (a)	Technology	Response Range	Response Time Range	Recovery Time Range	Alarms (Indicated
TIC/CWA <sup>(a)</sup>	Interferent	(bars)	(s)	(s)	Chemical)
AC	Control	8	2-4	185-299	10/10 (CY)
	Paint Fumes	8	2-4	347-590	10/10 (CY)
	Floor Cleaner	8	2-4	141-342	10/10 (CY)
	DEAE	8	1-4	123-448	10/10 (CY)
	Gasoline Engine	8	1-4	100-201	10/10 (CY)
	Exhaust Air Freshener	8	2-4	142-522	10/10 (CY)
CK	Control	5-8	2-8	14-24	6/10 (CY)
	00114101		_ 0	- · - ·	4/10 (CL2)
	Paint Fumes	8	2-6	12-37	10/10 (CY)
	Floor Cleaner	8	2-9	14-39	10/10 (CY)
	DEAE	8	2-6	12-38	10/10 (CY)
	Gasoline Engine	8	2-9	13-40	10/10 (CT) 10/10 (CY)
	Exhaust	Ü	2 )	15 40	10/10 (01)
	Air Freshener	8	2-5	13-35	10/10 (CY)
CG	Control	8	2-5	7-9	10/10 (CLX)
	Paint Fumes	8	1-5	5-9	10/10 (CLX)
	Floor Cleaner	8	3-5	6-10	10/10 (CLX)
	DEAE	8	2-4	6-9	10/10 (CLX)
	Gasoline Engine	8	2-5	6-9	10/10 (CLX)
	Exhaust				` /
	Air Freshener	4-8	1-5	6-9	10/10 (CLX)
Cl <sub>2</sub>	Control	8	3-15	24-35	10/10 (CL2)
- 2	Paint Fumes	4-5	6-17	0-24	3/10 (CL2)
		(7 runs - NR)			(- /
	Floor Cleaner	10 runs - NR	NA	NA	0/10
	DEAE	7-8	3-14	31->600	10/10 (CL2)
	Gasoline Engine	8	5-14	18-60	10/10 (CL2)
	Exhaust				
	Air Freshener	7-8	3-16	18-35	10/10 (CL2)
GB	Control	3	7-14	42-55	5/5 (GB)
	Paint Fumes	1-5	18-31	30->600	2/5 (GB)
		(3 runs - NR)			` /
	Floor Cleaner	6 runs - NR	NA	NA	0/6
	DEAE	4	8-11	25-47	5/5 (GB)
	Gasoline Engine	5	8-15	36-49	5/5 (GB)
	Exhaust		0 10	55.7	0,0 (02)
	Air Freshener	5 runs - NR	NA	NA	0/5
HD	Control	3-8	5-9	24-44	10/10 (HD)
	Paint Fumes	3-4	3-6	8-25	10/10 (HD)
	Floor Cleaner	6-7	5-6	26-38	5/5 (HD)
	DEAE	3-4	4-8	20-38	5/5 (HD)
	Gasoline Engine	3-4	5-10	26-35	5/5 (HD)
		J <del>-4</del>	5-10	20-33	SIS (IID)
	Exhaust				

<sup>(</sup>a) Data shown are for illustration, TIC results shown are from data at IDLH level only. NR - indicates "No Response"

The interference effects varied widely among the different TICs. For AC and CG, response was essentially unaffected by the presence of interferents along with the TIC. For CK, the RAID-M response was generally higher when both CK and an interferent were present than when CK alone was present. This was true for all five interferents tested. On the other hand, RAID-M response for Cl<sub>2</sub> was strongly suppressed by the presence of latex paint fumes and floor cleaner vapors. In fact, no Cl<sub>2</sub> response was seen at all when floor cleaner vapors and Cl<sub>2</sub> were present together. These interference effects for CK constitute a form of false positive, in that CK response is exaggerated due to the presence of an interferent. The interference effects for Cl<sub>2</sub> constitute false negatives, since the indication of Cl<sub>2</sub> by the RAID-M is suppressed partly or completely by the presence of an interferent.

