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DOMESTIC PREPAREDNESS PROGRAM
TESTING OF THE SCENTOSCREEN GAS CHROMATOGRAPH INSTRUMENT AGAINST CHEMICAL WARFARE AGENTS
SUMMARY REPORT

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This report characterizes the chemical warfare (CW) agent detection potential of the commercially available SCENTOSCREEN by Sentex. The instrument was tested against HD, GB, and GA vapor under various conditions. This report is intended to provide the emergency responders concerned with CW agent detection an overview of the detection capabilities of the SCENTOSCREEN.

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PREFACE

The work described herein was authorized under the Expert Assistance (Equipment Test) Program for the U.S. Army Soldier and Biological Chemical Command (SBCCOM) Program Director for Domestic Preparedness. This work was started in February 2001 and was completed in August 2001.

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. This report may not be cited for purposes of advertisement.

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FIGURE

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DOMESTIC PREPAREDNESS PROGRAM TESTING OF THE SCENTOSCREEN GAS CHROMATOGRAPH INSTRUMENT AGAINST CHEMICAL WARFARE AGENTS SUMMARY REPORT

1. INTRODUCTION

The Department of Defense (DoD) formed the Domestic Preparedness (DP) Program in 1996 in response to Public Law 104-201. One of the objectives is to enhance federal, state, and local capabilities to respond to Nuclear, Biological and Chemical (NBC) terrorism incidents. Emergency responders who encounter either a contaminated or a potentially contaminated area must survey the area for the presence of either toxic or explosive vapors. Presently, the vapor detectors commonly used are not designed to detect and identify chemical warfare (CW) agents. Little data are available concerning the ability of these commonly used, commercially available detection devices to detect CW agents. Under the DP Expert Assistance (Test Equipment) Program, the U.S. Army Soldier and Biological Chemical Command (SBCCOM) established a program to address this need. The Applied Chemistry Team (ACT), Aberdeen Proving Ground, Maryland, performed the detector testing. ACT is tasked with providing the necessary information to aid authorities in the selection of detection equipment applicable to their needs.

Reports of the instrument evaluations are posted in the Homeland Defense website (http://hld.sbccom.army.mil/) for public access. Instruments evaluated and reported since 1998 include:

- MiniRAE plus from RAE Systems, Inc.
- Passport II Organic Vapor Monitor from Mine Safety Appliance Co.
- PI-101 Trace Gas Analyzer from HNU Systems, Inc.
- TVA 1000B Toxic Vapor Analyzer (PID and FID) from Foxboro Co.
- Draeger Colorimetric Tubes (Thioether and Phosphoric Acid Ester) from Draeger Corp.
- Photovac MicroFID detector from Perkin Elmer Corp.
- MIRAN SapphIRe Air Analyzer from Foxboro Co.
- MSA Colorimetric Tubes (HD and Phosphoric Acid Ester) from Mine Safety Appliances Co.
- M90-D1-C Chemical Warfare Detector from Environics OY, Finland
- APD2000 Detectors from Environmental Technologies Group, Inc.
- SAW MiniCAD mkII from Microsensor Systems, Inc
- UC AP2C Monitor from Proengin Inc., France
- ppbRAE Photo-Ionization Detector from RAE Systems, Inc.
- SABRE2000 detector from Barringer Technologies, Inc.
- CAM (Type L) from Graseby Dynamics Ltd., UK

In 2001, the evaluation of instruments continued using test items that were loaned to the DP program by the respective manufacturers. Viable candidate instruments were required to pass a pre-screening test. In exchange, the instruments were evaluated under the DP protocol and the manufacturers were permitted to take data during the evaluations. Instruments evaluated included:

- VaporTracer System from Ion Track Instruments, Inc. (Wilmington, MA)
- HAZMATCAD from Microsensor Systems, (Apopka, FL)
- GC-FPD/MSD with Dynatherm System from Agilent (Columbia, MD)
- SCENTOSCREEN GC from Sentex Systems, Inc. (Ridgefield, NJ)

Each of these evaluations will be reported separately. This report pertains to the evaluation of the Sentex SCENTOSCREEN.

