November 2004

Environmental Technology Verification Report

MICROSENSOR SYSTEMS INC.
HAZMATCAD™ PLUS
SURFACE ACOUSTIC WAVE/ ELECTROCHEMICAL DETECTOR

Prepared by
Battelle

Battelle
The Business *of* Innovation

Under a contract with

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

November 2004

Environmental Technology Verification Report

ETV Safe Buildings Monitoring and Detection Technology Verification Program

Microsensor Systems Inc. HAZMATCAD™ Plus Surface Acoustic Wave/ Electrochemical Detector

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Notice

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 six verification 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 Technology 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

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List of Abbreviations

AC	hydrogen cyanide
AEGL	acute exposure guideline level
ANOVA	analysis of variance
Cl_2	chlorine
CG	phosgene
CK	cyanogen chloride
CW	chemical warfare
DEAE	N,N-diethylaminoethanol
EC	electrochemical
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
IDLH	immediately dangerous to life and health
L	liter
L/min	liter per minute
μg	microgram
$\mu g/m^3$	microgram per cubic meter
μL	microliter
mg/m^3	milligram per cubic meter
mL	milliliter
mL/min	milliliter per minute
MSD	mass selective detection
PE	performance evaluation
ppb	part per billion
ppm	part per million
ppmC	part per million of carbon
psig	pound per square inch gauge
QA	quality assurance

QMP	quality management plan
RH	relative humidity
SA	arsine
SAW	surface acoustic wave
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 Technology Verification Program, which is funded by EPA and conducted by Battelle. In this program, Battelle recently evaluated the performance of the Microsensor Systems Inc. HAZMATCAD Plus portable detector, which uses surface acoustic wave (SAW) and electrochemical (EC) technologies for detecting chemical warfare (CW) agents and toxic industrial chemicals (TICs), respectively.

Chapter 2
Technology Description

The objective of the ETV Safe Buildings Monitoring and Detection Technology 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 HAZMATCAD Plus portable SAW/EC detector made by Microsensor Systems Inc. Following is a description of the HAZMATCAD Plus, based on information provided by the vendor. The information provided below was not subjected to verification in this test.

The HAZMATCAD Plus is a hybrid system using SAW sensors and ECs to detect CW agents and TICs, respectively. SAW sensors are small, solid-state devices that are extremely sensitive to minute changes in mass. These devices are coated with specific polymers that selectively absorb contaminants. The polymer surface responds rapidly and reversibly to nerve and blister agents. Using an array of three coated SAW sensors provides a response pattern that is unique to CW agents. The architecture of the SAW system allows for a high level of specificity in a complex sample. A preconcentrator is used to prepare sample for delivery to the SAW array. In this test, the HAZMATCAD Plus was operated in its “Fast” response mode, which employs a 20-second preconcentration cycle. The ECs are semi-selective and provide a rapid response to contaminants. Amperometric ECs use an electrolyte that is sealed behind a gas-permeable membrane. Gases and vapors diffuse through the membrane and dissolve in the electrolyte. Subsequent oxidation/reduction processes release electrons that are collected at an electrode. The resulting current signal is proportional to the amount of gas or vapor sampled.

Figure 2-1. Microsensor Systems Inc. HAZMATCAD Plus

HAZMATCAD Plus software logs and date stamps all alarms or systems faults. The HAZMATCAD Plus weighs 1.5 kilograms (3.4 pounds) including batteries.

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 HAZMATCAD Plus, a commercially available, portable SAW/EC detector, was to evaluate its ability to detect toxic chemicals and chemical agents in indoor air. This verification focused on the scenario of a portable detector 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/QA plan(1) that was developed according to the requirements of the Quality Management Plan (QMP) for the ETV program.(2) The following performance characteristics of the HAZMATCAD Plus 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 HAZMATCAD Plus with known vapor concentrations of target TICs and CW agents. HAZMATCAD Plus 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 HAZMATCAD Plus was tested after a cold start (i.e., without the usual warm-up period) and after hot storage to evaluate the delay time before readings could be obtained and the response speed and accuracy of the HAZMATCAD Plus once readings were obtained. All readings of a target chemical were obtained with the HAZMATCAD Plus operated on battery power. Battery life was determined as the time until HAZMATCAD Plus 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 that all tests would be conducted with two HAZMATCAD Plus units (Units 22 and 27). However, as a result of occasional problems, testing continued in a few cases with only one HAZMATCAD Plus unit.

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 stages: 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 ($COCl_2$, designated CG)
- Chlorine ($C1_2$, no military designation)
- Arsine (AsH_3, designated SA).

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

It should be noted that the HAZMATCAD Plus was programmed to detect all of these compounds expect CK; the library in the HAZMATCAD Plus at the time of testing did not include an alarm for the presence of CK. However, the instruments were challenged briefly with CK to confirm absence of response. Also, it should be noted that the chemical identification that the HAZMATCAD Plus units were programmed to display upon detecting a chemical varied among the target compounds. For AC, the HAZMATCAD Plus identifiers were "BLOD" at higher concentration levels and "CHOK" at lower concentration levels, indicating the presence of a blood or choking agent. For CG, the HAZMATCAD Plus identifier was "CHOK," indicating the presence of a choking agent. For SA, the identifier was "HYDR," indicating the presence of a hydride. For Cl_2, the HAZMATCAD Plus identifier was "HALO," indicating the presence of a halogen. For the CW agents, GB was identified as "G," and HD was identified as "H." HAZMATCAD Plus Unit 22 did not alarm when challenged with GB, so the GB test results are based on Unit 27 responses only.

Table 3-1 summarizes the concentrations of each TIC and CW agent used in this verification test. For the TICS AC, CK, CG, SA, and Cl_2, tests were conducted at the immediately dangerous to life and health (IDLH) levels. For the CW agents GB and HD, testing was conducted at a concentration level that produced a mid-scale to full-scale reading on the HAZMATCAD Plus under normal temperature and humidity conditions. The concentration used for GB was 0.39 part per million (ppm) (2.2 milligrams per cubic meter (mg/m^3), which is 11 times the IDLH concentration of 0.035 ppm (0.2 mg/m^3). 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 of Table 3-1.

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

Chemical	Challenge Concentrations	Type of Level
AC	50 ppm (50 mg/m^3)	IDLH[(a)]
CK	20 ppm (50 mg/m^3)	IDLH
CG	2 ppm (8 mg/m^3)	IDLH
SA	3 ppm (10 mg/m^3)	IDLH
Cl_2	10 ppm (30 mg/m^3)	IDLH
GB	0.39 ppm (2.2 mg/m^3)	11*IDLH
HD	0.6 ppm (4 mg/m^3)	7*AEGL[(b)]

[(a)] IDLH value for CK estimated from value for AC.

[(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/m^3) for HD is based on a 10-minute exposure.

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. Cold-/hot-start behavior and battery life were tested only with AC.

3.2.3 Test Locations

Two laboratories were used to conduct the verification test. 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 detector and filter tests using CG, AC, CK, SA, and Cl_2 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.

Table 3-2. Evaluations

Performance Parameter	Objective	Comparison Based On
Response Time	Determine rise time of HAZMATCAD Plus response	HAZMATCAD Plus readings with step rise in analyte concentration
Recovery Time	Determine fall time of HAZMATCAD Plus response	HAZMATCAD Plus readings with step decrease in analyte concentration
Accuracy	Characterize reliability of identification of TICs or CW agents	HAZMATCAD Plus identifier display in testing each TIC or CW agent
Repeatability	Characterize consistency of HAZMATCAD Plus readings with constant analyte concentration	HAZMATCAD Plus readings with constant input
Response Threshold	Estimate minimum concentration that produces HAZMATCAD Plus response	Reference method results
Temperature and RH Effects	Evaluate effect of temperature and RH on HAZMATCAD Plus performance	Repeat above evaluations with different temperature and RH
Interference Effects	Evaluate effect of building contaminants that may interfere with HAZMATCAD Plus performance	Sample interferents and TICs/CW agents together (and interferents alone[(a)])
Cold Start	Characterize start-up performance after cold storage	Repeat tests with no warm-up[(a)]
Hot Start	Characterize start-up performance after hot storage	Repeat tests with no warm-up[(a)]
Battery Life	Characterize battery life and performance	HAZMATCAD Plus readings with constant input,[(a)] while depleting batteries in continuous operation

[(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 verification test is outlined in Figure 3-1. Since HAZMATCAD Plus 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 CK took only one day because this TIC is not detected by the HAZMATCAD Plus.

Test 1: Vapor challenge with TIC
Alternating clean air with immediately dangerous to life and health (IDLH) level concentration of TIC five times with HAZMATCAD Plus operating on battery power, fully warmed up per manufacturer's instructions prior to testing, and room temperature (22 ± 3°C) and 50 ± 5% RH.

Test 2: Vapor challenge with TIC at reduced concentration
Test 1 is repeated at a lower concentration giving mid-range on-scale readings (only if off-scale response at IDLH). The concentration that gives a mid-range on-scale reading is then referred to as the target concentration for all subsequent tests.

Test 3: Vapor challenge with TIC at increased concentration
Test 1 is repeated at roughly 10 times the IDLH concentration (only if no response at IDLH).

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: Target/low/clean air challenge
Test 1 is repeated by alternating target concentrations, a low concentration (either 0.1 IDLH or response threshold concentration) and clean air six times and alternating order of low concentration and target concentration.

Test 6: Vapor challenge with TIC at room temperature, low humidity
Test 1 is repeated at room temperature (22 ± 3°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 ± 3°C) and 80% 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 ± 3°C) and 50% 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 ± 3°C) and 80% 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 ± 3°C) and 50% 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: Room temperature, cold start behavior
Repeat Test 1 with the HAZMATCAD Plus at room temperature for a minimum of 12 hours and no warm-up.

Test 14: Cold-/cold-start behavior
Repeat Test 1 after the HAZMATCAD Plus has been kept refrigerated (5-8°C) overnight for a minimum of 12 hours, with no warm-up.

Test 15: Hot-/cold-start behavior
Repeat Test 1 after the HAZMATCAD Plus has been kept heated (40°C) overnight for a minimum of 12 hours, with no cool-down or warm-up.

