FAA-74-10

REPORT NO. FAA-RD-74-134

DEVELOPMENT OF AN ELECTRON-CAPTURE TECHNIQUE SPECIFIC FOR EXPLOSIVES DETECTION

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JULY 1974

INTERIM REPORT

Prepared for

U.S. DEPARTMENT OF TRANSPORTATION
FEDERAL AVIATION ADMINISTRATION
Systems Research and Development Service
Washington DC 20591

Technical Report Documentation Page

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.
FAA-RD-74-134		

4. Title and Subtitle	5. Report Date
DEVELOPMENT OF AN ELECTRON-CAPTURE TECHNIQUE SPECIFIC FOR EXPLOSIVES DETECTION	July 1974
	6. Performing Organization Code
7. Author(s) J.R. Hobbs	8. Performing Organization Report No. DOT-TSC-FAA-74-10
9. Performing Organization Name and Address U.S. Department of Transportation Transportation Systems Center Kendall Square Cambridge MA 02142	10. Work Unit No. (TRAIS) FA-426/R4103
	11. Contract or Grant No.
12. Sponsoring Agency Name and Address U.S. Department of Transportation Federal Aviation Administration Systems Research and Development Service Washington DC 20591	13. Type of Report and Period Covered Interim Report June 1972 - June 1973
	14. Sponsoring Agency Code

15. Supplementary Notes

16. Abstract

This document contains information on the design, fabrication, and testing of a prototype detector specific for explosives which employs electron-capture sensors. The technique used exploits the observation that the electronegative vapors from explosives, which are readily detectable by electron-capture at room temperature, decompose at 250°C and cease to be detectable by this means, whereas electronegative vapors from non-explosives remain stable and detectable at this temperature. A study of portable vapor concentrators for use with explosive detectors is presented in Appendix A.

17. Key Words	18. Distribution Statement
Explosives Detection, Electron Capture Detector, Gas Chromatography, Dynamite, TNT.	APPROVED FOR U.S. GOVERNMENT ONLY. THIS DOCUMENT IS EXEMPTED FROM PUBLIC AVAILABILITY BECAUSE INFORMATION MAY BE OF USE TO THOSE WHO WISH TO THREATEN THE TRANSPORTATION INDUSTRY. TRANSMITTAL OF THIS DOCUMENT OUTSIDE OF THE U.S. GOVERNMENT MUST HAVE PROIR APPROVAL OF THE F.A.A.

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price
Unclassified	Unclassified	38	

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PREFACE

The work described in this report was performed in the context of an overall program at the Transportation Systems Center concerned with the assessment of various techniques for Civil Aviation Security. The program is sponsored by the Department of Transportation through the Systems Research and Development Service of the Federal Aviation Administration.

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1. INTRODUCTION

A continuing need exists for techniques which will detect concealed explosives. Probably the most effective method found to date is the use of dogs trained to "sniff" the minute amounts of characteristic vapor given off by an explosive.[1] Such dogs are not generally available; both their training and maintenance are costly, since they usually work only with a particular handler, and, they can operate effectively for only relatively limited periods. Consequently, efforts have been under way for several years to develop machines which can electronically "sniff" explosives.

The principal requirements for such devices are high sensitivity and high specificity. Sensitivity is necessary to detect the minute quantities of the characteristic vapor. The amount of vapor given off varies greatly with the type of explosive; typically the concentration in the ambient air may be less than one part per billion. Specificity is needed to avoid response to non-explosive substances (false alarm). Additional desirable characteristics are simplicity, fast response, and low cost. Table 1 presents the various devices currently available for explosives-vapor detection. As can be seen, the electron-capture detector, except for poor specificity, has most of the features desired in an explosives-effluent detector. It was for this reason that work at TSC was devoted to further study of the electron-capture technique.

TABLE 1. CHARACTERISTICS OF EXPLOSIVES-VAPOR DETECTORS

	Sensitivity	Specificity	Complexity	Cost	Response
Gas Chromatograph	< ppb	Excellent	High	High	Slow
Mass Spectrometer	~ ppb	Fair	High	High	Fast
Bioluminescence Detector	~ ppb	Fair	Low	Low	Fast
Electron-Capture Detector	< ppb	Poor	Low	Low	Fast

2. ELECTRON-CAPTURE DETECTORS

An electron-capture detector (Figure 1) is basically an ionization chamber in which electrons are produced from a radioactive cathode (tritium or nickel-63). These electrons are injected into a stream of inert carrier gas (helium or argon) where they rapidly dissipate their energy to a thermal level by inelastic collisions with the molecules of the carrier gas. The thermal electrons are collected by a positive electrode (anode) and constitute a constant (standing) current, N_o electrons/second. When an electron-capturing substance is introduced into the carrier gas, the standing current decreases to a reduced level, N electrons/second, where

$$N = N_0 \exp (XKC).$$

X is a constant factor, K and C, respectively, are the electron-attachment coefficient and the concentration of the electron capturing substance. Substances known to be electron-capturing are alkyl halides, conjugated carbonyls, nitrates, nitriles, organometallics, oxygen and water.

Electron-capture detectors are fast-responding and low in cost. They have a sensitivity of about 1 ppb for most compounds, but are not specific. High specificity can be obtained by means of a gas chromatograph column on the inlet to the detector. This technique is used by the Hydronautics-Iseral Vapor Trace Analyzer (VTA) Model 103A (Figure 2). The chromatograph column permits time separation and analysis of the different compounds as they arrive at the detector. Thus, a "window" can be found for the detection of a given substance, such as an explosives effluent.

The Hydronautics VTA has a sensitivity to the effluent of dynamite [ethylene glycol dinitrate (EGDN)] of about 0.03 ppb.[2] One of the factors which contributes to this high sensitivity is the concentrator at the inlet to the gas chromatograph column. This concentrator is a 0.05 in. diameter platinum wire, 2.0 cm. long, mounted in a Teflon valve. In the "sampling" position of the valve, room air is passed over it for 5 seconds at a rate of 10 liters per minute. Explosive vapor is absorbed on the wire. The

