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EXPLORATORY ANALYSIS OF VAPOR IMPURITIES FROM TNT, RDX AND COMPOSITION B

W. F. O'Reilly, et al

Cold Regions Research and Engineering Laboratory
Hanover, New Hampshire

October 1973

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13. ABSTRACT

Vapors collected from TNT, RDX and composition B explosives maintained at 25°C and 70°C were analyzed using a gas chromatographic/mass spectrometric technique to identify the most prominent vapor components. A gas chromatograph equipped with electron capture and flame ionization detectors also was used to screen vapors for components present at concentrations below the sensitivity of the GC/MS. Cyclohexanone which could have significance in development of an explosive or mine detector was identified in vapors from RDX and composition B. Water vapor, carbon dioxide, probably nitrous oxide and a number of unidentified compounds were evolved from all samples. It may be possible to identify these unknown compounds present at trace levels, but extreme analytical care and precaution to avoid contamination would be required. In this regard, differentiation between compounds which are incorporated into the explosive matrix during manufacturing and those which are adsorbed from air by the explosive at a later time is also required.

14. Key Words

Composition B	Gas detectors	TNT
Explosives	Gases	Trace gas detection
Gas chromatography	RDX	

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iv

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CONTENTS

	Page
Introduction	1
Materials and methods	1
Description of samples	1
Collection of vapors	2
Analytical procedure	3
Results and discussion	4
Literature cited	6
Appendix A	7
Appendix B. Retention characteristics of chromatographic columns for analysis of organic compounds	7
Abstract	19

ILLUSTRATIONS

Figure

1. Inlet system for transfer of explosive vapors to gas chromatograph 2
2. Experimental arrangement for analysis of explosive vapors: GC/MS, GC . 3

TABLES

Table

I. Explosive samples 2

EXPLORATORY ANALYSIS OF VAPOR IMPURITIES FROM TNT, RDX AND COMPOSITION B

by

W.F. O'Reilly, T.F. Jenkins, R.P. Murrmann, D.C. Leggett and R. Barrierra

INTRODUCTION

Locating military explosives and mines by sensing vapors emitted into the atmosphere is an approach to detection that is currently under investigation.[1,6,7,10] In considering this concept, it is envisioned that the explosive vapor itself, vapors from impurities in the solid explosive, or volatile decomposition products of the explosive will form a unique chemical signature. However, very little is actually known of the composition of vapors from military explosives. Even values for vapor pressure of the explosive material in the ambient temperature range[1,7] have been extrapolated from data obtained at elevated temperature.[4,12] This general absence of pertinent information relevant to the development of trace gas detection systems is in no way due to a lack of interest in this area,[1,4] but rather to the fact that until recently techniques and instrumentation have not been available with sufficient sensitivity to analyze explosive vapors at extremely low concentrations. Decomposition products such as nitrogen dioxide, nitrous oxide, carbon dioxide and various ring-structured byproducts have been studied,[1,6] as have explosive components in the solid phase.[1,5] The identity and consequent behavior of those components and impurities in the vapor phase have, however, not been extensively studied.

This investigation was undertaken to identify the components in the vapor from military explosives: TNT, RDX and composition B. Initially, attempts at positive identification were made using gas chromatographic/mass spectrometric methods. Later work was conducted on an exploratory basis using only gas chromatographic techniques with higher sensitivity in order to provide a basis for more refined studies in the future.

MATERIALS AND METHODS

Description of samples

Samples of TNT, RDX and composition B (40% TNT, 59% RDX and 1% wax) were provided for this study by MERDC.* The characteristics of the samples are summarized in Table I. Part of the RDX sample, originally in the size of chips, was ground so that analysis was conducted on two types of RDX. The selection of the samples was based on a previous experiment conducted at MERDC† in which a continuous weight loss of the explosive material was observed at room temperature. This indicated the possibility that constituents in the vapor emitted by the samples might

*Charles L. Collins, Mine Detection Division, USA MERDC, Ft. Belvoir, Va.
†Personal communication, Charles L. Collins.

Table I. Explosive samples.
Charles I. Collins, USA MERDC, Ft. Belvoir, Va.
Source of samples: Picatinny Arsenal.

Sample	*Lot no.*	*Description*
RDX	HOL-SR-4-4624	Coarse form
TNT	BC-3476	Grade 1
Composition B	35-471	Grade A

indeed be detectable. While the samples were stored in closed containers, no unusual provisions were made at CRREL to prevent adsorption of contaminants from laboratory air. This precaution was thought to be unnecessary since the explosives had apparently received no special handling since manufacture.

Collection of vapors

One gram of explosive material was placed into the center of a 12.6-mm-OD by 15.24-cm-long quartz tube. To secure the sample, the open ends of the tube were plugged with silanized glass wool. The tube was fitted using Teflon ferrules and equipped with two stainless steel Whitey valves to provide gas flow control (Fig. 1). A helium source was connected to one valve while the outlet valve was connected directly to the inlet of a gas chromatograph. A tubular heater was used around the quartz tube to provide temperature control during experiments. Before the sample was placed in the tube, the entire assembly was baked out at high temperature and tested at 70°C using the same analytical procedures as for sample analysis. This was to minimize the degree of background contamination. After a sample was placed in a tube, the tube was flushed with zero helium for several minutes. The valves were then closed for one hour to allow accumulation of vapors from the explosive, after which the vapors were transferred by helium flow directly to the head of a chromatographic column maintained at -75°C. Initially, the explosive was maintained at room temperature (25°C). The entire sequence was then repeated with the sample at 70°C in order to increase vapor concentration for detection purposes. This temperature is below the melting point of the various explosives. The same sample was used and the sample assembly maintained intact throughout the analysis sequence to provide compatibility of results and avoid contamination by labor[illegible] after initiation of the analysis procedure.

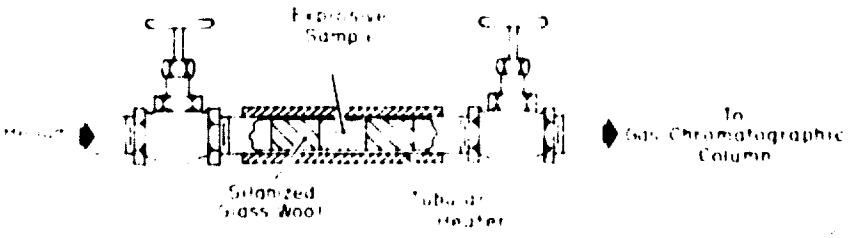

Figure 1. Inlet system for transfer of explosive vapors to gas chromatograph.

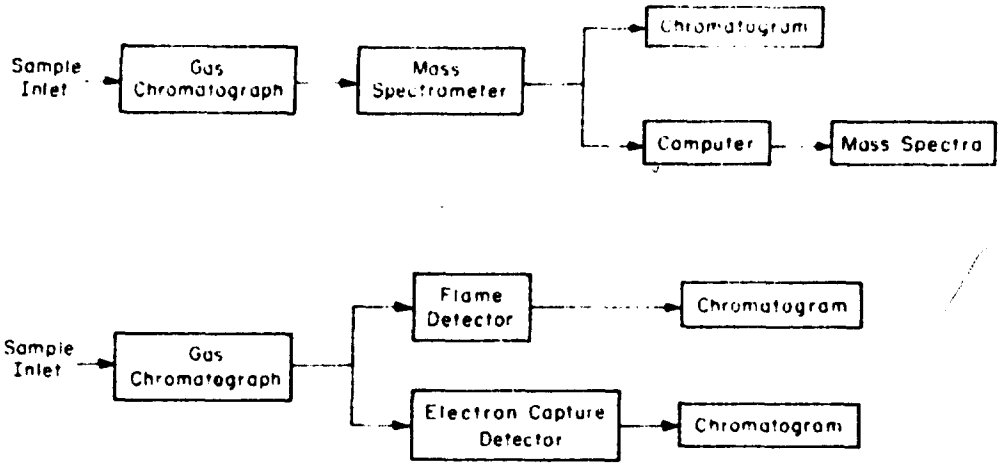

Figure 2. Experimental arrangement for analysis of explosive vapors: GC/MS (top), GC (bottom).

Analytical procedure

The three stainless steel chromatographic columns used were:

1. 0.61 m × 0.318-cm-OD tubing filled with 100-120 mesh Porapak-Q*
2. 2.74 m × 0.318-cm-OD tubing filled with 100-120 mesh Durapak* (Carbowax 400/Porasil C)
3. 0.91 m × 0.318-cm-OD tubing filled with 15 percent DC-200† on 100-120 mesh Anakrom Abs.

