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Portable Vapor Generator for the Calibration and Test of Explosive Detectors

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Abstract— *The explosive vapor trace detectors deployed in the field require frequent verification and calibration to guarantee their accurate operation. In this paper we describe the latest advancements in the use of precision micro-dispensing technology for explosive vapor generation. The portable explosive vapor generator uses digitally controlled ink-jet dispensing to precisely eject minute amounts of dilute explosive solutions and convert them into vapor by placing them on a heater. The amount of explosive delivered to the detectors can be controlled by the number of drops (dose mode – specified number of drops is generated) and the frequency of the droplet generation (continuous mode – droplets are generated continuously at fixed frequency. The control for the heater temperature allows setting specific temperatures, but also permits specification of temperature profiles. By using a small thermal capacitance heater it is possible to achieve very sharp temperature increases. The ability to precisely control the heater temperature over a wide range permits the generation of vapors for all common explosives.*

1. INTRODUCTION

A generator of precise and minute amounts of explosive vapors is needed to provide the standards required to maintain the accuracy and sensitivity of explosive trace detectors. [1,2,3] Such a generator can be used for periodic evaluation of field instruments and to support the continuous research and development for the improvement of the detection limit that requires a vapor source of very low concentration. In this area, the vapor generator provides the means to quantify the improvements in the development of the explosive detectors.

The vapor generator is based on the ability to generate small droplets of dilute explosive solutions that are deposited on a heater where they evaporate generating the explosive vapors.[2,4,5,6] Droplets are generated using piezoelectrically actuated ink-jet dispensers.[7]

The main advantages include: 1) very reproducible drops and thus very reproducible explosive amounts; 2) almost continuous variation through the accretion of droplets; 3) large dynamic range adjusted by the amount of explosive deposited on the heater; 4) digital control.[2]

Several prototypes using this technology were built and evaluated. It was demonstrated that the technology could be used for several explosives including RDX, TNT and PETN. The range provided by a vapor generator employing ink-jet microdispensers was evaluated and shown that the concentration can be varied almost continuously from 0 to hundredths of parts per trillion (v/v) when operating the dispenser in continuous mode.[5,8]

2. INK-JET MICRODISPENSING

The ink-jet dispenser that is employed in the vapor generator is drop on demand (DOD) meaning that it can produce a drop only when required. The actuation of the dispenser is done with a piezoelectric element. The dispenser consists of a glass tube having an orifice at one end and being connected by the means of Teflon™ tubing to the reservoir containing the solution to be dispensed. An annular piezoelectric element poled in the radial direction is bonded to the glass using a thin epoxy layer. When voltage is applied to the piezoelectric actuator, the actuator contracts or expands (depending on the direction of the electric field produced in the piezoelectric element relative to the poling direction) and this deformation is transmitted to the glass and then to the fluid. Because the structural response is very fast and the solution to be dispensed is in contact with the glass, the motion of the structure translates in a volumetric change in the solution. The change produces a localized pressure variation that travels as acoustic waves in the solution contained by the glass tube.

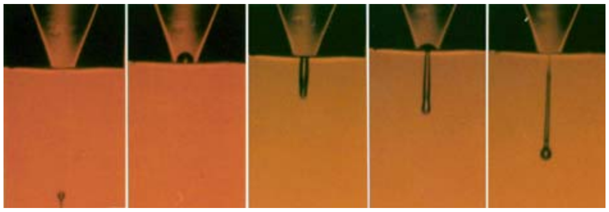

Figure 1 – Sequence of generation of 50μm droplets (ethylene glycol) at 4kHz

Figure 1 presents the sequence of generation of droplets using a piezoelectric ink-jet microdispenser. The visualization of the droplets is done using a CCD and stroboscopic illumination using a LED that is synchronized with the drop generation. By adjusting the time between the drop generator and the time the LED comes on, the droplet is captured at various locations along its trajectory.

For proper operation of a DOD ink-jet microdispenser, the fluid has to be flush with the orifice face (see leftmost picture in Figure 1). This condition is achieved by adjusting the backpressure in the fluid reservoir.

