

Miniaturized Explosives Preconcentrator for Use in a Man-Portable Field Detection System

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Abstract

We discuss the design and testing of a miniaturized explosives preconcentrator that can be used to enhance the capabilities of man-portable field detection systems, such as those based on ion mobility spectrometry (IMS). The preconcentrator is a smaller version of a similar device that was developed recently at Sandia National Laboratories for use in a trace detection portal that screens personnel for explosives. Like its predecessor, this preconcentrator is basically a filtering device that allows a small amount of explosive residue in a large incoming airflow to be concentrated into a much smaller air volume via adsorption and desorption, prior to delivery into a chemical detector. We discuss laboratory testing of this preconcentrator interfaced to a commercially available IMS-based detection system, with emphasis on the explosives 2,4,6-trinitrotoluene (TNT) and cyclotrimethylenetrinitramine (RDX). The issues investigated include optimization of the preconcentrator volume and inlet air flow, the use of different types of adsorbing surfaces within the preconcentrator, and preconcentrator efficiency and concentration factor. We discuss potential field applications of the preconcentrator, as well as avenues for further investigations and improvements.

Introduction

Trace chemical detection of explosives – the art of detecting explosive materials from minute quantities of vapor or microscopic particles – can be an important aspect of many physical security systems. Among the challenges currently confronting researchers in this area, the problem of how to collect the explosive sample and transport it to the detector without major losses, remains one of the most significant. In many applications, swipe collection of particles via direct physical contact with the person or object to be screened for explosives is considered either excessively invasive or too time consuming, so it is necessary to base the collection process solely on air flows. The vapor and/or airborne particle material that is collected in such air flows is usually far more dilute than is ideal for the detector to be utilized, and in many cases the air flow is too large to be directly accommodated by the detector. These disparities give rise to the need for devices referred to as preconcentrators. The role of a preconcentrator is to take a trace sample of an explosive, or other material to be studied, from a large incoming air flow, and concentrate the material into a much smaller volume before it is introduced into a trace detector. This paper discusses one such preconcentrator that has been developed recently at Sandia National Laboratories (SNL) with funding from the Department of Energy Office of Safeguards and Securities (OSS) and the Nuclear Emergency Search Team (NEST).

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The Sandia National Laboratories Preconcentrator

The preconcentrator considered in this paper is a miniaturized version of a preconcentrator developed earlier at SNL. That preconcentrator, intended for use in a trace detection portal for personnel screening, is protected under U.S. Patent # 5,854,431. The smaller version dealt with here is intended for field use in any type of man-portable explosives detection system, and it operates under similar principles. Figure 1(a) shows a schematic diagram of how the preconcentrator is interfaced to a trace chemical detector, and Figure 1(b) illustrates how it delivers a sample to the detector. The detector used with our preconcentrator is usually an ion mobility spectrometer or IMS, a technique that has been thoroughly reviewed elsewhere [1].

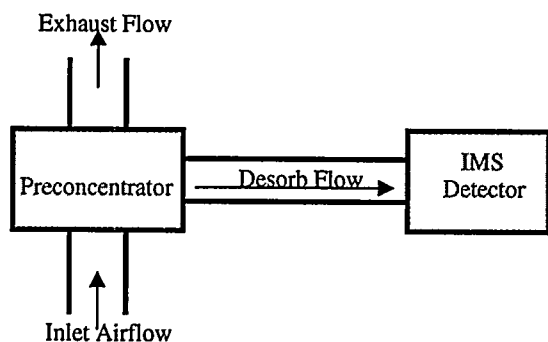


Figure 1(a)

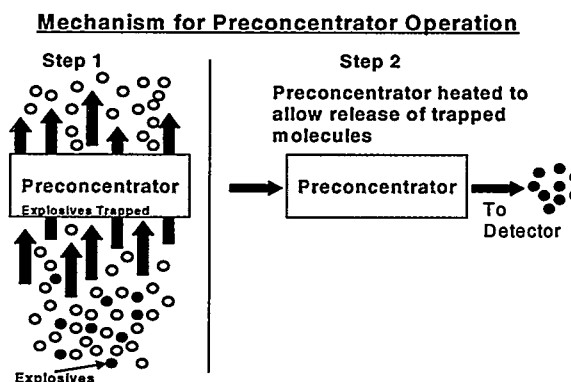


Figure 1(b)

