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MULTI-TEMPERATURE GAS CHROMATOGRAPHY USING ISOTHERMAL COLUMNS IN SERIES

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ABSTRACT

A computer-controlled three-oven gas chromatograph having a multi-position stream-switching valve has been used first to divide a wide-boiling mixture of n-alkanes into three cuts and then to fractionate each cut using a packed column at a different temperature. After initial injection of the sample into the highest temperature column, the low boilers were switched to a low-temperature oven. Then, the mid-boilers were switched to a column at an intermediate temperature. For routine repetitive analyses, the multi-oven approach offers an attractive alternative.

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I. INTRODUCTION

The advantages and problems of using combinations of selective gas chromatographic columns in series have been known for a long time (1-3). Recently, additional experiments with columns in series have included the switching of selected components from one column to another (4-9). These multi-demensional techniques have been reviewed by Bertsch (10). Generally, the emphasis has been on switching to columns having different liquid phases so as to enhance the separations of selected components. Schomberg et al. (11) have suggested a similar idea by recommending the venting or backflushing of slow-moving eluents using flow-switching techniques. Both fluidic and mechanical switching valves have been used, and frequently an intermediate cyrogenic trap has been employed (10).

The use of a change in column temperature has also been applied. Bertsch et al. (5) coupled two chromatographs, each capable of operating under optimum temperature conditions for the selected columns. Pretorius et al. (12) have pointed out that column polarity and, thus, the elution order of components, can change with temperature of the columns. By adjusting the temperatures of two columns in series, an optimized separation was indeed possible as shown by Kaiser and Rieder (13).

In the present paper, the use of three columns, each at a different temperature, has been explored and then compared to programmed-temperature analysis. Using the three-oven system, a sample was injected into a column held at the highest temperature so as to provide a preliminary separation of first the low-boilers and then the middle-boilers from the high boilers by means of a low-dead-volume, multi-position stream-switching valve.

An on-line computer provided precision timing for column switching, and it recorded data from three thermal conductivity detectors, each at the end of a column in a different oven. In that way, it was possible to perform more analyses in a given time than by programmed-temperature operation of only one column since reequilibration of the oven temperature was not required.

II. EXPERIMENTAL

A. Equipment

The system shown in Figure 1 consisted of three chromatographic columns, each in a different oven. A central oven at the highest temperature held the injection ports, the high-temperature column, a high-temperature thermistor detector, and a six-position column-selection valve. The left-hand oven held the lowest temperature column, and a conductivity detector. The right-hand oven held the third column and its detector at an intermediate temperature. These columns are later referred to as 1, 2, and 3, respectively.

The three ovens were formed from aluminum boxes 35.5 cm x 35.5 cm x 12.7 cm, and their inner walls were insulated using 3.2 cm asbestos board. Glass-fiber insulation, 3.8 cm thick, separated the ovens. Each oven contained a floor-mounted 1200-watt heating coil and a side-wall fan to circulate air. Two zero-crossing proportional temperature controllers (Thermotrol, Model 1053A, Hallikainen Instruments, Richmond, CA), fitted with platinum film sensors (Model 100R30, Omega, Stamford, CT), and one Valco Instrument Co. (Houston, TX) Model ITC-K10 temperature controller having a thermocouple feedback sensor were used.

Two Model 1100 and one high-temperature Model 1152 thermistor thermal conductivity detectors (Carle Inc. Anaheim, CA) each having cell volumes of 58 µl or less, were mounted on the inside of each oven and clamped to the aluminum wall. Two Model 100 Carle detector bridges and one Gow-Mac (Madison, NJ) Model 40-002 detector bridge were used to balance the signals from each pair of reference and sample channels.