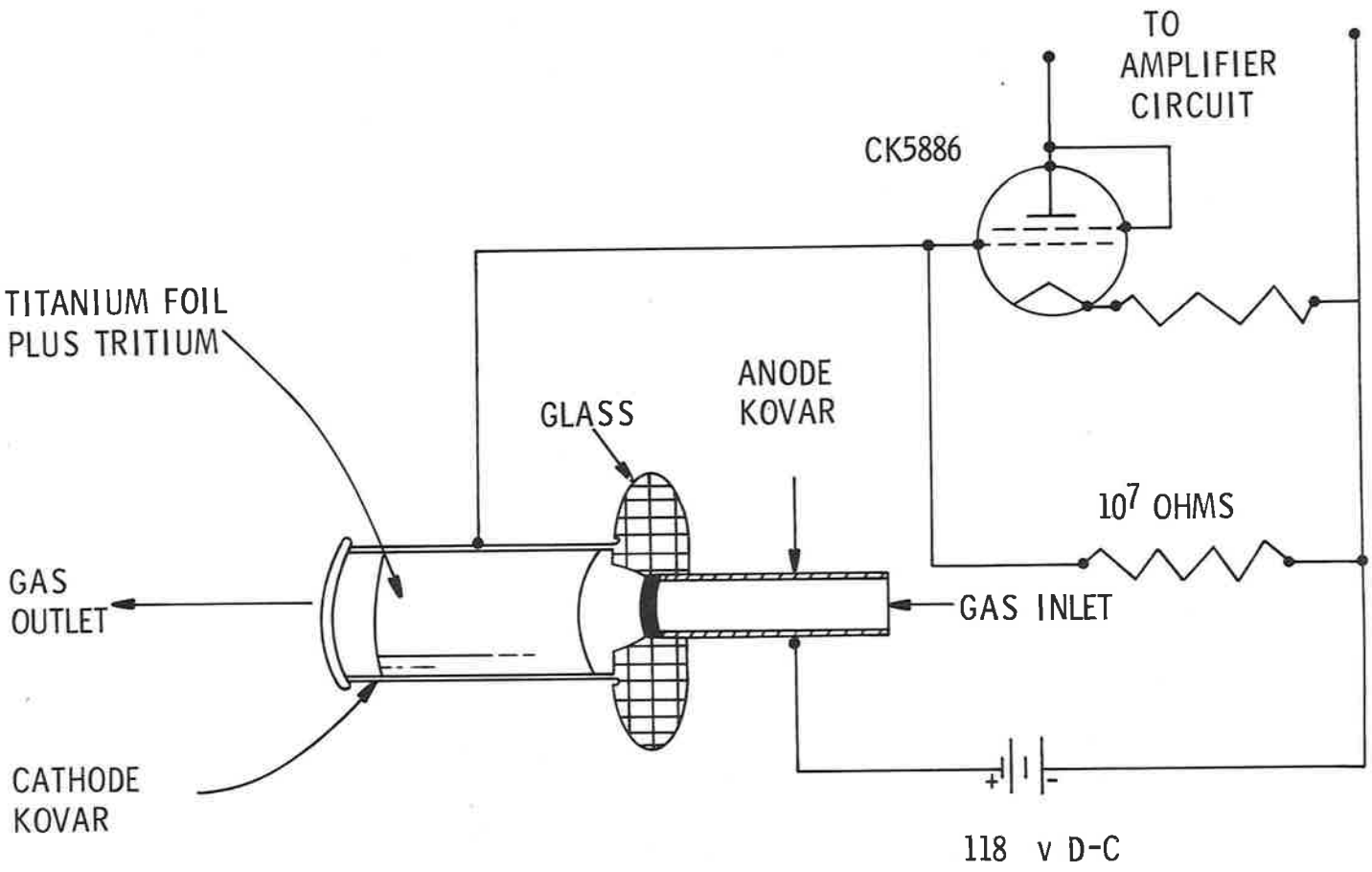

Figure 1. Electron-Capture Detector

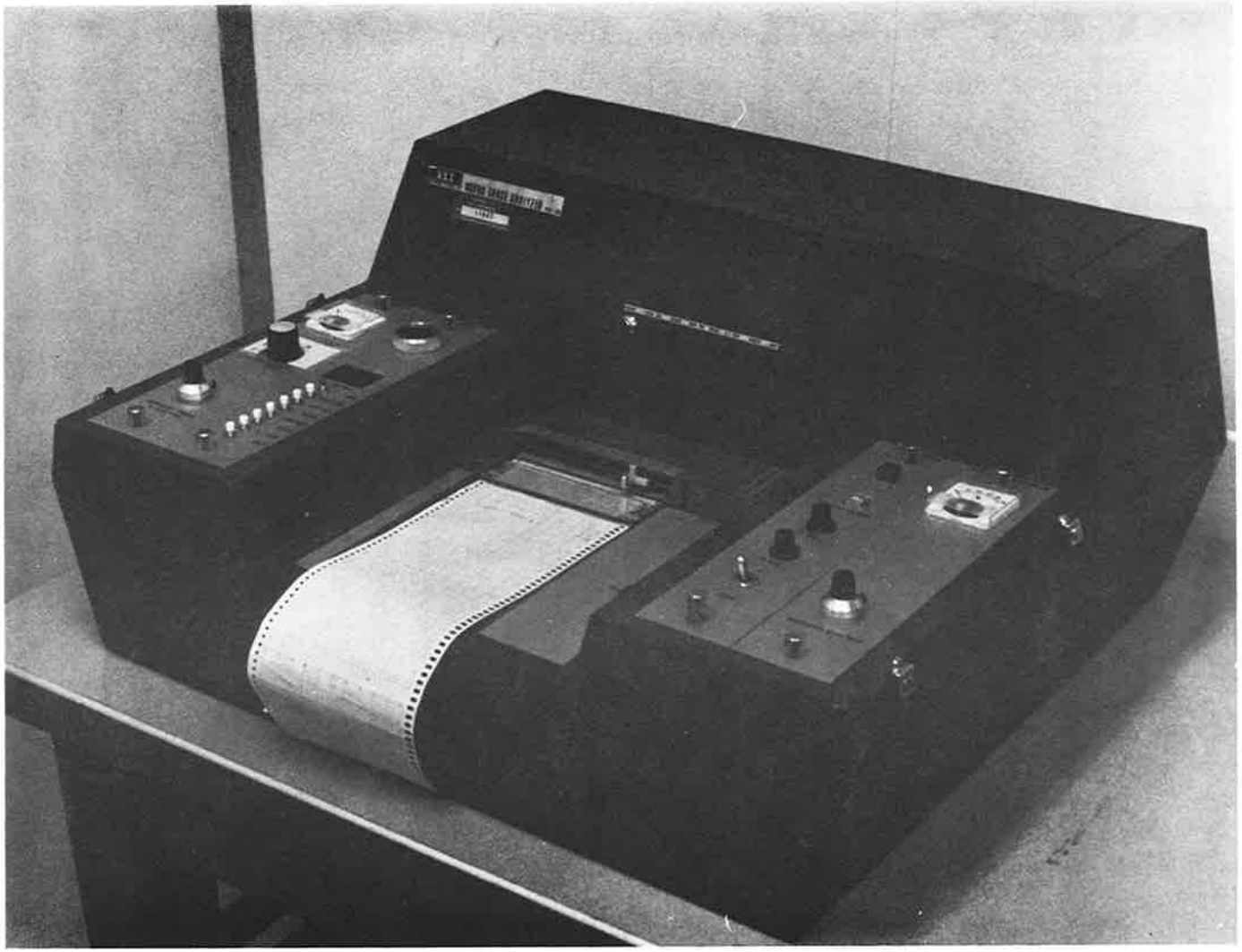

Figure 2. Hydronautics Vapor Trace Analyzer

valve then rotates 90° to the "detect" position where helium gas passes over the wire; the wire is heated, and the explosive vapor is desorbed and carried into the column for separation and detection. (A more detailed discussion of vapor concentrators is given in Appendix A).