3. VAPOR GENERATORS

Vapor Generation

The first instrument developed was designed for the determination of the threshold (sensitivity) of the human nose. Odorant droplets are generated and deposited onto a heater where they are evaporated. The vapors are carried by a flow and presented to the patient nose. The threshold level determined using with the ink-jet based olfactometer can be used for the early diagnostics of neurodegenerative diseases.

Previous Explosive Vapor Generators

The explosive vapor generators use a similar principle of operation as the olfactometer with the difference being that the dispensed solution is not an odorant but a dilute solution of explosives. The droplets of explosive solutions are generated using a piezoelectric ink-jet microdispenser and deposited onto a heater where they are evaporated and the vapors are carried towards the detector / sensor to be tested (Figure 2).

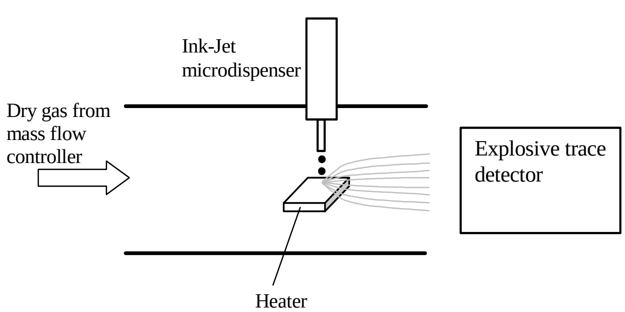

Figure 2 – Basic components of the vapor generator

Explosive vapor generators were built using the described principle including a first prototype built for NIST incorporating six different ink-jet dispensers for six different explosive solutions; a single solution system that incorporated a cooled reservoir and control of the temperature of the heaters; and a prototype of a portable system.[2]

4. PORTABLE VAPOR GENERATOR

Main Components

Figure 3 shows the diagram of the vapor generator and its main components. From a functional perspective there are two main modules: one for the printhead – dispenser and one that provides the operational support.

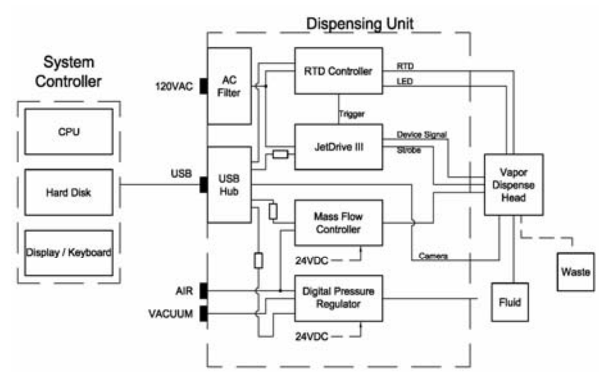

Figure 3 – Diagram of the vapor generator indicating the main components

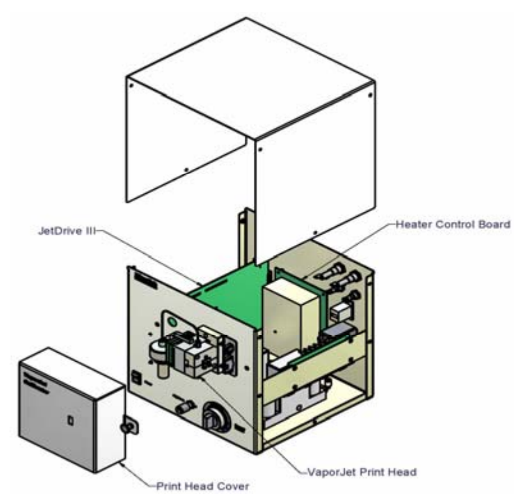

Figure 4 – Portable vapor generator and its main components

The ink-jet microdispenser, fluid reservoir, heater and observation camera are assembled in the printhead module. This module is mounted on the front of a support module.