A Valco six-position sampling valve (Model ACSF-6-HTa) was used for column selection. Figure 2 shows a more detailed view of the flow system before and after switching the valve from column 2 (low temperature) to column 3 (intermediate temperature). The valve was capable of operating at 400 psi and 300°C using an air-driven solenoid. It was connected through 0.15 cm zero-dead-volume fittings to both the low- and mid- temperature columns. At 300°C, the solenoid required more pressure to ensure switching than normally recommended (70 psi versus 60 psi). In addition, the valve was found to work best when the pressure to the solenoid was maintained during switching, an event which caused a momentary but rapid release of pressurized switching gas. By using a gas reservoir consisting of a 350 cm x 0.314 cm coil of tubing between the regulator and the switching solenoid, less than 0.1 sec was required to switch the valve. However, a delay of approximately 1 sec between switchings was found to be necessary in order for the switch mechanism to reset. The sequential switch positions were indicated by closure of electrical contacts.

Brooks controllers (Model 8744, Hatfield, PA) were used to adjust the flow to the high-temperature column and to the reference chambers of the detectors, which were linked in series. Milliflow regulators (Part 41310103, Veriflo Instruments, Richmond, CA) were used to control gas flow

to the other two columns. The carrier gas for each column was connected to injection ports that had been taken from Perkin-Elmer series 900 gas chromatographs and placed in the central oven. One port was connected directly to column 1 and was wrapped with heating tape whose current was controlled using a Powerstat. The ports for columns 2 and 3 were connected to both the pressure regulators and the column selection valve using 0.16 cm stainless steel tubing.

Samples were usually injected by a Hewlett-Packard auto-sampler (Model 7670A). It used a 10 µl Hamilton syringe to take either 0.5 or 1.5 µl of a liquid sample from a rubber-capped vial and inject it through a type W-9 septum (Applied Sciences, State College, PA) into the chromatograph. A Valco six-port sampling valve (ACV-6-HP), or a Hamilton 10 µl syringe were also used to introduce some liquid samples into the high-temperature column.

Connections between columns, valves, and detectors were made using 0.05 cm i.d. (0.16 cm o.d.) stainless steel tubing. Valves were equipped with 0.16 cm zero-dead-volume fittings. Column end fittings were 0.32 cm to 0.16 cm Swagelok^{R} reducing unions. Internal volumes were minimized using silanized glass wool. The total volume between any two columns, including detector, valve, and tubing, was approximately 300 µl.

A PDP 11/20, having real-time BASIC software, was used to control the chromatograph using a device panel (15), i.e., an 8-bit computer output port, 8-bit input port, and interrupt sensor. Five-volt signals at the output port turned on 120 VAC solid-state relays interfaced to the auto-sampler. After injection of a sample, the computer was signaled to start the collection of data.

Signals from the detectors were fed to instrumentation amplifiers (Analog Devices Model 6102 or Burr-Brown Model 3625A) to raise the maximum signal from 10 mV to 10 V. Signals

were then transmitted in parallel to a Linear Instruments recorder and to a Beckman Anscan (multi-channel analog-to-digital converter). Each signal to the Anscan was filtered using a simple low-pass filter having a time constant of 0.18 sec. Data were stored on DECtape.

B. Chemicals

n-Pentane, hexanes, and n-heptane were obtained from Fisher Scientific Co. (Pittsburgh, PA); n-hexadecane from Eastman Kodak (Rochester, NY); and n-octane, n-nonane, n-decane, and n-dodecane from Phillips Petroleum Co. (Bartlesville, OK). Samples for analysis were prepared by mixing equal volumes of the liquids.

Each of the three columns was 244 cm x 0.32 cm stainless steel that was packed with 5% SE-30 60/80 mesh acid-washed Chromosorb W. Before use, columns were conditioned overnight at 230°C using a low flow of helium.

Helium (Selox, Inc., Gainesville, GA) was used as the carrier gas after being passed through a trap that contained activated silica and 4 Å molecular sieve (Davison Chemical, Baltimore, MD). Nitrogen (Selox, Inc.) was used as the switching gas for the air-actuated valves and solenoids.

III. PROCEDURES

In the basic procedure, samples were injected into the high-temperature column and flowed through its detector and the column selection valve into the low-temperature column. After specified time intervals, the flow was first switched to the intermediate-temperature column and then to open air. The outputs of the three detectors were recorded simultaneously, and, to a first approximation, the last species eluted from each column at the same time.

