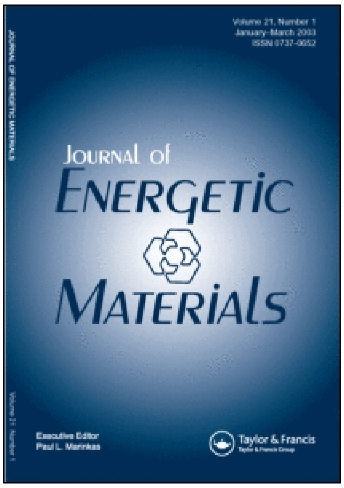

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RESEARCH PROGRAMME ON EXPLOSIVES VAPOUR DETECTION AT NRC

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ABSTRACT

The paper consists of two parts. In the first part, the rationale and considerations leading to the development of an explosives vapour detector suitable for screening suspect aircraft for hidden bombs is discussed. The detector is based on GC/ECD and consists of two components, the main analysing unit and a detachable sampling probe. A number of prototype detectors was fabricated at the National Research Council of Canada (NRC), and, after laboratory and field trials, the design became the basis of a commercially produced model.

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In the second part, the application of vapour sniffing technology for rapid screening of passengers and baggage at the airports is considered. In this scenario, trace explosive vapour must be preconcentrated in a rapid and selective manner. A novel device is described, termed a Continuous-Action Preconcentrator (CAP), which was tested and found capable of enhancement of vapour concentration by a factor of 10^3 in less than 6 seconds. Coupling of the CAP to a real-time sensor could provide the type of system required for the high-throughput airport application.

INTRODUCTION

General

The National Research Council of Canada (NRC), at the request of the Ministry of Transport, has conducted research for the past several years into the development of explosives 'sniffers', instruments designed to sample and analyze ambient air for characteristic vapour traces of hidden bombs. This type of instrument would appear to have potential in safeguarding the security of civil aircraft and airport terminals. Though the Canadian effort in the explosives detection field has been of a comparatively modest scale, the allocation of technological resources in a time of cost-consciousness makes it prudent to re-appraise the rationale of explosives sniffers in airport security. Moreover, it can be argued that, since their introduction some 15 years ago, sniffers have not made conspicuous inroads in the security field, as have, for example, metal detectors and x-ray monitors. The question then arises touching on the cost-effectiveness of R&D in the area of explosives detectors.

There would seem to be little argument as to the desirability of improving the quality of security measures at airports. If the number of real-bomb incidents has subsided in recent years, the frequency of hoaxes has not, hoaxes which often necessitate deplaning, resulting in lost time and revenue for the carriers and disrupted schedules for the passengers. Because flying tends to invoke a sense of vulnerability, at least in the minds of the general public, aircraft will likely remain a favourite target for extortionist activity.

There should also be little question concerning the principle of vapour sensing as a countermeasure to the bomb threat in airlines operation. It goes without saying, of course, that it is not impossible to hermetically seal a bomb and thereby prevent the escape of telltale vapours; by the same token, however, it is possible to shield, mask or disguise a bomb to avoid detection by virtually any means, including visual examination. In the interest of maximal security, there is clearly a need for a diversity of countermeasures, making use of as many different technologies as feasible.

The case to be made for undertaking and supporting research aimed at developing vapour sensing technology into a practical countermeasure to the bomb threat rests less on what has been accomplished with it to date and more on its inherent potential. There are today instrumental methods of chemical analysis which measure, with high selectivity, vapour concentration levels in air well below 1 part in 10^{12}, and instruments with projected detection limits reckoned in terms of femtograms (10^{-15} g). The transfer of such technology from the laboratory to the field in practical terms has not yet been achieved at this stage. Nevertheless, recognizing that trace vapour sensing is a relatively new analytical process finding use in more and more application areas, it would not be unreasonable to expect substantial progress in the near future of adaptation of such sophisticated techniques for field use. There is, finally, another outstanding reason for pursuing the present explosives detector programme: the successful development of a reliable, versatile and affordable vapour sensor for aviation security purposes has high application potential in many other areas as well.

Background

More than a dozen different models of explosives vapour detectors have been produced by a number of companies in Israel, the U.K., Canada and the U.S. Most of these instruments have in common the use of an electron-capture detector (ECD) as the basic sensing element, as well as the need of an inert gas supply. They may be categorized as either real-time or batch-type sampling devices.