2. OBJECTIVE

The objective of this report is to assess the capability and general characteristics of the SCENTOSCREEN to detect CW agent vapors. The intent is to provide the emergency responders concerned with CW agent detection an overview of the detection capabilities of the instrument.

3. SCOPE

This evaluation is an attempt to characterize the CW agent vapor detection capability of the Sentex SCENTOSCREEN detection instrument. Due to time and resource limitations, the agents used were limited to tabun (GA), sarin (GB), and mustard (HD). These were chosen as representative CW agents because they are believed to be the most likely threats. Test procedures follow the established DP Detector Test and Evaluation Protocol described in the Phase 1 Test Report[1]. However, due to the nature of the SCENTOSCREEN instrumentation, the DP protocol was amended slightly to accommodate the more exploratory and time consuming procedures required of the operator than the previously evaluated DP instruments. This instrument is intended to be operated in a mobile analytical lab where the temperature is controlled, therefore no temperature testing was deemed necessary. The system was evaluated using the following test protocol:

a. Determine the Minimum Detectable Level (MDL) where repeatable detection readings are achieved for each selected CW agent. The current military Joint Services Operational Requirements (JSOR)[2] served as a guide for detection sensitivity objectives.

b. Investigate the effects of humidity on instrument performance.

c. Observe the effects of potential interfering substances upon instrument performance both in the laboratory and in the field.

4. EQUIPMENT AND TEST PROCEDURES

4.1 INSTRUMENT DESCRIPTION

Sentex Systems, Inc. (http://www.sentexinc.com/scentoscreen.html, Ridgefield, New Jersey is the manufacturer of the SCENTOSCREEN. The system was loaned to the DP Program for inclusion in the 2001 detector evaluations. The SCENTOSCREEN is a portable gas chromatograph (GC) that weighs less than 30 lbs. Instrument description and operating procedures originate from the SCENTOSCREEN Operation/Instruction Manual[3]. The SCENTOSCREEN is a portable gas chromatograph designed specifically to complete an entire analysis without the need for additional equipment usually required for interpretation of the chromatographs.

The Sentex SCENTOSCREEN operates through a portable computer that can perform automatically either Gas Chromatographic Analysis or Total Hydrocarbon Analysis. Hydrocarbon analysis can only be performed if the instrument is equipped with a Photoionization Detector (PID). The instrument tested was equipped with a Micro Argon Ionization Detector (MAID). The MAID is an electron capture type detector utilizing argon gas. Results obtained in the report are specific with this MAID detector operating in the GC mode only.

In the Gas Chromatographic Mode (GC), the instrument will perform the following functions automatically:

- Sample concentration
- Sample injection through thermal desorption
- Chromatographic separation and detection using the MAID
- Peak identification and integration
- Display of chromatograms, retention times, concentration levels and operating conditions.
- Unattended, repeating analysis
- Recalibration at a preselected frequency

The Sentex SCENTOSCREEN performs GC analysis in two operational modes; the first of which is the Calibration Analysis. In this mode, the instrument analyzes an external calibration mixture for system calibration and displays the calibration chromatogram, the name, concentration level, and retention time of the calibration compound. The area under the peaks are integrated and assigned a predetermined concentration level corresponding to the peak area.

The second operational mode is the Sample Analysis, which is used to analyze ambient air, headspace, or liquid sample. In this mode, the instrument displays the analysis chromatogram above the calibration chromatogram and lists the names, concentration levels, and retention times of the compound peaks that match the compounds stored in the given Calibration Library. Compounds detected that do not match compounds identified by the Calibration Library are listed as “UNKNOWN”.

The instrument can operate from its internal gas supply and battery power for several hours. The unit is shown in the figure below. It connects to a portable laptop computer to control its operation, data processing and storage of all chromatograms. The computer, which fits on top of the unit, is detachable from the basic body of the SCENTOSCREEN for remote analysis review, hard copy printout, and operation of other MS-DOS programs. The SCENTOSCREEN can function unattended, perform analysis periodically, and calibrate at a chosen frequency with results automatically stored on disk for later review.