In developing the basic procedure, the temperatures of each of the three ovens and the times for the two column-selection changes had to be determined. The first variable to be adjusted was the temperature of the high-temperature oven by injecting samples that were vented through its detector to waste. The temperature was selected that provided adequate resolution for the components in the last 90% of the chromatogram in the minimum time.

Then, knowing that each 45-60C^{o} decrease in temperature will reduce the vapor pressure (and, hence, the corrected retention time) by approximately a factor of 10, oven temperatures for the mid- and low- boiling fractions were set at 45-60C^{o} and 90-120C^{o}, respectively, below that of the high-temperature oven. The switching valve was then set so that the effluent from the high-temperature oven (and detector) passed directly into the mid-temperature column. Next, a sample was injected and, after approximately 10% of the total time (corrected for "dead" time) required to elute the high-temperature fraction had passed, the eluent from the high-temperature column was switched to waste. By following a procedure similar to that used for the high-temperature oven, the column temperature and switching time for the middle boiling fraction was adjusted using successive samples so that adequate resolution was attained in roughly the same time required for the high-boiling fractions. Then, a similar procedure was followed for the low-temperature column by connecting it to the detector from the high-temperature oven and switching to waste after approximately 1% of the time required for completion of the chromatogram for the high- (and mid-) boilers. Later, it will be pointed out how the switching time can also be adjusted to advantage. Hence, both temperature and switching time can be adjusted so as to attain the desired resolution within a preselected time for completion

of the chromatogram.

Once the oven temperatures and switching times had been selected the final procedure was as follows. The switching valve was set so that the carrier gas passed from the high-temperature column into the low-temperature column. After a period that included the time to sweep the dead volume of the high temperature column plus approximately 1% of the time for the highest boiling component to elute, the eluent from the high-temperature column was switched to the entrance of the mid-temperature column. Next, after approximately 10% of the "total" time had passed, the eluent from the high-temperature column was switched to waste. In that valve position, all three columns had independent carrier streams that were being vented to waste after passing through a detector. Under such a regime, assuming that the pressures had been adjusted so that the flow rates were the same in all three columns, the chromatogram for each column should be completed in approximately the same time. Furthermore, the range of boiling points that could be separated would, in principle, be close to a maximum under the idealized conditions stated earlier.

In the special case of the n-alkane series, overlaps of sample species were relatively minor, and a change with temperature in the relative elution order due to different polarities of the species was not a consideration. Hence, it was possible to deviate quite widely from the 1% and 10% switching times and from the suggested differences in oven temperatures. In addition, because one could easily construct plots of the logarithms of the net retention times on the high-temperature column versus carbon number, switching times could be selected that cut close to halfway between any two components of the series, even when they did not resolve when eluting from the high-temperature column. Thus,

switching times, total analysis times, and temperatures were relatively easy to select once the conditions for the high-temperature column had been fixed.

Prior to starting an analysis of the n-alkane mixture, ovens 1, 2, and 3 were usually set at 205°C, 65°C, and 130°C, respectively, and the injection port, at 210°C. Those oven temperatures were the estimated minima since, in practice, it was faster to make adjustments by increasing rather than by decreasing the temperatures of an oven. Furthermore, those temperatures caused the most volatile test sample, pentane, and the least volatile, n-nonadecane, to have reasonable elution times (approximately 1 and 8 min, respectively) when injected into an appropriate column.

Flow rate of the gas stream that passed through the reference side of each detector in series was set between 20 and 30 ml/min. Flow rates to each of the columns were approximately the same, but those for columns 2 and 3 had to be adjusted so as to minimize baseline shifts and/or pressure-pulses resulting from column switching. (A small baseline shift usually occurred due to a change in flow rate.) At first, flow controllers were used for columns 2 and 3, but switching effects were larger than when pressure regulators were used. Obviously, the use of flow-insensitive detectors would have further decreased those effects. For columns 1, and 2 and for columns 1 and 3, the air peak took 66 sec.

Detector controllers provided 25-35 ma total current to the thermistor bridge. The sensitivity was adjusted so that a 0.2 µl injection of n-pentane produced a full-scale response.