Test 16: Battery test
Repeat Test 1 with the HAZMATCAD Plus operating on battery power. The TIC at target 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 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, perform Test 3, then go to Step 4.
Step 2b: If the response in Test 1 is on scale, skip to Step 3 and perform all subsequent tests at the IDLH concentration.
Step 2c: If the response in Test 1 is off-scale, perform Test 2. Establish the concentration that gives a mid-range on-scale response and proceed with Step 3, using that established concentration in all subsequent tests.
Step 3: Perform Test 4 (if not already done), Tests 5 through 10, and Test 12 at the concentration(s) determined above. For the first TIC, also perform Test 11 and Tests 13 through 16.
Step 4: Return to Step 1 and repeat Test 1 through 10 and 12 for all other TICs.
Step 5: Repeat Tests 1 through 10 and 12 for all CW agents

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

Table 3-3. Test Schedule

Chemical	Test Dates
AC	May 4–14, 2004
CK	May 17, 2004
CG	May 25–June 1, 2004
SA	May 18–24, 2004
Cl_2	June 2–14, 2004
GB	July 16-August 12, 2004
HD	August 16-27, 2004

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 used for each compound, and the applicable concentration range of each method. For AC and CK, samples were injected directly for determination by gas chromatography (GC) with flame ionization detection (FID). A

Table 3-4. Primary Reference Methods

Analyte	Concentration Range (ppm)	Sampling Method	Analysis Method
AC	0.05 to 100	Air sample injected directly	GC/FID
CK	2 to100	Air sample injected directly	GC/FID
CG	0.05 to 10	Collection in impingers with nitrobenzyl pyridine	Visible absorption at 475 nanometers
Cl_2	0.1 to 100	Continuous EC detector with chlorine-specific sensor	Continuous detection
SA	0.05 to 100	Capillary GC with syringe injection from bag sample	MSD
GB	0.01 to 100	Air sample collected in gas sample bag	GC/FPD
HD	0.01 to 100	Air sample collected in gas sample bag	GC/FPD

colorimetric method, using a liquid reagent solution in a small impinger train was used for CG.(3) Cl_2 was determined by a continuous EC analyzer with a Cl_2-specific sensor to allow rapid determination of Cl_2 levels delivered to the HAZMATCAD Plus 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 a gas sample bags and determined by GC with flame photometric detection (FPD), according to existing HMRC test procedures.

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

The analytical instrument used for AC and CK reference measurements 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 HAZMATCAD Plus.

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.(3) 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 nanometers using a spectrophotometer.

Cl_2 was monitored with a Draeger MiniWarn Multi-Gas Monitor, which was calibrated by preparing known concentrations of Cl_2 in gas sample bags.

SA was determined using an Agilent 6890 GC coupled to a 5970 mass selective detector. Air samples [100 microliter (µL)] were injected onto a GS-Q capillary column held at -30°C. SA was separated from xenon at a constant flow of 3 mL/minute (mL/min) helium using the following temperature program: hold at -30°C for 1 minute; ramp at 50°C/minute to 150°C; hold at 150°C for 5 minutes (total time 9.6 minutes). The injector temperature was maintained at 100°C, operated in splitless mode, and was purged after 0.5 minute at 5 mL/min. 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.

The reference method for GB and HD involved collecting the agents by flowing air from the test apparatus into gas sample bags. The agent concentrations were then determined using a capillary GC with FPD. Concentrations were determined based on a linear regression of peak area with the amount of agent.

A continuous FID was used for determining the total hydrocarbon (THC) content of interferent mixtures provided to the HAZMATCAD Plus 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 HAZMATCAD Plus to selected levels of the interferents in clean air, to see whether the interferents generated a positive response from the HAZMATCAD Plus when no TICs or CW agents were present. Each interferent also was introduced to the HAZMATCAD Plus along with each TIC and CW agent to determine false negatives, i.e., whether the interferent prevents the HAZMATCAD Plus 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. Interferent Test Concentrations

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

The appropriate concentrations of latex paint fumes were established directly by measurements in and around a freshly painted office. Samples were obtained using a 25-liter (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 the information cited in Section 3.2.6.1 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 an indoor air quality conference. 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 $\mu g/m^3$ 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 HAZMATCAD Plus. 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.(5) 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 10,020 ppm AC (Cylinder B0005506, Scott Specialty Gases); 991 ppm CG (Cylinder RR37345), 997 ppm SA (Cylinder KE-50368), and 5,811 ppm Cl_2 (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 hydrocarbon-free 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. DAAD13-03-H-0003.

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 AC, CK, CG, Cl_2, 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.

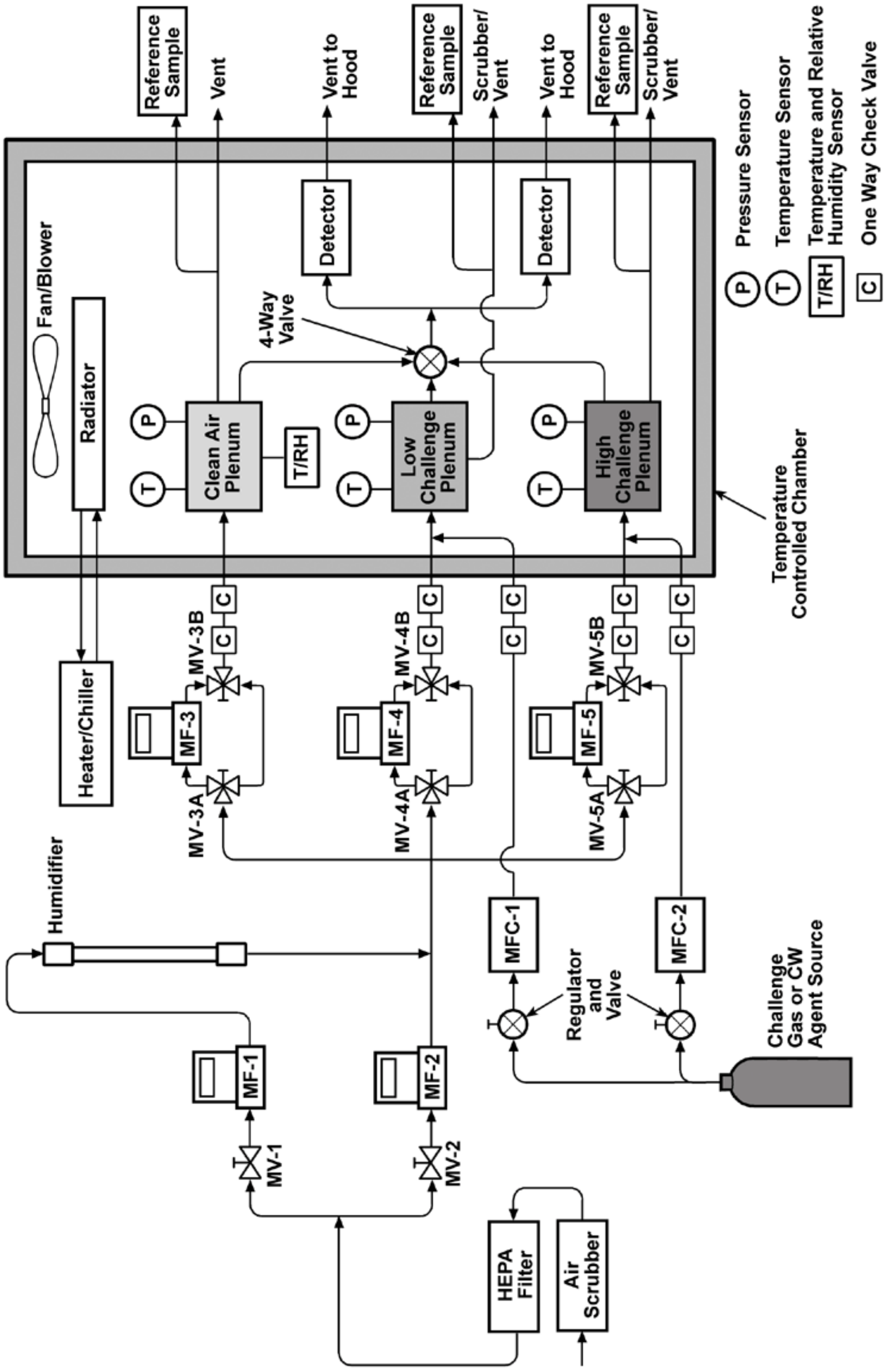

Figure 3-3. Test System Schematic

3.2.7.3 Temperature/Humidity Control

The HAZMATCAD Plus was evaluated at the temperature and humidity conditions indicated by an “X” in Table 3-6. Both the delivered air temperature and the HAZMATCAD Plus 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.

Table 3-6. Temperature and Relative Humidity Conditions

	Temperature (°C)		
RH (%)	**5 ± 3**	**22 ± 3**	**35 ± 3**
$\leq$ 20		X	
50 ± 5	X	X	X
80 ± 5		X	X

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 using approximately 0.1 liter per minute (L/min) flow of clean air. 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 µL of 51 neat liquid components and 250 µL 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 µL of liquid neat DEAE to a final pressure of 1,200 psig. In all cases these cylinder gases or concentrated vapor streams were diluted to the appropriate level by addition to the large flows of clean air passing through the test apparatus (Figure 3-3).

3.2.7.5 Performance Evaluation Audit Materials

As part of the QA effort in this verification test, a performance evaluation (PE) audit was performed on reference methods used to confirm the TIC concentrations provided to the HAZMATCAD Plus. 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 TIC PE audit was within 20%.

For the TICs, the PE audit standards were 10,000 ppm AC (Cylinder LL320), 1,043 ppm CG (Cylinder AS51117), 1,090 ppm SA (Cylinder AS73486), and 6,015 ppm Cl_2 (Cylinder LL23078), all from Linde Gas LLC.

A comparable PE audit could not be done for the CW agents because of the lack of independent standards. In lieu of a PE audit for the CW agents, check samples were prepared at the HMRC by an analyst other than the staff who conducted routine calibration of the reference method. These samples were analyzed by the same approach used for analysis of calibration samples from GB and HD testing, and the results were compared.

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 HAZMATCAD Plus units 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 HAZMATCAD Plus units' background, the four-way valves connected to the two HAZMATCAD Plus units were switched to the clean air plenum to collect baseline data.

After the background measurement, the four-way valves connected to the two HAZMATCAD Plus units were switched to one of the challenge plenums to allow the HAZMATCAD Plus units 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 HAZMATCAD Plus units. The reference methods described in Section 3.2.5 were used to confirm that the TIC concentrations in the challenge plenums were within ± 20% of the target level (or within 35% of the target level for the CW agents). Concentrations outside those tolerance ranges triggered a repeat of any test procedures conducted since the last analysis.