Because of the presence of the gas chromatography column and the concentrator, the response time of the Hydronautics VTA is slow (about 18 seconds for EGDN). The instrument is costly (about $18,000.00), heavy (90 lbs.), and fairly complex.

Another type of commercial instrument employing the electron-capture detector is the Ion Track Instruments Model 27 Gelignite Detector (Figure 3). This instrument has a semi-permeable membrane in the inlet ahead of the electron-capture detector. The membrane reduces the concentrations of oxygen, nitrogen, and other light gases sufficiently to permit real-time sampling of atmospheric air without valves or rubber septa such as those used in gas chromatography. The ITI Model 27 is less expensive (about $1800), lightweight (about 10 pounds), and easy to use. Its sensitivity to dynamite (EGDN) is 0.8 ppb. It responds to many classes of electron-capturing compounds. However, it recovers much more slowly when the compound in question is an explosive, so in the hands of a trained observer it does have some degree of specificity.

The ITI Model 58 (Figure 4), is identical in concept to the ITI Model 27, except that the inlet, membrane, and detector can be heated to 200°C. This feature gives the instrument greater sensitivity and a more rapid recovery, since explosives vapors do not condense on the walls of the inlet, membrane, and detector housing. Its sensitivity to EGDN is somewhat better than that of the Model 27. The instrument is portable, easy to operate, and costs $5,500.

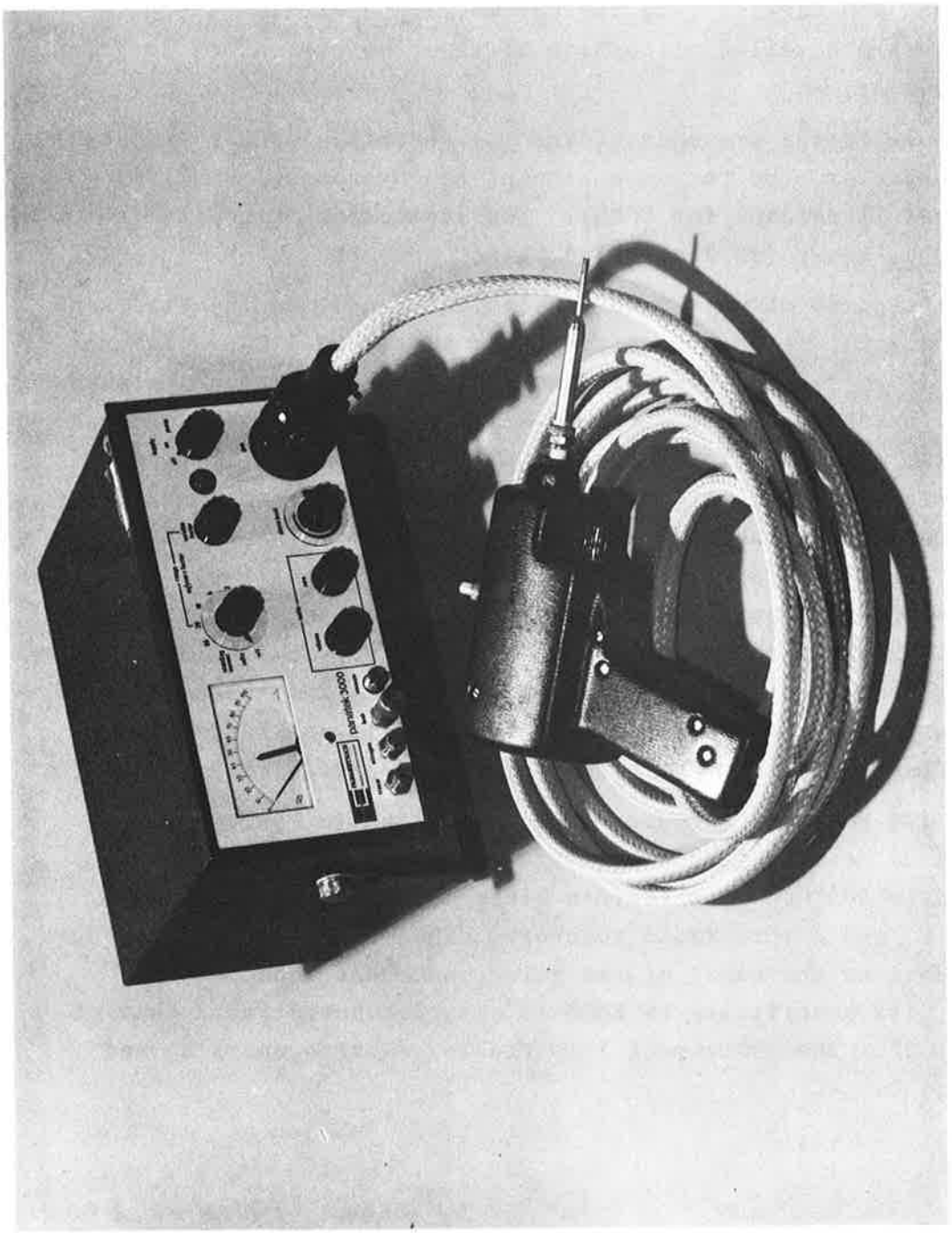

Figure 3. ITI Model 27 Gelignite Detector

Figure 4. ITI Model 58 Explosives Detector

3. DEVELOPMENT OF THE PROTOTYPE EXPLOSIVES DETECTOR

3.1 BACKGROUND STUDIES

In the course of a prior TSC study of commercially available explosives detectors,[2] a mass spectrometer with a field-ionization source and a membrane-separator inlet was used to produce a signature characteristic of dynamic effluent (Figure 5). The characteristic peaks at 30 and 46 atomic mass units were identified as being due to NO^+ and NO_2^+ ions respectively. The following questions had to be addressed: (1) whether some of these observed compounds were present in the natural effluent of dynamite; (2) how easily they could pass through the membranes; (3) whether there was any fragmentation of EGDN in passing through the membranes.

To answer these questions measurements utilizing various types of oxides-of-nitrogen detectors were carried out, described in detail in Reference 2, which readily established the following: (1) The amount of NO in an air sample saturated with dynamite effluent (assumed to be about 50 ppm EGDN)[3] was typically about 0.05ppm, and that of NO_2 less than 1 ppm. (2) Passage of a calibrated NO sample through a single membrane reduced the NO concentration by about 80%. (3) When an air sample saturated with dynamite effluent was applied to a single membrane, the NO concentration after passage was again only about 20% of the original 0.05 ppm level, so no significant fragmentation of EGDN into NO had taken place during its passage through the membrane. It was found, however, that heating the air stream containing EGDN to near 200°C produced a major effect, depending on the type of detector employed: the signal of an oxides-of-nitrogen detector increased; the signal of an electron-capture detector decreased, and at 220°C, disappeared completely. Other tests showed that the vapors of non-explosive, electro-negative compounds, such as perfume and nitrobenzene (in shoe polish), continued to be detectable by electron capture, even at temperature as high as 250°C. From these tests, it was concluded that EGDN was thermally decomposed into oxides of nitrogen; non-explosive vapors, on the other hand were stable. These observations led to