RAID-M response times for the TICs were not affected to any practical extent by the presence of the interferents, with the exception that Cl<sub>2</sub> response was completely eliminated by the floor cleaner vapors.

The interferents also had relatively little impact on RAID-M recovery times for the TICs, with the exception that Cl<sub>2</sub> response was completely eliminated by the floor cleaner vapors. At the IDLH concentrations used in interferent testing, the estimated mean recovery time for AC was about 230 seconds with no interferents present, and ranged from 140 to 270 seconds with all interferents except latex paint fumes. With paint fumes, the RAID-M estimated recovery time for AC was lengthened to nearly 500 seconds. Also, the estimated recovery times for Cl<sub>2</sub> were all less than about 30 seconds, except with DEAE present, when estimated recovery time was lengthened to nearly 100 seconds. Other than these two observations, the interferents had no effect on TIC recovery times.

Substantial interference effects were observed in detection of the CW agents. RAID-M response to GB was sharply reduced by the presence of air freshener vapors, floor cleaner vapors, and latex paint fumes. In fact, no response at all was observed to GB in the presence of air freshener vapors and floor cleaner vapors. However, the presence of those interferents caused the RAID-M to indicate the presence of other CW agents, specifically VX and GA. For HD, RAID-M response was reduced by about half by the presence of air freshener vapors, latex paint fumes, DEAE, and gasoline engine exhaust hydrocarbons. However, floor cleaner vapors had no effect on RAID-M response to HD. The presence of any of the interferents caused the RAID-Ms to indicate the presence of VX and, occasionally, GA or GB. These indications were in addition to the indications of HN, which often occurred in the GB and HD tests, with or without interferents.

The RAID-M response time for HD was little affected by the presence of the interferents. For GB, response time was unaffected by DEAE or gasoline engine exhaust; however, the presence of latex paint fumes lengthened the estimated response time (from about 9 to about 24 seconds). Note that no response at all was observed to GB in the presence of air freshener vapors and floor cleaner vapors.

In all cases but one, the RAID-M estimated recovery times for GB and HD were actually shorter in the presence of interferents than with the agent alone. Estimated mean recovery times for GB were about 50 seconds without interferents and about 30 to 42 seconds with DEAE, latex paint

fumes, or gasoline exhaust hydrocarbons present. Note that no response at all was observed to GB in the presence of air freshener vapors and floor cleaner vapors. Estimated recovery times for HD were about 33 seconds without interferents and about 19 to 30 seconds with DEAE, latex paint fumes, floor cleaner vapors, or gasoline exhaust hydrocarbons present. The only exception was that estimated recovery time for HD was 57 seconds in the presence of air freshener vapors.

False positives were observed only for floor cleaner vapors and DEAE. In 16 trials with floor cleaner vapors, a false positive response was observed five times, for a 31% false positive rate. In eight trials with DEAE, a false positive response was observed three times, for a 38% false positive rate. The RAID-M responses in these cases were relatively low, never exceeding 3 bars on the display. When the false positives occurred, the RAID-Ms erroneously identified the interferent as being the nerve agent VX.

#### 6.8 Cold-/Hot-Start Behavior

Analysis of the effects of insufficient warm-up time, under start-up conditions ranging from cold (5 to 8°C) to hot (40°C), are summarized here and detailed in Appendix B, Section B.7. Table 6-4 illustrates the data obtained in testing for cold-/hot-start effects, showing the RAID-M unit used, the start condition, sequential experiment number, delay time, response reading, response and recovery times, and indicated chemical. Such testing was conducted only with AC, at the IDLH concentration.