Figure 1. Top View of the SENTEX SCENTOSCREEN

4.2 SEQUENCE PARAMETERS

Operating parameters were established through series of experimental observations using surety materials (GA, GB, and HD) vapors. The various time, temperature, and chart duration settings were selected based on the recommendation from the manufacturer and the proposed JSOR requirement for instruments that could potentially detect the AEL concentration levels of these surety agents within 15 minutes. The company suggested that a six minute sample time would provide optimal performance at the sampling rate of approximately 250 milliliters per minute for the instrument. Sequence parameters were set as follows for this evaluation testing:

Initial Temp: 70°C
Final Temp: 70°C
Noise Threshold: 300
Analysis Tolerance: 3%
Analysis Method: Pre-concentrator
Sampling: Manual
Trap Name: Carboxen
Integrate Options: Manual

Sample Time: 360 seconds
Dry Purge Time: 60 seconds
Delay Time: 20 seconds
Desorption Time: 4 seconds
Inhibit Time: 0 seconds
Chart Duration: 5 minutes
Backflush: Off

The intake sample flow was approximately 250 cc/min as measured through a flow meter. The total run duration per analytical cycle requires 13 minutes.

4.3 CALIBRATION

Operating procedures were followed according to the operator's User's Manual. Identification using gas chromatography is based on the principle that at constant temperature and carrier gas flow through a set length of GC column, the retention time for a substance vapor eluted (the travel time from the time of injection to the time of detection) is consistent. In order to enable the instrument to identify certain vapors, samples of known vapors must be introduced into the system to determine the retention time and recorded into the system library. Thereafter, when an unknown sample is introduced to the system and its retention time is found to match one of the previously stored retention times in the library, the unknown sample is identified as the same chemical. Thus, the initial calibration of the system requires accurate determination of the retention time for each of the different agents (GA, GB, and HD). The retention times for this particular instrument were determined by exposure to the respective vapor from a humidity controlled vapor generator.

4.4 AGENT VAPOR CHALLENGE

The agent challenges were conducted using the Multi-Purpose Chemical Agent Vapor Generation System[4] using Chemical Agent Standard Analytical Reference Material (CASARM) grade or the highest purity CW agents available. Agent challenge followed successful instrument start up. The vapor generator system permits testing of the instrument with humidity conditioned air without agent vapor to assure background air does not interfere before challenging it with similarly conditioned air containing the CW agent vapor. With the instrument's inlet placed under the cup-like sampling port of the vapor generator, the SCENTOSCREEN is exposed to the conditioned air to establish a background trace and ensure that the instrument does not exhibit undesired response peaks before agent challenges. The trace is saved as the background trace by the instrument for that series of tests.

Agent challenge begins when the solenoids of the vapor generation system are energized to switch the air streams from conditioned air only to similarly conditioned air containing the agent. The unit was tested three times under each condition. The SCENTOSCREEN collected the sample and performed the analysis. The resulting trace is overlaid on the background trace to show the existing elution peaks. The peak that corresponds with the predetermined agent peak, with regard to retention time, is construed to be a positive detection. Absence of the expected peak would indicate no detection.

The instrument was tested with the agents GA, GB, and HD at different concentration levels at ambient temperatures and 50% RH in an attempt to determine the MDL.

4.5 AGENT VAPOR QUANTIFICATION

The generated agent vapor concentrations were analyzed independently. The vapor concentration was quantified by utilizing the manual sample collection methodology[5] using the Miniature Continuous Air Monitoring System (MINICAMS®) manufactured by O. I. Analytical, Inc., Birmingham, Alabama. The MINICAMS® is equipped with a flame photometric detector (FPD), and was operated in phosphorus mode for the GA and GB agents, and sulfur mode for HD.