The principle of preconcentrator operation is straightforward, with the device acting as a sort of molecular filter. A blower pulls air into the preconcentrator inlet. This inlet airflow is typically much larger than is suitable for the IMS detector being used. In the case of the preconcentrator used in the SNL personnel portal it is 160 liters/second, while in the miniaturized preconcentrator it can approach 2 liters/second. The inlet airflow is then pulled through a material known as metal felt, a high-density mesh of metal filaments. This material allows the air itself to pass through to an exhaust line, while trapping, via adsorption, high molecular weight organic molecules such as explosives. Collection of the latter molecules will occur with reasonable efficiency whether they are in vapor or particle form. Once this “adsorption cycle” is completed, typically after a few seconds of collection, valves are closed to isolate the preconcentrator from the ducts supplying both the inlet and exhaust airflows. At the same time, another valve opens in the duct between the preconcentrator and the detector, the metal felt is heated to 200 °C to desorb any collected explosive material back into the gas phase, and a much smaller air flow directs the desorbed explosive material into the detector. This “desorption cycle” also takes a few seconds, and the airflow is perpendicular to the inlet airflow. Thus the desorption airflow is parallel to the face of the metal felt, rather than perpendicular to it. Since the desorption airflow is very small compared to the inlet flow, the net result is to concentrate the trace explosive material into a much smaller air volume prior to its delivery to the detector. This concentration effect greatly increases the probability of detection with a concentration-sensitive detector such as an IMS.

The Miniaturized Preconcentrator

Figure 2(a) shows a photograph of the miniaturized preconcentrator, while Figure 2(b) shows a photograph of the miniaturized preconcentrator interfaced to a DC motor/impeller and a commercial, hand-held IMS detector. The detector shown is the Vapor TracerTM from Ion Track Instruments [2], and it was used in all of the experiments discussed in this paper. In Figure 2(a), the six inch long, one-inch diameter preconcentrator inlet tube can be seen protruding to the left. This long, narrow inlet tube allows sampling of material from recessed or otherwise hard to access surfaces. Also visible at the center of the circular main body of the preconcentrator is the metal felt that serves as the adsorbing material, which in this case has been folded into a pleated configuration to increase its surface area. In Figure 2(b), the motor/impeller is visible as the black circular object directly below the preconcentrator body. Power for the miniaturized preconcentrator is provided by a rechargeable, sealed lead acid battery rated at 12 volts and 7.2 ampere hours. The motor typically draws a current of 5 amperes during sampling, while during the desorption cycle the resistively heated metal screen can draw 20 amperes. Due to the small dimensions of the screen, the heating circuit only needs to be energized for 0.5-0.7 seconds per desorption cycle. The detector is powered by a separate battery pack attached to the detector case. The airflow through the preconcentrator can be varied from about 0.3 to well over 2 liters/second, while the air flow pulled in by the detector is typically 0.067 liters/second. A significant difference between this miniaturized preconcentrator and the larger preconcentrators that have been developed at SNL is that in the miniaturized unit the preconcentrator controls provide no airflow that pushes the desorbed contents of the preconcentrator towards the detector. Due to the small volume of the preconcentrator and its close proximity to the detector, the 4-liter/min inlet flow provided by the detector is sufficient to pull the desorbed material into the detector.

