Explosive Vapor Generator

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Table of content

List of Figures

Introduction

The need to detect very low levels of illicit substances (chemical and biological agents and explosives) has become, after September 11, 2001, a priority for the federal, state and local government agencies. Systems capable of detecting minute amounts of the above materials are required in the airports, border crossings and high security areas. Explosives represent one important class of illicit substances with the military explosives (e.g. TNT, RDX, PETN, HMX) being an important subclass that is currently targeted by the various trace detection methods. Trace detection – detection of very small amounts of the explosives – identifies people or things that have come in contact with explosives. The trace detection methods have been implemented in a variety of instruments ranging from hand held and portable to benchtop or portals.[1,2]

A vapor generator capable of producing vapors of the substances of interest in a wide range of precisely controlled concentrations has multiple uses in the area of trace detectors. The first and the most important application is the test and calibration of the trace detectors. To make sure that the sensitivity of a system is still acceptable, periodic evaluation and, if necessary, recalibration are required. By creating explosive vapors of known concentration, the vapor generator provides the means to verify the detection limit of the systems in the field and their recalibration.

The continuous research and development for the improvement of the detection limit requires a generator of very low concentration explosive vapors. It is desired that such a vapor source is portable, because a large number of the vapor trace detectors deployed in the field are fixed. Existing technologies[3,4] are not very precise and cannot be easily miniaturized thus the ink-jet based explosive vapor generator provides a much needed tool in the test and evaluation of explosive vapor trace detectors. Moreover, because the various levels within the range can be precisely controlled, the vapor generator based on ink-jet microdispensing is capable of quantifying the iterations in the development of new detection methods or improvement of existing ones.

Principle of operation of the vapor generator

For this application droplets of diluted explosive solutions are generated using a piezoelectric ink-jet microdispenser and deposited onto a heater. The droplets landing on the heater are evaporated and the vapors are carried towards the detector / sensor to be tested (Figure 1).

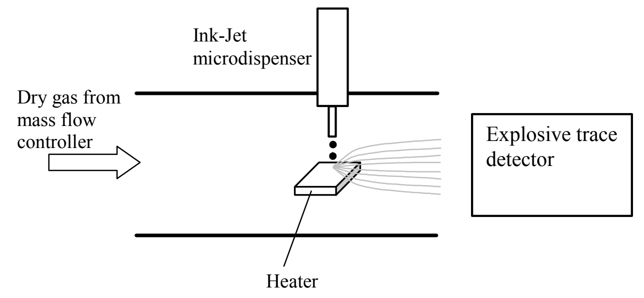

Figure 1. Principle of operation of a ink-jet based explosive vapor generator.

Ink-jet microdispensing

In the case of the drop on demand (DOD) ink-jet dispenser employed in this system, a volumetric change in the fluid is induced by the displacement of a piezoelectric material that is coupled to the fluid[5] This volumetric change causes pressure/velocity transients to occur in the fluid and these are directed to produce a drop that issues from an orifice.[6,7] Demand mode ink-jet printing systems produce a droplet only when desired. Figure 2 illustrates a piezoelectric based DOD system, in which a droplet is generated only when a voltage pulse is applied to the piezoelectric actuator. In traditional printing systems the drops are targeted to specific locations on the substrate/paper. For the vapor calibrator, the droplets land on the heating element.

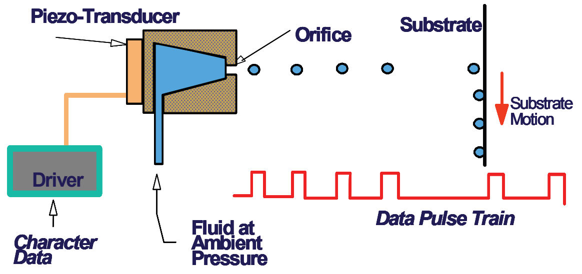

Figure 2. Piezoelectric DOD ink-jet printing system.