This system normally monitors air by collection through sample lines and subsequently adsorbing the CW agent onto the solid sorbent contained in a glass tube referred to as the pre-concentrator tube (PCT). The PCT is located after the MINICAMS® inlet. Then the concentrated sample is periodically heat desorbed into a gas chromatographic capillary column for subsequent separation, identification, and quantification. For manual sample collection, the PCT was removed from the MINICAMS® during its sampling cycle and connected to a measured vacuum source to draw the vapor sample from the agent generator. The PCT was then re-inserted into the MINICAMS® for analysis. This "manual sample collection" methodology eliminates potential loss of sample along the sampling lines and the inlet assembly when the MINICAMS® is used as an analytical instrument. The calibration of the MINICAMS® was performed weekly and checked daily using the appropriate standards for the agent of interest. The measured mass equivalent (derived from the MINICAMS chromatogram) divided by the total volume (flow rate x time) of the vapor sample drawn through the PCT produces the sample concentration that converts into mg/m^3.

4.6 LABORATORY INTERFERENCE TESTS

The laboratory interference tests were designed to assess the effect on the instruments of vapor exposure from potential interfering substances by determining if any of the substances would produce false identification as one of the surety materials. In these tests, no CW agent was present. The substances were chosen based on the likelihood of their presence during an emergency response by first responders.

The SCENTOSCREEN unit was tested against 1% of the headspace concentrations of vapors of gasoline, JP8, diesel fuel, household chlorine bleach, floor wax, AFFF, Spray 9 cleaner, Windex, toluene, and vinegar. The unit was also tested against 25 ppm NH_3 (ammonia). A dry air stream carries the headspace vapor of the substance by sweeping it over the liquid in a tube or through the liquid in a bubbler to prepare the interferent gas mixture. Thirty milliliters/minute of this vapor saturated air is then diluted to 3 liters/minute with the conditioned air at 23°C and 50% RH to produce the 1% concentration of interferent test mixture. The 25 ppm ammonia was derived by proper dilution of a stream from an analyzed 1% NH_3 vapor (10,000 ppm) compressed gas cylinder with the appropriate amount of the conditioned air.

4.7 FIELD INTERFERENCE TESTS

The instrument was tested in the presence of common potential interferents such as the vapors from gasoline, diesel fuel, jet propulsion fuel (JP8), kerosene, Aqueous Film Forming Foam (AFFF, used for fire fighting), household chlorine bleach, and insect repellent. Vapor from a 10% calcium hypochlorite solution (HTH slurry, a chlorinating decontaminant for CW agents), engine exhausts, burning fuels, and other burning materials were also tested. The objective was to assess the ability of the instrument to withstand outdoor environments and to resist false alarming indications when exposed to the selected substances. In these tests, no CW agent was present.

The field tests were conducted outdoors at M-Field, Edgewood Area, Aberdeen Proving Ground, in July 2001. These experiments involved open containers, truck engines, and fires producing smoke plumes, which were sampled by the instrument at various distances downwind. The SCENTOSCREEN unit was exposed to either the smoke or fume test plume to achieve moderate exposures (e.g. 2 - 15 ft for vapor fumes and 6-30 ft for smokes).

Testing continued with the next challenge after the instrument had recovered from prior exposure.

5. RESULTS AND DISCUSSION

5.1 MINIMUM DETECTABLE LEVELS

The MDLs for the tested SCENTOSCREEN are shown in Table 1 for each agent at ambient temperatures and 50% RH. The MDL values represent the lowest CW agent concentration that produced a peak that corresponded with the determined agent peak, consistently for three trials. The MDL concentrations are expressed in milligrams per cubic meter (mg/m^3) with equivalent parts per million (ppm) values also given. For comparison, the current military JSOR requirements for CW agent sensitivity for point detection alarms, the U.S. Army's established values for Immediate Danger to Life or Health (IDLH), and the Airborne Exposure Limit (AEL) are also listed in Table 1. The AEL values are equal to the safe TWA (time weighted average) concentration for unmasked workers in an agent environment for 8 hours. Army Regulation (AR) 385-61 is the source for the IDLH, AEL, TWA values for GA and GB, and the AEL, TWA values for HD. The AR 385-61 does not establish an IDLH for HD due to concerns over carcinogenicity.