The auto sampler was used to inject most of the liquid samples. A 0.5 µl sample was usually used, but when the sample contained many components, the sample size was 1.5 µl. However, for preliminary studies of the system, mixtures of a few hydrocarbons were used with a gas sampling valve. The

sample was placed in a heated, fritted bubbler through which helium passed. The helium containing the saturated vapor then passed through a 50 µl sampling loop in the air-actuated six-port sampling valve. The sampling loop and the 0.16 cm o.d. connection line to the high-temperature column were heated using heating tape and a powerstat.

To obtain a programmed-temperature chromatogram having a degree of separation roughly comparable to that from the multi-oven system, column 1 from the multi-oven gas chromatograph was transferred to the Perkin-Elmer 900 where a flame ionization detector was used. Column flow was set at 20 ml/min and the initial temperature of 50°C. At 0.5 µl sample of the n-alkane mixture was injected. The temperature increased at 24C°/min for 8 min and then held at the maximum for 2 min so as to allow elution of the last component. The automatic cooling-reset rate was set at the mid (normal) position and took 12 min.

A. Computer Control

The column-selection valve was switched under computer control, and its position was checked by reading the input from the device panel. By raising a bit at the output port of the device panel, a relay device was triggered which activated the switching solenoid. Timing for those switches were controlled using a programmable real-time clock, and they were programmed to fall between data points.

In the final operating program, the starting position of the sampling valve was moved by the computer to connect the high-temperature column to the low-temperature column. After a 1-min delay to allow for system equilibration, a signal to the auto sampler began its injection sequence. After injection of a sample, the auto sampler provided an interrupt signal for the computer to begin data acquisition and to

monitor the time for the purpose of making changes in column selection. Data collection and testing of valve location continued until all switching operations had been made and all of the data acquired.

B. Data Acquisition

Generally, 500 data points per channel were acquired at a minimum of 0.2 sec between readings (0.6 sec between readings of a given channel). Because the time between data readings also included the time to execute the BASIC commands to read the time and determine if the switching valve should be changed, the sampling time was actually 0.74 sec/datum for a single channel. The minimum time between readings could be changed, and 0.1 and 0.5 sec sampling times were also used.

IV. RESULTS

A. Preliminary Experiments

To determine if the dead volume in the switching valve affected the chromatograms to a noticeable extent, column 1 at 100°C was connected directly to column 2 at 50°C using an empty 15 cm x 0.05 mm i.d. (0.16 o.d.) stainless steel tube. This arrangement bypassed the detector for column 1 and the column-selection valve. Injection of samples containing pentane and nonane produced peak shapes that were indistinguishable when the digitized peaks were plotted and superimposed. Hence, the effect of the valve on peak shape was negligible.

Next, the repeatability of the switching operation under computer control was examined. For a 5-min interval, the timing could be controlled to better than 0.2 sec at the 95% confidence level. This meant that, for our system, where the minimum peak width for a non-retained compound was between

2 and 3 sec (the narrowest one in the series), the switching irreproducibility on that peak represented much less than 10% of its width and approximately 12% of its area. For retained compounds, the percentage would be smaller.

To test that prediction, column 1 was set at 100°C and columns 2 and 3, at 50°C. A sample of pentane was injected, and a switch made from column 2 to 3 after 41.5 sec, at the maximum of the pentane peak, as noted by detector 1. The two pentane peaks that resulted were detected by detectors 2 and 3. Those peaks had retention times of approximately 80 sec and baseline widths of approximately 5 sec. Peak areas were calculated from the digitized peaks after being scaled to adjust for the differences in detector sensitivities are shown in Table 1.

Table 1

REPRODUCIBILITY OF SWITCHING IN TERMS OF RELATIVE AREAS FOR A PENTANE PEAK SPLIT BETWEEN COLUMNS 1 AND 2 BY THE COLUMN-SELECTION VALUE

Percentage of Total Area	
Detector 2	Detector 3
56	44
52	48
52	48
50	50
51	49
52.2 ± 6.34^a	47.8 ± 6.34^a

12 13

[a] mean $\pm \delta, t$

where $\delta = 2.28$

and t = student's t - value at 95% confidence level = 2.78

As expected, pentane was split into two approximately equal fractions, each having 12-13% uncertainty in relative peak at the 95% confidence limit. That value represented the worse possible case compared to what one would obtain by making the switch at a valley between two peaks.