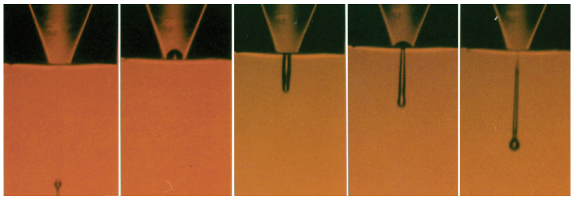

Figure 3. Sequence of generation of 50μm droplets (ethylene glycol) at 4kHz.

Figure 3 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 3). This condition is achieved by adjusting the backpressure in the fluid reservoir.

Advantages of the ink-jet based vapor generator

1. High precision: Ink-jetting produces highly repeatable drops that can create larger volume by accretion.
2. Continuous variation: The very small size of the individual drops (20-200picoliters) produces, from the perspective of this application, almost continuous variation of the total (accumulated) amount.
3. Range of concentration: The dynamic range of a vapor generator based on ink-jet microdispensers extends from almost zero (equivalent of several drops) to several thousands of parts per trillion. The low end resolution can be further increased by using more dilute solutions containing the substances of interest. Because of the digital nature of the vapor generation the explosive output level can be changed from one value to another almost instantaneously. This is a significant improvement over vapor generators that are based on the equilibration of releases from explosives in solution or solid form.
4. Data driven: The piezoelectric dispensers are electrically driven and they can be controlled from data files. This makes the ink-jet vapor generator easily adaptable for automatic testing and possibly for automated calibration.
5. Multiple solutions/explosives: An ink-jet vapor generator can be easily adaptable for multiple solutions. The cartridges containing the solutions can be all loaded in the system and the operator (or the automatic calibration program) can select between them.
6. Size: The proposed vapor generator can be developed in a portable format that is required by the fixed systems in the field. The system can be further miniaturized and possibly made as a modular component for incorporation in the vapor trace detectors.

Construction of vapor generator

Diagram

Figure 4 shows the diagram of the vapor generator and its main components.

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

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.

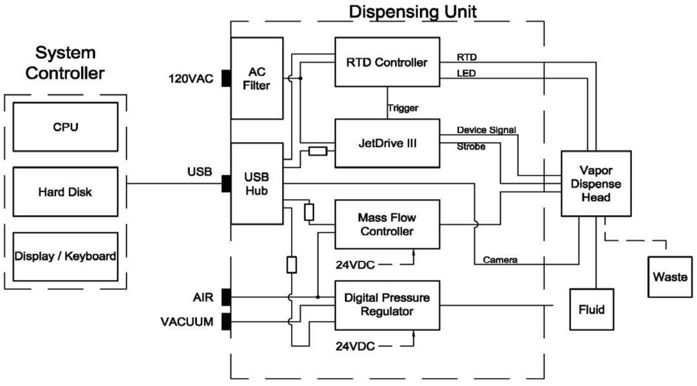

Figure 4. Diagram of the vapor generator

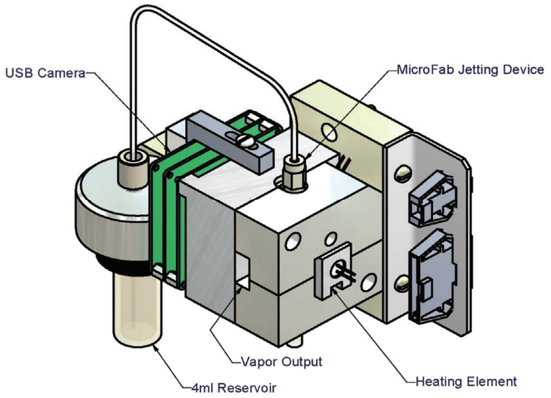

Figure 5. VaporJet printhead and its main components.

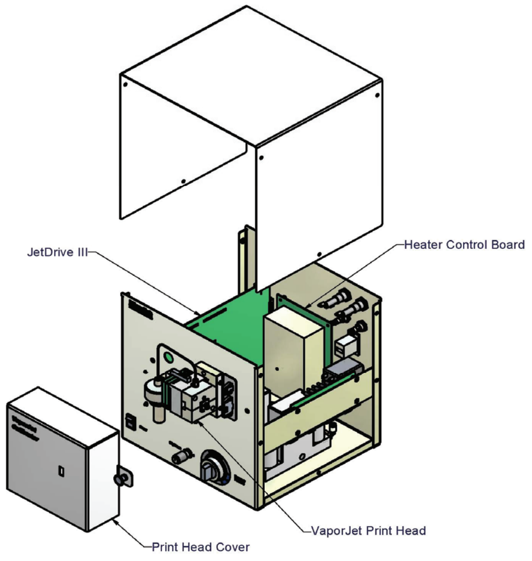

Figure 6. VaporJet – main components and assembly.