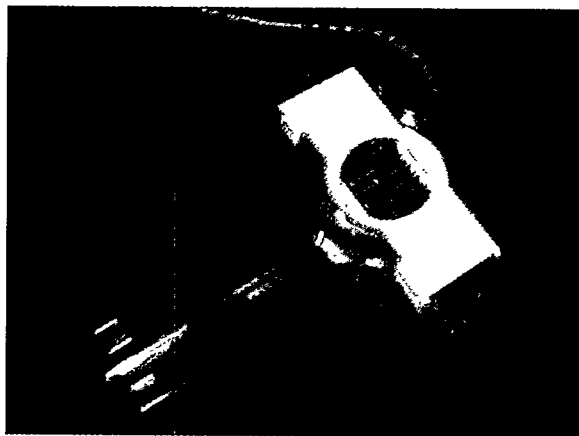


Figure 2(a): Inside the miniaturized preconcentrator, pleated adsorbing material exposed.

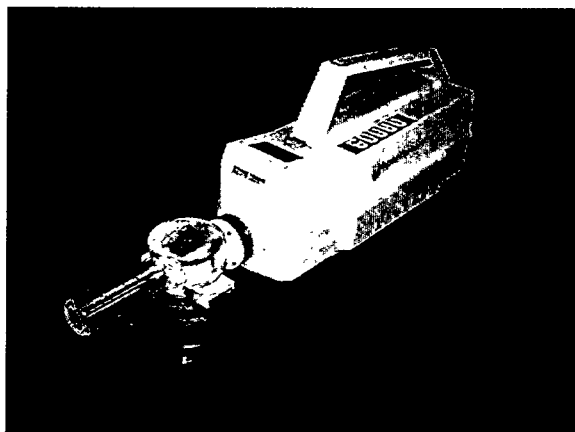


Figure 2(b): Preconcentrator interfaced to impeller and detector.

A fieldable prototype system utilizing the miniaturized preconcentrator and the ITI Vapor Tracer is shown in Figure 3, where a ruler is also shown to illustrate the approximate size of this system. The preconcentrator controls are contained within a backpack, and the weight of the entire system is approximately 23 pounds. These features make the system ideally suited to a number of field applications involving the detection of explosives.



Figure 3: Man-portable Explosive Detection System

Testing of the Miniaturized Preconcentrator

The preconcentrator has been tested using the high explosive compounds TNT and RDX. In most cases, the explosive material was desorbed into the preconcentrator inlet using a NiCr hotwire desorber. With this type of desorber, a small amount of explosive dissolved in a standard solution is placed onto a NiCr loop using a syringe. After the highly volatile solvent evaporates, the wire is resistively heated, desorbing the explosive into the preconcentrator. This technique allows reasonably quantitative results to be obtained concerning the preconcentrator's performance. However, it may underestimate the preconcentrator efficiency slightly because it does not take into account the fact that a small amount of the explosive may decompose on the hotwire while it is heated. In a few cases, experiments were also performed with particulate material. These experiments utilized C-4, a plastic explosive composed primarily of RDX. A number of parameters relating to preconcentrator construction and performance were investigated, including preconcentrator volume, screen configuration (flat versus pleated metal felt), screen porosity, inlet airflow, and preconcentrator efficiency. Many of these results have been discussed in greater detail elsewhere [3].

Experimental Results: Preconcentrator Design and Inlet Air Flow

Table I summarizes a number of experimental results that were obtained during testing of the miniaturized preconcentrator. The different experiments are discussed separately below.

Table I: Summary of experimental results relating to the configuration and inlet air flow of the miniaturized preconcentrator	
Volume Minimization	An internal volume of 0.51 cubic inches results in detector signals that are more than three times greater than those obtained with an internal volume of 1.77 cubic centimeters.
Screen Configuration	Pleated and unpleated metal felts produce approximately equal signals at the detector, but results are more consistent using the pleated felts.
Inlet Air Flow	Detector signal increases monotonically as the inlet air flow is lowered from approximately 1.88 to 0.37 liters/second.
Screen Porosity	A screen with 77% porosity performs better in C-4 particulate tests than screens with 87% and 90% porosity.