The SCENTOSCREEN was able to detect 0.006 mg/m^3 HD, 0.007 mg/m^3 GA, and 0.02 mg/m^3 GB using the 13 min cycle. Thus, the SCENTOSCREEN could detect well below the JSOR and IDLH concentration levels but was unable to do so within the time specified for point detection by the JSOR. The SCENTOSCREEN was unable to detect to the AEL values for HD, GA or GB.

Table 1. Minimum Detectable Level (MDL) at Ambient Temperatures and 50% RH for the SCENTOSCREEN

AGENT	Concentration in milligrams per cubic meter, mg/m^3, with parts per million values in parentheses (ppm), and Response Times			
	SENTEX SCENTOSCREEN MDL	JSOR*	IDLH**	AEL***
HD	0.006 (0.0009) in 13 min	2.0 (0.300) in 2 min 0.003 (0.0005) up to 15 min	N/A	0.003 (0.0005) up to 8 hr
GA	0.007 (0.001) in 13 min	0.1 (0.015) in 0.5 min 0.0001 (0.000015) up to 15 min	0.2 (0.03) up to 30 min	0.0001 (0.000015) up to 8 hr
GB	0.02 (0.0035) in 13 min	0.1 (0.017) in 0.5 min 0.0001 (0.000017) up to 15 min	0.2 (0.03) up to 30 min	0.0001 (0.000017) up to 8 hr

* Joint Service Operational Requirements for CW agent detectors (ACADA and JCAD).
** Immediate Danger to Life or Health values from AR 385-61 to determine level of CW protection. Personnel must wear either the full ensemble with SCBA for operations or full-face piece respirator for escape.
***Airborne Exposure Limit values to determine masking requirements. Personnel can operate unmasked for up to 8 hrs. Otherwise known as the safe TWA (time weighted average) concentration for unmasked workers in an agent environment for 8 hrs. AEL and TWA values are from the unclassified Army Regulation AR 385-61, Feb 1997.

5.2 HUMIDITY EFFECTS

The instrument was tested under varied humidity conditions to assess its behavior. Table 2 lists the respective MDL responses of the unit at the various test conditions. The tests were conducted at ambient temperatures and approximately 10, 50 and 90% RH. The results listed represent multiple challenges with the test unit at low agent concentrations.

At ambient temperatures, the humidity changes did not appear to adversely affect the performance of the SCENTOSCREEN, except with GA. At humidity <10% the sensitivity for detecting GA was decreased three-fold.

Table 2. SCENTOSCREEN Response to CW Agent Concentrations at Various Humidity Conditions

Agent	Concentration (mg/m^3)	Concentration (ppm)	Humidity, % RH	Temperature, °C
HD	0.0058	0.0009	4	25
HD	0.0060	0.0009	50	24
HD	0.0065	0.0010	93	26
GA	0.0225	0.0034	9	22
GA	0.0073	0.0011	50	25
GA	*Not Tested	*Not Tested	90	23
GB	0.0185	0.0032	8	23
GB	0.0203	0.0035	51	23
GB	0.0184	0.0032	88	23

*Not Tested: Testing was not completed due to instrument failure and insufficient time for further testing.

5.3 LABORATORY INTERFERENCE TESTS

Table 3 shows the established retention time for the agents tested. A range is noted since the retention time shifted due to unknown factors, possibly room temperature fluctuations.

Table 3. Established Retention Times for HD, GA, and GB

AGENT	Retention Time (RT) in secs
HD	55-57
GB	205-213
GA	240-246

Table 4 presents the results of exposing the instruments to several potential interferents without CW agent. Most of the interferents produced very high and broad peaks. Many of the peaks did not return to the baseline and therefore would mask any compounds with retention times following the initial peak. Since the peaks from the substances were seen to overlap with the established peaks determined for the agents, they are construed to have affected the detection capability of the instrument and are listed under the headings “Possible Interfered Agent” and “notes”.