In all cases of start-up without sufficient warm-up, whether from cold, room temperature, or hot conditions, the RAID-M response initially gave readings that were lower than those observed under fully warmed-up operation. This difference was most noticeable in the first few RAID-M readings and diminished as measurements continued, as the RAID-M warmed up during operation. The delay times of the RAID-Ms (i.e., the time after being powered on before the RAID-Ms were ready to give a reading) varied with start condition and from one RAID-M unit to another. Starting cold at room temperature, two RAID-M units each showed delay times of about 40 seconds. Starting from a cold-soak condition, the delay times of both units were longer, at 1 minute 2 seconds and 2 minutes 59 seconds, respectively. The longest delay time for RAID-M BW000701 occurred when starting after a hot soak, at 2 minutes 5 seconds; and the longest delay time of all was observed for RAID-M BW01335 after a hot soak, at 13 minutes 52 seconds. Recovery times were lengthened to over 600 seconds in operation from a cold temperature or room temperature cold start, but response times were not affected by start-up conditions. The RAID-Ms always correctly identified AC with the "CY" designation, regardless of start condition.

### 6.9 Battery Life

The battery life test was conducted by placing fully charged batteries in the RAID-Ms. The RAID-Ms were then powered on and allowed to warm up fully according to the manufacturer's directions. The delay or standby time before the RAID-M was ready to give a reading was 31 seconds and 24 seconds for the RAID-M BW000701 and BW01335 units, respectively. An initial response time test was conducted with AC at the IDLH concentration level. The

Table 6-4. Summary of Cold-/Hot-Start Test Data

RAID-M Unit	Start Condition	Experiment Number	Delay Time (mm:ss)	Response (bars)	Response Time (s)	Recovery Time (s)	Alarm <sup>(a)</sup> (Indicated Chemical
BW000701	Control	1		8	4	185	CY
	(Fully	2		8	4	203	CY
	Warmed Up)	3		8	3	227	CY
		4		8	4	229	CY
		5		8	2	234	CY
	Room	1	00:40	2	3	445	CY
	Temperature	2		1	2	>600	CY
	(Cold Start)	3		7	3	>600	CY
		4		7	5	>600	CY
		5		8	4	>600	CY
	Cold	1	01:02	4	2	440	CY
	Temperature	2	<u>-</u>	7	5	>600	CY
	(Cold Start)	3		8	5	>600	CY
		4		8	4	>600	CY
		5		8	2	>600	CY
	Hot	1	02:05	6	4	>600	CY
	Temperature	2	02.03	8	3	402	CY
	(Cold Start)	3					
				8	6	413	CY
		4		8	5	424	CY
		5		8	2	435	CY
BW001001	Control	1		8	4	220	CY
	(Fully Warmed Up)	2		8	4	256	CY
	warmed Op)	3		8	4	294	CY
		4		8	4	299	CY
		5		8	4	297	CY
	Room	1	00:44	3	5	>600	CY
	Temperature	2		6	6	>600	CY
	(Cold Start)	3		7	5	>600	CY
		4		8	4	>600	CY
		5		8	6	>600	CY
	Cold	1	02:59	2	5	>600	CY
	Temperature	2		7	5	>600	CY
	(Cold Start)	3		8	5	>600	CY
		4		8	2	>600	CY
		5		8	2	>600	CY
BW01335	Hot	1	13:52	5	5	321	CY
	Temperature	2		7	3	161	CY
	(Cold Start)	3		8	4	142	CY
		4		8	4	150	CY
	1	5		8	3	156	CY

<sup>(</sup>a) All tests conducted with AC as the challenge TIC, at the IDLH level.

RAID-Ms then sampled clean air for approximately 30 minutes, and then the AC mixture was sampled again. This procedure was repeated with the RAID-Ms operating continuously until the batteries were depleted and the RAID-Ms no longer responded to the presence of the AC.