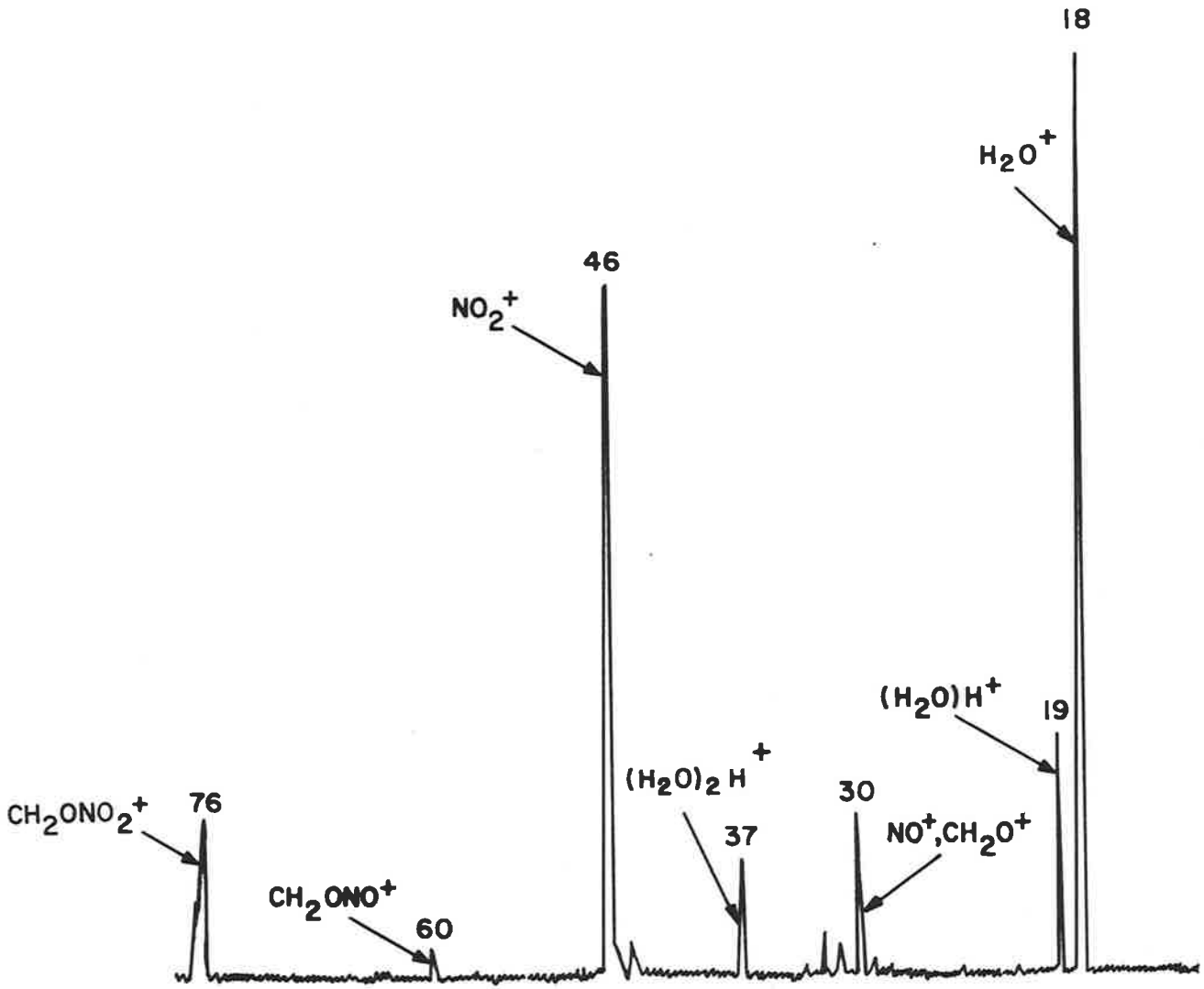

Figure 5. Field Ionization Spectrum of EGDN

Dynamite vapor was passed through the tube into an electron-capture sensor at various temperatures, monitored by a thermo-couple. It was established that a temperature of 250°C was sufficient to completely decompose the vapor. The length of the decomposition tube had to be adequate to ensure complete decomposition of the effluent at a sample flow rate of at least four liters per minute. This flow rate was necessary in order that dynamite could be detected at a distance of four feet from the inlet in a period of less than six seconds. This required flow rate was obtained with a Brailsford diaphragm pump. Various lengths of stainless-steel tubing were tried with it; 12 inches was found to be the minimum length for complete decomposition of dynamite vapor at 250°C.

Two Ion Track Instrument Model 27 electron-capture sensors were used, each consisting of a stainless-steel housing, one-half inch in diameter, containing a tritiated titanium radioactive foil mounted concentrically around a collector wire on the axis of the housing. The semi-permeable silicone membrane supplied with these sensors* is mounted in the sample inlet upstream of the detector (see Figure 6); it limits the concentration of oxygen reaching the detector to 10 ppm or less. This factor is important, because oxygen molecules are electron-capturing; their presence reduces the standing current and thus decreases the sensitivity. Pre-purified argon gas is the recommended carrier gas and the optimum flow rate is that which results in the maximum standing current.

3.3 PROTOTYPE EXPLOSIVES DETECTOR

A prototype explosives detector, shown in Figure 8, was fabricated based on the design as established above: it uses a dual-inlet system, with the heated inlet at 250°C, two ITI Model 27 electron-capture sensors with Philips semi-permeable membranes, argon carrier gas, and a sample flow rate of 4 liters per minute.

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*Philips Company, the Netherlands; General Electric's dimethyl silicone membrane material is equally effective.

