Testing & Evaluation Report Form

Report Date 07/15/2009

Project Information

Title: HazMatID FTIR Evaluation
Evaluation Type: Instrumentation
Stakeholder(s): Smiths Detection, NFSTC Mobile Laboratory Project
Start Date: 4/13/2009 End Date: 5/13/2009

Manufacturer Information

Manufacturer: Smiths Detection
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Evaluation Overview

The National Forensic Science Technology Center (NFSTC) conducted an assessment of the Smiths Detection HazMatID portable Fourier Transform InfraRed (FTIR) system. The purpose of this performance evaluation was to determine and establish the suitability of this chemical analyzer for use in the identification of forensically relevant chemical compounds. The samples used during the evaluation were selected to be a representative subset from each of the following groups: drugs of abuse, ignitable liquids, explosives and common household materials.

During the collection of the IR spectra from these compounds, the following observations concerning the instrument were noted:

- The instrument utilizes a stainless steel ATR sampling port with an embedded diamond that allows for non-destructive analysis of samples without the need for specialized sample preparation equipment. The beam splitter uses a ZnSe substrate that is very resistant to the environment.
- A library of FTIR spectra of more than 32,000 known compounds is included within the HazMatID system, which provides the user with an extensive database of known compound spectra to search against. A fully customizable library is also available for the addition and identification of user specific compounds.
- A Search Residual option within the HazMatID software allows for the identification of individual components within some mixture samples. When activated, this feature automatically subtracts the peaks from the spectrum corresponding to the primary identified component, and then compares the

difference or “residual” spectral peaks to the library for the secondary component identification.

- The HazMatID FTIR system accurately identified illicit drugs of abuse when tested in pure form, but results for drug mixtures and other compounds tested were less reliable, depending primarily on the IR characteristics of the primary component as well as the compound(s) of interest within the sample.
- The HazMatID system can be used to qualitatively identify materials such as powders, liquids, pastes, gels and solids.
- The instrument is fully portable and easy to transport given the unit’s rugged external casing with built-in handle.
- The time required from initial instrument setup to collection of the first sample spectrum is approximately 20 minutes, including automatic mirror alignment, beam power check and QC sample analysis.

Evaluation Team

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Product Specifications

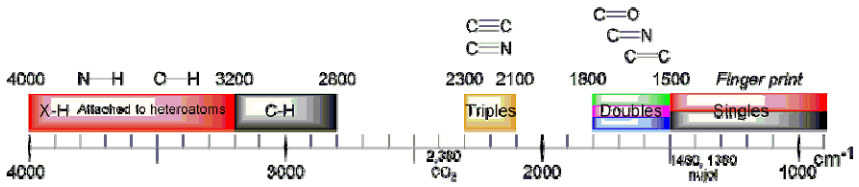
Photo(s):

Product Name: HazMatID System
Model No: N/A
Serial/Lot No: 026890908H

Brief Description and Background Information

The HazMatID system is a small portable chemical analyzer manufactured to allow safe transportation and quick setup for use in field operations or in mobile laboratory environments. The HazMatID operates based on Fourier Transform InfraRed (FTIR) spectroscopy coupled with an Attenuated Total Reflectance (ATR), non-destructive sampling technique.

In infrared (IR) spectroscopy, electromagnetic radiation is focused onto a sample. Some of the electromagnetic radiation is absorbed by the sample, and some of it is passed through or transmitted. An FTIR instrument examines the absorbance/transmittance over the entire infrared spectrum simultaneously. FTIR spectroscopy is based upon the theory that chemical functional groups will absorb IR radiation and then convert that energy into specific molecular vibrations through either stretching or bending of the chemical bond(s). Given the unique way the chemical bonds present within a compound absorb the IR radiation, no two compounds should produce the same infrared spectrum. The following chart represents some of the types of bonds and their corresponding wave numbers used to facilitate chemical identification in FTIR.



Product Uses

The Smiths Detection HazMatID FTIR can be used to qualitatively identify chemical compounds, including illicit drugs, explosives, TICs, WMDs and other organic compounds that produce an IR spectrum with sufficient discriminating detail.