Table 6-5 provides the results for the battery life test. The elapsed time and the response (and response time) for each test are provided in the table. Both units of the RAID-M gave a response of 8 bars and showed response times of a few seconds every time the AC challenge was sampled. RAID-M BW000701 responded in a normal fashion until the "EMPTY" battery indicator was given, which was quickly followed by the "ERROR E070" and finally the powering off of the detector. The battery life for this detector was 6 hours and 29 minutes. RAID-M BW01335 also responded in a normal fashion until the same battery indicator and error message were given. The battery life for this detector was 7 hours and 52 minutes. There was no degradation of response or response time with either RAID-M as the batteries approached

Table 6-5. Responses Recorded from the RAID-Ms in Battery Life Testing<sup>(a)</sup>

		F	RAID-M Identification Number					
		BW000	0701	BW01335				
Test	Time (from start-up)	Response (Response Time)	Battery Indicator	Response (Response Time)	Battery Indicator			
1	3 minutes	8 bars (2 s)	Full	8 bars (5 s)	Full			
2	33 minutes	8 bars (2 s)	Full	8 bars (3 s)	Full			
3	1 hour 4 minutes	8 bars (4 s)	Full	8 bars (2 s)	Full			
4	1 hour 34 minutes	8 bars (2 s)	Full	8 bars (2 s)	Full			
5	2 hours 5 minutes	8 bars (2 s)	Full	8 bars (3 s)	Full			
6	2 hours 35 minutes	8 bars (4 s)	Full	8 bars (4 s)	Full			
7	3 hours 6 minutes	8 bars (3 s)	Full	8 bars (3 s)	Full			
8	3 hours 36 minutes	8 bars (3 s)	Full	8 bars (3 s)	Full			
9	4 hours 7 minutes	8 bars (3 s)	Full	8 bars (4 s)	Full			
10	4 hours 37 minutes	8 bars (5 s)	Full	8 bars (2 s)	Full			
11	5 hours 8 minutes	8 bars (3 s)	Full	8 bars (3 s)	Full			
12	5 hours 37 minutes	8 bars (5 s)	Half	8 bars (3 s)	Full			
13	6 hours 8 minutes	8 bars (3 s)	Half	8 bars (5 s)	Half			
	6 hours 24 minutes		Empty					
	6 hours 28 minutes		Error E070					
	6 hours 29 minutes		<b>Power Off</b>					
14	6 hours 39 minutes			8 bars (2 s)	Half			
15	7 hours 9 minutes			8 bars (4 s)	Half			
16	7 hours 41 minutes			8 bars (4 s)	Half			
	7 hours 47 minutes				Empty			
	7 hours 50 minutes				Error E070			
	7 hours 52 minutes				Power Off			

All battery life tests were conducted with AC as the challenge TIC at the IDLH concentration of 50 ppm  $(50 \text{ mg/m}^3).$ 

38

depletion. However, the RAID-M battery indicator readings did not change from "Full" to "Half" until the useful battery life was nearly gone. Thus, this indicator should not be taken as a measure of the battery life remaining.

## **6.10 Operational Characteristics**

General performance observations noted during verification testing:

- Two Libraries—The RAID-M internal software consists of a Library A and a Library B. Library A is the CW agent library and opens automatically when powering up the RAID-M. Library B is the TIC library. Use of the RAID-M to detect unknown contaminants would require switching between Libraries A and B to assure monitoring of both CW agents and TICs. A few compounds, such as hydrogen cyanide, are detectable by both libraries; however, they do not have the same alarm identification in both libraries. Hydrogen cyanide alarms as AC in Library A and as CY (cyanides) in Library B. Also, the simulants utilized to ensure that the RAID-M is functioning properly are identified with different acronyms in each library.
- Simulants—Two simulants are used to test the RAID-M prior to any challenge test. The RAID-M gives a response to these simulants, which allows the user to determine whether the RAID-M is functioning properly. Testing with simulant prior to use provides the only indication that the detector is functioning properly, unless an error message is provided by the instrument.
- Display and Alarms—The liquid crystal display (LCD) visual display can be backlit, which allows easy reading of the display. The visual alarms include a strip of flashing red lights above the display window and a bar reading on the display window with an acronym identifying the chemical detected. The red lights flash slowly for an alarm reading of 1 to 4 bars and quickly for an alarm reading of 5 to 8 bars. The identification of the chemical is easy to read, but distinguishing the bars corresponding to the concentration of the chemical is more difficult, especially between 4 and 8 bars. The audible alarm is very loud and obvious. The audible alarm is slower for an alarm reading of 1 to 4 bars and more rapid for an alarm reading of 5 to 8 bars. The LCD display can provide information on up to three compounds at one time, with the compounds grouped by agent types in the display. Given that the RAID-M operates in both positive and negative ion modes essentially simultaneously, this ability to display multiple compound IDs is valuable.
- Push Switches—Two push switches on the RAID-M allow the operator to easily change parameters. The parameters that can be changed include turning the audible alarm on/off, changing from one library to the other, and resetting the RAID-M after a consumable has been changed.
- Consumables—Three consumables are used when operating the RAID-M: a backflush filter (carbon), a drying tube, and an ammonia dopant. All of these consumables, when depleted, affected the performance of the RAID-M, causing behavior such as longer clear-down times and lower concentration responses. The RAID-M provided error messages when these