Real-time sniffers generally incorporate a semi-permeable membrane or other means which allows passage of organic vapours to the detector while excluding oxygen. Though response time to explosives vapours is only a few seconds, this type of sniffer lacks specificity and is consequently prone to false alarms. The sensitivity or detection limit to organonitrate explosives is in the range of 0.5 - 2 ppb (parts per billion: 1 ppb = $1/10^9$).

The batch-type or non-continuous instruments generally make use of a chromatographic column to effect separation of oxygen and other interferants from the vapours of interest. A very high degree of specificity is achieved with such sniffers, which, together with the use of an adsorbent collector to preconcentrate the explosives vapours from a discrete volume of air sample, yields a much enhanced sensitivity, about 0.02-0.07 ppb. The trade-off to gain sensivitiy, however, results in a relatively long response time with this class of detectors, e.g., 20-60 seconds.

A departure from the electron-capture type of instrument has recently appeared which obviates the need of an inert carrier stream. In this case vapour sensing is effected on the basis of ion mobility of the explosives molecules, in real-time and in the sample air stream, resulting in a very compact and easy-to-use device. This type of sniffer, however, would probably be of little use in most search scenarios, exhibiting as it does relatively poor sensitivity (>5 ppb) while being subject to alarm falsely on fairly common items (e.g., cigarette smoke, colognes, deodorants, alcohol and acetone).

Nearly all of the commercial bomb detectors marketed to date have been tested in this Laboratory for their sensitivity to organonitrate vapours, in particular to EGDN. As well, three of the real-time detectors were utilized in an extensive field evaluation of various aircraft search methods. That program, conducted at Montreal International Airport by an interdepartmental team of scientists, engineers and security personnel, also involved visual and hand searching methods and the use of trained dogs (Ref. 1). Subsequent to these trials, a number of experiments were also performed at the Ottawa and Toronto airports using advanced mass spectrometric as well as gas chromatographic techniques to sample the ducted aircraft cabin air (Ref. 2). Among the findings which emerged from these trials were the following:

> the use of bomb detectors of the real-time variety in conducting a detailed, point-by-point search of an aircraft for hidden bombs is of little more than marginal value; this type of sniffer was found to be insufficiently sensitive, prone to false alarm, cumbersome and far less effective than a visual/hand search;
>
> GC-type sniffers used in a secondary or backup role to the visual search proved to be efficaceous; a significant probability was demonstrated that a hidden bomb missed in a visual/hand search of the aircraft could be revealed in a follow-up areal search with these devices;
>
> effective use of vapour detection technology can also be made through whole-cabin air monitoring as opposed to point-by-point sampling.

AIRCRAFT SCREENING APPROACH

Findings such as outlined above have influenced the direction of research in this laboratory on the development of an explosives detector suitable for aircraft screening. On the premise that the most reliable screening procedure for a suspect aircraft is a visual and hand search conducted by trained personnel, the rationale adopted here seeks to utilize a technological device to augment that type of search. While not intended to replace the hand search, such an instrument should lend a considerable measure of confidence to the search without adding unduly to the search time.

The instrument designed for this purpose is basically a gas chromatograph (GC). The GC is a powerful and well-established tool, having advantages of simplicity and low cost; when coupled to a vapour preconcentrator, the system can be adjusted to yield almost any level of sensitivity and specificity desired. The disadvantage in the use of gas chromatography is the seemingly long analysis time which would appear to conflict with the fast pace requirements of normal airline operation.

This feature, however, need not be a serious drawback, depending on the deployment of the device. Use of a GC-based sniffer to emulate a trained dog by examining one by one the numerous potential hiding places in an aircraft cabin is clearly an impractical approach. On the other hand, analysis time becomes of minor importance if relatively few but much larger air samples are collected - representative of the cabin as a whole - than is possible in a detailed scan of the aircraft. Such air samples could be obtained, for instance, by sampling the cabin air exhausted through the outflow and dump valves, or by sampling inside the cabin. Thus, although 1-2 minutes may be required for a sample collection and an additional 2-3 minutes for analysis, the full capabilities of the GC method with regards to sensitivity and resolution may be utilized to best advantage with this approach.

Another type of constraint associated with GC (or any other physico-chemical methodology) which the present approach seeks to obviate is that of physical size of the instrument. While considerable miniaturization over a laboratory-type instrument is fairly straightforward, the sizeable power requirements and the need for a compressed gas supply and ancillary hardware can result in an instrument too cumbersome for field use. Therefore, in the present design the vapour collection or air sampling is performed by means of a small, hand-portable aspirator probe, and the analysis of the sample is conducted subsequently with the GC which may be stationed as close to the search scene as convenient.