Droplet visualization

Droplet visualization is achieved 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 8). The visualization of the drops on the heater helps identify when the solvent is evaporated and carried away.

Figure 7. VaporJet with the printhead cover removed.

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 1). To produce a uniform flow, the carrier gas is introduced in the dispensing module through a frit/diffuser. The system is set-up such that mass flow controllers with different ranges can be employed.

Heater

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.

In the printhead module (Figure 5), the heater is mounted on a slide. The slide is pushed in such that the heater is under the microdispensing device during normal operation. 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.

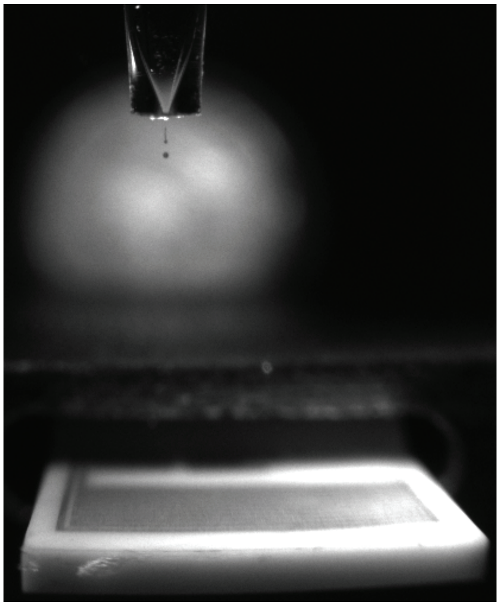

Figure 8. Image captured with the CCD camera showing the drop generation under stroboscopic illumination. The generated droplets are landing on the heater element.

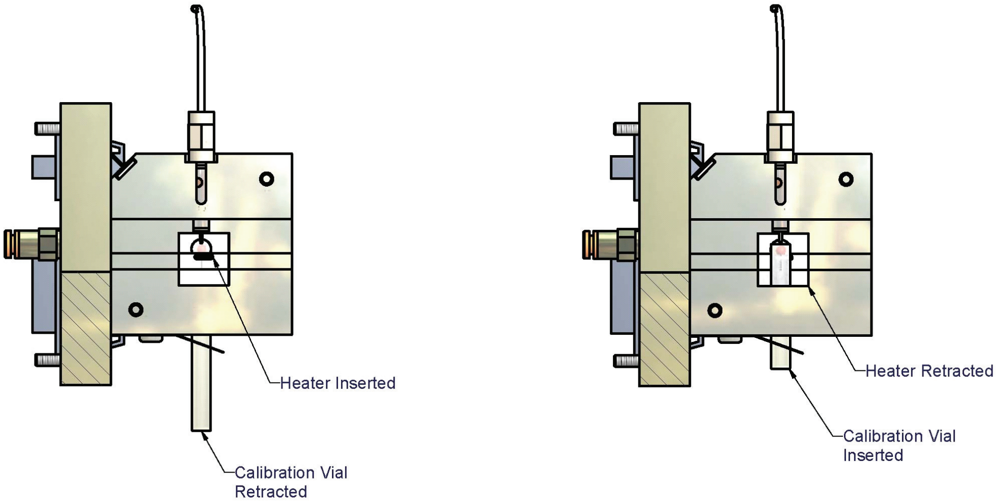

Figure 9. Dispenser head shown: left - with the heater in normal operation position; right – with the heater retracted and the collection vial pushed in.

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 (the fluid level in the reservoir is higher) 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.

Waveform to the piezoelectric actuator

A microdispensing device is actuated using a trapezoidal waveform (Figure 10). 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.