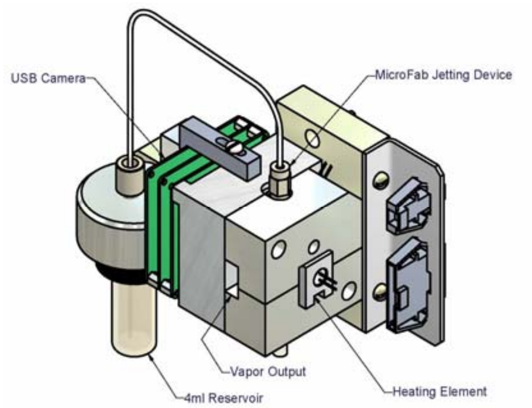

Figure 5 – Printhead module and its main components

The support module contains the drive electronics that generate the signal applied to the piezoelectric actuator, the mass flow regulator, the pressure/vacuum regulator and the board containing the heater temperature control.

Droplet Visualization

Droplet visualization is accomplished using a CCD camera that allows the observation of the generated drops by illumination with an LED that is turned on synchronized with the pulse sent to the piezoelectric actuator. The CCD camera is board based with a wide field of view that allows the visualization of the drops in flight and also when they land on the heater surface (Figure 7). The visualization of the drops on the heater confirms when the solvent is evaporated.

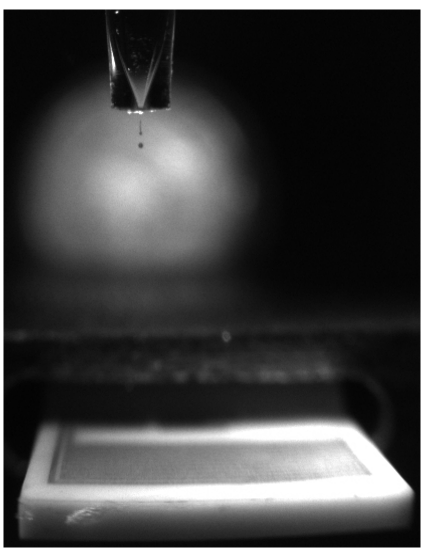

Figure 7 – Image captured with the CCD camera showing the drop generation under stroboscopic illumination. The generated droplets land on the heater element.

Carrier Flow

Dry air or nitrogen is input into the system through a mass flow regulator. The flow is used to carry the explosive vapors generated on the heater towards the explosive vapor detector (Figure 4). To produce a uniform flow, the carrier gas is introduced in the dispensing module through a frit/diffuser. The system is setup such that mass flow controllers with different ranges can be employed.

Heating Element

The heater consists of a 100 Ohms RTD flat element with very small thermal mass to permit rapid increases of the temperature. The RTD's platinum element is fabricated by screen printing and laser trimming. The element is covered by a thin layer of glass.

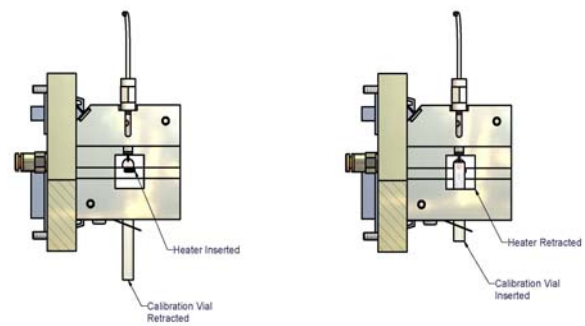

Figure 6 – Dispenser head shown: left - with the heater in normal operation position; right – with the heater retracted and the collection vial pushed in.

In the printhead module (Figure 5), the heater is mounted on a slide. During normal operation the slide is pushed in such that the heater is under the microdispensing device. During jetting set-up or when collecting explosive solution for analysis, the slide is moved out and a collection vial is pushed up from the bottom.

5. SYSTEM CONTROL

A GUI software incorporates all controls for the vapor generator. The software resides on an external laptop connected to the control module. Communication is done through USB ports.

Backpressure

The control of the backpressure balances the capillary and hydrostatic forces on the fluid at the orifice level. If the forces are not balanced, the fluid will either drip or be pulled back inside the glass tube. Either of these conditions prevents the generation of droplets on demand.