B. Mixtures of n-Alkanes

Before exploring how a hydrocarbon mixture could be analyzed by measuring simultaneously the effluents from three columns, held at three different temperatures, a mixture of n-alkanes from n-pentane through n-nonadecane was first injected into column 1 at 205°C and its entire chromatogram recorded by detector 1. Figure 3 shows the entire digital high-temperature column chromatogram with the switching locations marked by an "X".

The first valve switch (from column 2 to column 3) was made 44.4 sec after injection or, when subtracting the void retention time, 8.4 sec into the chromatogram. The second switch (from column 3 to open air) occurred after 74.4 sec or 45 sec into the chromatogram.

Figure 4 shows the resulting chromatograms of the n-alkanes as recorded by the three detectors. The low-temperature fraction contained pentane through octane and about 5% of the nonane peak (not shown). The intermediate fraction contained some octane, the remaining 95% of the nonane, and the alkanes through tetradecane. Although pentane to nonadecane eluted from the high-temperature column, the components that eluted only from that column are the ones shown in this figure.

As reported above, the switch location of 44.4 sec from column 2 to column 3 put 5% of the nonane peak in the low-temperature fraction. For comparison purposes, Figure 5 shows both a "complete" low-temperature output and a second chroma-

togram in which all of the n-octane and n-nonane were eluted from the low-temperature column. To determine the split ratio of those components that are reported in Table 1, the chromatograms were normalized using the unsplit n-heptane peak as a reference.

In the event quantitative information was needed and a correction factor for the "lost" nonane (in Figure 4) was not acceptable, the best procedure would be to shift the switching time so that the peak of interest was not split. Otherwise, the split ratio could be determined as above or the split peak could be analyzed on both columns. In the latter case calculations of peak area would have to be made twice, and differences in detector sensitivities would have to be taken into account as they were for the data in Table 1. Generally, no more than two components were split in the test mixtures, and it was usually possible to select the time of the switch so that the splitting of each peak was minimal, i.e., at the valley between the peaks.

Figures 4 and 5 emphasize the fact that the appearance of two (or more) components is more likely to occur for the first switching operation where the species are most volatile and where the time before switching is the shortest. Even so, one can see that a complete analysis took only 7 or 4 min, depending upon whether or not one measured for each sample the small amount of nonane that eluted from the low-temperature column. In contrast, Figure 6 shows that, although a faster programming rate might have completed the chromatogram (with acceptable resolution) in 4 min, the oven required an additional 12 min to cool down. Hence, one could have operated the isothermal system at significantly lower temperatures, so as to improve fraction cutting and resolution while still meeting or bettering the time required for the programmed-temperature run.

The sample of n-alkanes was, of course, a special case compared to a general sample made up of a wide variety of types of compounds. Figure 7 shows that the logarithms of net retention times versus carbon number gave the expected linear plots. The semilog plot of the net retention time versus carbon number of the high temperature fraction was useful in predicting switching times for cutting between hydrocarbons of the series. As a result, it was possible to select conditions for our figures to illustrate how cuts could advantageously be taken at switching times other than 1% and 10% of the total (i.e., 3% and 19% in Figure 4).

It is clear that when an n-alkane had passed through two columns, each at a different temperature, the overall retention time should be a composite of the individual retentions on the two columns. Figure 8 shows a contour plot obtained from the data in Figure 7. The inflections in the 125-145°C region reflected the fact that the retention times involved column 1 plus column 2 or 3. Such contour plots were useful in observing the effect of temperature on analysis time and in "guesstimating" the resolution of pairs of peaks.