These columns were selected for their ability to separate different classes of compounds. The Porapak-Q column will separate water and its performance is not degraded by water. It is useful for analysis of some inorganic gases as well as organic compounds in the C_1-C_5 molecular weight range. The performance of the Durapak column is not seriously affected by water but water does not elute as a defined peak; however, this column is useful for analysis of compounds in the C_4-C_{10} hydrocarbon range and also for C_1-C_4 alkylated aromatics. The DC-200 column was selected for its ability to separate aldehydes, ketones, other oxygenated compounds, and higher molecular weight hydrocarbons. The Porapak-Q column was programmed to increase temperature at 4 degrees per minute from −75°C to 170°C. The Durapak and DC-200 columns were programmed at the same rate from −75°C to 130°C and 170°C, respectively. Each column was heated at its final temperature for at least one hour and a blank run conducted before another explosive sample was run.

During the course of this work, two different instruments were used. The first was a Perkin-Elmer 270 gas chromatograph/mass spectrometer (GC/MS). The GC/MS is equipped for continuous scanning of the GC effluent with on-line computer processing of MS data (Fig. 2). The advantage of this instrument is that positive identification can be obtained by comparing mass spectra obtained for components eluted from the gas chromatograph with literature values. The sensitivity of the instrument, however, is lower than that of some other types of gas chromatographic detectors. The second instrument employed to obtain higher sensitivity was a Perkin-Elmer 900 gas chromatograph with both a flame ionization detector, which is highly sensitive to all organic compounds, and an electron capture detector which is ultrasensitive to electrophilic compounds such as those containing nitro groups. At this stage of the study no attempt was made to identify these compounds. Rather, the intent was to determine whether other components were present at levels below the

*Waters Assoc., Inc., Framingham, Mass.
†Dow-Corning, Midland, Mich.

detection limit of the mass spectrometer. The gas chromatograph was configured to split the effluent from the chromatographic column 1:1 to each type of detector. Use of the two detectors in parallel is an advantage in that relative responses give additional information about unknown components.

RESULTS AND DISCUSSION

Gas chromatograms typical of those obtained for each type of sample and analytical procedure are shown in Appendix A (Fig. A1-A7). The chromatograms obtained at 25 C and 70 C for each explosive using a given analytical column are aligned for purposes of comparison with that obtained at 70 C for a blank run without sample. Peaks believed to represent compounds originating from the explosives are shaded to distinguish them from those due to background contamination indicated by the 70 C blank run.

The chromatograms obtained using the gas chromatograph/mass spectrometer system are given in Figure A1. Using the Porapak-Q (PPQ) column, carbon dioxide and water were detected in the vapor from all samples in amounts in excess of those found during blank runs. The most water and carbon dioxide was evolved from composition B and the least from RDX. This difference may only reflect factors such as sample surface area or porosity. At 70 C, nitrous oxide (N_2O), presumably a decomposition product, was detected from RDX and composition B but not from TNT. While no N_2O was observed at 25 C, data obtained later using the higher sensitivity electron capture detector suggest that N_2O is evolved at room temperature even from TNT. Using the Durapak column, no compounds were observed except for one unidentified component from RDX at 70 C which appeared from the mass spectrum to be an alcohol. Using the DC-200 column, cyclohexanone was positively identified from mass spectra of vapors evolved from composition B at 70 C but no cyclohexanone could be detected at 25 C. More cyclohexanone was evolved from the chip form than from the ground form of composition B. Since the chips were used to make the ground form, the lower concentration of cyclohexanone in composition B (ground) was probably due to more volatilized loss during storage because of the higher surface area. A small amount of cyclohexanone was also detected from the RDX at 70 C. The occurrence of cyclohexanone in vapor from composition B could be of considerable significance in development of a mine detection device. Cyclohexanone is used in recrystallization and, consequently, it is a common impurity of RDX.* While this "tag" is present in solid composition B, it would be difficult to design a high sensitivity detector to discriminate cyclohexanone from other organic compounds. However, cyclohexanone is unique in the sense that it is not a naturally occurring trace gas present in the atmosphere. To further evaluate the potential of cyclohexanone as a "tag," the amount of cyclohexanone commonly evolved from composition B and composition B charged mines should be determined.

The results of the GC/MS work indicate that the major vapor constituents evolved from TNT, RDX and composition B were carbon dioxide, water vapor and nitrous oxide. Cyclohexanone was also emitted by composition B and, to a lesser extent, by RDX. However, it is not known whether the evolution of these components could account for the continuous loss of weight observed for the samples.* The absence of more peaks in the various chromatograms does not mean that other impurity compounds were not present in the vapor. Rather, it more likely means that additional compounds were present at concentration levels below the detection limit of the mass spectrometer total ion monitor, about 10^{-7} grams. The retention characteristics of the analytical columns used in this study are given in Appendix B. These compounds were used for column calibration and represent many of the types of chemicals which would have been detected if present at sufficient concentration.

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*Personal communication, Charles I. Collins.

Although vapor from the explosives was analyzed simultaneously using both flame ionization and electron capture detectors, it was not practical, because of the complexity of the chromatograms, to display the response of the two detectors together in the same illustration. Accordingly, chromatograms obtained using the flame ionization detector are grouped in Figures A2-A4 while those from the electron capture detector are represented by Figures A5-A7. With the PPQ column (Fig. A2) no peaks were observed that could not be accounted for by background contamination indicated by the blank chromatogram. Using the Durapak column, no compounds were observed which could be attributed to the sample of TNT (Fig. A3a). For the RDX and composition B samples (Fig. A3b-d), however, two peaks at longer retention times were found at 70 C which obviously originated from the samples. Judging from the peak area, the amount of these two compounds in the ground composition B was about a factor of 30 higher than in the vapor from RDX. The concentration in the vapor from the composition B chips was a factor of 10 greater than observed for the ground composition B. In the sample of chips, one compound was clearly evident even at 25 C. When the DC-200 column was used, a substantial number of peaks were present for all samples, both at 25 C and 70 C (Fig. A4). While the effort required to identify these peaks was beyond the scope and resources available for this project, it is believed that cyclohexanone is represented by the large tailing peak in the center of the chromatograms for RDX and composition B. This peak is present at 25 C for both RDX and composition B chips indicating that cyclohexanone can be detected at ambient temperature. In addition, several other organic compounds have been detected in the vapor from the explosives and are detectable at the sensitivity of a flame ionization detector. Thus, with the flame detector, it is feasible to detect these compounds, although a more detailed study is required for identification.

In using the electron capture detector, the complexity of the chromatograms increased considerably in conjunction with higher detector sensitivity. A casual glance at the chromatograms obtained for blank runs shows that contamination was a problem. The peaks are probably due to freons and other electrophilic compounds present in trace amounts in the purge gas used to transfer vapor to the chromatograph inlet. Using the PPQ column (Fig. A5), most peaks can be accounted for by contamination. The exceptions are the first peak at short retention time for both composition B samples (Fig. A5c, d) and the last peak at the longest retention time for all types of samples. The initial peak observed for vapor from composition B is thought to represent nitrous oxide observed earlier using the mass spectrometer detector. With the Durapak column there was little evidence for the presence of impurities in vapors from the explosives except for the case of the composition B samples (Fig. A6c, d) where higher molecular weight compounds are indicated at longer retention times. A number of peaks were observed for vapor from all samples when the DC-200 column was used. The first peak in each chromatogram is probably due to nitrous oxide. Peaks at longer retention times are unidentified. Based on retention time criteria, some of these peaks were initially thought to be due to DNT and TNT isomers detectable in TNT vapor[1] but their appearance in vapor from the RDX sample dismisses this possibility. The close similarity in appearance of the chromatograms of all of the samples seems to illustrate another type of contamination problem. Although some of the peaks undoubtedly represent vapors unique to a given explosive, many other compounds are probably due to adsorption of vapors by the explosives after manufacturing. Thus, care must be used to differentiate between compounds that are an integral part of the solid explosive matrix and those that result from exposure of the explosive following manufacture. Nevertheless, these results illustrate the presence of trace impurities in explosive vapors and show that analysis of explosive vapors using electron capture and flame ionization detectors is possible, provided that extreme care is used in development and application of proper analytical procedures, and that the importance of sample history is appreciated.