The backpressure adjustment and control is done using a computer controlled pressure regulator with a high repeatability (0.58 mm water column) and resolution (0.7 mm water column). The pressure is set in the main control program.

Electrical Waveform for the Piezoelectric Actuator

An ink-jet microdispenser is actuated using a trapezoidal waveform (Figure 8). During “rise time” the inner surface of the glass tube moves outward and a negative pressure wave is generated and starts to move both to the supply and orifice end. At the supply end the wave reflects as a positive pressure wave. The “dwell time” is selected such that the “fall” of the drive signal starts when the reflected positive pressure wave reaches the middle of the channel. The voltage “fall” corresponds to a compression of the fluid (inward motion) and thus reinforces the reflected wave for a minimization of the required voltage or maximization of the drop velocity at the same applied voltage. The “echo time” (time during which the signal is at the voltage minimum value) is chosen to cancel the residual pressure waves traveling in the channel after drop generation.

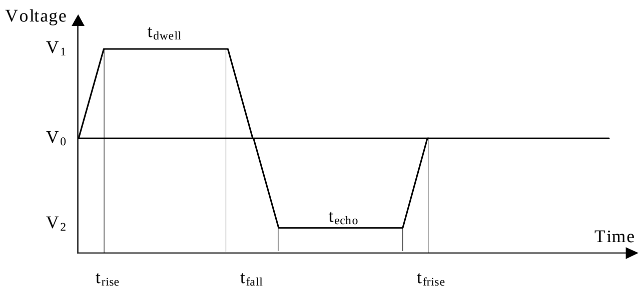

Figure 8 – Waveform applied to the piezoelectric actuator

The purpose of the “echo” part of the waveform is to eliminate satellites (smaller droplets trailing the main drop). Depending on the properties of the dispensed solution, the actuating signal can be simplified to the positive part only (no “echo”).

A drive electronics board is incorporated in the support module. This board is capable of generating the described signals and more complex ones defined by arbitrary points.

Mass flow regulator

The flow is required to carry the vapors to the output and it needs to be controlled for correlation with the explosive vapor trace detector that is tested. A computer controlled mass flow regulator is incorporated in the control module. The vapor trace detectors that are currently on the market have a wide range of flow for the sample intake. To accommodate this, the vapor generator comes with two options: low flow (0-50 cc per minute – accuracy of 0.5 cc per minute) or large flow option (up to 5 liters per minute – accuracy of 0.05 liters per minute). The flow is set and controlled from the main software.

Heater temperature control

It is possible to have the temperature of the heater follow a desired profile. The temperature control is done by applying voltage pulses to the RTD that is employed as a heater. Between these pulses the RTD is switched to a circuit that measures its resistance to determine its temperature. A PID controller algorithm is used to determine the length of the next power pulse. The measurement and the heating circuits are implemented in a single board that is incorporated into the support module. Because the applied voltage is fixed, the software provides for different values for the gains for different values of the temperature.

This approach can generate fast response for temperature increases, but also has the ability to control the temperature such that it follows specified profiles that can be correlated with the dispensing events. The profiles are specified as a series of ramps followed by a constant temperature segment. Each pair is defined by the ramp duration, constant temperature value and time for which the temperature is constant. A total of four such segments can be defined.

Software and Data Recording

A GUI interface is implemented to set-up the inkjet dispenser and the test for generation of vapors by choosing the operational mode (continuous or dose); setting the number of droplets and the generation frequency to be deposited on the heater; the heater temperature profile; the mass flow rate.

Data relevant to the ink-jet dispensing and the tests is recorded into a file.

Communication with Other Equipment

To facilitate integration with the detectors to be tested, the vapor generator also provides a trigger output. The software allows specifying two trigger pulses at any time during a heating-dispensing cycle in dose mode operation.

6. FABRICATION AND OPERATION

Fabrication

The prototype of the portable explosive vapor generator was built incorporating all the functions described above (Figure 9).