V. DISCUSSION

A three-oven system has been shown to be useful where a mixture having a wide boiling range of components needs to be analyzed. It should be especially useful in process control where repeated analyses need to be done in a minimum time. At the same time, the system design was very versatile because analyses could still be done on a single column or on two. Finally, a relatively small amount of additional hardware would permit expansion to a four-column system.

Other modifications could include the addition to the unused positions of the switching valve of a second series of

columns packed with a different liquid phase. If these were connected in parallel to columns 1, 2, and 3, and then to the reference channels of the detectors, analyses could be done using either of two sytems. Thus, the three-oven gas chromatograph is a promising way to minimize time for complex separations of routine, repetitive samples.

Chromatograms for the low and middle boiling fractions could be modified substantially by changing the times for switching the effluent from the high-temperature column and by the temperatures of columns 2 and 3. Therefore, it is important to note that the present study deviated quite substantially from the 1% to 10% guideline suggested earlier for the switching times. In fact, both the percentages of the cuts and the temperature differences between the ovens were larger. This serves to emphasize the flexibility of this overall approach.

If one made the initial fraction very small, it would enable the resolution of that fraction to be increased, but it would simultaneously cause a decrease in the resolution of a later fraction since more components would now have to be resolved in the same time. For the special case of the homologous n-alkanes, maximum resolution occurred when each fraction contained an equal number of components. Those results were consistent with a concept of information theory which states that the maximum information occurs when the probability of information is the same in all fractions.

For very complex samples, the identification of components in relationship to others will not be as obvious as for a programmed temperature analysis unless the data are plotted on a logarithmic time axis. However, when known compounds are involved, retention indices such as those published in Gas Chromatographic Data Compilation (17) will be useful because the analyses are being done under conditions close to

isothermal. How those retention indices vary with column temperature is known and can be used to identify compounds (18-20). For a completely unknown sample, arbitrary switching times and oven temperatures will have to be used first following which the times and temperatures can be fine-tuned.

Earlier, mention was made of the pressure pulses produced by the switching operation as a result of the detectors being sensitive to flow rate and pressure. However, the pressure pulse could actually be put to good use. One pulse appeared immediately in the chromatogram for the detector through which the sample had just passed, while the second pulse had to travel through the next column before it was detected. The time difference between the two pulses provided a good estimate of the void volume of the second column.

Pressure changes were the greatest in the high-temperature detector, and they depended upon the position of the column selection valve. Changing the exit from column 2 to 3 caused little change in pressure because those columns were carefully matched to give the same flow rate. However, changing from column 3 to open air significantly reduced the pressure in the high-temperature detector and caused a baseline shift in its response.

The first asterisk in figure 3 marks the point of the new baseline after the column switch to open air. The second asterisk marks where a manual adjustment was made to the old baseline using the detector bridge controls. Generally, that baseline shift was ignored.

Improvements can be made in the design and construction of the system. First, temperature isolation was inadequate. When the central oven was at 220°C, the side ovens were approximately 80°C due to thermal leakage when their heaters were off. As a result, the range of boiling points that could be separated was limited on the low end. Second, the poor

shape for the more strongly retained peaks was, probably due to the small temperature gradient in each oven from the top to the bottom. A secondary cause may have been that the detectors were held at the oven temperatures rather than above. Neither of these factors should be difficult to remedy.

VI. ACKNOWLEDGMENT

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FIGURE 1.

System layout

FIGURE 2.

Details of valve connections. Position A--High-temperature column and low-temperature column connected. Position B--High-temperature column and intermediate temperature column connected.

FIGURE 3.

Digital recording of n-alkanes by detector 1: "X" Marks switch locations; * indicates a baseline shift and electronic correction which occurred as a result of the second switch (Column 3 to open air).

FIGURE 4.

Outputs of detectors from three columns during an analysis of a mixture of n-alkanes from pentane through nonadecane. Column 2 at 65°C; Column 3 at 130°C; Column 1 at 205°C (after the last switching operation).

FIGURE 5.

Output from Detector 2 showing the nonane peak with and without a switching operation.

FIGURE 6.