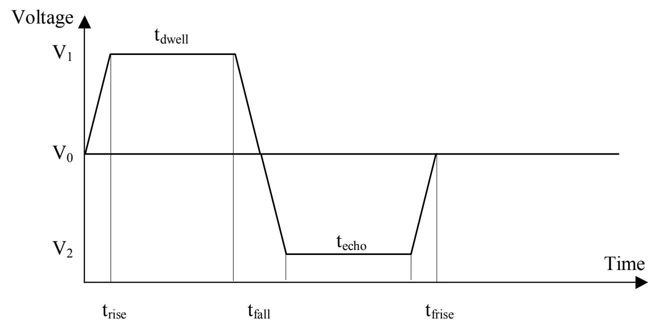

Figure 10. Waveform applied to the piezoelectric actuator.

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 reduced to the positive part only (no "echo").

A drive electronics board is incorporated in the control 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

The heater board controls the temperature of the heater such that it follows 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.

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 settings were: 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. The results indicate that the temperature follows very closely the specified profile. 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. The energy delivered to the heater is large enough to prevent the heater cool-off by the evaporating liquid.

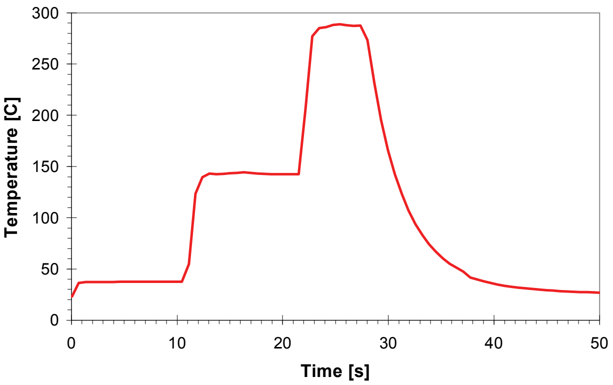

Figure 11. Temperature profile measured using a thermocouple in contact with the top surface of the heater.

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 including operator introduced information.

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.

Operating modes

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.

Dose operation

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 can be set initially at a relatively low temperature that ensures the evaporation of the solvent. 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.

Continuous operation

In continuous operation, the droplets landing on the heater are evaporated continuously and the output is constant in time. By adjusting the flow rate 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.

Output range

The VaporJet system is a fixed volume (dose mode) or fixed volumetric flow rate (continuous mode) system. In addition to the volume or flow rate of the solution containing the explosives that it dispensed onto the heater, the concentration of explosive vapor at the output depends on the following:

1. concentration of the explosive in the initial solution. A doubling of the concentration of the explosive in the solution will result in a doubling of the concentration of the vapor at the output.
2. flow rate of the carrier gas. Halving the flow rate of the carrier gas will result in doubling the concentration of the vapor at the output.

A wide range of output levels can be achieved with some limitations introduced by: a) the solubility limit of the explosive in the selected solvent, b) maximum dispense frequency of the explosive solution, c) maximum heater temperature (Leidenfrost effect) – also a solvent property, d) maximum explosive solution flow rate (introduced by the power available to the heater) and e) the maximum evaporation rate for the explosive (thermodynamic requirements for the vaporization of the explosive)[8].

The influence of the above factors depends on the mode of operation so the discussion is continued for each mode of operation.

Dose mode

The only limitation in dose operation is the concentration of explosive in the solution to be dispensed. Typical explosive standards are made by dissolving the explosive in acetonitrile. Because of its low boiling point, acetonitrile does not have very good jetting characteristics. Solvents that are better behaved from a jetting perspective are isopropyl alcohol, isubutanol, butanol and ethanol. The explosive solution can be prepared by dissolving the solid explosive in the chosen solvent or by combining a small amount of acetonitrile based standard with the solvent. Solutions with concentration of 10µg/mL can be prepared for RDX, TNT and PETN. The solubility limit is most likely higher than that.