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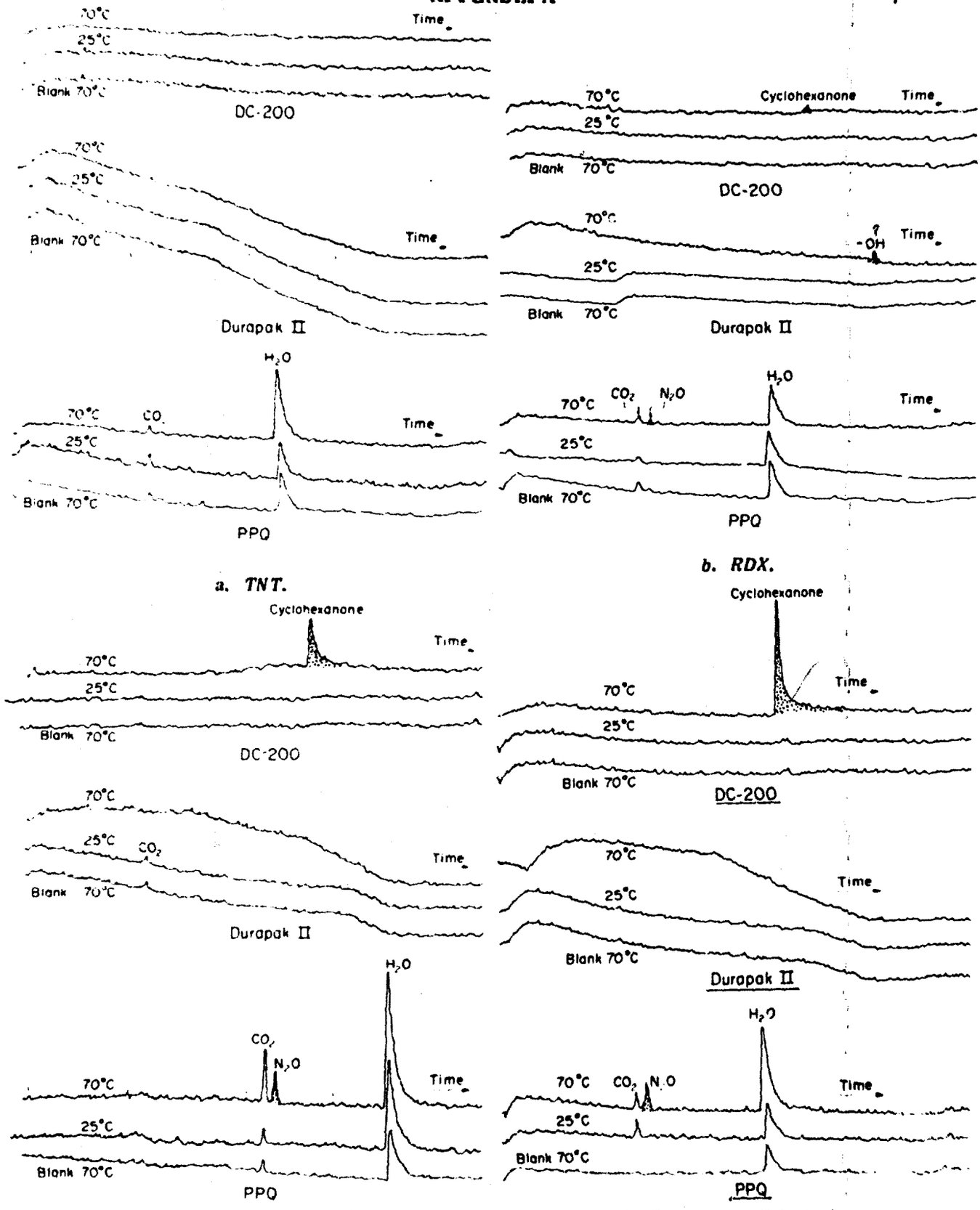


a. *TNT.*

b. *RDX.*

c. *Composition B (ground).*

d. *Composition B (chips).*

Figure A1. *Gas chromatograms obtained for vapors from various explosives using mass spectrometer total ion current monitor.*

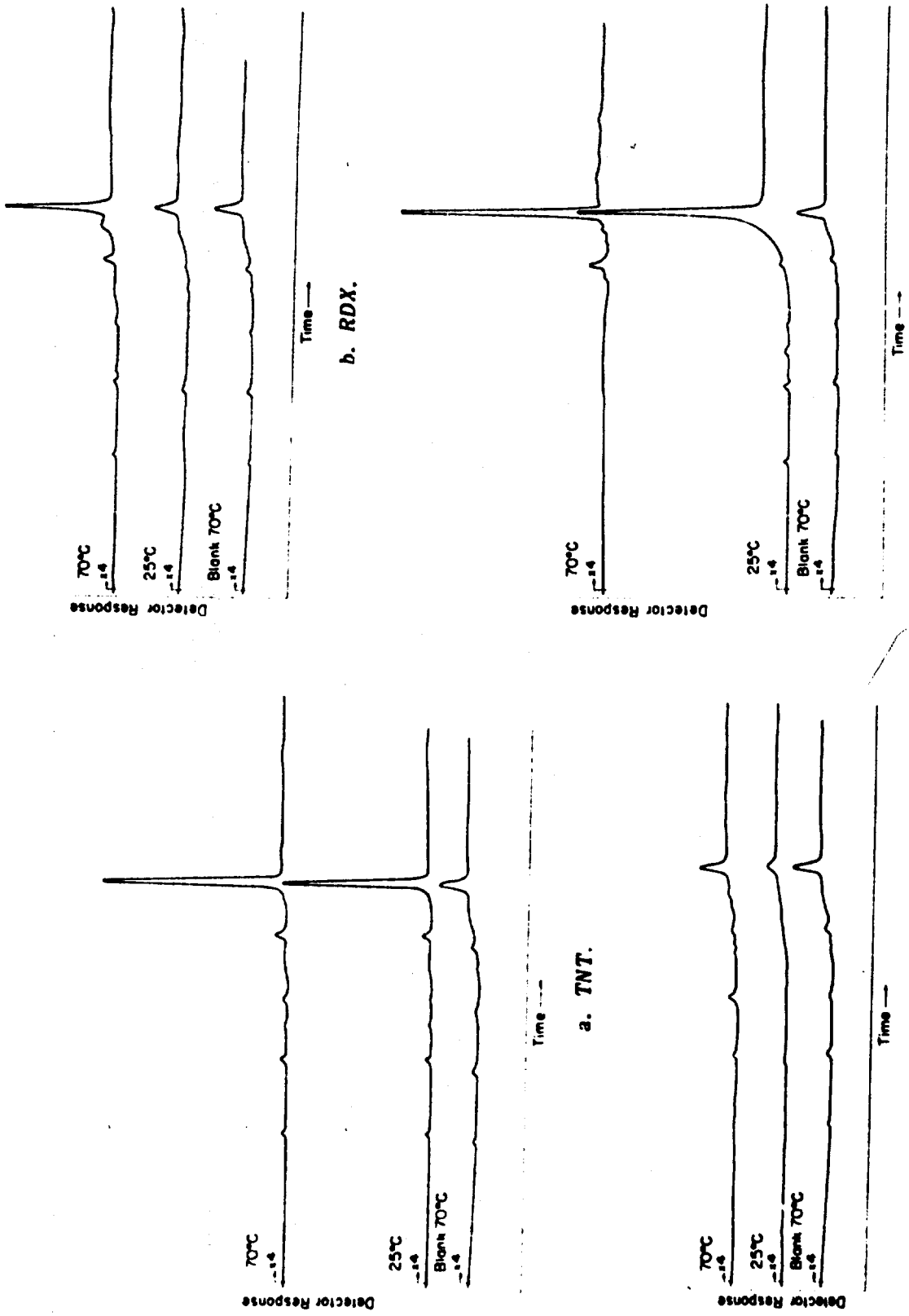


Figure A2. Gas chromatograms obtained for vapors from various explosives using Porapak-Q column and flame ionization detector.