consumables should be changed; however, these messages are based on metered time of use, not on the actual state of the consumable. Verification testing took place over a five-month period; the RAID-Ms were operating continuously (24 hours/day) for those five months, with challenge testing five days a week for the five-month period. The ammonia dopant was changed once during this five-month period. The backflush filter and carbon trap were changed much more frequently as needed. Changing consumables involved powering the detector off, unscrewing a cap, removing the expired consumable, replacing with a new consumable, replacing the cap, powering the detector on, and resetting the time on the display.

- Instrument Warm-Up—It was observed during testing that the detector response (bar reading shown) was strongly dependent on the length of time the RAID-M was allowed to warm up prior to use. The RAID-M did not give as high a concentration reading for a challenge when initially powered on as when the RAID-M was allowed to fully warm up.
- Positive/Negative Ion Identification—The RAID-M continuously switches between modes that detect and identify either positive or negative ions, at intervals of a couple of seconds. That is, the RAID-M will detect positive ions for 2 to 3 seconds and then will switch and detect negative ions. This is a continuous cycle that allows the rapid identification of chemicals producing both positive and negative ions.
- Double Alarms—If the RAID-M produced two simultaneous identifications of different chemicals during a verification test procedure, two possible responses were provided. If one of the chemicals provided a positive ion and the other a negative ion, both chemicals would be identified in the display window. For example, if both GB and HD were present, a response would be provided for both in the display window. On the other hand, if both chemicals present provided a positive (or negative) ion, the chemical with the higher alarm concentration (i.e., more bars indicated) would be identified in the display window, and the other chemical would not be identified.
- Errors—Several error readings were provided by the RAID-Ms over the course of verification testing. Some of the errors encountered included indications that the backflush filter or carbon trap needed to be changed, that the battery was empty, and that an electrical fault had occurred.
- Instrument Failure—A total of three RAID-Ms were used in the verification test. Two of the three RAID-Ms failed at some point in the test. One RAID-M gave an electrical fault error message and was unusable. The other RAID-M displayed an error message and could not be recovered without connecting the instrument to a laptop computer and overriding the fault. Verification testing took place over five months; the RAID-Ms were operated continuously, with challenge testing five days a week.
- Vendor Support— Before the verification, a vendor representative trained three Battelle employees to operate the RAID-Ms. Testing proceeded according to the representative's recommendations on how to operate the RAID-M for testing. The vendor supplied all of the consumables necessary for verification testing and responded promptly when information was needed or an instrument needed to be replaced.

■ Back-Flush—The RAID-M went into a "back-flush" mode on certain chemicals when the instrument reading reached 8 bars. These chemicals included the two simulants used to verify instrument operation and the CW agents HD and GB. In this mode, the intake airflow is reversed to flush the detected chemical out of the RAID-M. The "back-flush" mode does not apply when a TIC is detected.

### **6.11 Cost**

The purchase price of the RAID-M portable IMS, as used in this verification test, is approximately \$13,000.

