

*J. E. Chrostowski,¹ B.S.; R. N. Holmes,¹ B.S.; and
B. W. Rehn,¹ B.S.*

The Collection and Determination of Ethylene Glycol Dinitrate, Nitroglycerine, and Trinitrotoluene Explosive Vapors

The identification of an explosive during a bomb scene investigation is a difficult and tedious task. The debris obtained from a bomb scene is usually first examined by microscopic means [1], with the hope of detecting some unconsumed explosive. Particles of suspected explosives are analyzed by using color spot tests or by solvent extraction procedures, with subsequent concentration and verification by thin-layer chromatography (TLC) or infrared spectroscopy (IR). This technique is time consuming if large quantities of debris are to be examined. One approach by which analysis time may be reduced is to scan samples from a bomb scene with a commercially available explosive detector such as a special purpose automated gas chromatograph vapor trace analyzer (VTA) [2]. Samples on which a positive response is obtained are retained for further analysis. A positive response on the VTA is an indication that an explosive vapor of commercial nitroglycerine dynamite has been detected. These selected samples should be further analyzed to confirm the presence of other related explosives [2]. Methods for analyzing explosive vapors by methods other than the VTA have also appeared in the literature [3,4].

Methods for the analysis of trace organic constituents in air [5,6] and the analysis of headspace volatiles have recently been published [7,8]. In these methods, air and vapor were drawn through a tube containing gas chromatographic (GC) column packing materials, such as porous polymer beads, to concentrate or trap trace organic constituents. This method of trapping was preferred over methods such as dry ice/acetone baths and other cryogenic methods or the use of charcoal as an absorbent. Following the trapping or concentration step, GC was used to analyze the trace constituents. In other techniques chemically treated glass beads were used to collect and detect explosive vapors [9]. This paper describes the use of a short column containing porous polymer beads to collect explosive vapors of ethylene glycol dinitrate (EGDN), nitroglycerine (NG), and trinitrotoluene (TNT), with subsequent analysis by TLC.

Apparatus

Absorbent and Collection Column Preparation

Ten-gram quantities of Chromosorb 102 (Johns-Mansville) 80/100 mesh were

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¹ Senior chemist, senior chemist, and chief chemist, respectively, Philadelphia Laboratory, Bureau of Alcohol, Tobacco and Firearms, Department of the Treasury, Philadelphia, Pa.

washed twice with 100 ml of acetone and then filtered on a fritted glass filter. The washed absorbent was dried at 100°C (212°F) for 45 min with a high efficiency fluidizer in a stream of nitrogen gas. After 45 min, the temperature was raised to 200°C (392°F) for 1 h for final conditioning. The column for the collecting of the explosive vapors consisted of a disposable pipet² that was packed with approximately 0.35 g (about 2 in. or 51 mm in length) of absorbent between glass wool plugs.

Explosive Vapor Collecting System

The system used to collect and concentrate the explosive vapors is shown schematically in Figs. 1 and 2. It consists of a paint can and lid, a 60-deg funnel, collection column, and a vacuum pump capable of drawing 2 to 3 litres of air per minute through a filled collection tube.

Thin Layer Chromatography Materials

The TLC plates used were from Analtech, Inc. and were composed of precoated silica gel G, 250 μm thick. Capillary tubes delivering approximately 8 μl were used for spotting the TLC plates. Standard solvent systems were used to develop the TLC plates

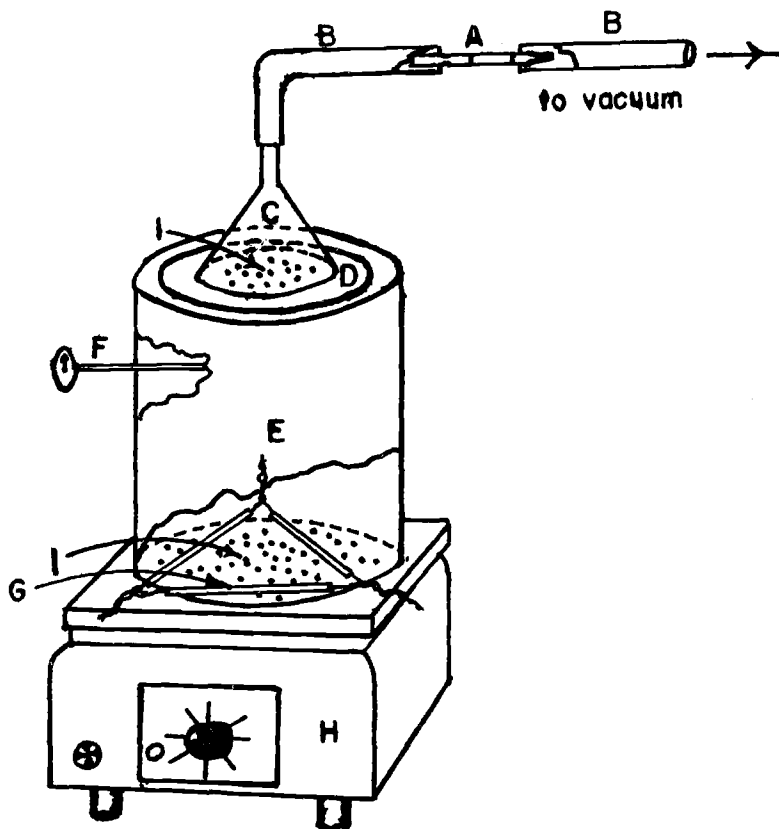


FIG. 1—Explosive vapor collection system: A—collection column; B—Tygon[®] tubing; C—funnel; D—container lid; E—unused metal container (paint can in pint, quart, or gallon size); F—thermometer; G—clay triangle; H—hot plate; and I—8-penny nail holes.