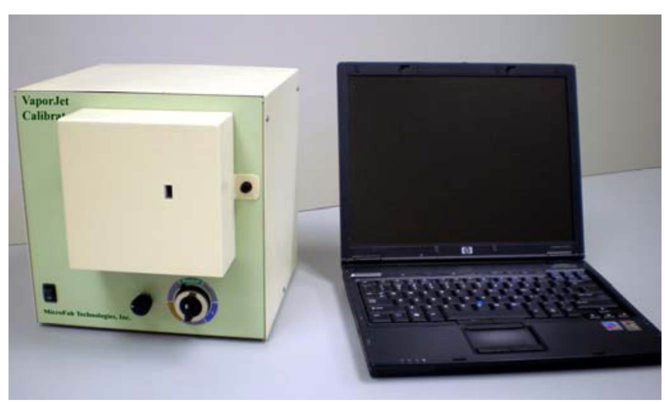

Figure 9 – Vapor Generator and control laptop

Figure 10 – Support module (box) and the dispensing module (front of the box)

Operation

The vapor generator can operate in two modes: a continuous mode in which the droplets are generated continuously at a selectable fixed frequency and a dose mode which consists of the generation of a specified number of drops at a selected frequency.

Continuous operation—In continuous operation, the droplets landing on the heater (maintained at a set and constant temperature) are evaporated continuously and the output is constant in time. By adjusting the flowrate of the carrier gas and/or the frequency at which the droplet are generated, the level of the output can be set to different values. The droplets can be generated

Dose mode—This function limits the amount of explosives that is output from the vapor generator and might be desired in the case of detectors that are sensitive to the solvent that is used to dissolve the explosives. In this case, the heater is set initially at a relatively base temperature that ensures the instrument is independent of the room temperature. The next temperature level is also a low temperature; this temperature is where the evaporation of the solvent occurs. Once the solvent is driven away, the detector is exposed to the gas stream coming out of the vapor generator while the heater temperature is increased to values that drive off the explosive. A final heating at high temperature ensures that all the residuals on the heater are burned off. The values for the various temperature levels are determined by the type of explosive used in the test and the solvent used to dissolve the explosive.

The dose is defined by the volume of the fluid dispensed on the heater. Another parameter

7. Test Results

Temperature Evaluation

The temperature profile for the heater was evaluated by measuring the temperature with a thermocouple in contact with the top surface of the RTD / heater. Figure 11 presents the temperature on the top surface of the RTD heater as measured with a thermocouple. The results indicate that the temperature follows very closely the specified profile. The flat areas in the temperature curve are an artifact of the data acquisition board.

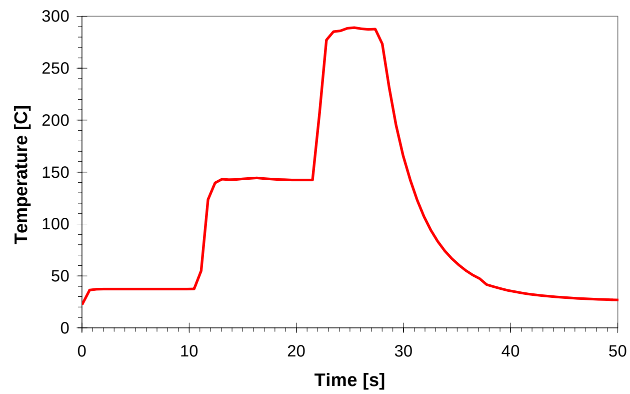

Figure 11 – Temperature profile measured using a thermocouple in contact with the top surface of the heater. Settings: ramp 1 – 1 second; temperature 1 – 40ºC for 10 seconds; ramp 2 – 1 second; temperature 2 – 150ºC for 10 seconds; ramp 3 – 1 second; temperature 3 – 300ºC for 5 seconds

An important observation is the fact that, due to the small thermal mass of the heater, the cooling off period is also very short and thus allows tests to be run at very close intervals.