General Description

The instrument is a portable, self-contained, single-column GC equipped with an electron-capture detector. It is comprised of two separate parts, the hand-held Probe and the main Analyzer unit, a photograph of which appear in Fig. 1.

The GC operates with high-purity grade nitrogen gas, and carries sufficient supply for 40 hours of continuous use in the 'Operate' mode and 400 hours in the 'Standby' mode. The internal cylinder can be easily re-filled in situ by connecting high pressure hose to a large cylinder of compressed N_2. The GC may also be run with an external source of N_2 regulated at 30 psig; a quick-connect fitting is provided for coupling to an external bottle. A Type K cylinder (Linde) of N_2 is sufficient for over 2½ years of Standby operation.

The electrical and electronic components are designed for 12 V DC. The instrument may thus be

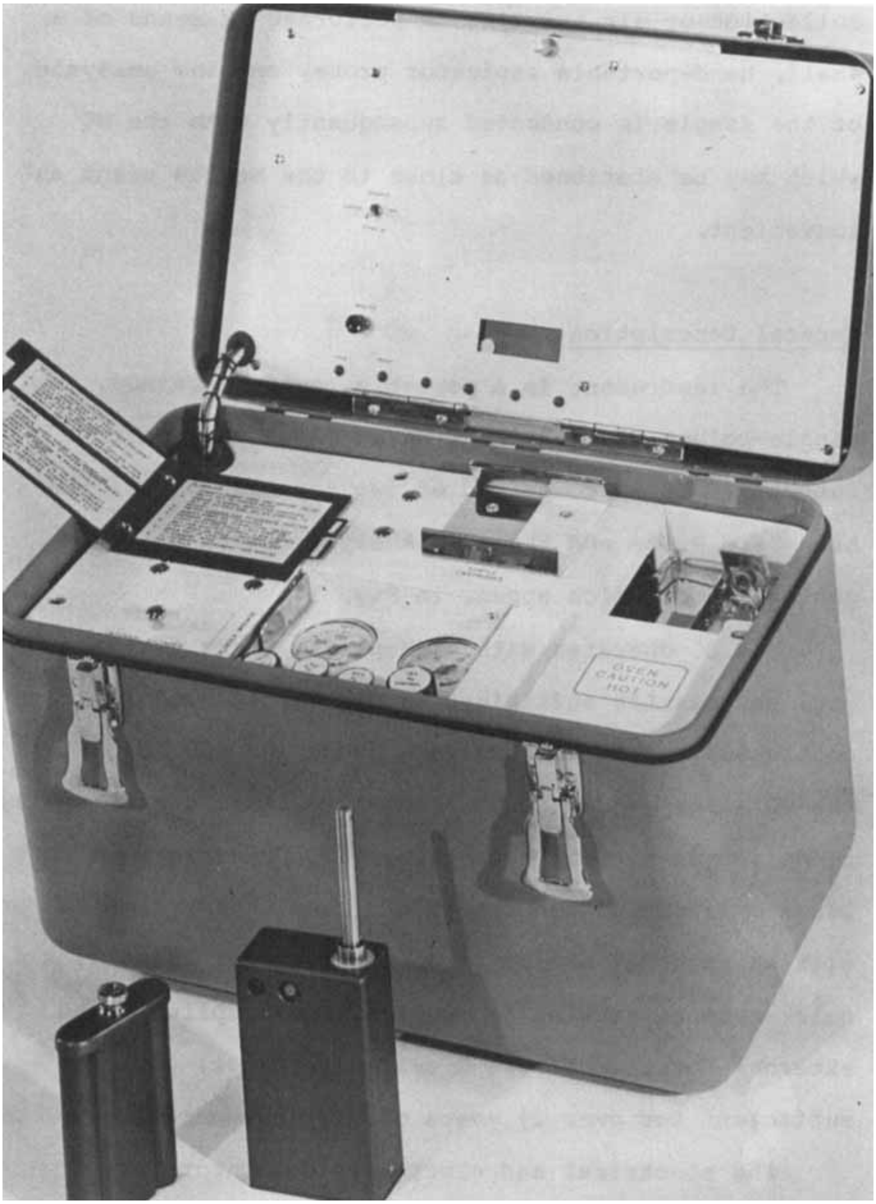


FIGURE 1

NRC Explosives Vapour Detector, showing Analyzer Unit, Sampling Probe with Adsorber Cartridge, and Magazine.

powered by connection to the cigarette lighter receptacle of a car or truck, or by means of an auxiliary battery pack, or may be plugged directly into the 110 V AC mains.