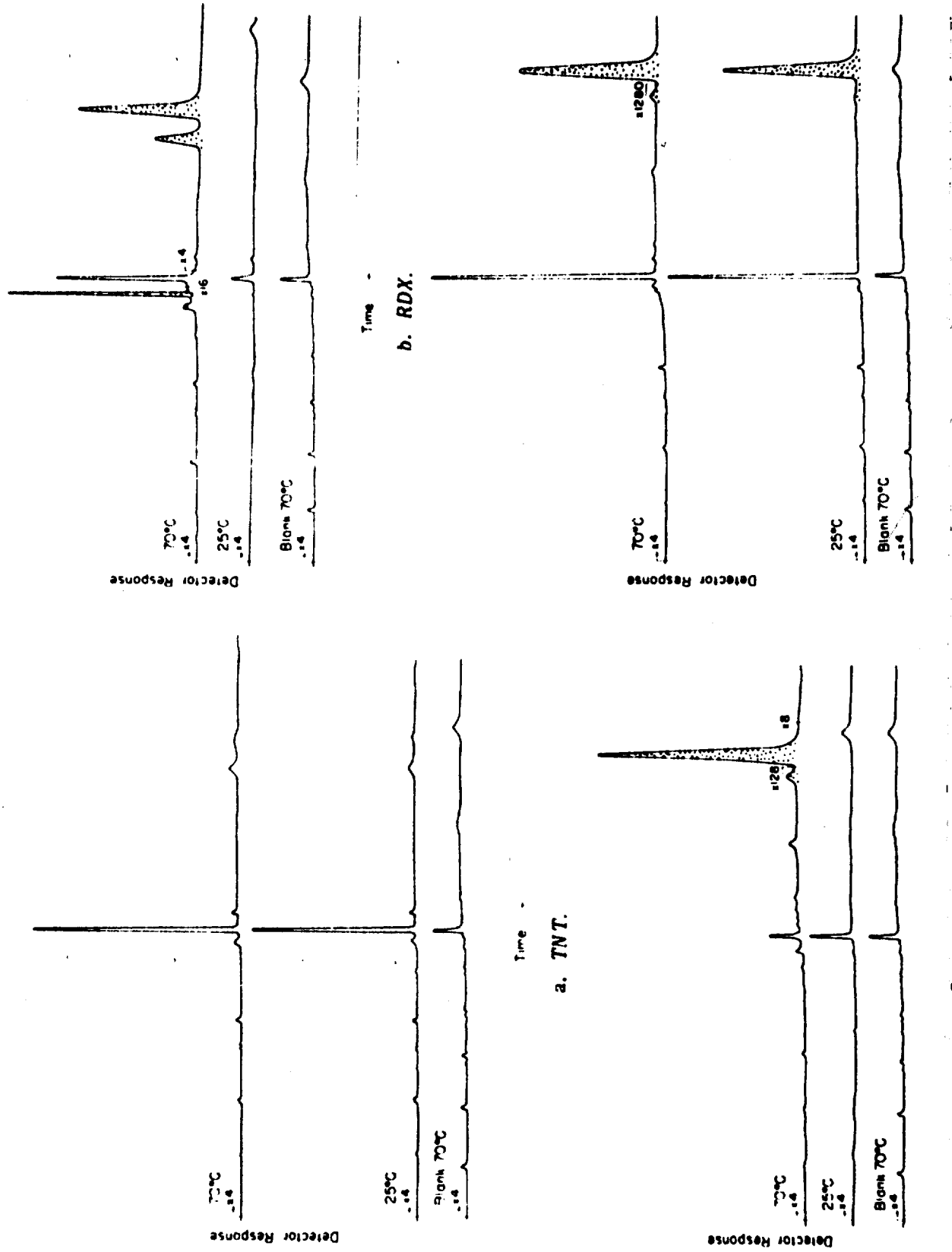


Figure A3. Gas chromatograms obtained for vapors from various explosives using Durapak column and flame ionization detector.

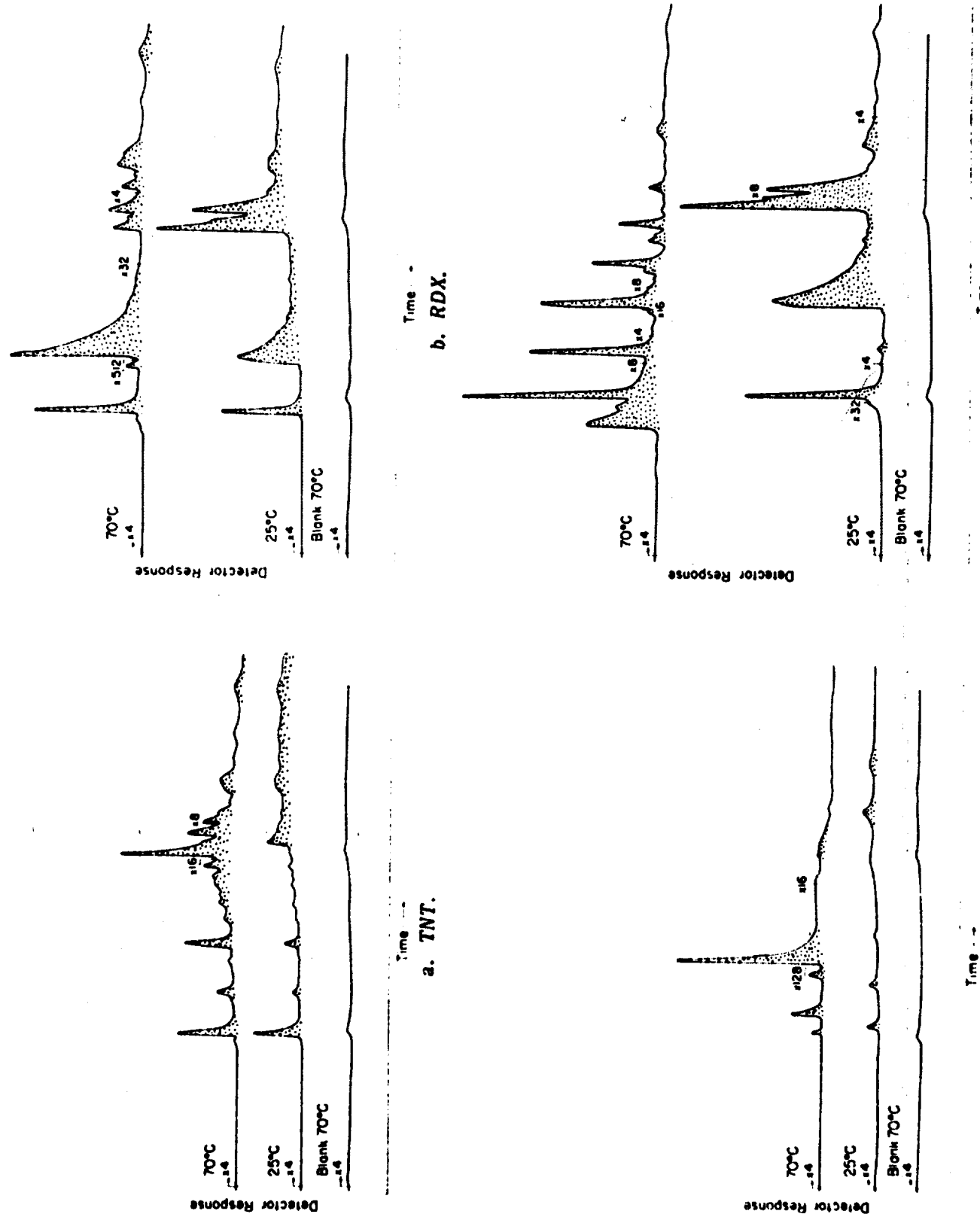
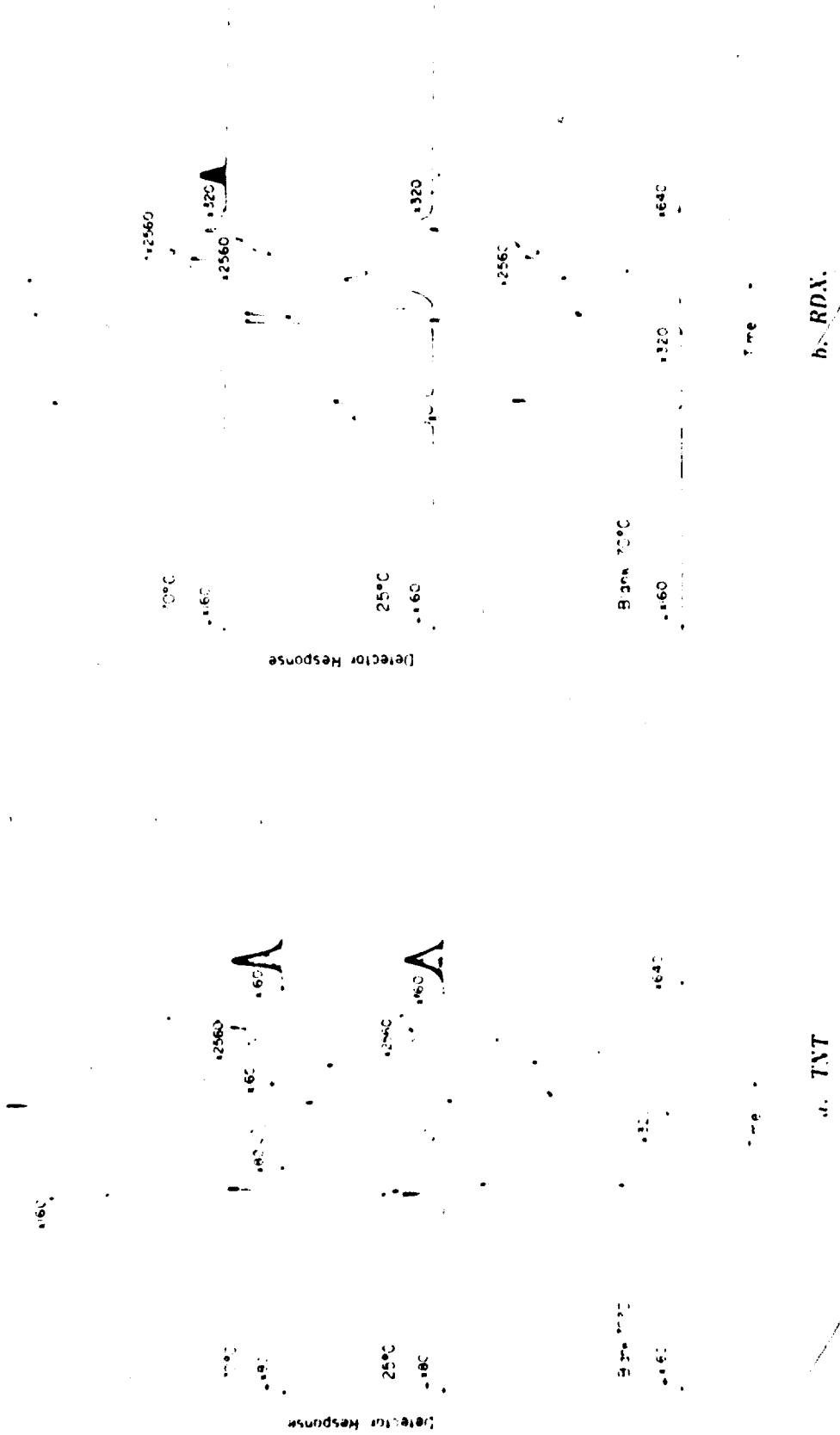