Evaluation of the Dispensing

The analysis determined the volume of solution dispensed by the piezoelectric ink-jet dispenser. This combined with the analysis of the solution just after preparation has indicated that there are no explosive losses in the reservoir/cartridge and the tubing.

The energetic materials that are used in the vapor generator are known to be thermally labile and subject to decomposition at elevated temperatures. Compounds to be vaporized include nitroaromatics, nitroesters, nitramines, and others. In order to have a trace explosive detector vapor calibrator, the concentration of an explosive vapor being emitted per unit time must be a known value for each compound to be used. The unstable nature of energetic compounds needs consideration.

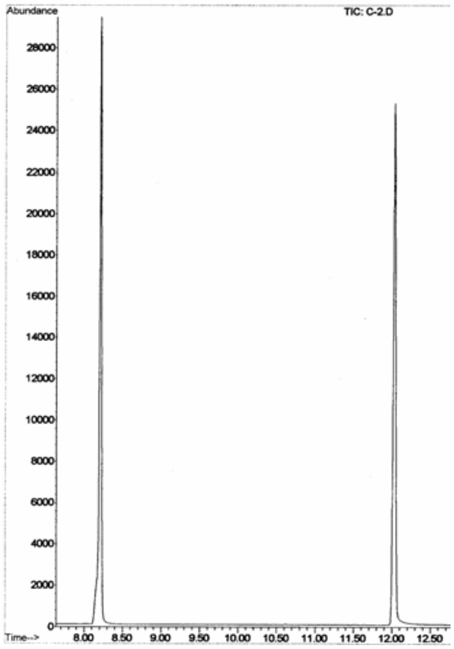

Figure 12 – The spectrum of the solution standard (before jetting)

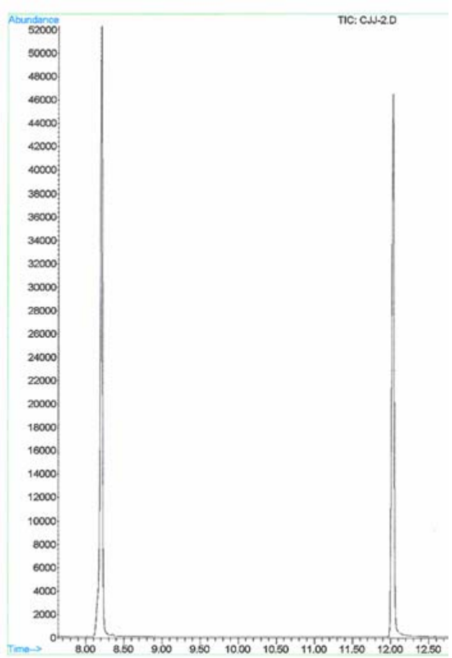

Figure 13 – The spectrum of the solution standard (after jetting)

Precision jet dispensing of solutions of the important explosives and their subsequent vaporization is a new approach to calibration of trace detectors. Jetting is a very low energy process, but the effect of its action on energetic materials can be questioned. The initial step in developing jettable explosive solutions is to make certain that jetting is not causing decomposition. Also, we want to make sure that the starting and jetted compositions are the same, i.e. no losses in the system.

The initial experiment was the jetting of a solution of the energetic compound 2,6-Dinitrotoluene (DNT) in 2-methyl-1-propanol using the same jetting device as the vapor generator. The solution contained 54 micrograms/mL of the DNT and 22 micrograms/mL of 1,2,3,4-Tetrahydronaphthalene (THN), which was used as a stable internal standard. As the jetted drop is approximately 50 picoliters in volume, which would contain 54 femtograms of DNT, a million drops were collected for a sample. Solutions were analyzed using a Hewlett-Packard 5890 series II GC with a VB-5 30m capillary column, a 5976 MSD, EI, and in the single ion mode with 104 and 165 m/z primary ions for THN and DNT respectively. Ion chromatograms for the initial and jetted solutions are shown below. THN retention is 8.2 minutes and DNT 12.0. No differences between the spectra for solution after jetting (Figure 12) and the solution standard before jetting (Figure 13) were observed, indicating that no changes occur.