Probe

The Probe houses a battery-powered aspirator pump which draws air through the first-stage adsorber cartridge or preconcentrator. The cartridge is a thin-walled steel tube 0.8 cm O.D. by 8 cm long which contains a filter material treated to collect explosives vapours. Flow rate through the adsorber tube is about 1 L/min when packed with a rolled strip of 80-mesh nickel gauze. The gauze is coated by dipping in a 1% solution of OV-17 silicone oil in chloroform, air drying, then conditioning at 300°C in a N_2 stream.

With the adsorber installed in the Probe, the air pump is activated by depressing a momentary pushbutton switch. A timing circuit in the unit limits the pump action to 15 sec; longer sampling times may be obtained by repeated depression of the switch.

Vapour sample collected in an adsorber is subsequently thermally desorbed and analyzed in the Analyzer unit; following desorption, the cartridge is ready for re-use.

Explosives vapour is retained on the adsorber packing for several hours without serious loss. In one test a known quantity of EGDN was deposited on the packing and the adsorber was stored overnight in a sealed container. Eighteen hours later roughly half of the original amount of EGDN was recovered on analysis.

A small magazine in which four cartridges are stored separately has been found convenient for multiple-sample use.

Analyzer

The Analyzer unit houses an automated GC, with attendant control and data processing circuitry and carrier gas support system. The instrument has been designed for use by non-technical personnel. After connecting an adsorber cartridge in the Analyzer for analysis, the operator is instructed to initiate the 'Analyze' cycle by means of a pushbutton switch; completion of the cycle, about 2 minutes later, is signalled by an indicator light. The presence of explosives in the sample is signified by the reading of a three figure LCD display, higher reading representing larger vapour concentrations.

Figure 2 is a schematic of the GC configuration. The 6-port valve, column and detector are thermostated in a small oven. The column is a 30 cm length of nickel tubing 3.2 mm O.D. packed with 3% OV-17 on Chromosorb W, 80-100 mesh, and controlled at 125°C.

Adsorber I represents the (detachable) Probe cartridge installed for analysis, Adsorber II is a smaller second-stage preconcentrator which is permanently incorporated in the system. Both adsorbers are located outside the GC oven and heated in sequence, as required, by means of surrounding heater windings.

The 6-port valve is of the tapered plug type, having a filled teflon plug and nickel body. It is rotated bi-directionally over an angle of 60° by means of a stepping motor. Two positions of the valve are shown in Figure 2, to illustrate the flow of the sample and the carrier gas during the second preconcentration stage (sample transfer) and during analysis (analysis/standby mode).

The two-adsorber technique has proven to be advantageous on several counts. In addition to providing convenience and ease of sampling, use of the first-stage adsorber avoids vapour losses frequently observed with the use of long sampling lines. Moreover, it virtually eliminates the possibility of gross system contamination and consequent cleanup, inasmuch as such contamination would be restricted to and necessitate, at worst, replacement of the adsorber packing.

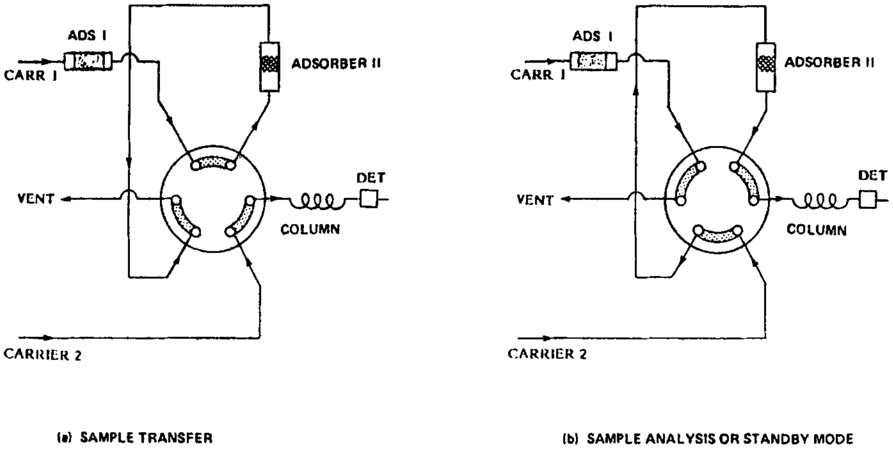


FIGURE 2

Schematic of GC Analyzer.