3.3.1 Response Time

To evaluate HAZMATCAD Plus response time, the environmental conditions were established at the target conditions of 22 ± 3ºC and 50 ± 5% RH. Initially 10 L/min of the clean humidified air passed through the clean air plenum. The HAZMATCAD Plus sampled the clean air for a minimum of 30 seconds or until a stable reading was indicated, but not exceeding 10 minutes, to obtain a baseline for the HAZMATCAD Plus. The clean air plenum also was sampled with the appropriate reference method. This sampling took place after the HAZMATCAD Plus readings had 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. For the TICs, the generator operating conditions and the dilution flow were adjusted as needed to establish a challenge concentration within ± 20% of the IDLH or other target. For the CW agents, a delivered concentration within 35% of the target level was acceptable. Reference samples were collected and analyzed immediately to establish the challenge concentration and demonstrate stability. 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_2 when the continuously monitoring reference method reached a stable reading.

After a stable reading was obtained from the HAZMATCAD Plus on background air, and the challenge mixture was stable and at the target concentration, the four-way valve at the HAZMATCAD Plus inlet was switched to sample from the challenge plenum. The response of the HAZMATCAD Plus was recorded and the time to produce an alarm was considered the response time. When feasible, based on the time response of the reference method, the challenge vapor concentration also was determined by reference method sampling periodically during the procedure. The HAZMATCAD Plus sampled from the challenge plenum for a minimum of 30 seconds, up to a maximum of 10 minutes. The high challenge vapor concentration was determined by the reference method as frequently as possible during the procedure. For AC, CK, and Cl_2, a reference sample was taken prior to, during, and after every set of tests with the HAZMATCAD Plus. For CG and SA, a reference sample was taken prior to and at the conclusion of every set of tests.

After the challenge sampling, the sample inlet four-way valve was switched to again sample from the clean air plenum. The time required for the HAZMATCAD Plus to clear, i.e., the time to return to starting baseline or non-alarm readings, was recorded as the recovery time. After a maximum of 10 minutes, regardless of whether the HAZMATCAD Plus had returned to baseline, subsequent cycles of alternating challenge/clean air sampling were carried out, controlled by the four-way valve. A total of five such challenge/clean air cycles were completed.

The same sampling procedure was carried out at different temperature and RH conditions or challenge concentration to evaluate temperature and RH effects and response thresholds. For each TIC or CW agent, the initial test was conducted at the levels shown in Table 3-1. If the instrument gave an over-scale reading when challenged at the initial level at the normal temperature and RH conditions (22ºC and 50% RH), a lower challenge concentration was chosen that provided an on-scale reading. All subsequent tests for that TIC or CW agent used that lower challenge concentration. If the instrument did not respond to the IDLH or other initial concentration selected, then the response threshold procedure in Section 3.3.5 was conducted; but, all subsequent tests planned for that TIC or CW agent were eliminated. Otherwise, testing proceeded as described.

Following the five challenge/clean air cycles, six cycles were conducted in which the HAZMATCAD Plus sampled sequentially from the high, low, and clean air challenge plenums. The high challenge plenum provided the IDLH or other primary target concentration, and the low challenge plenum provided a concentration of approximately 0.1 times that level, or the response threshold (see Section 3.3.5), whichever was greater. Clean air was sampled alternately with sampling from the challenge plenums, and the order of sampling from the high (H) and low (L) challenge plenums was reversed, i.e., the order of sampling was clean air/H/L/clean air/L/H/clean air/H/L/ . . ,. for a total of six such cycles. This procedure simulated use of the HAZMATCAD Plus in locations having different degrees of contamination.

3.3.2 Recovery Time

The time for the HAZMATCAD Plus 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. Those measurements confirmed that the target TIC or CW agent was present at the appropriate challenge concentration. The degree to which the HAZMATCAD Plus correctly identified the challenge TIC or CW agent was evaluated as the measure of accuracy.

3.3.4 Repeatability

Repeatability was assessed using data obtained from the five repeated challenge/clean air cycles or the high challenge/low challenge 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 HAZMATCAD Plus unit was evaluated by repeating the procedure in Section 3.3.1 at successively lower (or if necessary, higher) concentrations. The response threshold was determined at the baseline environmental condition of 22 ± 3ºC and 50 ± 5% 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 at least 10 times lower that the IDLH or target concentration was chosen. If there was no response at the starting test concentration, then the concentration of the challenge was increased by a factor of two. Similarly, if the HAZMATCAD Plus units responded to the starting concentration, then the challenge concentration was decreased by a factor of two. The increase or decrease in concentration was continued accordingly, until the response threshold had been bracketed. The minimum concentration producing a HAZMATCAD Plus unit response was denoted as the response threshold. If the duplicate HAZMATCAD Plus unit tested simultaneously produced different instrument responses, the concentrations were varied as needed to assess the response thresholds of each HAZMATCAD Plus unit.

3.3.6 Temperature and Humidity Effects

The tests described in Section 3.3.1 were repeated at the target concentrations shown in Table 3-1, over the range of environmental conditions shown in Table 3-6. 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 HAZMATCAD Plus 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. The test chemical generation was independently controlled to generate interferent in the absence or presence of the test chemical. This allowed interference effects to be evaluated with the interferent alone and with each interferent and TIC or CW agent together. Testing with the interferent alone allowed evaluation of false positive responses, and testing with the interferent and chemical together allowed evaluation of false negatives. The test procedures also allowed observation of interferent effects on the response time and recovery time of the HAZMATCAD Plus. The target concentrations of the planned interferents are shown in Table 3-5. Those concentrations are shown in terms of the equivalent THC concentration in ppmC. These target concentrations are based on actual indoor measurements by Battelle or on published data, as described in Section 3.2.6.

Interferent testing involved only one interferent at a time. Testing was done by alternately sampling clean air and the interferent mixture, 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. Testing with interferents alone involved alternately sampling from the clean air plenum and then from the challenge plenum, to which only the interferent in clean air was delivered. The same process was used for testing with interferents and TICs or agents together, with the two compounds diluted together in humidified air delivered to the challenge plenum. The same TIC and CW agent concentrations used in the initial testing under Section 3.3.1 were used in this test, i.e., the levels shown in Table 3-1.

A response from the HAZMATCAD Plus with the interferent alone was recorded as a false positive; and 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.

The replicate test runs conducted with the interferent plus TIC or agent also allowed the response time and recovery time of the HAZMATCAD Plus to be assessed with interferents present. Differences in response and recovery times, relative to those in previous tests with only the TIC or 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 only with the TIC AC in a manner similar to the response time test in Section 3.3.1. Prior to these tests, however, the HAZMATCAD Plus units were not allowed to warm up per the manufacturer's recommendation.

The cold-start test was conducted twice, once with the HAZMATCAD Plus units at room temperature and, subsequently, at reduced temperature, prior to start-up. In the former test, the HAZMATCAD Plus units were stored with the power off at 22 ± 3˚C for at least 12 hours prior to testing. The cold-start effect was assessed at the baseline conditions of 22 ± 3˚C and 50 ± 5 % RH. The time from powering up the HAZMATCAD Plus units to their first readiness to provide readings was considered start-up delay time. The response time (as defined in Section 3.3.1) was measured, followed by the recovery time. Repeatability and accuracy in five replicate clean air/challenge cycles with AC were also noted.

For the reduced temperature cold start, HAZMATCAD Plus units were placed in a refrigerated enclosure (5 to 8˚C) with the power off for at least 12 hours overnight. At the start of the next test day, the cold-start test was repeated, again using AC and using the same baseline conditions (22ºC and 50% RH) and again recording the start-up delay time and other performance parameters.

For the hot-start test, the HAZMATCAD Plus units were placed in a heated enclosure at 40 ± 3ºC for at least 12 hours overnight. At the start of the next test day, the hot-start test was conducted in the same fashion as in the cold-start test, with AC at the baseline test conditions (22ºC and 50% RH). Only one cold-/hot-start test was performed per day.

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 HAZMATCAD Plus units were turned on and allowed to warm up, and an initial response time test was performed (see Section 3.3.1). A single TIC (AC) was used in this evaluation. The indicated concentration signal from the HAZMATCAD Plus units was recorded. At each sampling of the AC mixture, the battery level of the HAZMATCAD Plus units was recorded. The HAZMATCAD Plus units sampled clean air for 30 minutes, and then the AC mixture was sampled again. This procedure was repeated with each HAZMATCAD Plus unit operating continuously until it 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 HAZMATCAD Plus were evaluated by means of the observations of test operators and inquiries to the HAZMATCAD Plus 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 HAZMATCAD Plus units 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 HAZMATCAD Plus 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 HAZMATCAD Plus as tested. Estimates for key maintenance items also were requested from the vendor.

Chapter 4
Quality Assurance/Quality Control

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

4.1 Equipment Calibration

4.1.1 Reference Methods

The reference methods used for determining TICs and CW 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_2 testing, blank reference samples were run before, during, and after each set of tests. The sequence of reference sampling thus included running a blank on clean air and then switching to challenge gas and taking a reference sample immediately prior to challenging the HAZMATCAD Plus units with the challenge gas, and again running a blank when the HAZMATCAD Plus units were once more sampling clean air. Blank reference samples were taken before and after, but not during, SA and CG testing, because the methods used were not conducive to running the large number of blank reference samples analyzed during the other TIC tests. In testing with GB and HD, blank gas sample bags were run at the start of each test day.

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

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 12.5 to 50 ppm AC were prepared and analyzed over a three-day period. The regression of peak area versus AC standard concentration had the form Peak Area = 0.7192 x (AC, ppm), with an r^2 value of 0.9961. 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 12.5 to 50 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 = 1.4292 x (CK, ppm), with an r^2 value of 0.9931.

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 3 ppm. Approximately 23 L of the CG/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 r^2 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 CG concentration of 2 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, a Draeger Pac III Single Gas Monitor also was used to provide a real-time indication of CG. This device was new, and its factory calibration was used in all monitoring.

Cl_2 was monitored with a Draeger MiniWarn Multi-Gas Monitor, which was calibrated by preparing standard known concentrations in sample bags. The Cl_2 calibration was done 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 calibration were within 5% of the expected standard concentrations.

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, 0.03, 0.3, 5.0, and 50 ppm and one method blank yielded a linear calibration plot having an r^2 value of 1.000.

Calibration for GB and HD was conducted by diluting stock agent to µg/mL concentrations, and then injecting a 1-µL volume of each standard into the GC-FPD. Regression of peak area versus amount of agent was prepared.