Product Specifications

Dimensions: 18” x 12” x 7” (L x D x H)
Weight: 23 pounds
Storage Conditions: -20°C to +80°C
Operational Conditions: -7°C to +50°C; system is waterproof, however battery power should be used in wet conditions
Power Requirements: 100-240V, 15 Amp, 50-60Hz; AC or battery (included)
Cost: approximately $55,000
User replaceable components: Fuse, cat #0234018 (pk of 5) $11
IR source, cat #0234014, $9,050
Battery, cat #0234008, 1 pair, $385

Note: Prices are approximate and subject to change.

Evaluation

Objectives

1. Determine the portability, robustness, and stability of the instrument in both a fixed and mobile laboratory environment. Evaluate the manufacturer's level of customer support.
2. Determine the ability of the instrument to accurately identify samples of known drugs, pharmaceuticals, ignitable liquids, explosives, and other substances commonly encountered in forensic scenarios.
3. Determine the ability of the instrument to detect and identify components of interest in a mixture, e.g., illicit drugs mixed with common 'cutting' agents.

Instrument Setup Performed by:

☐ Manufacturer ☐ Manufacturer & Evaluator ☒ Evaluator Only

Instrument Setup Comment(s):

The instrument was shipped in a ruggedized Pelican™ 1620 foam-lined case. The following components were provided with the HazMatID:

- HazMatID system with power supplies (two batteries and AC power adapter)
- HazMatID application software
- USB storage device
- Accessories kit containing liquids well, volatile cover, spatula, pipette and alcohol swabs
- A separate container with a keyboard, mouse and battery charger kit

The HazMatID system can operate on one battery or by using the provided power supply. Only the power supply provided with the HazMatID system should be used. Once turned on the Status screen should be always checked to ensure the instrument is functioning properly. If all internal checks are passed, the screen will display "Ready". If the status screen shows "Energy" and turns yellow, the Automatic Alignment must be performed. While Automatic Alignment is in process, the HazMatID must remain motionless. Prior to analysis of any samples, the user should assure that the sensor area is clean and clear of any particles and/or samples.

A performance validation should be performed periodically (preferably after movement and vibrations) to validate the spectral quality and overall performance of the HazMatID system.

Minimum Skill Level of Operator as Set By Manufacturer

☒ Non-Scientist ☐Technician ☐Scientist

Standards, Controls and Samples Used in Evaluation

Controls: Polystyrene film, Polyethylene chip

Controlled Substances: Cocaine Base, Cocaine HCl, Heroin, Methamphetamine, Quinine, Alprazolam, Chlordiazepoxide, Temazepam, Diazepam, Tylenol 3, Oxycontin, Oxycodone, Hydrocodone

Uncontrolled Substances: Table Sugar, Baking Powder, Baking Soda, Caffeine, Benzocaine, Guaifenesin, Water, Vinegar, Hydrogen Peroxide, Powder Sugar, Sodium Chloride, Mannitol, Boric Acid, Dimethyl Sulfide (DMS), Niacinamide, Procaine, Urea

Ignitable Liquids/Explosives: Diesel, Lamp Oil, Lighter Fluid, Kerosene, Acetone, Ammonium Nitrate, Ammonium Perchlorate, Aluminum Powder, Nitrocellulose, Magnesium Powder

Equipment and Consumables

- HazMat ID System
- Isopropyl Alcohol/Methanol/Acetone (cleaning agents)
- Tissue
- Spatula
- Screwdriver (flat head)
- Performance Validation Sample (polyethylene chip)
- Polystyrene film for verification

Experiments

All experiments were designed to evaluate the capability of the HazMatID FTIR system to accurately identify compounds either in pure form or as a component of a mixture. The system includes an extensive IR spectral library of known compounds and a search function that provides the user with library match results and appropriate correlation values. The software also includes a function that allows the user to search residual from questioned sample spectra in an attempt to identify other compounds that may be present within the sample. There were four different sets of experiments conducted during the evaluation; and they are as follows:

Set 1: Analysis of known, pure compounds listed above in Controlled and Uncontrolled Substances

Set 2: Analysis of materials listed in Ignitable Liquids/Explosives

Set 3: Analysis of controlled pharmaceuticals

Set 4: Analysis of mixtures (20:80, 40:60, 50:50, 60:40, 80:20) of controlled substances and uncontrolled substances

IR spectra were recorded from each sample in replicate and library searches (including residual peak searches) to determine the capability of the HazMatID instrument/software to identify the compound of interest in each sample. Powders, pastes, tablet contents, and solids were tested by placing a small amount of the sample onto the ATR sampling port using a spatula and lowering the 'Force applicator arm' onto the sample to ensure contact between the sample and the ATR port. Liquid and volatile samples were tested by pipetting one to four drops onto the sampling port.

Results

Objective 1: Instrument Portability, Robustness and Customer Support

The instrument was setup in the NFSTC laboratory to gather information about instrument setup, gain familiarity with the instrument software, and generate standard spectra from the included polystyrene standard sample and known samples. The instrument performed well in all of these tests. The instrument was then packed into the Pelican™ case, which includes spaces for the instrument, battery packs and other accessories. The HazMatID was easily transported to and from the NFSTC warehouse, up and down stairs and around the main laboratory complex. After each move or restart, the Automatic Alignment procedure built into the software was performed to ensure instrument performance. The instrument was also operated in a mobile laboratory environment with people entering and leaving the mobile laboratory, causing rocking of the unit. Collection of spectra under these conditions was slightly compromised. During some of the testing, the vendor's support line was called to evaluate the level of reachback support that would be available. The vendor was able to troubleshoot the problem and provide assistance. During this evaluation, the instrument was powered by either the rechargeable battery or the power supply.

Objective 2: Identification of Known Materials

Set 1: Controlled and Uncontrolled Substances: Table 1 lists the results from the library and residual peak search from these materials. The instrument was able to accurately identify the illicit drugs and some of the uncontrolled substances tested, but several of the other materials tested were incorrectly identified with the automated library search function.

Table 1: Controlled and Uncontrolled Materials

Material	Identified as	Residual ID
Polystyrene control	Polystyrene	Not required
Cocaine HCl	Cocaine HCl	Not required
Heroin	Heroin	Not required
Methamphetamine	Methamphetamine	Not required
Table Sugar	Pure Cane Sugar	Not required
Baking powder	Onion salt	Cerium III sulfate
Baking Soda	Onion salt	Cerium III sulfate
Caffeine	Caffeine	Not required
Benzocaine	Benzocaine	Not required
Guaifenesin	Guaifenesin	Not required
Water	Water	Not required
Vinegar	Water	Gluconic acid
Hydrogen Peroxide	Water	Hydrogen peroxide
Powder sugar	Sugar powdered	Not applicable
Sodium Chloride	Tellurium	Tin Oxide
Urea	Urea	Not required

Set 2: Ignitable Liquids/Explosives: Table 2 lists the results from the library and residual

peak search from these materials. The instrument was unable to correctly identify any of the ignitable liquids other than acetone. Three of the five materials used in explosives were accurately identified.

Table 2: Ignitable Liquids/Explosives

Samples	Identified as	Residual ID
Polystyrene control	Polystyrene	Not required
Diesel	Light Mineral oil	Petrolatum
Lamp oil	Light Mineral oil	Petrolatum
Lighter fluid	Light Mineral oil	Petrolatum
Kerosene	Light Mineral oil	Petrolatum
Acetone	Acetone	Not required
Ammonium Nitrate	Ammonium Nitrate	Not required
Ammonium perchlorate	Ammonium Perchlorate	Not required
Aluminum powder	Tin Oxide	Tin Oxide
Nitrocellulose	Nitrocellulose	Not required
Magnesium powder	Tin oxide	Tin Oxide

Set 3: Pharmaceuticals: Table 3 lists the results from the library and residual peak search from these materials. These prescription drugs were tested at multiple concentration levels to determine the capabilities and sensitivity of the HazMatID instrument. None of the active drug ingredients in the pills and capsules was correctly identified using the library or residual peak search function of the instrument.