Testing of the Prototypes

A number of prototype Explosives Detectors described above has been fabricated at the NRC and, after laboratory tests, were also tried on several occasions in the field. The units were programmed for detection of EGDN (although, if required, they could also be adjusted for detection of other explosives vapours, such as NG and DNT).

The laboratory tests with the Detectors consisted mainly of sensitivity evaluations. In the testing, use was made of special vapour sources developed in this laboratory (Ref. 3,4), in which a continuous stream of the vapours at the ppt-ppb concentration levels could be reproducibly generated. One such type of a vapour source (single dilution type) is shown in Figure 3. Since it utilizes unpurified room air with all its contaminants, this vapour generator provided a more meaninful test of the Detectors' utility.

Using the calibrated vapour source, it was established that concentrations of 0.005 ppb or less of EGDN in ambient air could be routinely detected by the prototype sniffers, often by using a single 'sniff' (a 15-second sampling at the flow-rate of 1 L/min). Detection limit of the units was affected by the level of air contamination; when larger samples could be

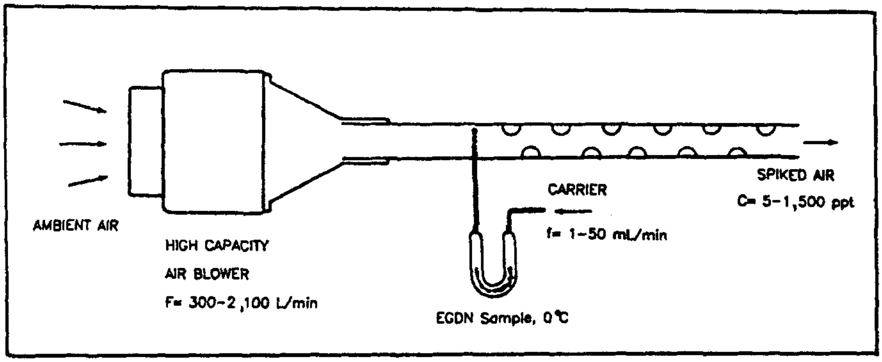


FIGURE 3

Single-Dilution Trace Vapour Generator. Nitrogen carrier passing over small quantity of thermostated EGDN sample mixes with diluent air stream in all-glass system. Concentrations less than 5 ppt obtained by thermostating sample below 0°C.

collected, concentrations as low as 0.001 ppb were detected.

Field trials with aircraft were focussed mainly on sampling cabin air vented through the dump valves of the aircraft. Numerous tests involving various types of passenger jets (DC-9s, DC-8s, 727s, 767s and 747s) have been conducted, in which explosive samples were placed on board the aircraft and, with the cabin door sealed and the air conditioning system operating, characteristic vapours were detected at the exhaust port outside the aircraft. Samples were placed in overhead luggage racks, under seat cushions, in lavatories and other areas readily accessible to the passenger. The success rate of detection using dump valve sampling ranged from 0 to virtually 100%, depending on the type of explosive used; overall, the probability of detecting an unknown bomb by this method is estimated to be between 20-40%. An internal, areal screening of the cabin using the prototypes allowed the detection of more explosives of lower volatility, and the probability of detection in an actual situation with this search method is thought to be 50-70%.

Some field experience with the units in non-airport scenarios was obtained during the Papal and Royal visits to Canada in the Fall of 1984, when they were used by the RCM Police to search buildings, cars and luggage. Several 'hits' were actually reported with the Detectors on those occasions (having to do with ammunition and firearms being inadvertantly placed in some of the suitcases by security personnel).

Concluding Remarks

The Detector technology developed at NRC has been transferred to industry, and the first commercial units, called EVD-1 (Ref. 5) appeared on the market in 1985. Based on the GC concept described here, the commercial sniffers have been re-engineered to meet special specifications on ruggedness and electromagnetic shielding for the Ministry of Transport.

An extensive laboratory evaluation of the EVD-1 was carried out recently by the RCM Police and the range of explosives which could be detected with the sniffer has been investigated (Ref. 6). In addition, over 70 different substances (including cosmetics and various solvents) were tested for potential interference. The EVD-1 was found to meet the specified performance requirements of high sensitivity for explosives detection with an extremely low false alarm rate.

Presently, there are about 26 EVD-1 units deployed at all major international airports in Canada. EVD-1 operators are selected and undergo special training on proper use of the detectors in the field for maximum efficiency.