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 HAZMATCAD Plus units were operated and maintained according to the vendor's instructions throughout the verification test. Maintenance was performed according to a preset schedule or in response to predefined HAZMATCAD Plus instrument diagnostics. Daily operational check procedures for the HAZMATCAD Plus units were performed with a vendor-supplied simulant tube. Proper response of the HAZMATCAD Plus units to the simulant was required before testing could proceed. Both units were operated in the "Fast" mode at all times.

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 a standard that was independent of the standards used during the testing. The acceptable tolerance for each target TIC was ±20%. Table 4-1 shows that the results of the PE audit were all within the target tolerances. For the CW agents, standards of GB and HD were prepared in the same manner as for the normal calibration standards, but by a different analyst. The reference data obtained for these standards were compared. For GB, four standards were prepared at concentrations of 5, 2.5, 1.0, and 0.5 µg/mL. For the average responses, all results were within 7% for the standards made by the two different individuals. For HD, five standards were prepared at concentrations of 10, 5, 2.5, 1.0, and 0.5 µg/mL. For the average responses, all results were within 4% for the standards made by the two different individuals.

Table 4-1. Performance Evaluation Audit Results[(a)]

TIC	Sample	Date of Audit	Concentration	Result	Agreement (%)
AC	Standard (Cylinder B0005506)		10,020 ppm	43.2 ppm	
	PE Audit Std (Cyl. LL320)	7/12/04	10,000 ppm	47.8 ppm	9.8
CG	Standard (Cylinder RR37345)		991 ppm	2.18 ppm	
	PE Audit Std (Cyl. AS51117)	7/13/04	1,043 ppm	2.63 ppm	12.7
SA	Standard (Cylinder KE50368)		997 ppm	267332 PA[(b)]	
	PE Audit Std (Cyl. AS73486)	8/5/04	1,090 ppm	277556 PA	5.3
Cl_2	Standard (Cylinder RA64239)		5,811 ppm	9.85 ppm	
	PE Audit Std (Cyl. LL23078)	7/12/04	6,015 ppm	9.60 ppm	6.2

[(a)] As noted in Section 3.2.7.5, PE audits were not performed for CK (no independent standard available).

[(b)] PA = Peak area; based on comparison of neat (undiluted) standards.

4.2.2 Technical Systems Audit

The Battelle Quality Manager conducted a technical systems audit (TSA) to ensure that the verification test was performed in accordance with the test/QA plan(1) and the ETV QMP.(2) 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,(1) 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 records concerning the TSA are permanently stored with the Battelle Quality Manager. The single deviation from the test/QA plan was the inability to complete the PE audit (Table 4-1) within the period of TIC testing as a result of delays in receiving some PE audit standards. However, as shown in Table 4-1, all PE audit results were within the acceptance criteria, so no impact on the test resulted from this deviation.

A separate TSA was also conducted by EPA QA staff on June 7, 2004. That TSA produced two findings: (1) the need to ensure traceability of electronic records from the test, and (2) the need to complete the PE audit of the TIC challenge gases, as was noted above.

The first finding was addressed by improving the documentation of electronic files placed in the electronic archive for this test. The second finding was motivated by delays in obtaining some of the PE audit gases. That finding was addressed by completing the PE audit immediately once the final audit gases were obtained. The actions taken to address these findings were documented to the EPA Quality Manager, and records related to this TSA are stored with the Battelle Quality Manager.

4.2.3 Data Quality Audit

At least 10% of the data acquired during the verification test was audited. 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.

4.3 Quality Assurance/Quality Control 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 the HAZMATCAD Plus 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 the HAZMATCAD Plus 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 HAZMATCAD Plus units' responses, alternating with the HAZMATCAD Plus units' baseline readings, were recorded in each step of the test procedure. That is, for each HAZMATCAD Plus unit and each TIC or CW agent, such readings were recorded with each concentration, at a range of temperature and RH conditions (Table 3-6), with the exception that data at high temperature and high humidity (35°C/>80% RH) were used in evaluating performance, but not incorporated in statistical analyses. Response to each TIC and CW agent was also determined at medium temperature and humidity, both with and without each interferent. In addition, readings were obtained at the normal temperature and RH conditions at different HAZMATCAD Plus unit starting conditions. These data were the basis for the statistical analysis of HAZMATCAD Plus unit 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 following sections detail the statistical methods used for the TIC analysis. Methods used for the CW analysis were similar. However, it must be noted that only one of the HAZMATCAD Plus units responded to GB during testing with that CW agent. Consequently, data for testing with that agent were limited, as compared with data from HD or TIC testing. Because of concentration differences between chemicals (Table 3-1), the effects of concentration and chemical on the behavior of the HAZMATCAD Plus units cannot be separated. For this reason, the statistical analyses were performed on a per chemical basis. In descriptive comments about figures that summarize results in Appendix A, differences that appear across chemicals are highlighted. It should be kept in mind that the differences noted may be the result of a difference in TIC/agent identity, a difference in concentration, or a mixture of both effects.

5.1.1 Analysis of Response, Response Time, and Recovery Time

The effects of temperature and humidity on the actual response were investigated using the Jonckheere-Terpstra test.(6) This non-parametric method tests the hypothesis of no association between response and temperature or humidity versus the hypothesis that the response increases or decreases as temperature or humidity increases. The test accommodates the categorical nature of the dependent (response) and independent (temperature or humidity) variable and is appropriate when both the dependent and the independent variables have a natural ordering (low, medium, and high in this case). Furthermore, the Jonckheere-Terpstra test is appropriate when the sample size is small or the data are sparse.

Unlike temperature and humidity, start state has no natural ordering. The Kruskall-Wallis test(6) was used to determine whether start state has an effect on machine response. This test is equivalent to an analysis of variance (ANOVA) performed on the ranked data. Unlike the Jonckheere-Terpstra test, the Kruskall-Wallis test simply tests for differences in response among the start state alternatives (i.e., the alternative hypothesis is not ordered). Like the Jonckheere-Terpstra test, it accommodates small sample sizes and sparse data.

For the analysis of response time, a standard ANOVA was used. This allowed testing for the effect of temperature, humidity, and start state on the response time. To investigate the effect of temperature, for example, the following model was fit:

$$Y_{ij} = \mu + \alpha_j + \epsilon_{ij} \qquad (1)$$

Here Y_{ij} denotes the log of the i^{th} response time for a given TIC under temperature j. The term μ represents the mean overall treatment levels, the term α_j denotes the effect of temperature j, and the term ϵ_{ij} accounts for all variation not explained by the model.

The log response time was modeled because time-to-event measurements are typically skewed to the right. The log transformation is a standard technique used to achieve normality of error(7) effects when the data are skewed in such a manner. This model provided the average log response time under a given set of conditions. This average was transformed back into the original scale (as opposed to log scale) by exponentiating it. Thus, the modeled geometric mean of the response times was reported under the given set of conditions. The significance of effects of interest was tested by evaluating the corresponding coefficients in the model. Thus, to test whether temperature had an effect on log response time, a standard F test was used to test whether α_j is equal to zero for all j. For more information on the ANOVA approach, see Kirk.(7)

The analysis of recovery time was similar to that of response time unless there were recovery times that were "censored." When the HAZMATCAD Plus did not recover within the maximum allotted time of 600 seconds, that particular recovery time was considered censored. In a censored model, instead of assuming that the log recovery times, Y_{ij}, have a joint normal density function, the likelihood for the vector of recovery times, Y, is assumed to be:

$$f(Y) = \prod_{C^*} g\left(Y_{ij}\right) \prod_{C} S\left(Y_{ij}\right) \qquad (2)$$

where C is the collection of censored observations and C^* is the collection of uncensored observations. Here g is a normal density function and S is the "survival" function:

$$S\left(Y_{ij}\right) = 1 - \Phi\left(\left(Y_{ij} - \left(\mu + \alpha_j\right)\right) / \sigma\right)$$

where Φ is the standard normal distribution function and σ is the standard deviation for the recovery times. The parameter μ represents the common mean; the parameter α_j represents the effect of treatment j. Once again, effects were investigated by testing the parameters of the model. Because the model addressed the log recovery times, the geometric mean of the recovery times was reported.(8)

5.1.2 Analysis of Accuracy

The HAZMATCAD Plus response was defined as "accurate" under a given set of conditions if the HAZMATCAD Plus:

1. Alarmed in the presence of a TIC or CW agent challenge
2. Correctly identified the TIC or CW agent.

The HAZMATCAD Plus accuracy was modeled under a given set of conditions via a binomial logit model.(9) The significance of an effect can be determined by investigating the corresponding coefficient(s) in the model. For example, to investigate accuracy under different temperatures, the following model applies:

$$\log(p_i / (1-p_i)) = \mu + \alpha_i \qquad (3)$$

where p_i is the proportion of accurate responses under temperature i. Here α_i denotes the effect of temperature i and μ is the common mean. By testing the significance of the α_i's using a likelihood ratio test, the effect of each factor was tested.

As noted in Section 3.2.1, the HAZMATCAD Plus displayed specific identifiers when a chemical was detected. For AC, the correct identifiers were “BLOD” or “CHOK.” For CG, the correct identifier was “CHOK,” while the correct identifiers for Cl_2 and SA were “HALO” and “HYDR,” respectively. The correct identifers for GB and HD were “G” and “H,” respectively. Any response with an incorrect identifier was counted as inaccurate.

5.1.3 Analysis of Repeatability

For testing the repeatability of response and recovery times for the HAZMATCAD Plus, a test of equal variances was used. Where there is a difference between the variability in response or recovery times for the different levels of temperature or humidity, there is evidence that temperature or humidity has an effect on the repeatability of the response or recovery time. The specific test used to test for equal variances was the Brown-Forsythe test.(7) This test is essentially an ANOVA run on the absolute deviation from the treatment (level of temperature or humidity) medians.

For testing repeatability of the HAZMATCAD Plus response, an approach was used that took into account the categorical nature of the response data. For all responses observed under a given set of conditions, the mode (the most common response) was computed. The number of observed responses that equaled that mode was then determined. Thus, the proportion of responses equaling the most common response was the measure for the HAZMATCAD Plus response repeatability. This proportion was modeled using a binomial logit model.

5.1.4 False Negatives and Positives Analysis

To test whether interferents caused false negatives in the HAZMATCAD Plus response, Dunn’s non-parametric multiple comparisons procedure was used.(10) To employ this procedure, the responses for all interferent tests are ranked (ties receive average ranks). The test statistic, which is asymptotically normal, is then:

$$\frac{R_i - R_C}{\sqrt{\left(\frac{N(N+1)}{12}\right)\left(\frac{1}{n_i} + \frac{1}{n_C}\right)}} \qquad (4)$$

where R_i is the average rank for interferent i, R_C is the average rank for no interferent, n_i is the number of tests for interferent i, n_c is the number of tests for no interferent, and $N = n_i + n_c$. The smaller this test statistic is, the greater the evidence that the given interferent is creating a false negative response.