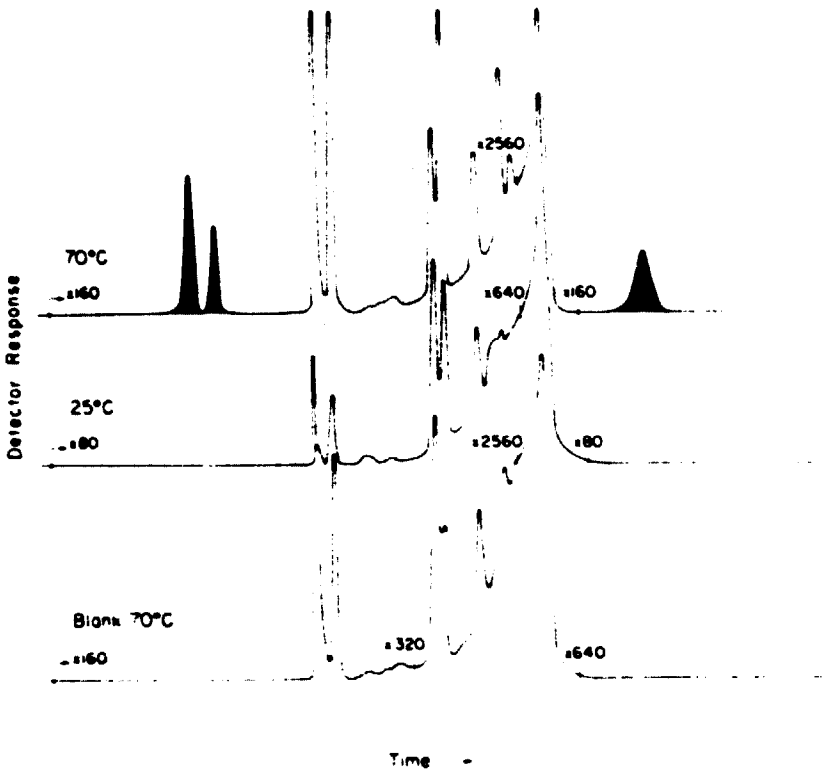
Figure A4. Gas chromatograms obtained for vapors from various explosives using DC-200 column and flame ionization detector.



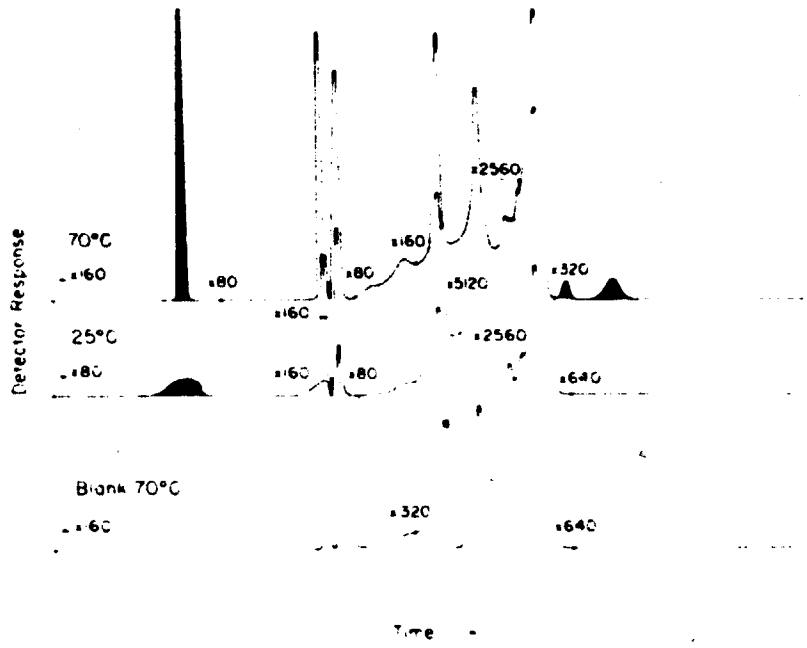
a. TNT

b. RDX.

Figure A5 Gas chromatograms obtained for vapors from various explosives using Porapak-Q column and electron capture detector.



c. Composition B (powder).



d. Composition B (chips).

Figure A5 (cont'd). Gas chromatograms obtained for vapors from various explosives using Porapak-Q column and electron capture detector.