## Chapter 7 Performance Summary

This chapter summarizes the overall performance results found in testing of the RAID-M portable IMS with both TICs and CW agents. This summary focuses on aspects of the performance that are most important in field use of the RAID-M by first responders. Consistent with that use, most test procedures were conducted with challenge levels of the TIC or CW agent that were at or near IDLH concentrations. Section 3.2.1 describes the challenge levels used. Response thresholds were tested by stepping downard in concentration from these challenge levels. A general observation is that the RAID-Ms were not programmed to respond to arsine, so no results are reported for that TIC.

RAID-M response to AC, CK, and Cl<sub>2</sub> was very sensitive. Full-scale readings were obtained even at concentrations of a few percent or less of the IDLH concentrations for these TICs.

The RAID-Ms provided an audible and visual alarm within 3 to 5 seconds response time after exposure to CG, CK, and AC, and within about 9 seconds for Cl<sub>2</sub>. Response times for GB and HD were about 10 seconds and 5 to 8 seconds, respectively. Over the ranges of 5 to 35°C and <20 to >80% relative humidity, temperature and RH had no effect on response time for any TIC or CW agent, with the sole exception that at 5°C the response time for GB was lengthened to about 20 seconds. Response times for AC also were unaffected by operating the RAID-M from a cold start (i.e., with insufficient warm-up time).

RAID-M recovery times (i.e., the time needed for the RAID-M to return to baseline after the end of exposure to a TIC or CW agent) varied widely, depending on the TIC or CW agent sampled and also on the sampling conditions. Average recovery times for CG, CK, and Cl<sub>2</sub> were relatively consistent under all conditions and were always less than 10 seconds for CG and generally 10 to 40 seconds for CK and Cl<sub>2</sub>. Recovery times for AC ranged from 15 seconds to over 600 seconds, with the fastest recovery times occurring at low concentrations and high temperatures. Recovery times for GB and HD averaged about 50 seconds and about 34 seconds, respectively, at normal temperature, with average recovery times reduced by about half at higher temperatures. The overall ranges of all observed recovery times were about 15 to 70 seconds for GB and about 10 to 100 seconds for HD. Relative humidity had minimal effect on recovery times. In operation from a cold start, the recovery time for AC was lengthened to at least 600 seconds.

The RAID-Ms were 100% accurate in identifying the TIC being sampled under almost all test conditions. Accuracy in identifying the CW agents also was high: overall accuracy for GB was 97.5% (excluding data from interferences that suppressed GB response), and for HD was 99.4%,

when all test data were included. In addition to correctly identifying GB and HD, the RAID-Ms usually gave a secondary indication of "HN" when testing with these agents. Accuracy below 100% occurred primarily for CK, with the lowest accuracy (~50%) at high humidity and low temperature. The inaccuracy for CK occurred in the form of misidentification of CK as chlorine gas (Cl<sub>2</sub>).

The accuracy of RAID-M response when alternating between different challenge concentrations also was essentially the same as that when alternating between clean air and a challenge concentration.

The repeatability, or consistency, of RAID-M response, response times, and recovery times also was evaluated. AC showed the most consistent responses and also the lowest %RSD of response times and recovery times. In fact, repeatability of response for AC was perfect, as full-scale readings consistently resulted at the test concentrations. The %RSD of recovery times was low for AC primarily because of the long average recovery times for that TIC under many conditions (see above). Response and recovery times were most variable for CK. RAID-M readings and recovery times for Cl<sub>2</sub> were strongly affected by relative humidity, with the most variability at high humidity. For the CW agents, the repeatability of RAID-M response to HD improved as temperature increased, but the repeatability of response and recovery times for HD lessened. Repeatability of response for GB did not vary substantially with test conditions, and the only effect on repeatability was that recovery times for GB were less repeatable at high humidity.

The response thresholds of the RAID-M were <0.06 ppm for AC, <0.6 ppm for CK, 0.08 to 0.33 ppm for CG, and 0.25 to 0.5 ppm for  $Cl_2$ , where the ranges are for two RAID-M units. The response threshold for GB was 0.0035 to 0.007 ppm, and for HD was 0.01 to 0.02 ppm.