To investigate the proportion of false positives, a Clopper-Pearson approach(11) was used. To estimate the rate of false positives, the sample proportion was used (i.e., the number of false positives divided by the number of trials). Along with this point estimate, a measure of its uncertainty was calculated in the form of a 95% confidence interval. Simply because the process did not register a false positive for a particular interferent does not guarantee that it would never register a false positive for that interferent. This methodology makes an effort to quantify such a possibility by determining bounds for the false positive rate estimate based on its value and the number of trials. By assuming that the response obtained was representative of HAZMATCAD Plus performance, the individual tests may be modeled as a binomial distribution, and standard methods of confidence interval estimation may be employed. The Clopper-Pearson “exact” interval is commonly used in such instances. Its endpoints are directly calculated from the binomial distribution without approximation.

5.1.5 Analysis of Response to Alternating Concentrations

This analysis addressed the HAZMATCAD Plus response to varying concentrations of the target TIC or CW agent. As described in Section 3.3.1, the test procedure involved sequentially sampling clean air or a high (H) or low (L) target concentration in varying order (i.e., clean air/H/L/clean air/L/H/clean air/H/L. . .). The data analysis involved two separate analyses. In the first analysis, the association between the order in which the challenge levels was presented and the HAZMATCAD Plus response was investigated after adjusting for the level of challenge. This was accomplished using a Cochran-Mantel-Hansel statistic.(9) Empty cells for this contingency table analysis were filled with counts of 0.01 to allow for convergence of the test statistic. In the second analysis, the difference between the response for the two challenge levels was investigated. More specifically, when challenged by a high concentration after being challenged by a lower concentration, the machine response should increase. Similarly, when challenged by a low concentration after being challenged by a higher concentration, the machine response should decrease. The proportion of tests exhibiting this behavior for each TIC was recorded. Clopper-Pearson bounds were placed on the probability that the machine response would increase or decrease as appropriate. Results of this analysis are presented in Section 6.3.

5.2 Other Analyses

The data used to evaluate the response threshold were the replicate HAZMATCAD Plus 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 HAZMATCAD Plus response in all replicate runs. In this evaluation, any positive HAZMATCAD Plus response was taken as detection of the target TIC or CW agent, i.e., HAZMATCAD Plus response of L (low) was sufficient in terms of the response threshold evaluation.

Battery life was assessed. Battery life is reported as the total time from start-up to battery exhaustion when a HAZMATCAD Plus 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 when the HAZMATCAD Plus no longer responded to a challenge mixture of AC in air.

Chapter 6
Test Results

As discussed in Chapter 5, statistical approaches were used to test for the effects of different conditions on the HAZMATCAD Plus performance. The following sections summarize the statistical results from this verification. A more extensive presentation of the modeled statistical results is included as Appendix A of this report. Note that the statistical evaluations reported in this chapter were not applied to CK since the HAZMATCAD Plus did not respond to that TIC. Two HAZMATCAD Plus units were used in all testing except for GB (as a result of the failure of Unit 22 to alarm for GB during testing).

6.1 Response Time

Results of the response time analysis are summarized here and detailed in Appendix A, Section A.1. Table 6-1 summarizes data used for the analysis of response time and other performance parameters. This table is for illustration purposes, and the TIC results shown are drawn from data obtained at the target concentrations (see Table 3-1).

The HAZMATCAD Plus produced an alarm and a visual display of response typically within a few seconds after the initiation of sampling of one of the TICs. Over the range of temperature settings, modeled geometric mean response times varied from a low of about 8 seconds for AC to a high of about 12 seconds for CG. Temperature had no effect on HAZMATCAD Plus response time. Over the humidity range settings, modeled geometric mean response times varied from a low of about 6 seconds for AC to a high of about 14 seconds for CG. A statistically significant difference was found among the response times for the different levels of humidity for CG; however, the difference (12 to 14 seconds) does not seem practically significant. For GB and HD, over the range of temperature and humidity settings, temperature and humidity had no effect on the HAZMATCAD Plus response time. The data collected for HD at the low temperature setting were not used in the statistical analysis because the target concentration for that test was 1 mg/m^3, while the target concentration for all other HD tests was 4 mg/m^3. For GB, HAZMATCAD Plus Unit 22 did not alarm in any tests, while Unit 27 provided no response for one of the five challenges at room temperature and high humidity and for four of the five challenges at high temperature and high humidity. (The Microsensor Systems vendor attributed this instability to a possible decrease in the collection efficiency of the concentrator used in the SAW part of the HAZMATCAD Plus caused by exposure to the TICs during testing.)

Table 6-1. Illustrative Data

TIC/CW Agent(a)	Environmental Conditions	HAZMATCAD Plus Response Level	Response Time Range (s)	Recovery Time Range (s)	Alarms (Indicated Chemical)
AC	Control (22˚C – 50% RH)	H	3-13	78-227	10/10 (BLOD)
	22˚C - <20% RH	H	5-10	64-152	10/10 (BLOD)
	22˚C – 80% RH	H	3-10	64-99	10/10 (BLOD)
	35˚C – 50% RH	H	5-14	73-124	10/10 (BLOD)
	35˚C – 80% RH	H	4-10	58-105	10/10 (BLOD)
	5˚C – 50% RH	H	4-10	142-537	10/10 (BLOD)
CG	Control (22˚C – 50% RH)	H	9-16	36-46	10/10 (CHOK)
	22˚C - <20% RH	H	10-16	33-40	10/10 (CHOK)
	22˚C – 80% RH	H	12-18	27-40	10/10 (CHOK)
	35˚C – 50% RH	H	8-13	39-48	10/10 (CHOK)
	35˚C – 80% RH	H	11-20	25-39	10/10 (CHOK)
	5˚C – 50% RH	H	9-18	46-70	10/10 (CHOK)
SA	Control (22˚C – 50% RH)	H	6-12	18-29	10/10 (HYDR)
	22˚C - <20% RH	H	6-12	16-29	10/10 (HYDR)
	22˚C – 80% RH	H	5-11	19-28	10/10 (HYDR)
	35˚C – 50% RH	H	5-11	20-28	10/10 (HYDR)
	35˚C – 80% RH	H	5-11	16-27	10/10 (HYDR)
	5˚C – 50% RH	H	6-11	19-30	10/10 (HYDR)
Cl_2	Control (22˚C – 50% RH)	M	4-12	54-75	10/10 (HALO)
	22˚C - <20% RH	M	7-13	53-65	10/10 (HALO)
	22˚C – 80% RH	L (8)-M (2)	6-12	52-66	10/10 (HALO)
	35˚C – 50% RH	M (9)	6-14	36-69	9/10 (HALO) 1/10 (XECM FAIL)
	35˚C – 80% RH	L	8-69	31-55	10/10 (HALO)
	5˚C – 50% RH	M	5-11	58-89	10/10 (HALO)
GB	Control (22˚C – 50% RH)	M	22-39	28-41	5/5 (G)
	22˚C – <20% RH	M	34-40	18-26	5/5 (G)
	22˚C – 80% RH	M (4)	21-42	0(b)	4/5 (G) 1/5 NR(c)
	35˚C – 50% RH	M (4)-H (1)	21-33	26-44	5/5 (G)
	35˚C – 80% RH	H (1)	21	22	1/5 (G) 4/5 NR
	5˚C – 50% RH	H	23-36	66-92	5/5(G)
HD	Control (22˚C – 50% RH)	H	40-94	267-517	10/10 (H)
	22˚C - <20% RH	H	21-69	338-527	10/10 (H)
	22˚C – 80% RH	H	15-80	215-394	10/10 (H)
	35˚C – 50% RH	H	31-125	58-265	10/10 (H)
	35˚C – 80% RH	H	22-177	35-486	10/10 (H)
	5˚C – 50% RH(d)	M	82-114	349-600(e)	10/10 (H)

(a) TIC/CW agent results shown are from data at target concentration level (see Table 3-1). All results are from two units of the HAZMATCAD Plus, except for GB (Unit 22 did not respond to GB during testing).

(b) Cleared during challenge resulting in zero recovery time.

(c) NR = No response.

(d) Challenge concentration for HD cold temperature test was 1 mg/m^3.

(e) HAZMATCAD Plus unit did not return to a cleared response within 600 seconds.

6.2 Recovery Time

Results of the recovery time analysis are summarized here and detailed in Appendix A, Section A.2. Recovery time results also are illustrated in Table 6-1.

In general, recovery times differed considerably from one target chemical to another. For AC, modeled geometric mean recovery times ranged from 76 to 361 seconds; for CG, from 36 to 57 seconds; for Cl_2, from 49 to 73 seconds; and for SA, from 23 to 25 seconds (see Figures A-2 and A-3). Modeled geometric mean recovery times for GB ranged from 20 to 80 seconds and for HD ranged from 143 to 434 seconds.

Temperature has a significant effect on recovery time for every TIC except SA. While differences in modeled geometric mean recovery time among the levels of temperature are evident for AC, CG, and Cl_2, the greatest differences are apparent for AC. In this case, recovery time increases as temperature decreases. The modeled geometric mean recovery time for AC under low temperature at medium humidity was 361 seconds, which is nearly four times as long as that for AC at higher temperatures.

For GB and HD, temperature has a statistically significant effect on the HAZMATCAD Plus recovery time. The longest modeled geometric mean recovery time for GB (80 seconds) was at the low temperature setting. For HD, only the medium and high temperature settings were analyzed. The medium temperature setting resulted in a much longer modeled geometric mean recovery time (357 seconds) than the high temperature setting (143 seconds). In general, for the agents, recovery time appears to be greater for lower temperatures.

Humidity also had statistically significant effects on the recovery time for AC, CG, and Cl_2, but these small effects were of minimal importance in a practical sense. The recovery time for AC was longest for medium humidity, with a modeled geometric mean recovery time of 131 seconds. With that exception, all TICs (including AC) had modeled geometric mean recovery times below about 90 seconds for all humidity conditions.

For both GB and HD, humidity had a statistically significant effect on the HAZMATCAD Plus recovery time. The modeled geometric mean of recovery time for GB was minimally longer at 50% RH than at 20% RH (31 vs. 20 seconds). No recovery time information for GB at high humidity is available because of unstable responses or no response to the presence of GB. For HD, the opposite trend was apparent for the modeled geometric mean of recovery time, with longer recovery times (mean 434 seconds) at 20% RH compared to 281 seconds at 80% RH.