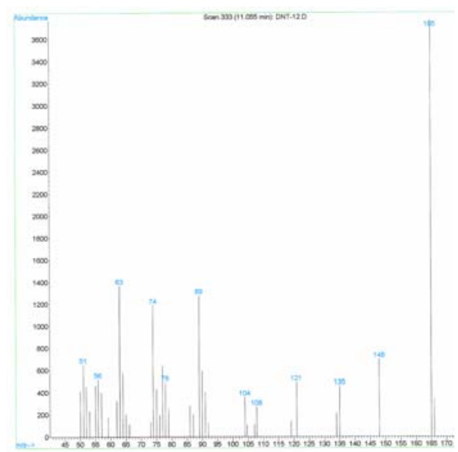

Figure 14 – Spectrum of DNT

DNT was chosen for the initial test. It proved difficult to analyze and very sensitive to technique due to decomposition at elevated temperatures. The DNT/2-methyl-1-propanol stock solution was run 48 times with the area percentage for the 165 m/z ion being measured against the area for the 104 m/z ion (Figure 13). This data was then calculated as a Ratio % of DNT to THN, which was always 100. Unless a very large concentration of DNT was injected, no trace of its molecular ion (182 m/z) could be seen. The Ratio % for the Standard was 95.2 ± 1.0, and for the jetted Sample 94.9 ± 1.0. However, the Relative Standard Deviation, %RSD, was no better than 1.07, which required a significant amount of technique to achieve. Improvements planned for the GC are a different column and NICI for the MSD.

The conclusions are that jetting does not damage the DNT and the repeatability of the jetting is good.

Evaluation of Function of the Reservoir and Dispenser

To facilitate the transfer from the solutions prepared in the lab to the vapor generator we have selected the reservoir/cartridge to be an amber glass vial of 4mL in volume. The solutions can be prepared in the bottles and the loading on the system only consists in removing the cap of the new solution bottle and screwing it in. The function of the reservoir – dispenser combination has been verified in the vapor generator.

The repeatability was verified by weighing the output from the device finding a standard deviation of less than 1.7%. The output from various batches of solutions/bottles has been tested by GCMS as described above.

Evaluation with Explosive Detectors

Preliminary evaluation was conducted using RDX and TNT solutions in dose mode. a TNT standard (in acetonitrile) as 1:99 (ethanol, isobutanol) and TNT standard (in acetonitrile) as 1% standard and 99% solvent (IPA, ethanol, isobutanol) and RDX standard 10% standard and 90% isobutanol. This lead to final concentrations of 1µg TNT per 1mL and 10µg RDX per 1mL solution. Based on the concentration of the explosive in the solution and the drop volume the amount of explosive dispensed is 6.5 picograms of TNT and 65 picograms of RDX per 100 drops.

The tests consisted in:

1. deposit the desired number of droplets (from 25, 100 or 250 – all give good signals for TNT; at least 250 drops for RDX) onto the heater at a frequency of 240Hz.
2. wait for 10 seconds (this is the period during which the solvent is evaporating)
3. move the detector in the output path of the vapor generator
4. turn on the heater with a very short ramp time (1.0 seconds) to a temperature of 150ºC for TNT and 200ºC (a higher temperature was necessary to vaporize RDX) for RDX.
5. remove the detector from the flow path
6. turn on the heater at high temperature (300ºC) for 30 seconds to burn off the residue.

The results indicated good reproducibility based on the measurement value reported by the detector and ability to control the output even at very low levels.

8. Conclusions And Future Directions

The preliminary testing of the vapor generator with the explosive trace detectors has indicated that it is possible to use the portable prototype to evaluate the vapor trace detectors. The ability to control the temperature levels permits the use of the vapor generator for a variety of explosives. By controlling the amount of explosive solution dispensed onto the heater it is possible to change the output accurately even at low levels.

More extensive evaluations with explosive vapor trace detectors will be performed including operation in continuous mode and the use of solutions prepared with different explosives.

Acknowledgement

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