Temperature and relative humidity had little effect on RAID-M response to the TICs and CW agents. Higher readings for CK were generally found at lower temperatures, and higher readings for CK and Cl<sub>2</sub> were generally found at lower humidity. Slightly higher readings for both CW agents also were found at lower temperatures.

Interferents likely to be present indoors had large effects with individual TICs and CW agents. In terms of false negatives, RAID-M response for Cl<sub>2</sub> was sharply reduced by latex paint fumes and floor cleaner vapors; the floor cleaner vapors resulted in zero response for Cl<sub>2</sub>. Response to GB was sharply reduced by latex paint fumes, floor cleaner vapors, and air freshener vapors; the latter two interferents resulted in zero response for GB. Response for HD was reduced by about half by latex paint fumes, air freshener vapors, DEAE, and gasoline engine exhaust hydrocarbons. The interferents also caused the RAID-Ms to incorrectly report the presence of other agents, such as VX or GA. False positive responses occurred only with floor cleaner vapors and DEAE. Both of these interferents produced small positive responses in about one-third of the trials; in those cases the RAID-Ms incorrectly identified the interferent as the nerve agent VX. The interferents had little effect on response or recovery times for the TICs or CW agents.

Operating the RAID-M with insufficient warm-up time reduced the initial responses to AC, regardless of whether the cold start occurred after storage at 5°C, at room temperature, or at 40°C. The response time for AC was not affected by operating from a cold start, but the recovery time was lengthened in such operation. The delay time (time for the RAID-M to be ready for a

first reading after start-up) ranged from 40 seconds to about 3 minutes under cold-start conditions, except for one unit that showed a delay time of nearly 14 minutes after a 40°C soak and cold start.

The useful operating lives for fully charged batteries in two RAID-M units in continuous operation were found to be 6 hours 29 minutes and 7 hours 52 minutes, respectively.

Several operational characteristics of the RAID-M were noted during testing. In general, the RAID-M was easy to use, gave clear alarms and a readable and informative display, and provided error and diagnostic messages. The RAID-M automatically switched between positive and negative ion detection modes at intervals of a few seconds, allowing detection of a wide variety of chemicals. Among the most important other operational characteristics are

- The use in the RAID-M of two separate software libraries, one for TICs and one for CW agents, necessitating switching between libraries to detect both types of chemicals.
- The need for three types of consumables (carbon backflush filter, drying tube, and ammonia dopant), the first two of which needed to be replaced several times during the nearly fivemonth test period. RAID-M error messages calling for replacement of consumables are based on metered time of use, not on the actual state of the consumable.
- The need for proper warm-up of the RAID-M before use, to assure that full response is achieved when monitoring starts.
- The failure during testing of two of the three RAID-Ms used in this verification, one due to an electrical fault, and the other to an apparently incorrect error message that required overriding the message by connection to a laptop computer.

# **Chapter 8 References**

- 1. Test/QA Plan for Verification of Portable Ion Mobility Spectrometers for Detection of Chemicals and Chemical Agents in Buildings, Battelle, Columbus, Ohio, July 2003.
- 2. Environmental Technology Verification Program Quality Management Plan, Version 2.0, December 2002.
- 3. "Determination of Phosgene in Air," in *Methods of Air Sampling and Analysis*, Third Edition, J. P. Lodge, ed., Lewis Publishers, Chelsea, Michigan, 1989.
- 4. Spicer, C. W., Gordon, S. M., Holdren, M. W., Kelly, T. J., and Mukund, R., *Hazardous Air Pollutant Handbook: Measurements, Properties, and Fate in Ambient Air*, ISBN 1-56670-571-1, CRC Press, Boca Raton, Florida, 2002.
- 5. Turpin, J. "Direct Steam Injection Humidification: Is It Safe for Building Occupants?" *Engineered Systems*, www.esmagazine.com/CDA/ArticleInformation/features/BNP\_\_\_ Features\_\_Item/0,2503,23246,00.html.