PASSENGER AND BAGGAGE SCREENING

CAP Approach

The problem of screening passengers and checked baggage for hidden explosives by vapour detection is certainly one of the more formidable technological ones. The GC approach described in the preceding Section, which has proven feasible for aircraft and luggage screening on a spot-check basis, is not readily adapted to the high-throughput requirements of routine airline operations. These requirements, for example as seen by the U.S. Federal Aviation Administration, call for a detection system capable of sensing 0.01 ppt levels of explosives vapour (approximately 10^{-13} g/L) in 6 seconds or less, with a false alarm rate below 2%.

No sensor is available at present that can respond to such vapour levels, *per se*; clearly, some form of sample preconcentration is required prior to analysis. Moreover, the sample preconcentration step must be very rapid and, preferably, provide some selectivity in order to reduce the background signal from other gases and vapours.

A device which has been under development at NRC for this purpose, known as CAP or Continuous-Action Preconcentrator (patent pending), is based on an adsorption - thermal desorption technique, a proven preconcentration method in trace vapour analysis. The novel feature of the device is that, where, conventionally, discrete volumes of air are scrubbed of the pertinent vapours by a stationary bed of adsorbent material, so that the concentrating process is discontinuous and time-consuming, the CAP achieves a similar concentrating effect on a continuous and nearly real-time basis.

The device utilizes adsorbent powder, of the type used in GC work, which is repeatedly cycled through a cool (adsorption) region to a hot (desorption) region, and back again. The large surface area of the powder combined with small thermal mass leads to an effective and fast-responding vapour handling process.

The CAP is shown schematically in Figure 4. In the cold region, the adsorbent is exposed to the sample air stream. In the desorption region, maintained at selected higher temperatures, a carrier flow purges the collected vapours and vents to a suitable analyzer. By proper selection of the adsorbent powder and temperatures, some degree of specificity can be attained with regard to the vapours collected from the air.

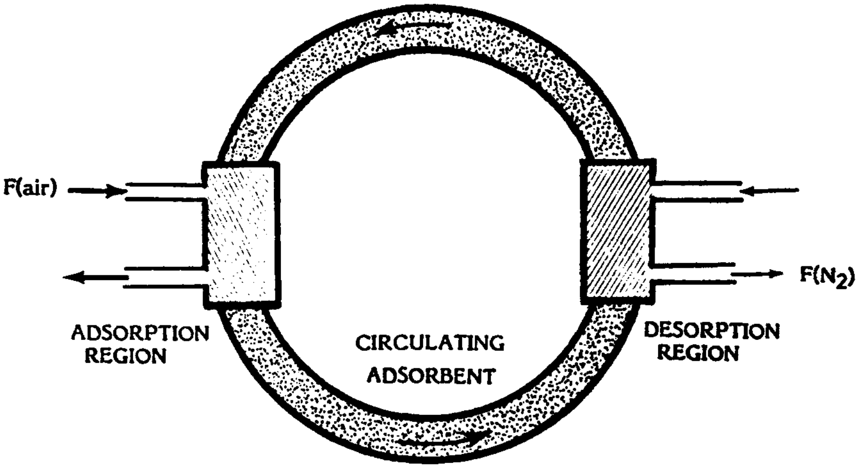


FIGURE 4

Schematic of Continuous-Action Preconcentrator (CAP).

Concentration Enrichment

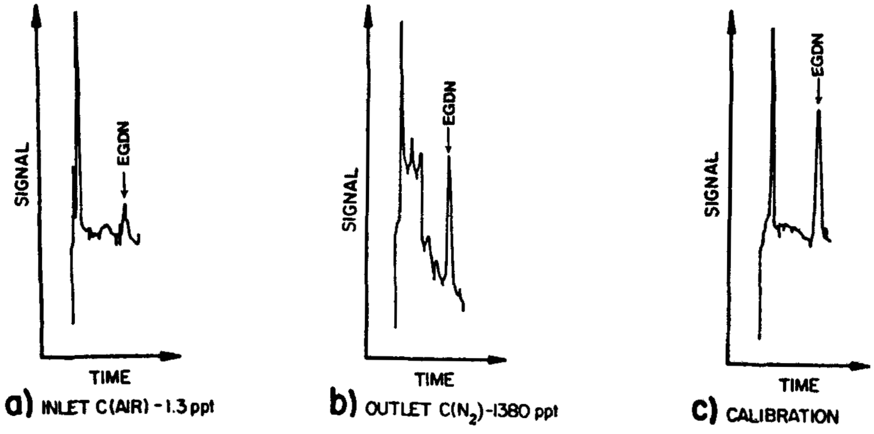
Vapour enhancement is achieved by transferring the vapours from a large flow of air, where they are present at low concentration, to a small flow of nitrogen, or other carrier gas. If the adsorption and desorption processes are complete, and no losses occur, the mass flow of a particular trace component *i* in the incoming air stream will be equal to the mass flow of the same component in the outgoing (nitrogen) stream; thus,