² Kimble disposable pipets, 5¾-in. (146-mm) long, Catalog #7200.

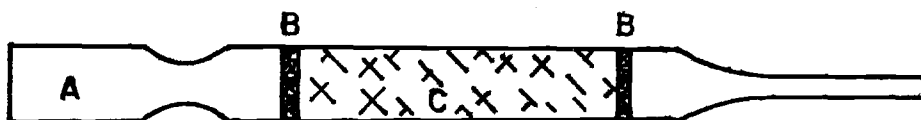


FIG. 2—Vapor collection column: A—disposable glass pipet; B—glass and wool; and C—washed Chromosorb 102 (Johns Mansville) 80/100 mesh.

[10,11]. For visualization of the spots the plate was lightly sprayed with a solution consisting of 3% diphenylamine in 95% ethanol and then exposed to ultraviolet radiation for approximately 10 min.

Explosive Standards

For experimental purposes the following explosives were used: 50% straight dynamite containing EGDN and NG, Pentolite[®] primer consisting of TNT and PETN (pentaerythritol tetranitrate), NG in lactose, and smokeless powder containing NG.

Experimental

Samples of 5 to 50-mg quantities of explosives were mixed thoroughly with approximately 300 g of sand, placed in a paint can over a thin bed of cotton, and sealed with a lid. Holes the size of an 8-penny nail were made in the bottom and top of the can to allow air to flow through the sample. A 60-deg glass funnel was placed over the top holes and the rim of the funnel was sealed to the can with tape. A collection tube containing the absorbent was fitted between the funnel and the vacuum pump with Tygon[®] tubing, with the tapered end of the glass tube toward the vacuum side to prevent the absorbent from being pulled through the tube. With the pump on, air was pulled through the system and vapors were collected for a 30-min period. After vapor collection, the column was removed and extracted three times with 1-ml quantities of acetone. The 3 ml of acetone was then reduced to approximately 30 μ l with a stream of air. A volume of the concentrated extract was spotted on TLC plates along with standard solution and developed by the TLC methods mentioned above.

Results and Discussion

At ambient temperatures (about 25°C or 77°F) and an air flow of 2 to 3 litres/min through the system, sufficient quantities of EGDN vapors were retained on the collection column in less than 5 min to give a positive response to a diphenylamine/concentrated sulfuric acid (DPA) spot test. However, after 30 min NG or TNT vapors were not detected. From the data of others, published by Urbanski [12], the vapor pressure of EGDN at 60°C (140°F) is shown to be 20 to 70 times greater than that for NG and considerably greater than that for TNT. It would be necessary and therefore impractical to use much longer collection times to detect NG and TNT vapors at ambient temperature. However, in the temperature range of 50° to 75°C (122 to 167°F) and an air flow of 2 to 3 litres/min, NG and TNT were detected within 15 min. In some instances where a DPA spot test was negative, such as short collection times or small samples of dynamite, the more sensitive TLC method on the same sample indicated the presence of explosive oils.

The ability of the short collection column to retain explosive vapors was demonstrated by drawing air through a large sample of dynamite (50 mg in 500 g of sand) at 75°C (167°F) for 30 min. Two collection columns were placed in series in the system. The TLC analysis showed that all explosive vapors were retained on the first collection column.

Experiments conducted with dynamite in wet sand showed that water had no adverse

effect on the collection of the explosive oils. This result may be attributed to the fact that Chromosorb-polymer beads have a high affinity for organic compounds and are extremely hydrophobic. During the collection of explosive vapors from wet samples, moisture collected in the column. The moisture was removed by disconnecting the column from the funnel and allowing the pump to draw air through the column.

This technique was used successfully to detect explosive vapors from several actual cases. Explosives were detected in cinder blocks, insulation, wire, crater debris, and paper. However, in several of these cases, NG was not detected by the conventional microscopic/spot test methods. Although not intended to replace microscopic and physical methods of bomb debris analysis, this method considerably reduces analysis time. If the debris consists of asphalt and resinous materials and must be extracted with organic solvents to complete the analysis, difficulty is encountered in the analysis because of the solubility of these materials in organic solvents. Using this method the solvent extraction procedure is unnecessary.

The method can also be used in conjunction with a commercial VTA to isolate and identify explosive vapors from samples that register a positive VTA response.

The frequency of explosive detection could be increased if personnel engaged in explosion investigation were trained to place bomb debris in containers such as tin cans that could be sealed with a lid to preserve any residual explosive vapors.

The method of collecting explosive vapors presented in this paper could readily be adapted for collecting EGDN vapors at the scene of an explosion. The system would consist of a funnel, a collection tube, tubing, and a portable vacuum pump. Vapors could be collected by placing the funnel directly into areas of the crater and near the seat of the explosion. The vapor collection tubes could be sealed and preserved until time of analysis.

Summary

The method presented in this paper demonstrated that minute quantities of explosives such as EGDN, NG, and TNT can be easily detected and determined in relatively large amounts of debris after collecting their vapors on porous polymer beads. The method of collecting explosive vapors is simple, inexpensive, sensitive, and relatively fast compared to microscopic and physical methods of analysis.

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Department of the Treasury
Bureau of Alcohol, Tobacco and Firearms
Room 1104 U.S. Customs House
Second and Chestnut Sts.
Philadelphia, Pa. 19106