6.3 Accuracy

Results of the accuracy analysis are summarized here and described in Appendix A, Section A.3. Results of tests that involved alternating different challenge concentrations, as opposed to alternating clean air and a single challenge concentration, are summarized below and detailed in Appendix A, Section A.8. Accuracy results also are illustrated in Table 6.1. The HAZMATCAD

Plus was nearly 100% accurate in identifying the target TICs under all levels of temperature and humidity, the only erroneous reading among nearly 250 data points (Table 6-1) being one failure indication during testing with Cl_2.

For HD, the HAZMATCAD Plus identified the agents with 100% accuracy under all levels of temperature. For HD, the HAZMATCAD Plus also performed with 100% accuracy under all humidity conditions. For GB, Unit 22 was inaccurate in all challenges (i.e., did not alarm for GB), whereas Unit 27 of the HAZMATCAD Plus performed with 100% accuracy under low and medium humidity, but at high humidity was inaccurate as a result of no response or the lack of a stable response. It should be noted that at room temperature/high humidity the HAZMATCAD Plus Unit 27 did alarm as "G" in four out of five challenges. However, the alarm cleared before the challenge was completed, resulting in an unstable response. (As noted in Section 6.1, the Microsensor Systems vendor attributes this high humidity behavior to a possible effect of the TICs on the concentrator used in the SAW part of the HAZMATCAD Plus.)

The HAZMATCAD Plus identified the target TIC or CW agent as effectively when sampling alternating concentrations (Section 5.1.5; Appendix A, Section A.8) as when sampling a single concentration. The HAZMATCAD Plus responded as expected to the sequential sampling of clean air, low, and high challenge concentrations.

6.4 Repeatability

Results of the repeatability analysis are summarized below and detailed in Appendix A, Section A.4.

Repeatability addressed the consistency of the Low, Medium, and High readings of the HAZMATCAD Plus units. Repeatability of response was always perfect under all levels of temperature and humidity for AC, CG, and SA. Cl_2 exhibited more variability in response. Typically the HAZMATCAD Plus units registered a Medium response with Cl_2. For one of the 10 runs at high temperature and medium humidity, HAZMATCAD Plus Unit 27 responded with a Low reading before showing an instrument failure alarm, and for eight of the 10 runs at high humidity and room temperature it responded with a Low reading. However, neither of the deviations was statistically significant. Given the mix of concentrations and TICs used in the repeatability analysis, there appears to be no evidence for a temperature or humidity effect on HAZMATCAD Plus response repeatability.

Repeatability of response was perfectly consistent for both GB and HD at each temperature level. Repeatability of response was also perfectly consistent for HD over all humidity levels. For GB, the repeatability of response from Unit 27 was perfectly consistent at the low and medium humidity levels. However, for GB at the high humidity level, there was either no response or an unstable response from Unit 27, as discussed in Sections 6.1 and 6.3. Unit 22 did not alarm for GB during testing.

The modeled repeatability of response times for both TICs and CW agents showed that there were no significant differences among the variabilities in response time for the different levels of temperature or humidity.

The modeled repeatability of recovery times for both TICs and CW agents showed that there were no significant differences among the variabilities in recovery time for the different levels of temperature for AC, CG, SA, GB, and HD. The effect of temperature on the repeatability of recovery time was significant for Cl_2. Recovery time for Cl_2 was most repeatable under medium temperature. The modeled repeatability of recovery times showed that there were no significant differences among the variabilities in recovery time for the different levels of humidity for CG, SA, Cl_2, GB, and HD. The effect of humidity on the repeatability of recovery time was significant for AC. The greatest variability in recovery time occurred under medium humidity for AC.

6.5 Response Threshold

Response threshold was determined by challenging each HAZMATCAD Plus unit with successively lower concentrations of each TIC and CW agent until it no longer responded. Table 6-2 provides the results for each TIC and CW agent for the response threshold test.

The concentrations used in each of these tests are given in the table and, for the TICs, are well below the IDLH concentrations used in the other tests. The responses listed in the table give the results for three successive challenge/clean air cycles. For the CW agents, the concentrations used are below the target concentrations used in the other tests; however, the concentrations are above the IDLH for GB and above the AEGL-2 level for HD.

For AC, the response threshold was between 0.6 and 1.25 ppm on both HAZMATCAD Plus units. At lower concentrations, the HAZMATCAD Plus units alarmed as CHOK for AC, whereas, at higher concentrations, the units alarmed as BLOD. According to the HAZMATCAD Plus manual, both alarms are correct when AC is present. For CG, the response threshold was between 0.3 and 0.6 ppm. For SA, the response threshold was between 0.2 and 0.4 ppm, and for Cl_2, the response threshold was between 0.5 and 1 ppm. According to the HAZMATCAD Plus manual, Cl_2 should alarm as HALO. At low Cl_2 concentrations, the alarm response changed to CHOK. This was not a correct alarm for the presence of Cl_2; however, the HAZMATCAD Plus units still identified the presence of a threat, so the CHOK response was used to determine the response threshold for Cl_2. In all cases, the two HAZMATCAD Plus units showed the same TIC thresholds.

For GB, the response threshold was between 0.6 and 1.1 mg/m^3 (0.11 and 0.2 ppm) for Unit 27. A response threshold for GB was not determined for Unit 22 because that unit failed to respond to GB. For HD, the response threshold was between 0.6 and 1.6 mg/m^3 (0.09 and 0.24 ppm) for Unit 22 and between 1.6 and 4 mg/m^3 (0.24 and 0.6 ppm) for Unit 27. Unit 22 responded to only two of the five challenges with HD at 1.6 mg/m^3, suggesting its response threshold is close to that level.

Table 6-2. Response Threshold Data

	HAZMATCAD Plus Response Level (ID)	
TIC/CW Agent (Concentration)	**Unit 22**	**Unit 27**
AC (5 ppm)	L (BLOD)	L (BLOD)
AC (2.5 ppm)	H (CHOK)	H (CHOK)
AC (1.25 ppm)	M (CHOK)	L (CHOK)
AC (0.6 ppm)	No Response	No Response
CG (0.6 ppm)	L (CHOK)	L (CHOK)
CG (0.3 ppm)	No Response	No Response
SA (0.4 ppm)	L (HYDR)	L (HYDR)
SA (0.2 ppm)	No Response	No Response
Cl_2 (1 ppm)	L-M (CHOK)	L-M (CHOK)
Cl_2 (0.5 ppm)	No Response	No Response
GB (1.1 mg/m^3) (0.2 ppm)	NA	L-M (G)
GB (0.6 mg/m^3) (0.11 ppm)	NA	No Response
HD (4 mg/m^3) (0.6 ppm)	H (H)	H (H)
HD (1.6 mg/m^3) (0.24 ppm)	L (H)	No Response
HD (0.6 mg/m^3) (0.09 ppm)	No Response	No Response

NA = Not applicable; no response to GB from this unit during testing.

6.6 Temperature and Humidity Effects

The results of investigating temperature and RH effects on the HAZMATCAD Plus response are summarized here and are detailed in Appendix A, Section A.5.

Table 6-1 also illustrates temperature and RH effect data. The effects of temperature and RH on the HAZMATCAD Plus response were small. The HAZMATCAD Plus response to each TIC was tested under medium humidity at low, medium, and high temperature. A High response was recorded for all tests under all temperatures for every TIC except Cl_2. Typically the HAZMATCAD Plus units registered a Medium response for Cl_2. For one of the ten Cl_2 runs at high temperature and medium humidity, a Low response was observed before HAZMATCAD Plus Unit 27 showed an instrument failure alarm. However, there is no statistically significant evidence that temperature had a consistent effect on the HAZMATCAD Plus units' response for the TICs.

For GB at low temperature, the response from Unit 27 was consistently High, while at medium and high temperature, the response was consistently at the Medium level. These results provide sufficient statistical evidence to conclude that temperature has an effect on the HAZMATCAD Plus response to GB. For HD, the response was consistently High for each temperature level. Thus, for HD, there was no evidence of an effect of temperature on the HAZMATCAD Plus response.

The HAZMATCAD Plus units' response to each TIC also was tested under medium temperature at low, medium, and high humidity. As in the temperature tests, a High response was recorded for all tests under all temperatures for every TIC except Cl_2. Typically the HAZMATCAD Plus units registered a Medium response for Cl_2, but for eight of the ten runs at high humidity and room temperature, the two HAZMATCAD Plus units registered a Low response. Statistically, humidity had a significant effect on the HAZMATCAD Plus units' response for Cl_2, i.e., the typical HAZMATCAD Plus unit response was lower for the highest level of humidity.

For GB at high humidity, there was either no response from Unit 27 or the response was not stable (Table 6-1). At low and medium humidity, the Unit 27 response was consistently at the Medium level. These results provide sufficient statistical evidence to conclude that, under the conditions of this test, humidity has an effect on the HAZMATCAD Plus response to GB. For HD, the response was consistently High for all runs within each humidity level. Thus, for HD there was no evidence of an effect of humidity on the HAZMATCAD Plus units' response.

6.7 Interference Effects

The results of investigating interference effects on HAZMATCAD Plus response are summarized here and are detailed in Appendix A, Section A.6. Table 6-3 summarizes data used for the analysis of interference effects. This table is for illustration purposes.

A false positive response would occur if the HAZMATCAD Plus responded and provided an alarm in the presence of an interferent and in the absence of a TIC or CW agent. A false positive was defined as any alarm under those conditions. For the five interferents tested, no false positive responses occurred. The HAZMATCAD units provided no response to the presence of any of the interferents when no TIC or CW agent challenge was present.

False negative responses would occur if the presence of an interferent masked the presence of a TIC or CW agent and the HAZMATCAD Plus provided a lower response or did not respond to the TIC or CW agent. The HAZMATCAD Plus responded to all TIC challenges when interferents were present; however, the HAZMATCAD Plus did not respond to all CW agent challenges when interferents were present. Changes in response, response time, and recovery time due to interferences are discussed in the following paragraphs.

The interferents did not show an effect on the response for AC, CG, and SA. For Cl_2, the interferents had a very small effect on the HAZMATCAD Plus response, in that air freshener slightly reduced the level of the response to Cl_2, while floor cleaner slightly increased the response level. However, neither of the effects for these two interferents is statistically significantly different from the control (i.e., the response to Cl_2 in the absence of interferents).