$$F(\text{air}) \times C_i(\text{air}) = F(N_2) \times C_i(N_2), \text{ or}$$

$$\frac{C_i(N_2)}{C_i(\text{air})} = \frac{F(\text{air})}{F(N_2)}$$

where F and C_i are flowrates and concentrations, respectively, of the air and carrier streams, as indicated. This relation can be used to estimate the upper limit of concentration enhancement for typical flowrates. For example, if F(air) = 100 L/min and $F(N_2)$ = 100 mL/min, the maximum concentration enrichment is 10^3.

To test the concept, a CAP apparatus was constructed and used to study the preconcentration of EGDN vapour. EGDN in the sample air and nitrogen carrier streams was determined by collection in Tenax adsorber tubes and subsequent GC analysis. Figure 5 shows chromatograms from such a study.



a) INLET C(AIR) - 1.3 ppt

b) OUTLET C(N_2)-1380 ppt

c) CALIBRATION

FIGURE 5

Preconcentration of EGDN Vapour with CAP: Chromatograms of EGDN Analysis. EGDN retention time 1.74 min; (a) 3.2 pg, sample volume 400 mL; (b) 87 pg, sample volume 10 mL; (c) 86 pg, 8 μL standard solution.

In the case shown, the CAP input was an air stream F(air) = 45 L/min spiked with 1.3 ppt EGDN, which was provided by the high-volume vapour generator of Figure 3. The CAP output was a nitrogen stream $F(N_2)$ = 12 mL/min with an EGDN concentration of 1,380 ppt. While the efficiency of the process was less than 30%, based on the gas flow ratio, the measured concentration ratio demonstrated that a thousand-fold vapour enhancement could be achieved even with a non-optimized CAP.

Response Time

Speed of response of the CAP was investigated in two separate experiments conducted with two different detectors attached to the device, a flame ionization detector (FID) and a tandem mass spectrometer (MS/MS).

In the first experiment, the outlet nitrogen stream was connected to a Gow-Mac Model 12-100 FID, and the signal from the detector was monitored on a chart recorder. The time response of the CAP-FID system was measured as a delay between a rapid syringe injection of a vapour sample into the air stream at the CAP inlet and the appearance of the signal in the outlet stream. The results with the vapours of propane and acetone are shown in Figure 6, where it can be seen that in each case the signal arrived approximately 3 seconds after injection of the sample, and reached its maximum in less than 6 seconds.

A similar result on the speed of response was also obtained in the second experiment using EGDN vapour, in which the CAP was coupled to a Sciex TAGA 6000 tandem mass spectrometer. The TAGA MS/MS was operated in the negative mode with an atmospheric pressure chemical ionization source, and with chloroform vapours as a source of reactive Cl^- ions. Specific detection of EGDN vapours was achieved by monitoring daughter ions from the EDGN.Cl^- parent ions. The EGDN vapours were again generated by means of the single dilution vapour source. The concentration of EGDN in the spiked test stream from the source was modulated by stopping and starting the source carrier stream (cf. Fig. 3) and the effect of the modulation was followed, in real-time, by the CAP-TAGA combination.

Figure 7 shows the intensities of some characteristic ions as a function of time when the vapour source was alternatively switched on (600 ppt) and off (Background, abbreviated as Bkgd.). It can be seen that, after switching the vapour source on the signal is observed with about 5-10 seconds delay. Part of this delay is probably due to the somewhat sluggish method of modulation used to vary the EGDN concentration in the vapour generator.