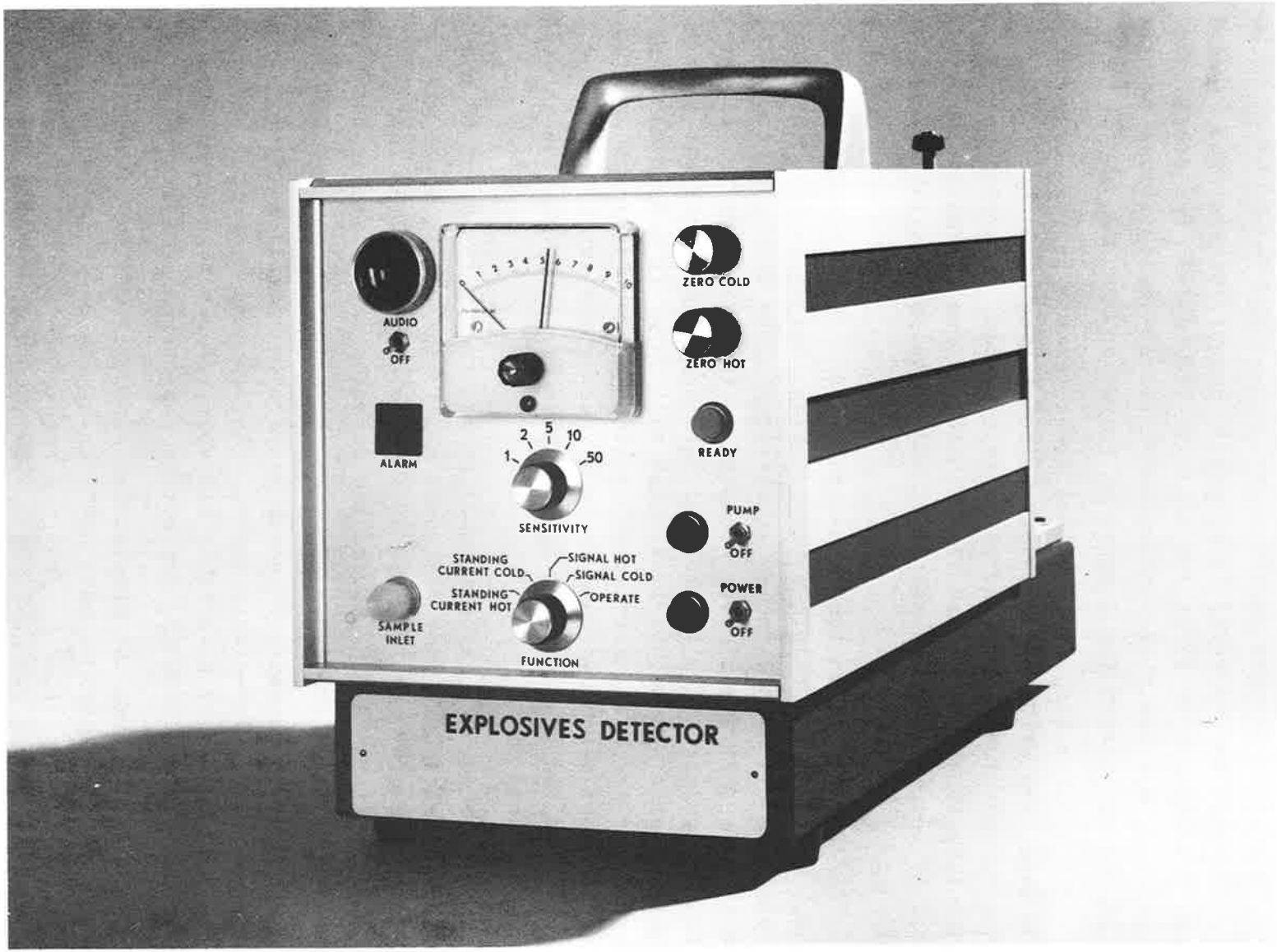

Figure 8. Prototype Explosives Detector

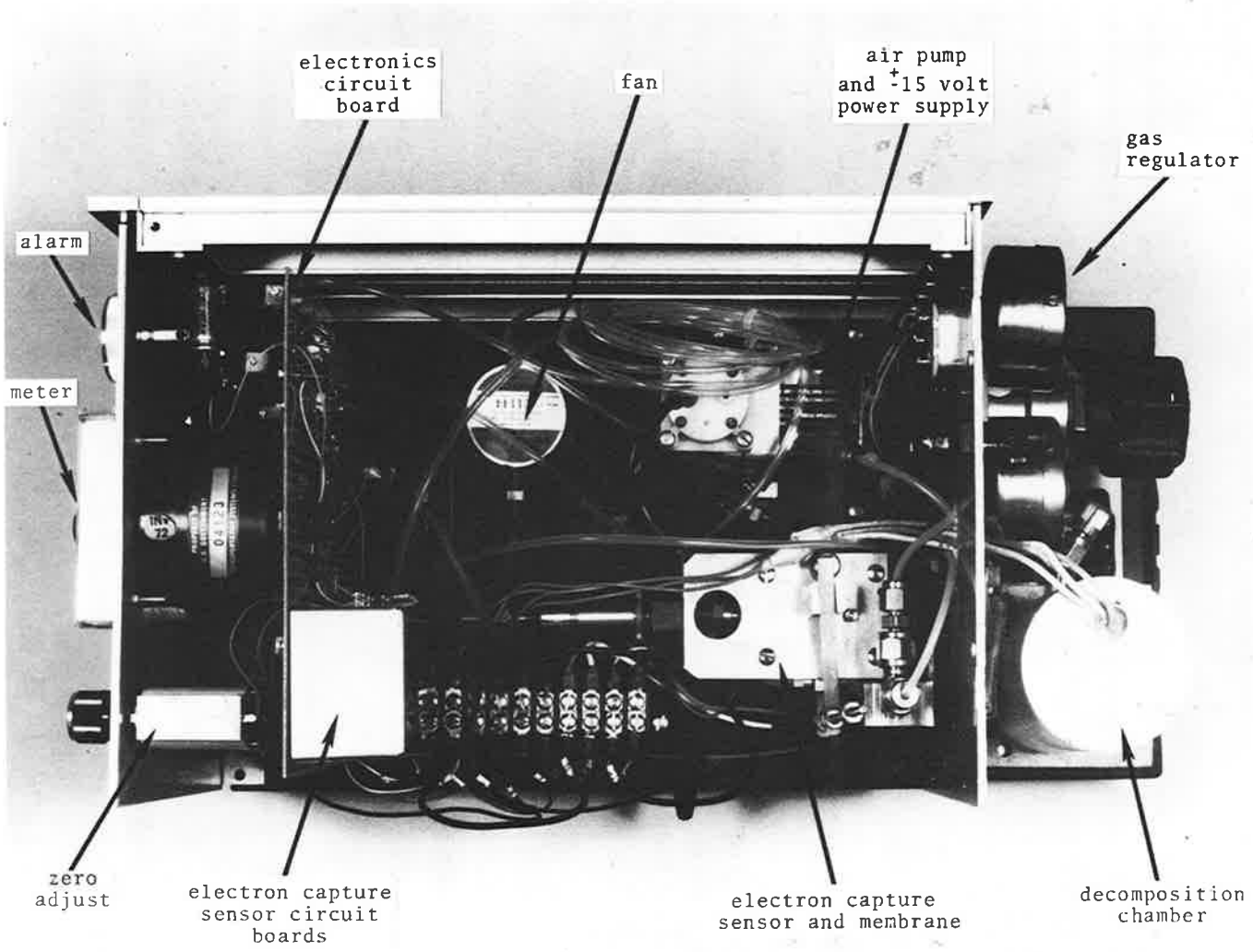

Figure 9. Top View of Components of Prototype Explosives Detector

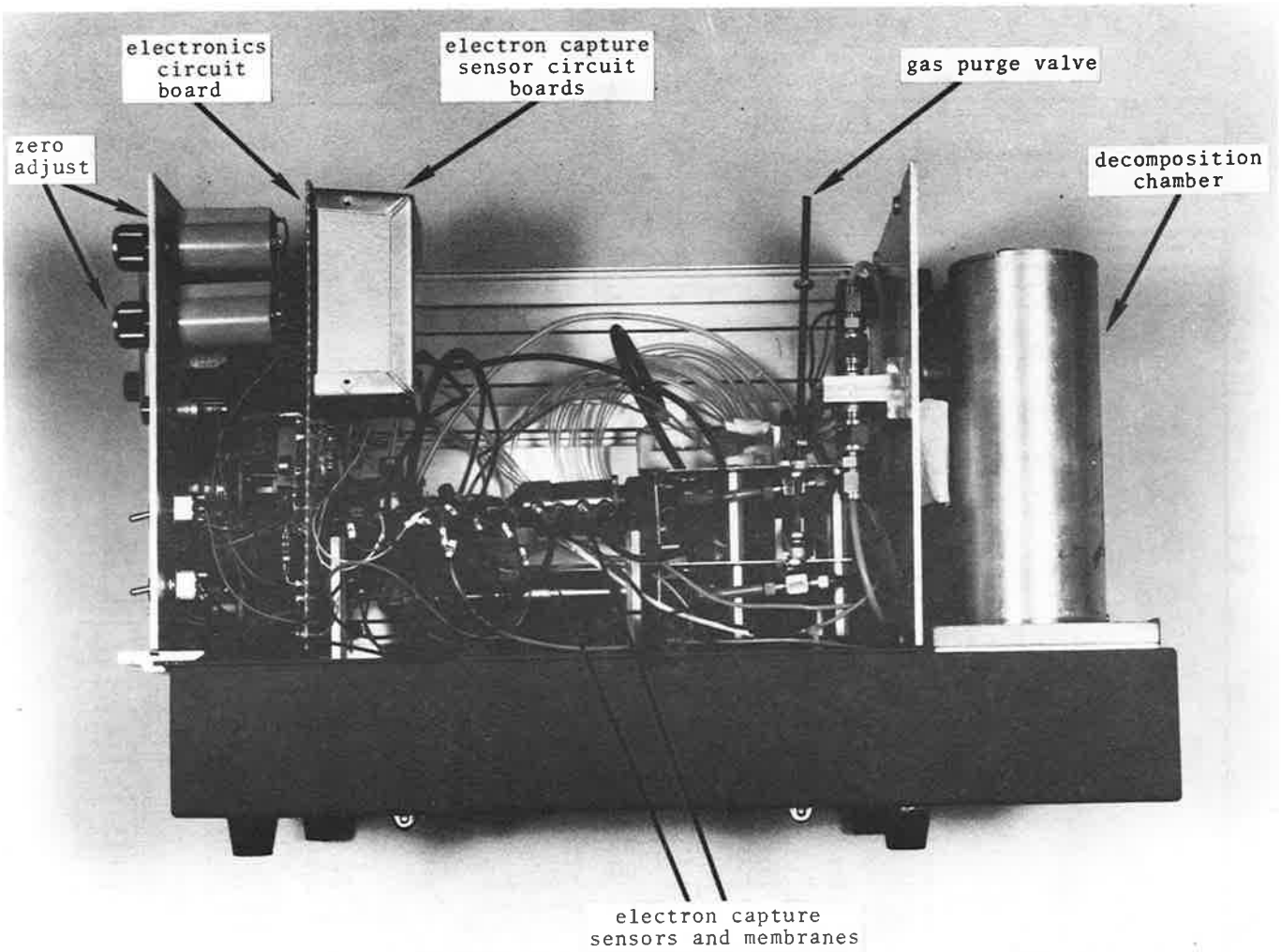

Figure 10. Side View of Components of Prototype Explosives Detector