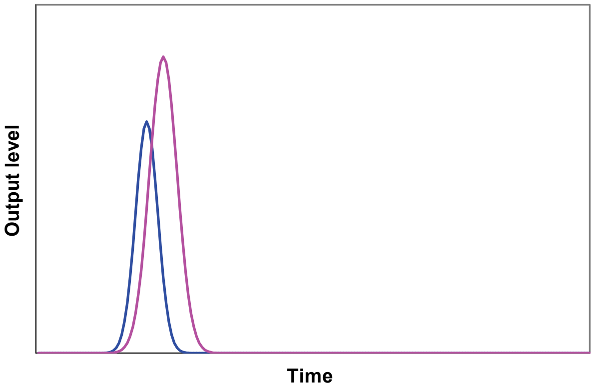

Figure 12. Output evolution in dose mode for two different doses. Blue curve is for a lower dose than magenta curve.

When the temperature of the heater is increased to levels that evaporate the explosive, the explosive vapors will become part of the output. Their concentration increases, reaches a maximum value and then decreases. The concentration of the explosive vapors has a time evolution as shown in Figure 12. The width of the peak at the base and the height of the peak will depend on: the type of explosive used (for the same dose, RDX will have a wider and shorter peak), temperature of the heater during explosive vapor release (higher temperature narrower and taller peak), amount of explosive – at the same solution concentration – in the dose (more explosive solution will result in a taller). Figure 14 shows that the response (peak) of the explosive trace detector follows an almost linear behavior indicating that the peak height is proportional to the amount of explosive with insignificant peak widening.

The area under each peak is proportional with the amount of explosive that is deposited on the heater and typical peaks, as measured with the explosive trace detectors, are about one second wide. The following calculation is done for a 10µg/mL solution of TNT in isobutanol. When using a 50µm orifice dispenser, one thousand droplets deposited on the heater contain 650 picograms of TNT, which, assuming the peak to be a triangle leads to a peak of 1300 picograms/second. For a carrier flow of 10 cc/min (0.000167 L/s) the maximum output concentration is 7.8µg explosive per liter of carrier gas. For the given concentration of the explosive solution (10µg/mL) and carrier gas flow rate (10 cc/min) the output can be varied almost continuously by changing the number of drops from 1000 down to one.

Figure 13 summarizes the output ranges when two different explosive solution concentrations and three carrier gas flow rates are employed. The values used in the figure are within the actual values used initially, but the range can be further extended by increasing/decreasing the explosive solution concentration and/or increasing/decreasing the flow rate. By using the large mass flow controller option (up to 5 L/min) the concentration range can reduced by a factor of up to 1000.

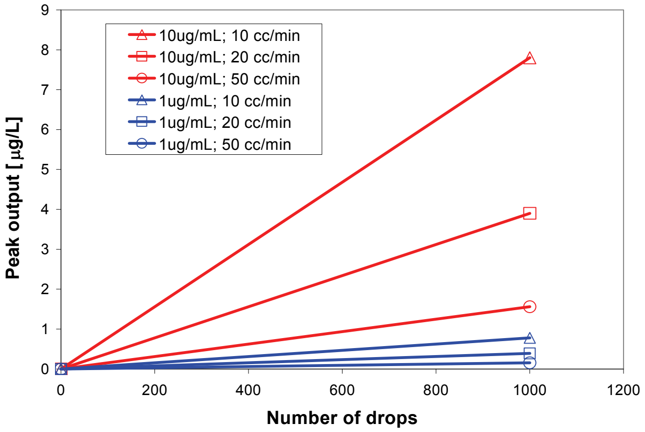

Figure 13. Output values for different number of drops for two explosive solution concentrations and three different carrier gas flow rates.

Continuous mode

In continuous mode the output is constant in time and is determined by the combination of explosive solution concentration, volumetric flow of the explosive solution (equal to the product between the drop volume and the drop frequency) and the volumetric flow rate of the carrier gas. The possible limits discussed earlier are detailed next in the context of continuous mode of operation. Note that these limits are reflected more in the way the vapor generator is operating with a wide range of output levels. Moreover some of them are outside the range of interest or can be eliminated by adjusting other operational parameters.

Solubility limit

The discussion at dose mode still applies. 10µg/mL is a concentration that can be reached for all explosives.