Table 3: Pharmaceuticals

Tablet/Capsule	Identified as	Residual result
Alprazolam 0.5mg TS-013 tablet	1) Beta Lactose 2) D-Lactose Monohydrate	1) Beta cyclodextrine hydrate 2) Sparteine SO4
Alprazolam 1.0mg TS-014 tablet	1) Alpha Lactose 2) D-Lactose Monohydrate	1) Cellulose powder 2) Piperazine Hexahydrate
Alprazolam 2.0mg TS-015 tablet	1) D-Lactose monohydrate 2) D-Lactose Monohydrate	1) Cellulose powder 2) Quanoline-N-Oxide Hydrate
Chlordiazepoxide 5mg TS-016 capsule	1) Beta-L-lactose 2) Beta Lactose	1) Rauwolfia serpentina 2) Cellulose powder
Chlordiazepoxide 10mg TS-017 capsule	1) Beta Lactose 2) Beta Lactose	1) Rauwolfia serpentina 2) Rauwolfia serpentina
Temazepam 15mg TS-010 Capsule	1) Cellulose powder 2) Cellulose powder	1) Temazepam 2) Temazepam
Diazepam 10mg TS-021 tablet	1) Rauwolfia serpentina 2) Cake mix	1) Rauwolfia Serpentina 2) Cellulose powder
Diazepam 5mg TS-020 tablet	1) Rauwolfia serpentina 2) Cellulose powder	1) Rauwolfia serpentina 2) Nontranite, Garfield Wash
Tylenol 3 / APAP	Acetaminophen	Brewer's Yeast
Oxycontin 20mg	a)Alfredo sauce b)Alfredo sauce	1 Tetradecanol
Oxycodone 5mg / APAP 325mg	Acetaminophen	Brewer's Yeast

Hydrocodone 5mg / APAP 500mg	Acetaminophen	Brewer's Yeast

***results labeled 1) were obtained on HazMatID prior to shipping back for repair**
****results labeled 2) were obtained on "loaner" HazMatID system on 5 May 2009**

Objective 3: Identification of Mixture Components

Set 4: Mixtures: Table 4 lists the results from the library and residual peak search of illicit drugs mixed with secondary materials. Drugs collected in criminal investigations are often reduced in purity by the introduction of a "cutting" agent. The ratio of the drug to the 'cutting' agent is shown in the first row of the table. The ability of the HazMatID instrument to detect the drug of interest depended on the IR characteristics of the secondary material and the relative purity of the drug in the mixture. In mixtures where the secondary material or cutting agent produced a strong IR spectrum, the minimum concentration of drug that could be detected was lowered.

The lowest level of cocaine base was the 20:80 mixture when mixed with mannitol, but only in the 80:20 mixture when mixed with caffeine. Cocaine HCl could be identified in the residual peaks in the 20:80 mixture when mixed with mannitol and procaine, but could not be identified at less than the 50:50 mixture when mixed with caffeine or boric acid. Even then it was a residual peak rather than the primary spectra obtained from the sample. Methamphetamine was accurately identified as a residual peak in the 50:50 mixture with caffeine. When mixed with niacinamide, methamphetamine was identified as the primary component in the 80:20 mixture, but the HazMatID was unable to accurately identify it at lower levels, even as a residual peak. Heroin was identified in a residual peak in the 20:80 mixture after the known 80:20 heroin:quinine spectra was added to the instrument's spectral library. Similar addition of known heroin:caffeine (50:50) and heroin:procaine (80:20) spectra to the library allowed identification of the heroin in the 50:50 mixture with caffeine and in the 20:80 procaine mixture.