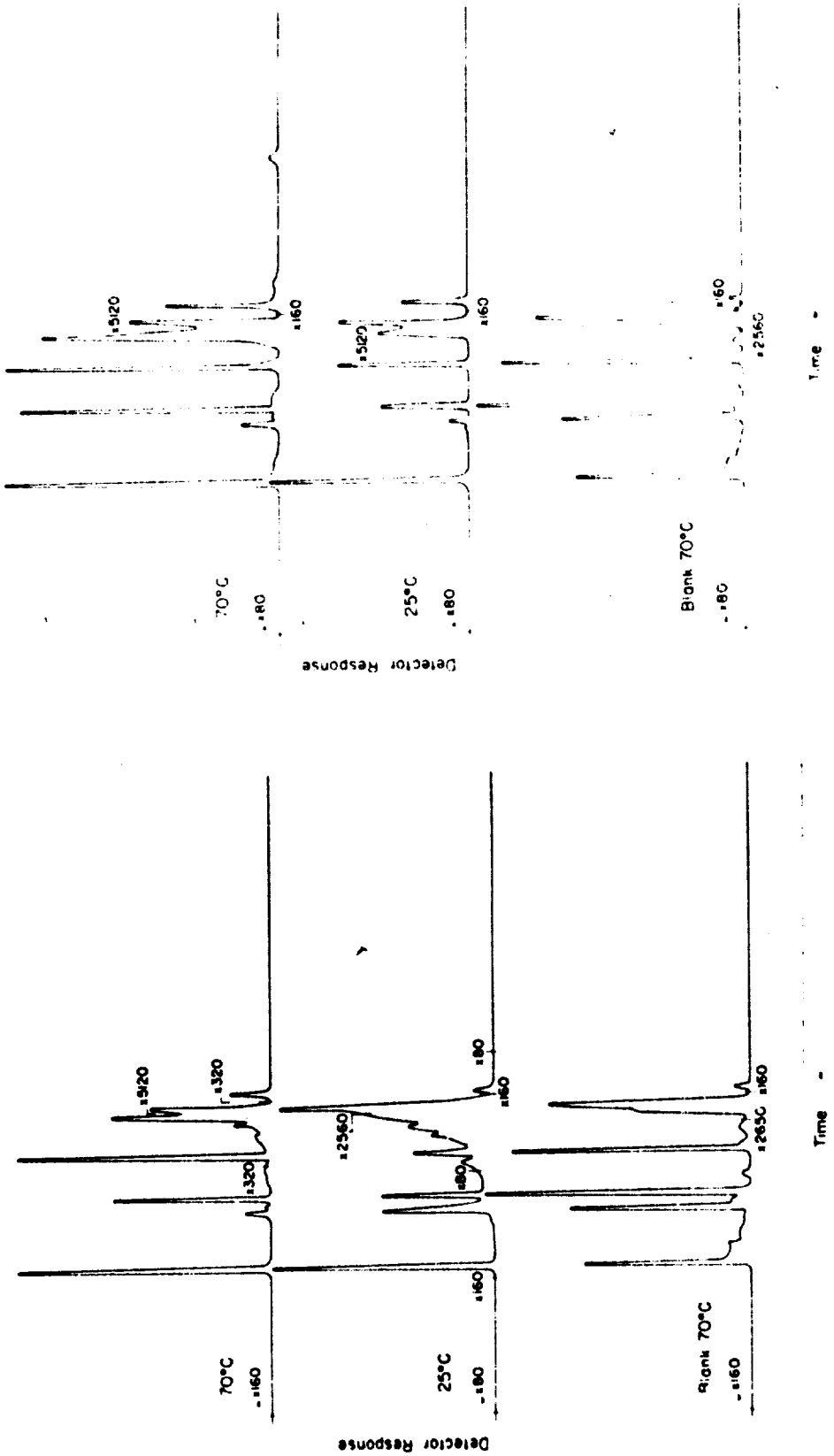
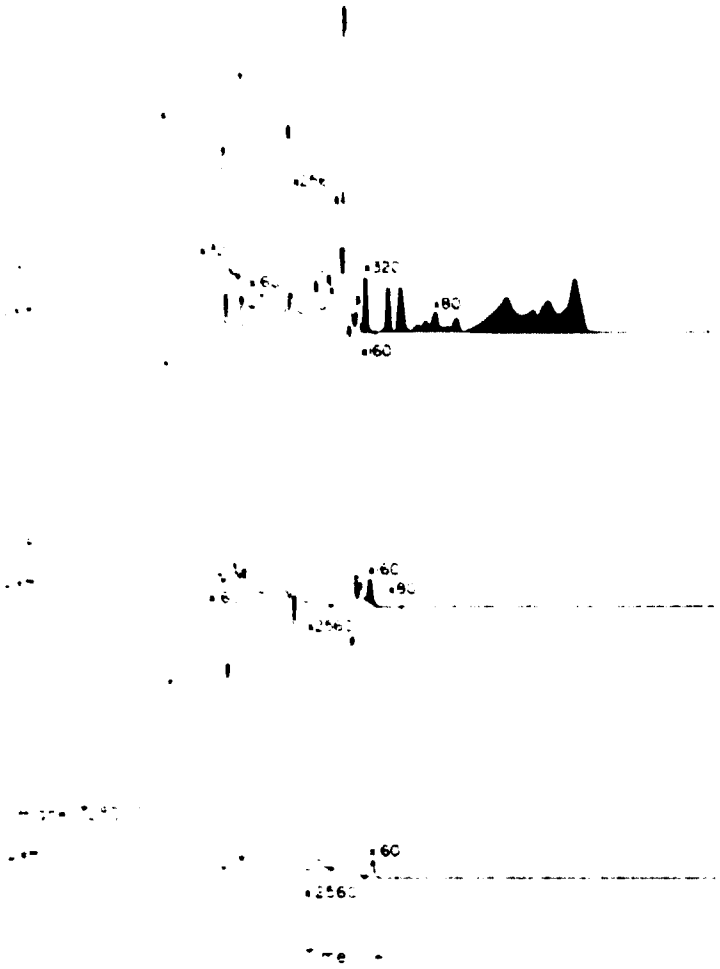
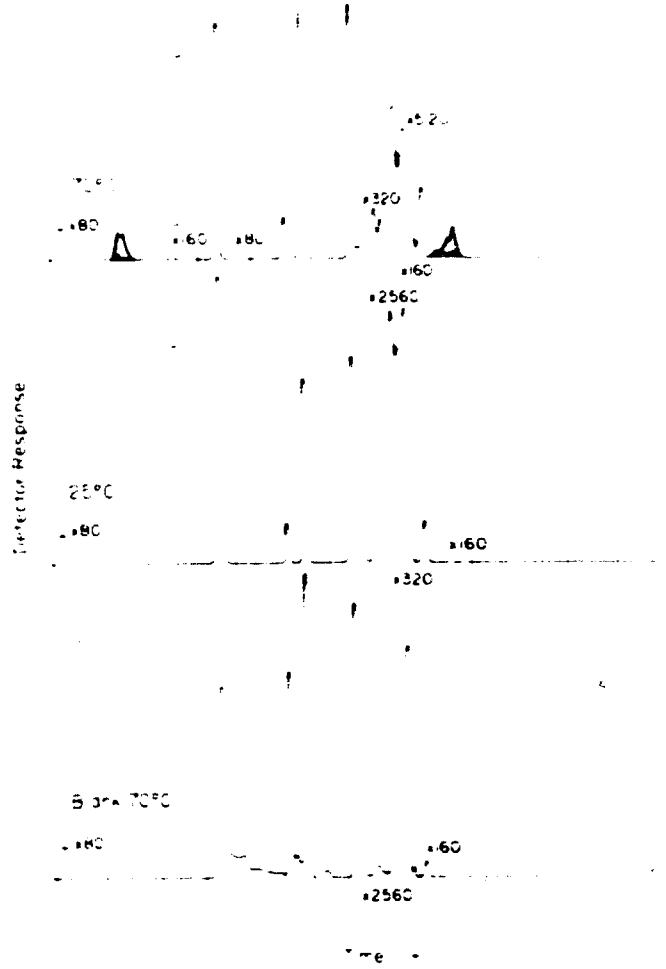


Figure A6. Gas chromatograms obtained for vapors from various explosives using Durapak column and electron capture detector.

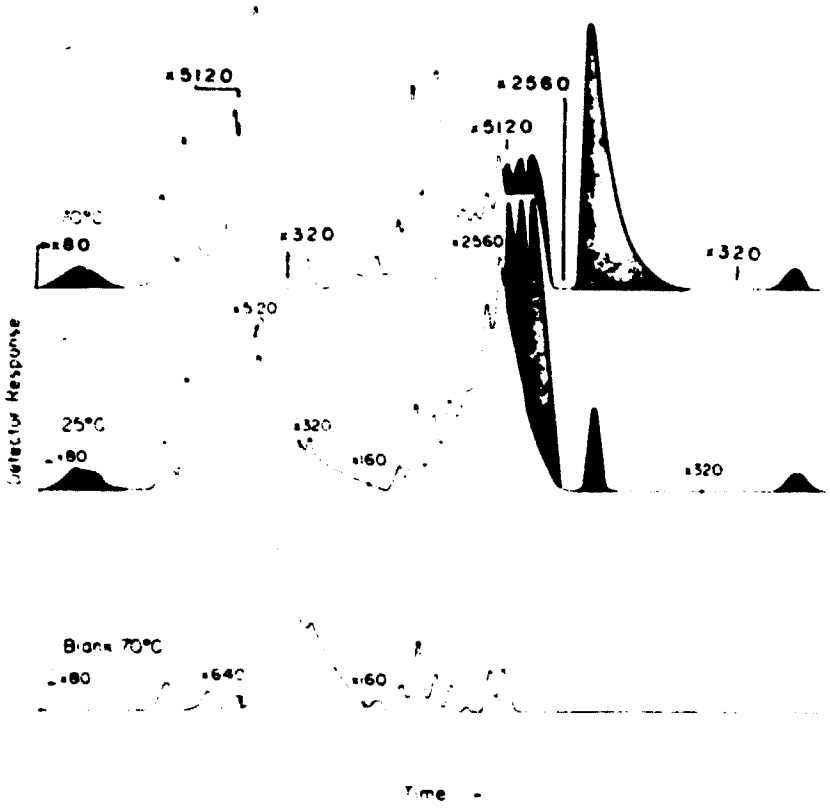


c. Composition B (ground).