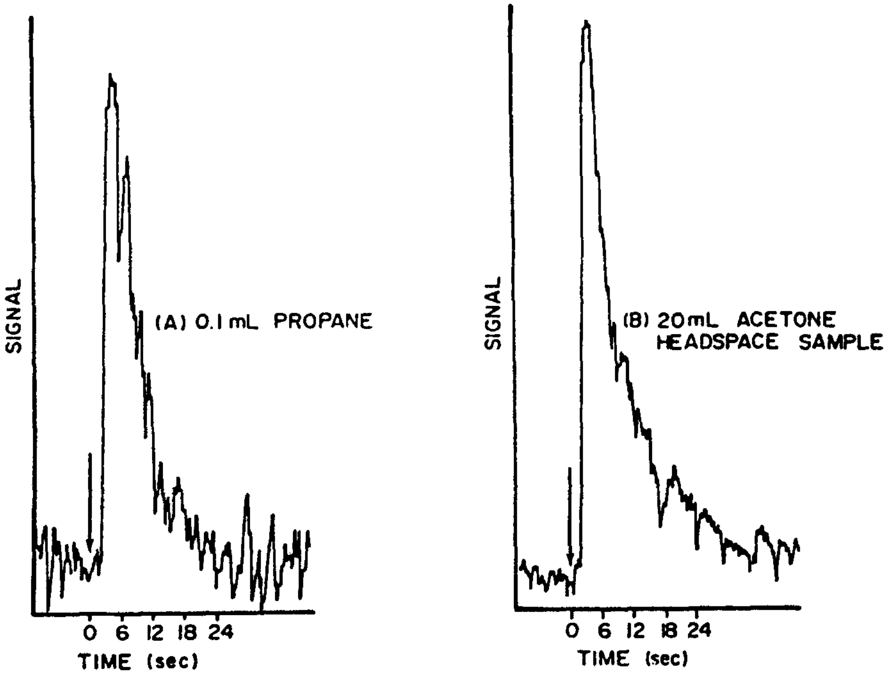


FIGURE 6

Speed of Response of CAP-FID to Hydrocarbons. Arrows indicate point of sample injection into CAP inlet.

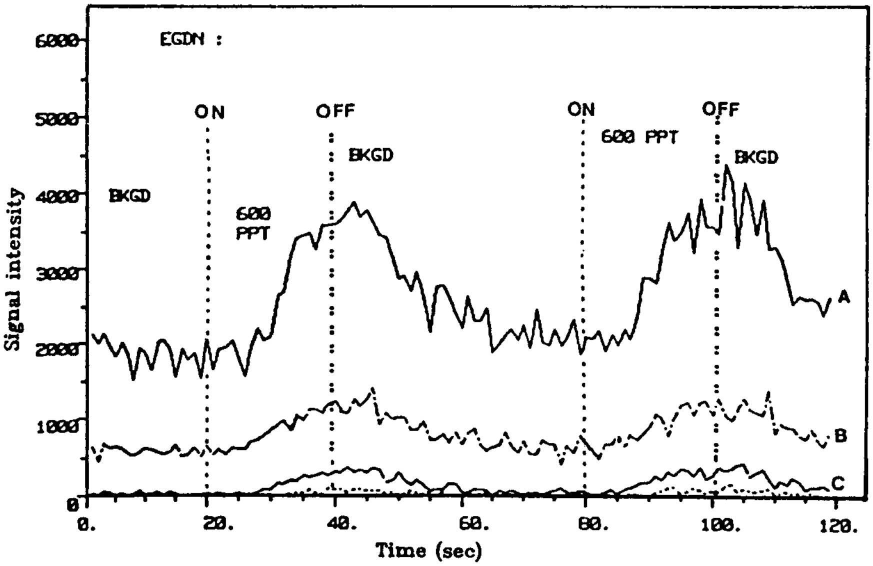


FIGURE 7

Speed of Response of CAP-TAGA System to EGDN from Vapour Source. EGDN switched alternatively ON and OFF. Profiles of daughter ions generated from EGDN.Cl^- parent ions (m/e^- 187 and 189): (A) $^{35}Cl^-$; (B) $^{37}Cl^-$; (C) NO_3^-.

Future Work

The CAP concept has been tested and shown to produce a 10^3-enrichment of concentration levels in 6 seconds or less. An apparatus of this type, therefore, could well serve as the front end of a sensitive and rapid-responding explosives vapour detector for passenger and baggage screening.

Apart from efficiency and speed of operation, which have been studied, the inherent capability of the CAP to selectively preconcentrate target vapours has not as yet been fully investigated. Selectivity requirements will vary, of course, depending on the type of analyzer used; with very specific detectors (e.g., tandem MS/MS), the selectivity of the preconcentration step will be less important than with other detectors (e.g., ECD, FID, etc.). Work continues on this project, and further progress is anticipated.

ACKNOWLEDGEMENTS

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