Table 4: Drug mixtures tested using HazMatID system

Mixture	20/80 mix	40/60 mix	50/50 mix	60/40 mix	80/20 mix
Cocaine Base/ Benzocaine	ID as Benzocaine Residual: pyridimol dichromate	ID as Cocaine Base Residual: benzocaine	ID as Cocaine Base Residual: benzocaine	ID as Cocaine Base Residual: benzocaine	ID as Cocaine Base Residual: benzocaine
Cocaine base/ Caffeine	Sample was not available.	Sample was not available.	ID as Caffeine Residual: chloroform	ID as Caffeine Residual: chloroform	ID as Cocaine Base
Cocaine Base/ Mannitol	ID as Mannitol Residual: Cocaine Base (crack)	Sample was not available.	ID as Mannitol Residual: Cocaine Base (crack)	Sample was not available.	ID as Cocaine Base (crack) Residual: D-Mannitol
Cocaine HCl/Caffeine	Sample was not available.	ID as caffeine Residual: Gluconic Acid	ID as Caffeine Residual: Cocaine HCl	Id as Caffeine Residual: Cocaine HCl	ID as Cocaine HCl
Cocaine HCL/ Mannitol	ID as Mannitol Residual: Cocaine HCl	ID as Mannitol Residual: Cocaine HCl	ID as Cocaine HCl	Sample was not available.	ID as Cocaine HCl Residual: Mannitol
Cocaine HCl/ Procaine	ID as Procaine Residual: Cocaine HCl	ID as Cocaine HCl	Sample was not available.	Sample was not available.	ID as Cocaine HCl
Cocaine HCl/Boric Acid	ID as Boric acid Residual: Tungstic acid	ID as Boric acid Residual: Tungstic acid	ID as Boric Acid Residual: Cocaine HCl	ID as Boric Acid Residual: Cocaine HCl	ID as Boric Acid Residual: Cocaine HCl
Methamphetamine/Caffeine	Sample was not available.	ID as Caffeine Residual: O-chlorocilline	ID as Caffeine Residual: Desoxyephedrine HCl (Methamphetamine)	ID as Caffeine Residual: Desoxyephedrine HCl (Methamphetamine)	Sample was not available.
Methamphetamine/DMS	Sample was not available.	Sample was not available.	Sample was not available.	ID- Methamphetamine	Sample was not available.
Methamphetamine/ Niacinamide	Sample was not available.	Sample was not available.	ID as Niacinamide Residual: Phenyl Trimethyltin	ID as Niacinamide Residual: Phenyl Trimethyltin	ID as Methamphetamine
Heroin/Quinine	ID as Quinine Residual: Heroin and Quinine 80/20 mix	Sample was not available.	Sample was not available.	ID as Quinine Residual: Heroin and Quinine 80/20 mix	ID as Heroin
Heroin/Caffeine	ID as Caffeine Residual: Chloroform	Sample was not available.	ID as Heroin/Caffeine 50/50 mix	Sample was not available.	Sample was not available.
Heroin/Procaine	ID as Procaine Residual: Heroin HCl	ID as Procain Residual: Heroin	ID as Heroin/Procaine 80/20 mix	Sample was not available.	ID as Heroin

Findings

Strengths

- Multiple options exist to provide power to the HazMatID instrument: rechargeable batteries, power supply, or an optional automobile 12V 'cigarette lighter' interface. The battery pack and power supply were both used in the testing without difficulty.
- The instrument is suitable for use in portable laboratory environments based on the testing done in this evaluation. The diamond ATR allows non-destructive sample analysis and was not damaged by any samples tested during this evaluation. The instrument is heat-and humidity-resistant. If the instrument is to be used in wet conditions (rain, splash, etc.), it is suggested that batteries be used so that the power compartment is sealed. No negative effects on spectral quality were observed after moving the HazMatID instrument. The Automatic Alignment procedure was successfully performed after each move.
- The instrument performance is verified by using the performance validation sample included in the case (hard chip) labeled 'Performance Validation Sample'. Only about 5 minutes is required to complete the performance validation checks. The system software includes readings of the energy level status and will automatically prompt the user to perform an automatic alignment procedure if the energy level falls below a preset level.
- The library included with the HazMatID instrument consists of 32,000 entries performed by the vendor; however, the analyst can make his/her own library of compounds and mixtures that are not found in the Smiths Detection library. This can be completed by adding known mixtures or compounds to the user library. The user library is empty when the instrument is shipped from the vendor and must be unlocked to be populated by the end user. The vendor-supplied libraries are locked by the vendor and prevent the user from making changes. The software includes administrative functions that allow an administrator to set different privileges for users. Once results are obtained, the analyst can save them on the system hard drive, an external USB flash drive or an external USB floppy drive.
- The manufacturer's contact number (866-442-0628) was called for reachback support following an instrument failure. Although the customer service representative was unable to resolve the system failure over the phone, a loaner instrument was shipped overnight to NFSTC. In the event that a sample can't be identified or the analyst requires additional technical support, the spectra from the sample can be emailed to Smiths Detection for assistance.
- The instrument accurately identified the known polystyrene standard sample prior to analysis of any test samples on the HazMatID. The instrument accurately identified pure standards of illicit drugs, some over-the-counter drugs and some household items. Other known substances were not correctly identified. Some common components of explosives such as ammonium nitrate, nitrocellulose, and ammonium perchlorate could be positively identified. Military grade explosives were not evaluated.