The original housing of the miniaturized preconcentrator had an internal volume of 1.77 cubic inches. An adjustable piston was added to the housing so that the volume could be varied from that value to as low as 0.51 cubic inches. Experiments were then performed in which one nanogram (ng) of TNT was desorbed into the preconcentrator inlet airflow, and subsequently desorbed into the detector. In ten replicate runs with internal volumes of 1.77 and 0.51 inches, an average detector signal for TNT of about 300 counts was obtained with the larger volume, while an average signal of about 1000 counts was obtained with the smaller volume. Thus the smaller volume produced a higher signal by slightly greater than a factor of three, which is about equal to the volume ratio of $(1.77/0.51) = 3.47$. In general, the signal should increase as the volume decreases, since the desorbed explosive material will be more highly concentrated in the smaller air volume. However, internal volumes much below 0.51 cubic inches are not practical due to other considerations, including the need to draw in a large inlet airflow.

Extensive experiments were also performed comparing detector signals obtained with pleated metal felts to those obtained with unpleated metal felts. Pleated metal felts, which are folded into a ridged pattern, have a larger surface area and thus might be expected to adsorb explosive material more efficiently, but the corrugated structure of a pleated felt might also interfere with the desorption process and hence hinder delivery of the desorbed explosive to the detector. In ten replicate experiments with pleated and unpleated felts, the average TNT signal at the detector was found to be nearly the same, perhaps reflecting a trade-off between these two factors. However, there was much less scatter in the data for the pleated felts. While the reasons for this more consistent data are at present unclear, these data lead to a preference of the pleated felt geometry over the unpleated geometry.

Inlet airflow experiments were performed in which the inlet airflow was varied in five steps from 0.37 to 1.88 liters/second. In these experiments, 3 ng of RDX vapor was desorbed into the inlet airflow, and after a full adsorption and desorption cycle the RDX signals at the detector were compared. The signal was found to increase monotonically with decreasing inlet airflow. At a flow of 0.37 liters/second it was approximately four times as great as at a flow of 1.88 liters/second. At high flow rates, it is possible that incoming explosive material is lost by being pulled through or around the metal felt. These results suggest that it might be worthwhile to investigate even lower inlet air flows, but that is not practical given the current system configuration and the need to have a significant inlet flow in order to sample as much material as possible.

Finally, particulate tests were performed that investigated the effects of varying the porosity of the metal felt. Felts with different porosities are available "off the shelf," and felts with porosities of 90%, 87%, and 77% were investigated. The tests were performed using muslin patches doped with 100 or 1000 ng of C-4 that were provided by the Federal Aviation Administration (FAA). These patches were placed in front of the system inlet and agitated, and following a desorption cycle it was determined whether or not an RDX signal could be observed at the detector. It was found that an RDX signal was detected for all patches when the 77% porosity felt was used, while a 90% porosity felt yielded detections for less than 40% of the 1000 ng patches and less than 20% of the 100 ng patches. Not surprisingly, the performance of the 87% porosity felt was found to be intermediate. These results suggest that the 77% porosity felt allows better adsorption of the explosive material, due to the fact that a larger air flow can pass through it more efficiently.

Experimental Results: Efficiency and Concentration Factor

The two most important figures of merit for a preconcentrator are the preconcentrator efficiency and the concentration factor. The efficiency (E) is simply the fraction of explosive molecules in the inlet airflow that are collected by the preconcentrator. This number can be estimated by taking the ratio of the signal obtained at the detector when a certain mass of explosive is desorbed into the preconcentrator inlet airflow to the signal obtained when the same mass is deposited directly onto the preconcentrator felt. Figure 4 shows the results of a series of experiments of this type. In these tests, one ng of TNT was either desorbed into the inlet airflow, or deposited directly onto the felt, and the felt was then heated to approximately 185 °C to desorb the material into the IMS detector. For the tests involving desorption into the inlet flow, the inlet flow was approximately 1.88 liters/second. It can be seen from the figure that the signal for the TNT desorbed into the inlet flow is well over half of the signal obtained for direct deposit onto the felt, and an average over all ten data sets shows that the precise value of E is 72%. Since a perfect preconcentrator would have an E value of 100%, this corresponds to over two thirds of the theoretical maximum value, and it indicates that further improvements in E can lead to only marginal improvements in overall system performance.