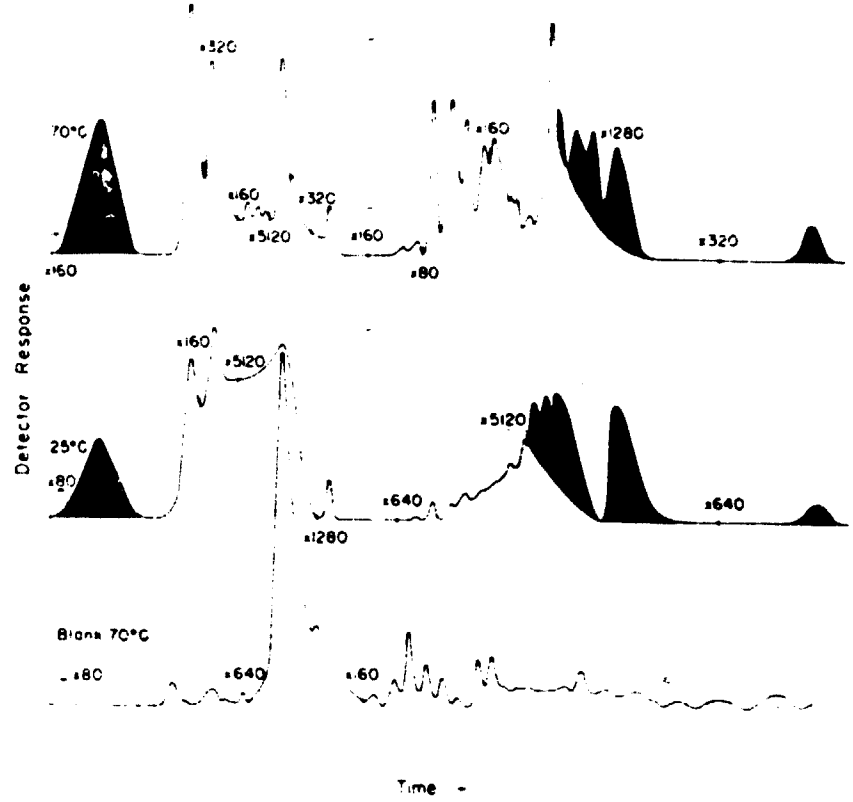


d. Composition B (chips).

Figure A6 (cont'd). Gas chromatograms obtained for vapors from various explosives using Durapak column and electron capture detector.

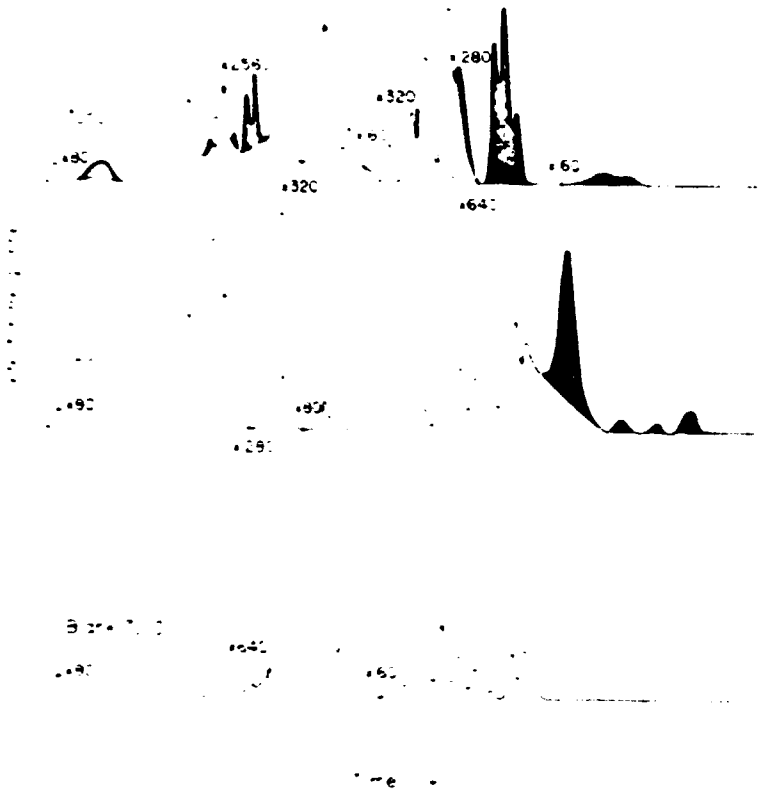


a. TNT.

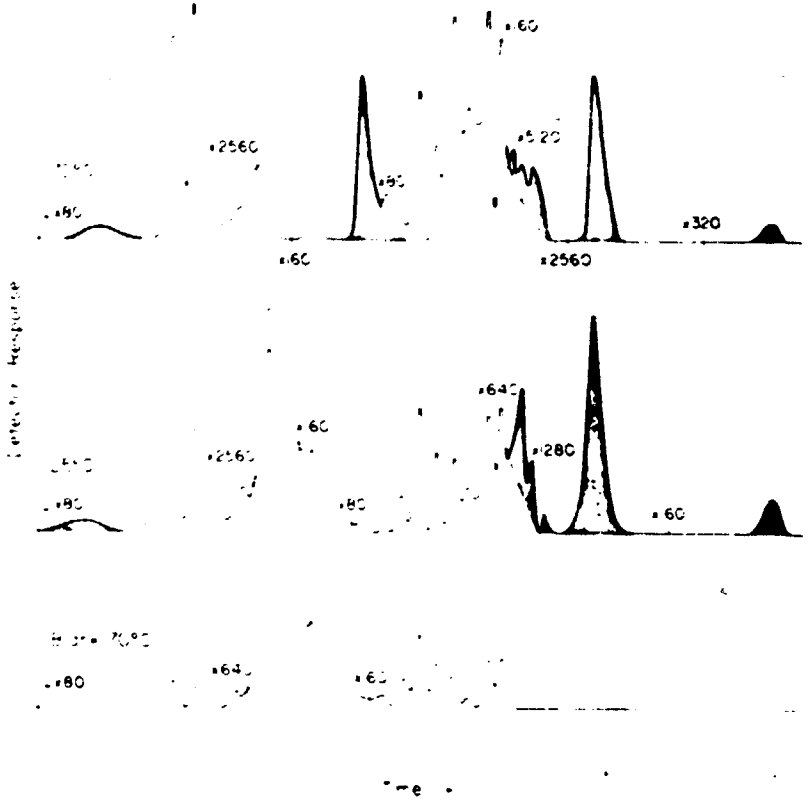


b. RDX.

Figure A7. Gas chromatograms obtained for vapors from various explosives using DC-200 column and electron capture detector.



c. Composition B (powder).



d. Composition B (chips).

Figure A7 (cont'd). Gas chromatograms obtained for vapors from various explosives using DC-200 column and electron capture detector.

APPENDIX B. RETENTION CHARACTERISTICS OF CHROMATOGRAPHIC COLUMNS FOR ANALYSIS OF ORGANIC COMPOUNDS

Table BI. Retention characteristics of Porapak Q chromatographic column.

Flow rate 20 milliliters/min, temperature -75 C to +170 C, program rate 10 C/min.