- While evaluating mixtures at different concentrations, some of the mixtures that were not available in the library were added, allowing identifications of the components in the mixtures to occur in subsequent testing. The Search Residual software feature could be used to identify secondary or minor components of a mixture. This feature was more successful after a known mixture was added to the user library.
- The quantity of sample placed on the ATR must be sufficient to cover the embedded diamond, but not so large as to interfere with the instrument's ability to record absorbance/transmittance of the IR beam. Homogenous, pure samples of illicit drugs, over-the-counter drugs and explosive components produced reproducible and expected results. Non-homogenous samples produced inconsistent and unpredictable results, often resulting in incorrect identification of compounds based on spectral library or residual searches.

Opportunities for Improvement

- When using this instrument in a deployed laboratory setting, vibration or movement of the laboratory creates difficulties in obtaining clear, usable spectra when testing samples. Prompting the user to analyze the included polystyrene standard film after realignment is performed to verify the instrument is in calibration would improve the accuracy and reliability of the data. The capability of the instrument to identify mixtures would be improved by adding known mixtures that are commonly encountered in forensic settings to the library.

Limitations of the Technology

- FTIR analysis is most suitable for organic compounds. Inorganic compounds such as sodium chloride and potassium chloride produce essentially no IR spectra because of their chemical structure. Aluminum and magnesium powders do not produce usable results. Discrimination between diesel, lamp oil, and kerosene is not possible because of their hydrocarbon base. The capability of this instrument platform is entirely dependent on the quality and contents of the known compound spectral library used for comparison of unknown substances. As more compounds and mixtures are included in the library, the probability that unknown compounds will be accurately identified increases.

- The HazMatID will correctly identify most commonly used illicit drugs (cocaine, methamphetamine, heroin) if encountered in pure form. However, in actual forensic samples that are often mixed with other compounds, the likelihood of identification depends entirely on the IR characteristics of the mixture components. This HazMatID instrument may fail to identify drugs or other materials of interest in mixtures because of the IR characteristics of the other components in the mixture.

Training Requirements

- The depth of training will depend on the desired level of the end user. An operator with limited or no chemistry knowledge could easily use this instrument. The analyst should be trained to visually compare each potential library hit on a

chemical and residual peak to ensure accurate identifications are made. For best results, the training should consist primarily of hands-on exercises. A Powerpoint® presentation is helpful when explaining the dynamics of the instrument performance and theory. Proper shutdown procedure is required for this instrument, otherwise the operating system will not restart normally. The Users Manual provided is very useful, easy to read, but it is also very bulky to be carried around. A copy of the NFSTC Standard Operational Procedure is available.

Health and Safety Issues

- To avoid shock hazard, the analyst should never allow liquid to run into the HazMatID battery compartment door. The IR Source gets very hot, and must be handled with care. When changing the removable IR source, adequate time must be allowed for the housing and old source to cool before touching. The operating temperature and temperature immediately after shutdown are extremely hot and will burn on contact.
- The analyst must not look directly into the laser.
- The analyst should always wear proper PPE when handling unknown materials and toxic chemicals.

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