The HAZMATCAD Plus Unit 27 did not respond to GB in the presence of air freshener and paint and in two of the exhaust hydrocarbon interferent tests the response was not stable (Unit 22 did not alarm in any test with GB). The HAZMATCAD Plus also did not respond to HD in the presence of ammonia cleaner and paint. However, after the HD/interferent challenge ended (after they were switched back to sample from the clean air plenum), both HAZMATCAD Plus units alarmed for HD.

Table 6-3. Interference Effects Data

TIC/CW Agent[a]	Interferent	Response Range	Response Time Range (s)	Recovery Time Range (s)	Alarms (Indicated Chemical)
AC	Control	H	3-13	78-227	10/10 (BLOD)
	Paint Fumes	H	4-9	96-452	10/10 (BLOD)
	Floor Cleaner	H	4-10	96-424	10/10 (BLOD)
	Air Freshener	H	6-11	84-423	10/10 (BLOD)
	Gasoline Engine Exhaust	H	5-11	91-365	10/10 (BLOD)
	DEAE	H	4-10	108-477	10/10 (BLOD)
CG	Control	H	9-16	36-46	10/10 (CHOK)
	Paint Fumes	H	9-17	43-52	10/10 (CHOK)
	Floor Cleaner	H	11-18	39-53	10/10 (CHOK)
	Air Freshener	H	10-16	42-54	10/10 (CHOK)
	Gasoline Engine Exhaust	H	8-17	39-54	10/10 (CHOK)
	DEAE	H	9-16	34-47	10/10 (CHOK)
SA	Control	H	6-12	18-29	10/10 (HYDR)
	Paint Fumes	H	5-9	17-30	10/10 (HYDR)
	Floor Cleaner	H	5-11	22-31	10/10 (HYDR)
	Air Freshener	H	6-11	23-35	10/10 (HYDR)
	Gasoline Engine Exhaust	H	7-11	18-29	10/10 (HYDR)
	DEAE	H	5-11	18-33	10/10 (HYDR)
Cl_2	Control	M	4-12	54-75	10/10 (HALO)
	Paint Fumes	M	6-11	60-83	10/10 (HALO)
	Floor Cleaner	M(2)-H(8)	8-16	44-136	10/10 (HALO)
	Air Freshener	L(1)-M(9)	5-10	62-86	10/10 (HALO)
	Gasoline Engine Exhaust	M	5-12	55-70	10/10 (HALO)
	DEAE	H(1)-M(4)	6-12	55-75	10/10 (HALO)
GB	Control	M	22-39	28-41	5/5 (G)
	Paint Fumes	NR[b]	NA[c]	NA	–
	Floor Cleaner	H	29-41	30-43	5/5 (G)
	Air Freshener	NR	NA	NA	–
	Gasoline Engine Exhaust	M	32-35	0[d]-22	5/5 (G)
	DEAE	H(1)-M(4)	28-38	21-36	5/5 (G)
HD	Control	H	40-94	267-517	10/10 (H)
	Paint Fumes	H(1)-NR(11)	22	0[d]	1/12 (H)
	Floor Cleaner	NR	NA	NA	–
	Air Freshener	H	57-112	137-600[e]	10/10 (H)
	Gasoline Engine Exhaust	H	22-129	274-459	10/10 (H)
	DEAE	H	23-65	238-447	10/10 (H)

[a] Data shown are for illustration, TIC/CWA results shown are from data at target concentration level only. All results are from two units of the HAZMATCAD Plus, except for GB (Unit 22 did not respond to GB during testing).
[b] NR = No response.
[c] NA = Not applicable.
[d] Cleared during challenge resulting in zero recovery time.
[e] HAZMATCAD Plus did not return to a cleared response within 600 seconds.

The interferents also showed no effect on the response time for AC, CG, and SA. The presence of floor cleaner vapors, however, had a significant effect on the response time for Cl_2. When floor cleaner vapors were present, the modeled geometric mean response time for Cl_2 increased from about 7 seconds to about 12 seconds.

The interferents showed no effect on the response time for GB. However, there was a statistically significant effect on response time for HD in the presence of interferents. Both DEAE and exhaust hydrocarbons decreased the response time for HD (to 40 and 48 seconds) compared to the control (61 seconds), and air freshener vapors increased the response time for HD (to 74 seconds).

The interferents showed an effect on the recovery time primarily for AC. The modeled geometric mean recovery time for the AC control was about 131 seconds. In the presence of interferent, the modeled geometric mean recovery time for AC ranged from about 299 seconds when DEAE was present to about 207 seconds when exhaust hydrocarbons were present. Upon review of the interferent recovery times, a trend was apparent for AC, in that recovery times tended to increase with each successive challenge within an individual test for all five of the interferents tested. Statistically significant effects of interferents on recovery time were also found for CG and Cl_2, but these effects were too small to be of any practical significance in actual use of the HAZMATCAD Plus.

In addition to the false negatives observed with GB in the presence of paint and air freshener vapors, the data showed a statistically significant effect on recovery time in the presence of one interferent. Recovery time in the presence of exhaust hydrocarbons was shorter than that for GB without an interferent. For HD, there was no evidence of an interferent effect on the HAZMATCAD Plus units' recovery time.

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 described in Appendix A, Sections A.1.3, A.2.3, and A.5.3. Table 6-4 illustrates the data obtained in testing for cold-/hot-start effects, showing the HAZMATCAD Plus 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. Results from the two HAZMATCAD Plus units were consistent, as shown in Table 6-4, except for recovery times exceeding 10 minutes observed with Unit 22 in the room temperature cold start.

The effect of start state of the HAZMATCAD Plus was tested with AC at the IDLH level and was found to have no effect on response time, but a strong effect on recovery times (data shown in Table 6-4). Under fully warmed-up conditions, the modeled geometric mean recovery time for AC was about 131 seconds. The longest mean recovery time resulted from a cold-start condition after maintaining the HAZMATCAD Plus at room temperature overnight with a mean recovery time of 315 seconds. Under cold-start conditions, after an overnight cold soak (5ºC), the modeled

Table 6-4. Cold/Hot-Start Effects Data[a]

HAZMATCAD Plus Unit	Start Condition	Experiment Number	Delay[b] Time (mm:ss)	Response	Response Time (seconds)	Recovery Time (seconds)	Alarm (Indicated Chemical)
22	**Control (Fully Warmed Up)**	1		H	3	171	BLOD
		2		H	4	227	BLOD
		3		H	7	220	BLOD
		4		H	6	143	BLOD
		5		H	8	178	BLOD
	Room Temperature (Cold Start)	1	0:18	H	12	156	BLOD
		2		H	10	219	BLOD
		3		H	6	600[c]	BLOD
		4		H	8	600[c]	BLOD
		5		H	7	600[c]	BLOD
	Cold Temperature (Cold Start)	1	0:40	H	6	166	BLOD
		2		H	8	209	BLOD
		3		H	9	283	BLOD
		4		H	5	223	BLOD
		5		H	7	277	BLOD
	Hot Temperature (Cold Start)	1	0:20	H	9	95	BLOD
		2		H	10	174	BLOD
		3		H	10	318	BLOD
		4		H	9	311	BLOD
		5		H	9	379	BLOD
27	**Control (Fully Warmed Up)**	1		H	13	78	BLOD
		2		H	8	97	BLOD
		3		H	5	109	BLOD
		4		H	7	82	BLOD
		5		H	8	101	BLOD
	Room Temperature (Cold Start)	1	0:16	H	12	90	BLOD
		2		H	4	198	BLOD
		3		H	6	373	BLOD
		4		H	8	287	BLOD
		5		H	7	317	BLOD
	Cold Temperature (Cold Start)	1	0:40	H	6	158	BLOD
		2		H	5	164	BLOD
		3		H	8	213	BLOD
		4		H	7	182	BLOD
		5		H	6	215	BLOD
	Hot Temperature (Cold Start)	1	0:20	H	9	73	BLOD
		2		H	5	120	BLOD
		3		H	5	336	BLOD
		4		H	6	405	BLOD
		5		H	4	568	BLOD

(a) All cold-hot-start tests conducted with AC.

(b) Delay time is duration from powering up the instrument to first indication that the instrument is ready to take a reading.

(c) HAZMATCAD Plus unit did not return to a cleared response within 600 seconds. The next challenge was initiated prior to clear-down.

geometric mean recovery time was about 205 seconds. Under cold-start conditions, after an overnight hot soak (40ºC), the recovery time was also long, with a modeled geometric mean recovery time of 230 seconds. Comparing the responses by start state (cold, room temperature, and hot soak) with the responses from the control runs, no effect of start state on response could be detected for AC at IDLH.

6.9 Battery Life

The battery life test was conducted by placing fully charged batteries in the HAZMATCAD Plus units. The HAZMATCAD Plus units were then powered on and allowed to warm up fully according to the manufacturer's directions.

An initial response time test was conducted with AC at the IDLH concentration level. The HAZMATCAD Plus units then sampled clean air for approximately 30 minutes, and the AC mixture was then sampled again. This procedure was repeated with the HAZMATCAD Plus units operating continuously until the batteries were depleted, and the HAZMATCAD Plus units no longer responded to the presence of the AC.

Table 6-5 provides the results for the battery life test. The time of each AC challenge, and the response (and response time), are provided in the table for both HAZMATCAD Plus Unit 22 and Unit 27. Both HAZMATCAD Plus units gave a response of H BLOD and showed response times of a few seconds every time the AC challenge was sampled. HAZMATCAD Plus Unit 22 responded in a normal fashion until the low-battery light began flashing at 15:42. Several minutes later the low-battery light became a solid light and, within seven minutes, the all clear light went out. During the next AC challenge (at 16:00), Unit 22 did not respond to the AC challenge. The unit powered off at 16:12. The battery life for this unit was 9 hours and 49 minutes. HAZMATCAD Plus Unit 27 responded in a normal fashion until the low-battery light began flashing at 16:52. During the next AC challenge, Unit 27 responded as H BLOD within 5 seconds. However, during this challenge, the low-battery light became a solid light. Six minutes later the all clear light went out, and the unit powered off at 17:16. The battery life for this unit was 10 hours and 53 minutes. There was no degradation of response or response time with either HAZMATCAD Plus unit as the batteries approached depletion. Even when the low-battery light was flashing and when it was solid, Unit 27 responded normally for the IDLH level AC challenge. However, when the all clear light was extinguished, Unit 22 failed to respond.