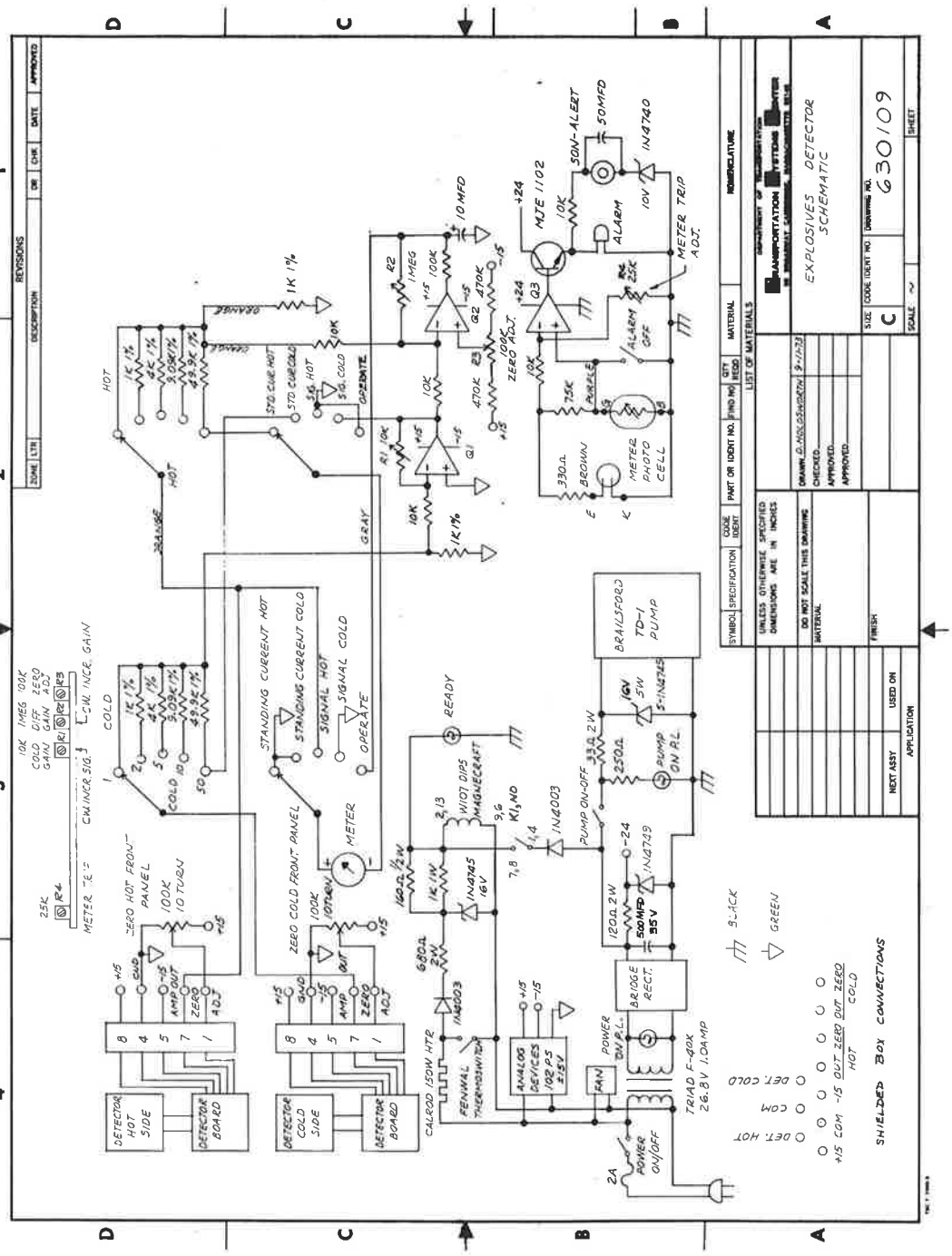

Figure 11. Schematic of Prototype Explosives Detector

4. RESULTS AND CONCLUSIONS

Table 2 compares the responses of the "hot" and "cold" electron-capture sensors. It can clearly be seen that a difference exists only for explosive electronegative compounds.