Time (min)	*Compound*	*Time (min)*	*Compound*	*Time (min)*	*Compound*
15.6	Water	22.2	2-Pentene	26.7	Heptene
17.3	Formaldehyde	22.2	Isoprene	27.0	Dimethylpentane
18.2	Isobutane	22.7	Cyclopentane	27.0	Heptene
18.2	Acetaldehyde	23.3	2, 3-Dimethyl-1-Butene	28.2	N-Heptane
18.5	Butene	23.4	Cyclopentadiene	28.9	Heptene
18.8	N-Butane	23.5	2, 2-Dimethylbutane	29.6	Toluene
19.1	Butenyne	23.9	2-Methylpentane	33.2	Octanes
19.1	Butene	24.4	3-Methylpentane	33.2	Octenes
19.7	Butyne	24.5	Methylcyclopentane	33.2	Octynes
19.9	Butadiene	24.8	N-Hexane	38.5	Xylenes
20.5	Neopentane	25.0	2-Methyl-1, 3-Pentadiene	42.2	Nonanes
21.1	Isopentane	25.3	Benzene	42.2	Nonenes
21.3	Carbon disulfide	25.3	Cyclohexane	42.2	Nonynes
21.6	1-Pentene	26.0	Trichloroethylene		
21.8	N-Pentane	26.5	2, 2, 3-Trimethylbutane		

Table BII. Retention characteristics of DC-200 chromatographic column for analysis of organic compounds.

Flow rate 20 milliliters/min, temperature -75 C to +170 C, program rate 10 C/min.

Time (min)	*Compound*	*Time (min)*	*Compound*	*Time (min)*	*Compound*
7.5	Chloroacetylene	13.8	Methylethylketone	18.9	Methylthiophene
7.7	Isobutane	13.8	Butyraldehyde	19.0	Toluene
8.2	Acetaldehyde	14.0	Crotonaldehyde	19.3	3-Methylheptane
8.7	Isobutene	14.1	2, 3-Dimethylbutane	19.6	Trimethylcyclopentane
8.9	N-Butane	14.2	2-Methylpentane	19.8	Furylmethylketone
9.6	Butenyne	14.5	Chloroform	20.2	Nonane
9.6	1-Butene	14.5	Methylethylketone	21.0	Ethylbenzene
9.7	Methanol	14.6	3-Methylpentane	21.2	M-Xylene
9.9	Formaldehyde	15.1	N-Hexane	21.2	P-Xylene
9.9	2-Butene	15.6	Methylcyclopentane	21.7	Styrene
10.8	Acetone	16.3	Benzene	21.7	O-Xylene
10.9	Propanal	16.8	2-Methylhexane	21.7	Nonene
11.0	Acetonitrile	16.8	Heptene	22.1	Cumene
11.0	2-Methyl-2-Butene	16.8	N-Heptane	23.0	Ethylmethylbenzene
11.1	Isopentane	16.9	2-Pentanone	23.0	N-Propylbenzene
11.5	Acetone	17.0	Dimethylcyclopentane	23.2	Ethylmethylbenzene
12.2	Methylene chloride	17.0	3-Methylhexane	23.2	N-Propenylbenzene
12.2	Furan	17.3	Trichloroethylene	23.8	B-Methylstyrene
12.4	2-Methyl-1, 3-Butadiene	17.5	N-Heptane	23.8	Trimethylbenzene
12.6	Pentene	17.5	Dimethylfuran	24.4	Trimethylbenzene
12.6	Carbon disulfide	17.6	N-Heptane	24.9	Dimethylethylbenzene
12.6	Ethanol	17.6	Heptene	25.5	Dimethylethylbenzene
12.7	Dichloroacetylene	17.6	Heptyne	25.6	Ethylstyrene
12.9	Pentene	18.0	Methylpyrrole	26.3	Dimethylethylbenzene
12.9	Isobutyraldehyde	18.2	Heptene	28.1	Naphthalene
13.2	2, 3-Dimethylbutane	18.5	Octene		

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Table BIII. Retention characteristics of Durapak II chromatographic column.
Flow rate 20 milliliters/min, temperature −75°C to +130°C, program rate 10°C/min.

Time (min)	Compound	Time (min)	Compound	Time (min)	Compound
0.7	Methane	18.8	Acetone	23.1	Octatriene
5.2	Ethylene	19.0	Heptadiene	23.3	Furylmethylketone
5.4	Ethane	19.2	Hexyne	23.4	2-Pentanone
5.8	Carbon monoxide	19.2	Heptene	23.7	Methylpyrrole
9.3	Propane	19.2	Heptane	24.4	Nonene
9.4	COS	19.3	Methylpentadiene	24.4	Nonadiene
9.7	Propene	19.4	Hexatriene	24.4	Trimethylcyclopentadiene
9.8	Hydrogen sulfide	19.4	Cyclohexane	24.4	Trimethylpentadiene
12.2	Isobutane	19.5	Benzene	24.5	N-Butanenitrile
12.5	Methylchloride	19.5	Isobutanal	24.5	Nonane
12.5	Butene	19.6	Methylpropanal	24.5	Nonene
12.7	N-Butane	19.7	Heptane	24.6	Thiophene (?)
12.9	Butene	19.7	Heptyne	24.7	Nonadiene
13.3	Butene	19.8	Heptene	24.8	3-Hexanone
13.4	Butene	19.8	Heptadiene	25.4	Nonane
13.6	Butadiene	20.0	Heptene	25.2	Ethylbenzene
13.8	Methanethiol	20.1	Heptadiene	25.7	Nonene
14.2	Ethylene chloride	20.1	Heptane	25.7	Nonadiene
14.5	Carbon disulfide	20.2	Heptane	25.7	Nonane
15.1	Isopentane	20.2	Isoheptane	25.7	Xylene
15.2	Pentene	20.3	Trichloroethylene	26.2	Nonene
15.4	N-Pentane	20.3	Butanal	26.4	Xylene
15.5	Carbon suboxide	20.4	3-Methyl Hexanone	26.4	Nonane
15.7	Pentene	20.5	Heptene	26.7	Nonene
15.7	Pentadiene	20.5	Heptadiene	27.0	Styrene
15.8	Acetaldehyde	20.5	Heptane	27.1	Pentanenitrile
15.8	2-Methyl-1-Butene	20.5	Ethanol	27.6	Methylisobutylketone
15.9	Pentadiene	20.6	Vinylacetate (?)	28.6	Isopropylbenzene
16.0	Chloroacetylene	20.8	Methylethylketone	28.7	Decene
16.1	Pentene	20.9	Heptane	28.7	Decadiene
16.1	2-Methyl-2-Butene	20.9	Heptadiene	28.8	Pyridine
16.1	Pentyne	20.9	Heptene	29.0	Cyclopentanone
16.2	Furan	20.9	Dimethylfuran	29.2	Methylcyclopentanone
16.2	Pentene	21.3	Methylcyclopentadiene	29.8	Decene
16.3	Dimethylsulfide	21.3	Octane	29.9	N-Propylbenzene
16.3	1,3-Cyclopentadiene	21.3	Octadiene	30.3	Ethylmethylbenzene
16.3	Pentadiene	21.4	Dimethylsulfide	30.5	Hydrocarbon C10
16.9	Pentadiene	21.4	Octene	31.3	Ethylmethylbenzene
17.5	Isopentane (?)	21.5	Dimethylcyclopentadiene	31.7	Furfural
17.4	Hexene	21.8	2-Methylbutanal	31.7	Decane
17.6	Isohexane	21.9	Octynes	31.7	Decene
17.6	Hexene	21.9	Octenes	31.7	Decadiene
17.7	2-Methyl-Pentene	22.0	Propanenitrile	31.7	Ethylmethylbenzene
17.8	Cyclohexane	22.1	Toluene	31.8	Methylstyrene
17.8	Methylpentadiene	22.2	3-Methylbutanal	32.1	Decene
17.9	Hexyne	22.5	Crotonaldehyde	32.3	Ethylmethylbenzene
18.0	N-Hexane	22.6	Methylisopropylketone	32.3	Hydrocarbons C10
18.0	Propanal	22.6	Nonane	32.7	1-Methylpyrrolidine
18.1	Hexene	22.7	Octane	33.5	Benzofuran
18.2	Methylcyclopentene	22.7	Octadiene	35.7	Methylisopropylbenzene
18.3	Hexene	22.7	Octyne	36.3	T-Butylbenzene
18.3	Hexyne	22.8	Octene	35.9	Indene
18.3	Hexadiene	22.8	Octatriene	37.9	Dimethylstyrene
18.5	Methylacetate	22.8	Nonene	38.1	Ethylstyrene
18.6	Methylfuran	22.9	Octene	40.1	Decane
18.6	Hexene	23.0	3-Pentanone	40.5	C1 Aromatic
18.6	Hexadiene	23.0	Methylpropylnitrile	40.8	Hydrocarbon C10
18.7	Methylcyclopentadiene	23.1	Octene	15.6	Methylbenzofuran
18.7	Methanol	23.1	Octadiene	15.8	Benzonitrile

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