6.10 Operational Characteristics

General performance observations noted during verification testing:

- Instrument Operation—The HAZMATCAD Plus has a button that is pushed to turn the detector on. It also has a separate button to turn the detector off. Other buttons include an ALARM SILENCE button that can be used to turn off the audible alarm and a MODE SELECT button that can be used to switch between fast and sensitive modes.

Table 6-5. Responses Recorded in Battery Life Testing[a]

		HAZMATCAD Plus Unit			
		Unit 22		Unit 27	
Test	**Time**	**Response (Response Time [seconds])**	**Battery Indicator**	**Response (Response Time [seconds])**	**Battery Indicator**
Start-up	0623				
1	0630	H BLOD (10)	Full	H BLOD (2)	Full
2	0700	H BLOD (5)	Full	H BLOD (6)	Full
3	0730	H BLOD (9)	Full	H BLOD (7)	Full
4	0800	H BLOD (7)	Full	H BLOD (9)	Full
5	0830	H BLOD (5)	Full	H BLOD (8)	Full
6	0900	H BLOD (8)	Full	H BLOD (5)	Full
7	0930	H BLOD (10)	Full	H BLOD (5)	Full
8	1000	H BLOD (5)	Full	H BLOD (9)	Full
9	1030	H BLOD (7)	Full	H BLOD (10)	Full
10	1100	H BLOD (5)	Full	H BLOD (10)	Full
11	1130	H BLOD (7)	Full	H BLOD (7)	Full
12	1200	H BLOD (5)	Full	H BLOD (5)	Full
13	1230	H BLOD (9)	Full	H BLOD (5)	Full
14	1300	H BLOD (8)	Full	H BLOD (5)	Full
15	1330	H BLOD (7)	Full	H BLOD (5)	Full
16	1400	H BLOD (6)	Full	H BLOD (9)	Full
17	1430	H BLOD (7)	Full	H BLOD (6)	Full
18	1500	H BLOD (6)	Full	H BLOD (5)	Full
19	1530	H BLOD (8)	Full	H BLOD (7)	Full
	1542		Low battery (flashing)		
	1550		Low battery (solid)		
	1557		All clear light out		
20	1600	No Response		H BLOD (6)	Full
	1612		**Power Off (9 hours, 49 minutes)**		
21	1630			H BLOD (11)	
	1652				Low battery (flashing)
22	1700			H BLOD (5)	Low battery (solid)
	1706				All clear light out
	1716				**Power off (10 hours, 53 minutes)**

[a] All battery life tests were conducted with AC as the challenge TIC at the IDLH concentration of 50 ppm (50 mg/m^3).

- Instrument Indicators—The HAZMATCAD Plus has several lighted indicators to show the status of the detector. These indicators include ALARM, ALL CLEAR, LO BAT, and SYS OK. The HAZMATCAD Plus also has an LED display with large letters that indicate the type of chemical detected and the level of alarm (high, medium, low) detected. The display flashes the type of chemical and the level of alarm alternately until the detector clears.

- Warm-Up—The HAZMATCAD Plus units generally reach a ready state within one minute after turning the detectors on.

- Batteries—The HAZMATCAD Plus unit uses two batteries that are easy to obtain and easy to install. The batteries fit only one way into the detector. Also, low batteries can be changed during operation by changing one battery at a time so that the detector does not need to be shut down to change the batteries.

- Errors—One of the HAZMATCAD Plus units occasionally showed an XECM failure on the LED display. The HAZMATCAD Plus unit would remain in this state until it was rebooted. After rebooting, the HAZMATCAD Plus unit operated correctly. One of the HAZMATCAD Plus units also would not alarm for GB.

- Vendor Support—Before the verification, vendor representatives trained three Battelle employees to operate the HAZMATCAD Plus units. Testing proceeded according to the vendor's recommendations on how to operate the HAZMATCAD Plus units for testing. The vendor also responded promptly when information was needed.

- Cost—The list price of the HAZMATCAD Plus units, as used in this verification test, is approximately $8,349 each.

Chapter 7
Performance Summary

This chapter summarizes the overall performance results found in testing of the HAZMATCAD Plus portable SAW and EC detector with both TICs and CW agents. This summary focuses on aspects of the performance that are most important in field use of the HAZMATCAD Plus by emergency first responders. Consistent with that use, most test procedures were conducted with challenge levels that were at or near IDLH or comparable concentrations. Response thresholds were tested by stepping downward in concentration from these challenge levels. The HAZMATCAD Plus was not programmed to respond to CK, so no results are reported for that TIC. Results summarized here are from two units of the HAZMATCAD Plus in all cases, except GB testing (as a result of the failure of one unit to alarm for GB).

The HAZMATCAD Plus response for AC, CG, and SA at IDLH concentration was a full-scale (high alarm) reading. The response for Cl_2 at IDLH concentration was a mid-scale (medium alarm) reading. The HAZMATCAD Plus response for GB was a mid-scale reading at 2.2 mg/m^3 (11 times the IDLH concentration). The response for HD was a full-scale reading at 4 mg/m^3 (7 times the AEGL-2 level for this agent).

In nearly all cases, the HAZMATCAD Plus provided an audible and visual alarm within 3 to 20 seconds after exposure to AC, CG, SA, or Cl_2 over the range of temperature and humidity tested (5 to 35°C; <20 to >80% RH). Over the different temperatures and humidities, response time ranges for GB and HD were from 21 to 42 seconds and 15 to 177 seconds, respectively. Over the ranges of 5 to 35 ºC and <20 to >80% RH, temperature and RH had no practically significant effect on response time for any TIC or CW agent. Response times for AC were unaffected by operating the HAZMATCAD Plus from a cold start (i.e., with insufficient warm-up time).

HAZMATCAD Plus recovery times (i.e., the time needed for the HAZMATCAD Plus 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. Recovery times differed considerably from one TIC to another. For AC, modeled recovery times ranged from 76 to 361 seconds; for CG, from 36 to 57 seconds; for SA, from 23 to 25 seconds; and for Cl_2, 49 to 73 seconds. The effect of temperature on recovery time was small, except for AC, for which recovery times increased by about a factor of four as temperature decreased from 35°C to 5°C. Temperature had an effect on recovery time for GB and HD, with recovery times at 35°C less than half of those at 5°C or 22°C. All TICs had recovery times less than about 90 seconds under all RH conditions, with the exception of a modeled mean recovery time for AC of 131 seconds at medium (50%)

RH. Recovery time for GB was slightly longer at higher humidity, whereas HD showed the opposite trend, with recovery time increased by about 50% at low (<20%) RH relative to high (>80%) RH. In operation from a cold start at normal temperature and humidity, the recovery time for AC was lengthened to over 600 seconds.

The HAZMATCAD Plus was nearly 100% accurate in identifying the TIC being sampled under all temperature and humidity conditions, with only one erroneous reading among nearly 250 data points. For HD, the response was 100% accurate under all temperature and humidity settings. For GB, one unit of the HAZMATCAD Plus did not alarm during testing. With the other unit, 100% accuracy was achieved for all conditions except the high humidity tests, where unstable responses or no responses were recorded. (This behavior at high humidity is attributed by the Microsensor Systems vendor to a possible decrease in the collection efficiency of the concentrator in the SAW part of the HAZMATCAD Plus as a result of exposure to the TICs during testing.)

The accuracy of the HAZMATCAD Plus in identifying the TIC or CW agent was the same when alternating between different challenge concentrations as that when alternating between clean air and a challenge concentration.

The repeatability, or consistency, of HAZMATCAD Plus response, response times, and recovery times also was evaluated. Repeatability of response was always perfect under all levels of temperature and humidity for AC, CG, and SA. Cl_2 exhibited more variability in response. Repeatability of response for GB and HD was unaffected by temperature level. At the different humidity levels, HD had a consistent response. For GB, the response from the one HAZMATCAD Plus unit was consistent at low and medium humidities, but either no response or an unstable response was reported for GB at the high humidity, as noted above. The modeled repeatability of response times for both TICs and CW agents showed no effect from the different levels of temperature or humidity. The modeled repeatability of recovery times for AC, CG, SA, GB, and HD showed no effect from the different temperature levels. The effect of temperature on the repeatability of recovery time was significant for Cl_2, with the recovery time most repeatable under medium temperature. The modeled repeatability of recovery times for CG, SA, Cl_2, GB, and HD showed no effect from the different humidity levels. The effect of humidity on the repeatability of recovery time was significant for AC, with the greatest variability for recovery time under medium humidity.

The response thresholds of the HAZMATCAD Plus were 0.6 to 1.25 ppm for AC; 0.3 to 0.6 ppm for CG; 0.2 to 0.4 ppm for SA; 0.5 to 1 ppm for Cl_2, 0.6 to 1.1 mg/m^3 for GB; and 0.6 to 1.6 mg/m^3 and 1.6 to 4 mg/m^3 for HD on Units 22 and 27, respectively.

The effects of temperature and RH on the HAZMATCAD Plus TIC response were small, with the largest effect that, at high temperature or high humidity, Cl_2 produced some Low rather than Medium responses. For GB at low temperature, the response was consistently High, while at medium and high temperature, the response was consistently Medium. For HD, the response was consistently High for each temperature level. For GB at high humidity, the response was either an unstable response or no response, as noted above. For HD, the response was consistently High for each humidity level.

Interferents likely to be present indoors had large effects with individual TICs and CW agents. No false positive responses occurred. In terms of false negatives, however, neither HAZMATCAD Plus responded to GB in the presence of air freshener and latex paint fumes or to HD in the presence of ammonia cleaner and latex paint fumes. Interferents had almost no effect on response times for the TICs. The interferents showed an effect on the recovery time primarily for Cl_2, with all interferents increasing the Cl_2 recovery time substantially.

Operating the HAZMATCAD Plus with insufficient warm-up time showed no effect on the instrument response or response times for AC, regardless of whether the cold start occurred after storage at 5ºC, at room temperature, or at 40ºC. There was a strong effect on recovery time depending on start state, with the longest recovery time occurring at the room temperature cold-start condition. The delay time (time for the HAZMATCAD Plus unit to be ready for a first reading after start-up) ranged from 16 to 40 seconds.

The useful operating lives for fully charged batteries in two HAZMATCAD Plus units in continuous operation were found to be 9 hours and 49 minutes and 10 hours and 53 minutes, respectively. Both units showed rapid response and consistent readings throughout this test until the last few minutes of useful battery life.

In general, the HAZMATCAD Plus was easy to use, gave clear alarms and a readable and informative display, and provided error messages. Batteries are easy to obtain and install, and new batteries can be installed without interrupting continuous operation. The one operational limitation was the failure of one unit to respond during GB testing.

Chapter 8
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