The unit's sensitivity was determined by injecting 1 cc of air saturated with dynamite vapor (52 ppm* of EGDN) into the inlet. Taking into account the dilution of the sample and the magnitude of the response, the instrument's sensitivity to EGDN was found to be 0.1 ppb.

The prototype instrument has been shown to respond rapidly to dynamite, smokeless powder, TNT, C-4, and Swiss sheet explosive. Its reliability as a detector of bombs in suitcases has been tested at some length; three sticks of 40% dynamite placed within a packed suitcase for 40 minutes can be detected, and an alarm sounded, in less than 6 seconds.

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*At room temperature the saturated vapor pressure of dynamite is 4×10^{-2} torr; this pressure corresponds to a concentration of 52 ppm in air at atmospheric pressure.[3]

TABLE 2. RELATIVE RESPONSES OF ELECTRON-CAPTURE SENSORS

Substance	Sensor Response: No. 1 Room Temp	No. 2 250°C	Response Difference (No. 1 - No. 2)
Explosives			
Dynamite	900	0	900*
TNT	8	0	8*
Swiss Sheet	96	0	96*
Smokeless Powder	600	0	600*
C-4	120	0	120*
Non-Explosives			
Electronegative:			
Nitrobenzene	695	695	0
m-dinitrobenzene	100	100	0
2,4-dinitrotoluene	0	0	0
p-nitrotoluene	725	725	0
Trichloroethylene	800	800	0
Carbon Tetrachloride	800	800	0
Bath Lotion ("Fabulous")	150	150	0
Perfume ("Jeuvelle")	390	390	0
Nitric Acid Vapor	600	600	0
Non-Electronegative:			
Methyl Ethyl Ketone	0	855	negative #
Ethyl Alcohol	0	0	0
Isopropyl Alcohol	0	0	0
Acetone	0	465	negative #
Ethyl Acetate	0	205	negative #

* Response difference positive. Detector alarms.

\# Response difference negative (Due to formation of electronegative decomposition product in sensor No. 2). Detector does not alarm.

REFERENCES

1. Bell, R. Neil, "Field Evaluation of the Contribution of the Trained Dog/Handler Team to Airport Security," Federal Aviation Administration Report FAA-RD-73-5, January 1973.

2. Cline, J.E., Hobbs, J.R., and Barrington, A.E., "Laboratory Evaluation of Detectors of Explosives Effluents," Department of Transportation Report DOT-TSC-OST-72-27, November 1972.

3. Dravnieks, A., "A Bomb Detection System Study." Federal Aviation Administration Report FAA-ADS-81, October 1966.

4. Cline, J.E., Hobbs, J.R., and Barrington, A.E., "An Electron-Capture Technique Specific for Explosives Detection," Carnahan Conference on Electronic Crime Countermeasures, Lexington, Ky., April 1973.

APPENDIX

VAPOR CONCENTRATORS

A.1 BACKGROUND

The successful performance of a vapor concentrator in the Hydronautics VTA suggested that a vapor concentrator might be a valuable accessory to other explosives detectors. The concentrator as an independent sampling device is inexpensive and has a wide potential application in the screening of selected areas in airports and of the interior of airplanes in the event of a bomb threat. Furthermore, under appropriate conditions, it can also enhance the sensitivity of explosives detectors. For this reason, work was initiated on a study of concentrators.

A.2 CONCENTRATOR CONFIGURATION

Two concentrator configurations which could be attached to a "gun" containing a sampling pump and subsequently to the prototype explosives detector, were designed and tested. The first configuration, termed "wire" concentrator (Figures A-1, A-2), consisted of a teflon housing with a central platinium wire, 2 inches long. The wire could be heated electrically by a pulsed supply (Figures A-3, A-4) to release adsorbed material. The second configuration, termed "column" concentrator, shown in Figure A-5, consisted of a glass tube, 0.25 inches in diameter, 3 inches long, which could be filled with a column of various adsorbents, packed between cotton plugs. To release adsorbed material, the tube was placed in an aluminum block, sandwiched between two resistance coils powered by a variable transformer.

A.3 TESTING AND RESULTS

All tests were performed with the prototype explosives detector.

(a) Wire Concentrator

A 5-second sampling of EGDN vapor by the concentrator produced a detector reading of more than 5,000 divisions. However,