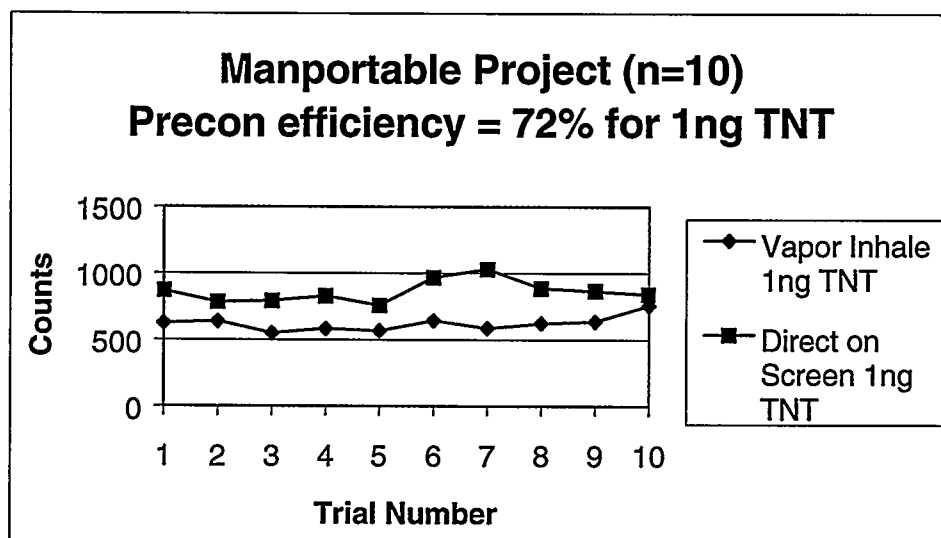


Figure 4: Preconcentrator efficiency for 1 nanogram of TNT

The concentration factor (CF) is the ratio of the concentration of explosive in the sample delivered to the detector to the concentration originally present in the inlet airflow. It is equal to the ratio of the associated air volumes multiplied by the efficiency:

$$CF = E [V(\text{air in inlet flow})/V(\text{air delivered into detector})].$$

Based on the above discussion, the value of CF can be easily calculated, if we assume equal times (of a few seconds) required for both the vapor inhalation into the preconcentrator, and the desorption of the material into the detector. The ratio of the volumes is then equal to the ratio of the associated flow rates, and recalling that the air flow into the detector is approximately 0.067 liters/second, we have

$$CF = 0.72 [1.88/0.067] = 20.3$$

Thus the sample delivered into the detector is approximately 20 times more concentrated than the sample initially taken into the preconcentrator. For a concentration sensitive detector such as an IMS, this leads to a significant increase in detection probability.

Summary and Future Work

This paper has presented information on the design and testing of a miniaturized explosive preconcentrator intended for use with man-portable trace detectors. When used in a complete man-portable system such as that shown in Figure 3, this preconcentrator should be useful in many field applications, including but not limited to investigation of suspicious objects or packages, search of vehicles or rooms, monitoring of background contamination, and perhaps (if properly adapted) mine detection. While this preconcentrator has been designed with explosives detection in mind, there is no reason it should not work for other types of heavy organic molecules, such as drugs. Future work with the preconcentrator will include tests with additional types of explosives such as

pentaerythritoltetranitrate (PETN), and further miniaturization and better packaging of the system controls, thus rendering the system more robust for field applications.

Acknowledgments

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References

- [1] G. A. Eiceman and Z. Karpas, "Ion Mobility Spectrometry," CRC Press, Boca Raton, 1994.
- [2] Ion Track Instruments, Inc., (978) 658-3767.
- [3] D. W. Hannum, K. L. Linker, F. A. Bouchier, and J. E. Parmeter, "Development and Testing of a Miniaturized Explosives Preconcentrator for Use in a Man-portable Field Detection System," 1999 NDIA conference proceedings.