Table 4. Results of Laboratory Interference Tests at Ambient Temperature and 50%RH

INTERFERENT (All at 1% concentration)	SAMPLE TIME (sec)	RT PEAKS IN AGENT WINDOW (sec)	POSSIBLE INTERFERED AGENT	NOTES
Vinegar	30	52	HD	Large, wide peak
AFFF	30	54	HD	Large, wide peak
Diesel	30	55, 215, 235	All agents	Large, wide peaks
Windex	30	55	HD	Large, wide peak
Toluene	30	55,193	All agents	Many cycles to purge
Spray 9	30	61	HD	Large, wide peak
Floor Wax	30	None	None	None
Bleach	30	None	None	None
JP8	30	54, 192, 253	All agents	Many cycles to purge
Gasoline	30	42, 214, 259	All agents	Many cycles to purge
Ammonia	30	None	None	None

5.4 FIELD INTERFERENCE TESTS

The results of the field test interferent exposures are presented in Table 5. Field test conditions included ambient temperatures in the range of 26-31 ^{o}C with relative humidity levels between 53-76% and gentle winds from 3 to 10 mph. The unit was exposed using the 6 min sampling time against the first five listed substances, but the sample time was decreased to 60 sec, to minimize the potential degree of contamination. As shown, every interferent was only tested once against the unit due to overwhelming signals caused by the interferents and testing time constraints. After each exposure the unit was allowed to run until cleared of residual peaks before next exposure that, sometimes, took nearly an hour to clear.

Referring to the retention times listed in Table 3, the potential false positive agent identifications, as the result of exposures to the substances, are summarized in Table 5. The entries under the heading “Possible Interfered Agent” and “Notes” are the judgements of the test personnel. As shown under “Notes”, the SCENTOSCREEN false alarmed with identification of substance peaks as HD.

Table 5. SCENTOSCREEN Field Interference Testing Summary

INTERFERENT	SAMPLE TIME (secs)	RT PEAKS IN AGENTWINDOW (secs)	POSSIBLE INTERFERED AGENT	NOTES
Gasoline Exhaust, Idle	360	None	None	None
Gasoline Exhaust, Revved	360	None	None	None
Diesel Exhaust, Idle	360	None	None	None
Diesel Exhaust, Revved	360	57	HD	Identified as HD
Gasoline Vapor	360	One Big Peak	All agents	3 cycles to purge
Burning Gasoline Smoke	60	54	HD	None
Diesel Vapor	60	54, 244	HD, GA	None
Burning Diesel Smoke	60	54	HD	None
JP8 Vapor	60	55, 242	HD	Identified as HD
JP8 Burning	60	54	HD	None
Kerosene Vapor	60	54, 242	HD, GA	None
Burning Kerosene Smoke	60	54	HD	Identified as HD
Burning Cotton Clothes	60	54	HD	None
AFFF (6%) Vapor	60	55	HD	Identified as HD
Clorox (6% Bleach) Vapor	60	55	HD	Identified as HD
HTH (10% Calcium Hypochlorite) Vapor	60	55	HD	Identified as HD
OFF Insect Repellent 10% DEET	60	55	HD	None
Burning Wood Fire Smoke	60	55	HD	None
Doused Wood Fire Smoke	60	55, 210	HD, GB	None

Post field test challenges against agents were not conducted due to gross contamination of the instrument. After more than a dozen blank runs, the instrument background baseline still showed gross contamination. Further testing was discontinued due to these problems.

6. CONCLUSIONS

Conclusions are based solely on the results observed during this testing. Aspects of the instruments other than those described were not investigated.

Civilian first responders and HAZMAT personnel use Immediate Danger to Life or Health (IDLH) values to determine levels of protection selection during consequence management of an incident. The SCENTOSCREEN unit was able to detect HD, GA, and GB at concentrations well below the JSOR and IDLH values with sampling times of 6 minutes at a 250 cc/min flow rate, which required a total of 13 minutes for the complete analysis cycle. Attempts

to qualify the instrument as an AEL detection device failed to materialize because of the lengthy analysis cycle. The instrument was unable to detect down to the AEL values for HD, GA or GB during this testing.

The instrument required connection to a laptop computer to operate. The instrument becomes contaminated easily when exposed to the tested interferents. The detection traces collected from interference testing showed that this instrument is subject to false agent indications (16 of 19 tested) on sampling of vapor from many different substances.

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