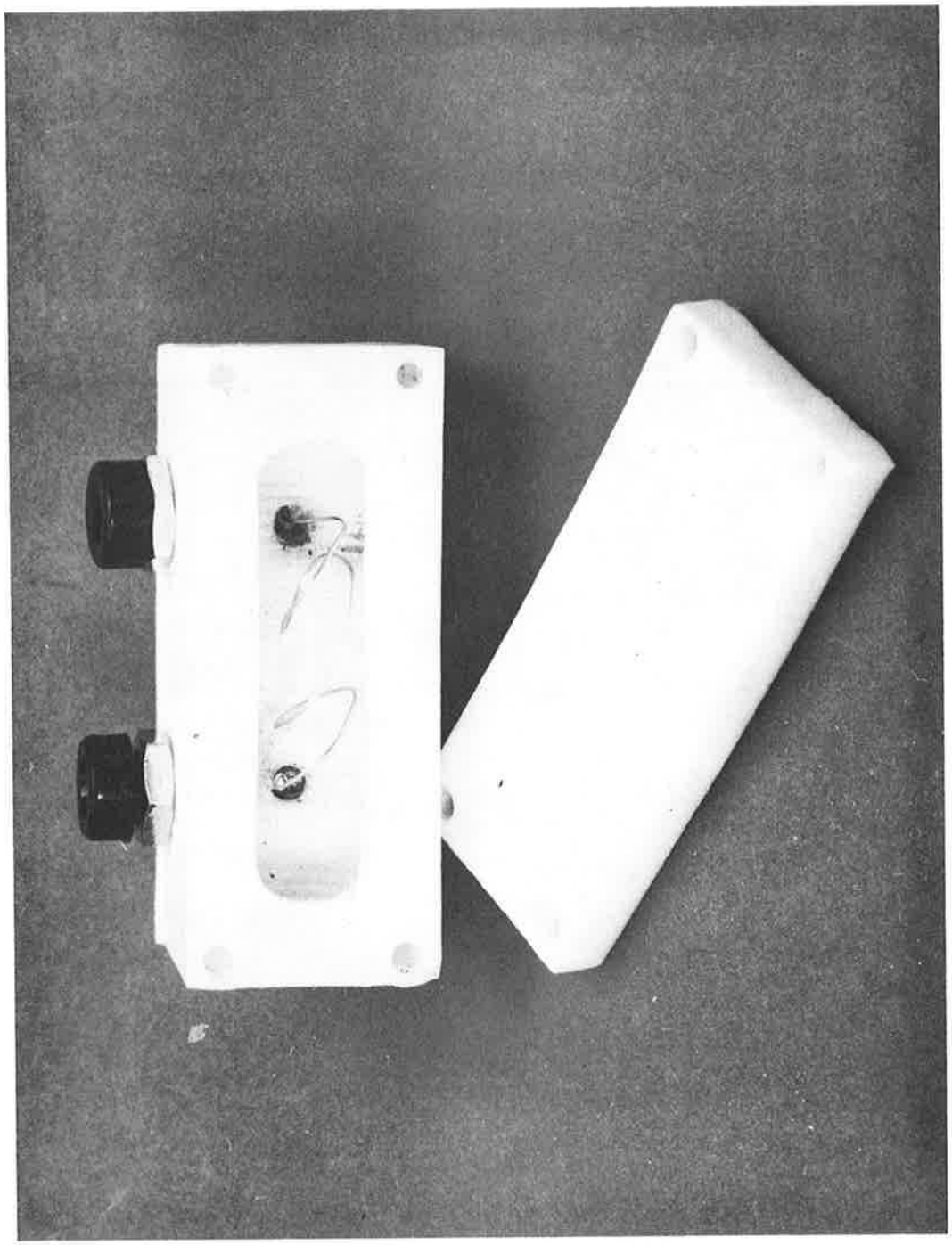

Figure A-1. Wire Concentrator

when the 5-second sampling was followed by 30-seconds sampling of air, the detector reading decreased to 200 divisions; after a 2-minute sampling of air, no detection whatever was registered. Thus, although platinium was a good adsorber, it did not retain adsorbed material on prolonged exposure to a flow of air. (It should be noted that the concentrator of the Hydronautics VTA operates only on a 5-second sampling period prior to the desorption cycle, with no subsequent sampling exposure.)

(b) Column Concentrator.

Adsorbent materials tested included Johns Manville Chromosorbs 101, 102, 104, and glass wool. Of the three chromosorbs, 102 was superior; however, there were serious drawbacks: desorption upon heating was non-uniform and the detector response was broadened; also, desorption continued at a finite rate after the heating was discontinued, which produced a non-zero detector response. Glass wool performed best. Retention of adsorbed material was not degraded seriously by subsequent flushing with air, release on heating was rapid and uniform, and ceased quickly when heating was discontinued. Figure A-6 shows the results of desorption on heating the glass wool after one minute of sampling at the seam of an attache case containing an empty plastic bag in which water-gel explosive (15% EGDN) had been stored. (The empty bag had been left in the attache case for two months.) The solid line represents the output of the cold sensor; the dotted line that of the "hot" sensor. The signal was sufficient to peg the prototype explosives detector on the medium sensitivity range.

A.4 CONCLUSIONS

The results of the tests demonstrate the feasibility of using a concentrator at a site remote from an explosive detector. The detector must be specific for explosives, since the concentrator will also collect vapors of innocuous non-explosive materials. Such a concentrator would be useful at airports, where a large number of searches might be required, (e.g., after receipt of a

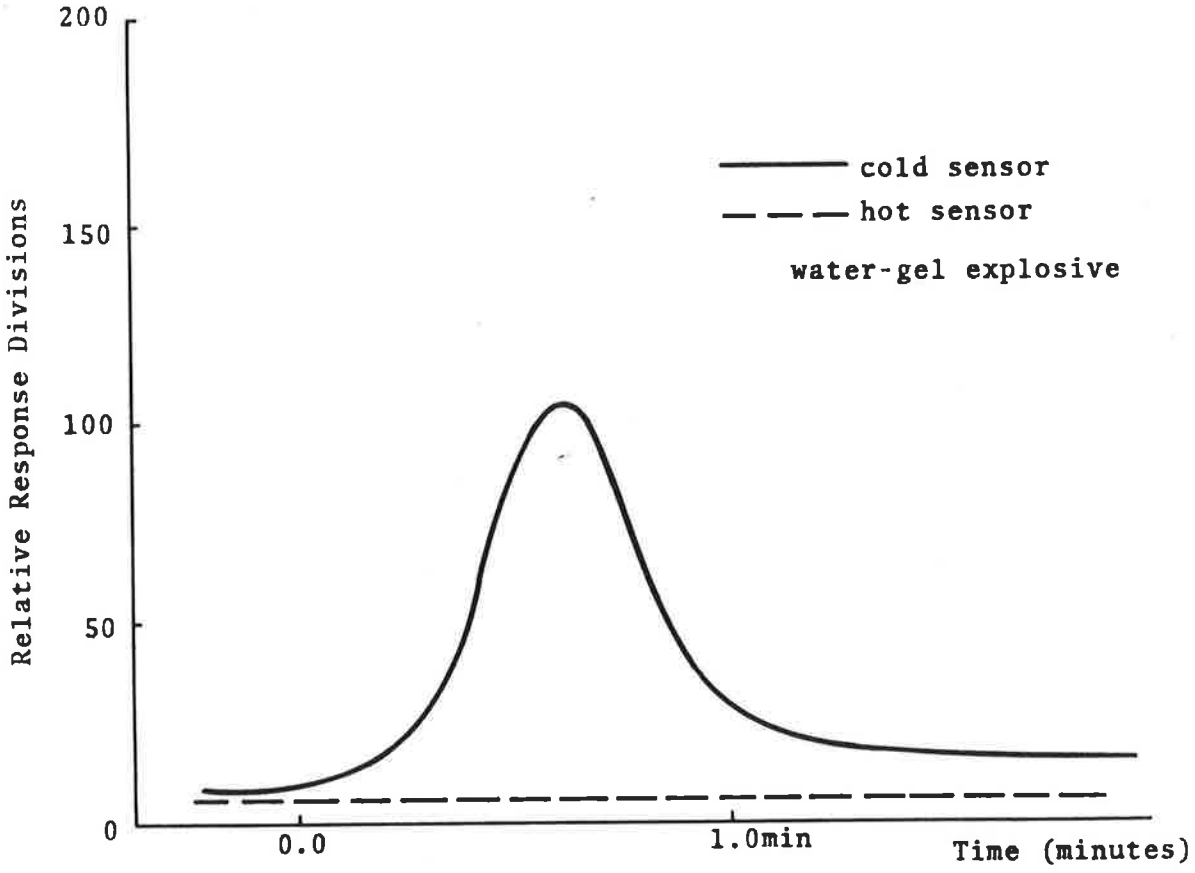

Figure A-6. Sensor Response of Prototype Explosives Detector to EGDN from Glass-Wool Column Concentrator

bomb threat). In such a case, concentrators on sampling "guns" could be used to check a large number of suitcases at the airport's various baggage facilities, and then be returned to a central explosives detector for testing. Sampling personnel could carry as many as a dozen concentrators, and rapidly sample at many locations, before returning to the detector. This procedure would be more convenient and considerably less expensive than the use of multiple detector units.