Dispense frequency of the explosive solution

The maximum frequency is mostly a function of the solvent that is used to dissolve the explosive. As the frequency increases wetting effects could result in fluid accumulation at the orifice. For the solvents mentioned earlier the maximum frequency will be at least in the kilohertz range.

The reduction in the output level towards small values cannot be fully accomplished by the reduction in frequency. It is expected that at a certain frequency value (probably tens – hundred of hertz) the output will have a wavy appearance. The output can be further reduced by increasing the carrier flow rate and/or by decreasing the explosive concentration in the solution.

The ability to adjust (increase or decrease) the concentration of the explosive in the solution and the carrier gas flow rate can move the required (by the output level) dispense frequency within the acceptable range for the solvent employed.

Maximum heater temperature

When droplets land on a heated surface there is a temperature (Leidenfrost) where the heat transfer between the droplets and the surface is minimum which translates into a maximum in the evaporation time.[9] The decrease in heat transfer is produced by a vapor film that forms between the drop and the substrate. That vapor film can cause the droplets to slide or bounce from the substrate so, in continuous mode, the heater temperature has an upper bound. The maximum temperature that does not result in the drop bouncing off the heater will be solvent dependent and in the range of 100-150ºC. Typically the temperature limit will be higher than the boiling point of the fluid by 20-30ºC. This is not a true limitation, but needs to be considered during operation.

Maximum explosive solution flow rate

While converting the explosive into vapor the heater also vaporizes the solvent used to dissolve the explosive. If the energy/power required to evaporate the solvent exceeds the heater power it could result in heater cool-off. Considering the generation of drop of 50 picoliters at 10,000 Hz, an estimate for the required power of vaporization is under 0.5W. The power available is from 48V on a 100ohms resistance at 80% which is of the order of 20W. The maximum explosive flow will not be a limitation for the vapor jet.

Maximum evaporation rate for the explosive

This was estimated based on the thermodynamic principles of mass diffusion across the interface.[8] It was determined that, for a heater temperature of 130ºC, the maximum mass diffusion for the explosives dissolved in isobutanol was 2.5 ng/s (RDX), 42 ng/s (PETN) and 550 ng/s (TNT) which are typically higher than the amount of explosive deposited on the heater. In that work the concentration at the outlet was 60 ng/L (PETN), 250 ng/L (PETN) and 250 ng/L (TNT) when using 1 L/min carrier flow rate. The output can be adjusted up by a factor of more than 100 by reducing the carrier flow to 10cc/min. Additional increase can be achieved by increasing the solution concentration. If interested in a smaller concentration range the explosive concentration can be reduced and/or the drop generation frequency can be reduced and/or the carrier flow rate can be increased (by up to a factor of 5).

Preliminary results

NIST has used a first prototype fabricated by MicroFab to evaluate the potential range provided by a vapor generator operated in continuous mode for several explosives (RDX, TNT and PETN) and has shown that the concentration can be varied almost continuously from zero to hundredths of parts per trillion (v/v).[8,10] Experiments have also shown the ability to step up the output by stepping up the drop generation frequency by almost three orders of magnitude; the changeover from one level to the next is done in couple of seconds.[11]

Preliminary testing was done in dose mode with some results included in Figure 14.

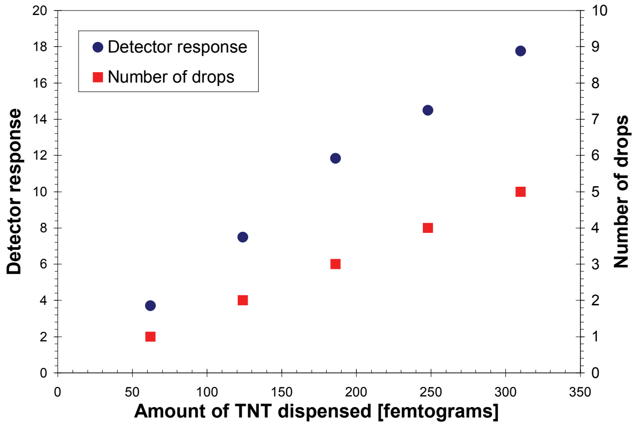

Figure 14. Response of a vapor trace detector to the output of the vapor generator operating in dose mode.

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