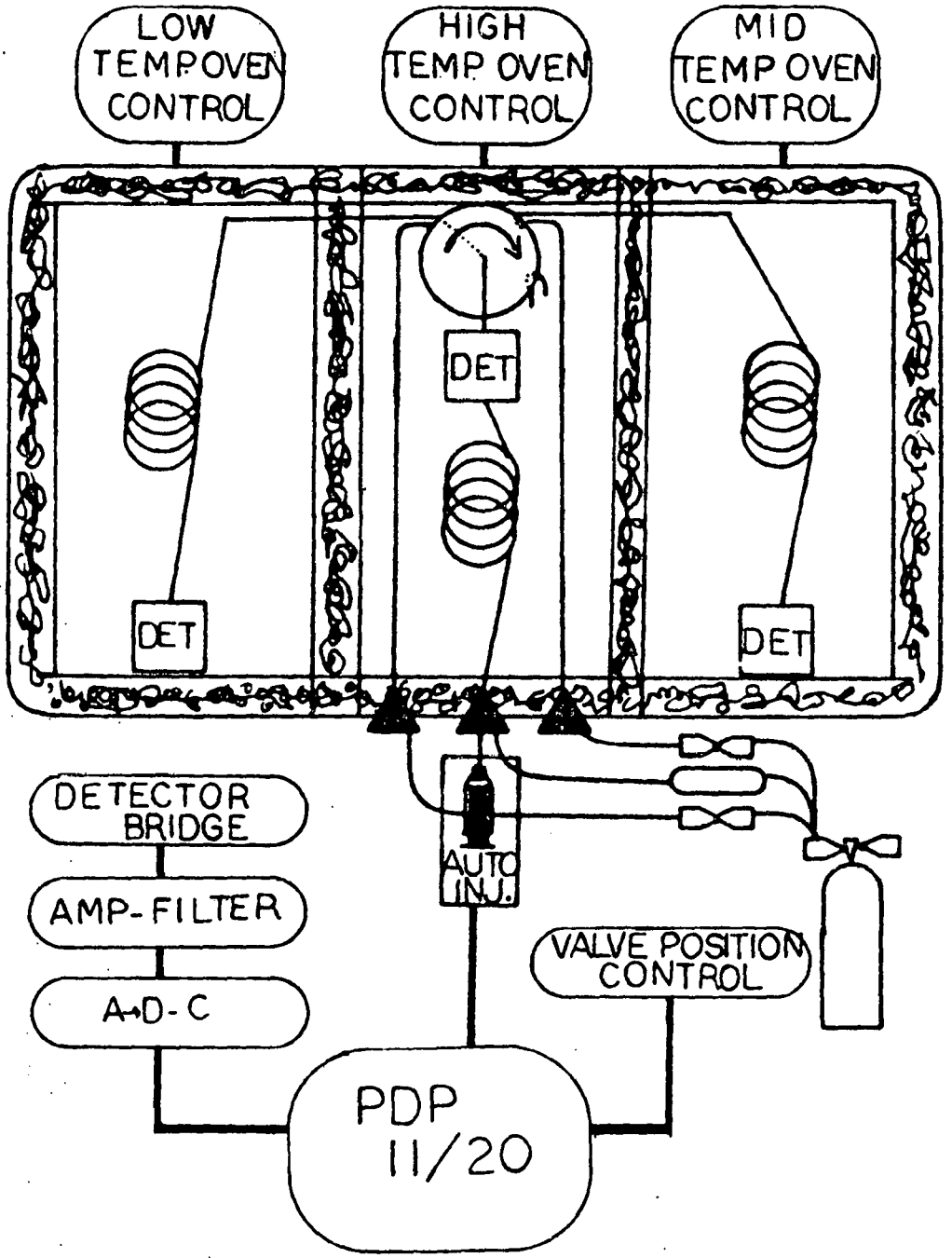
Temperature program of the sample of pentane through nonadecane from 50°C to 250°C and back to 50°C. Programming rate: 24C°/min.

FIGURE 7.

Net retention times of n-alkanes *versus* carbon number for Column 1 at 205°C and (a) Column 2 at 60°C (o), 85°C ( ), and 110°C (Δ); (b) Column 3 at 130°C (*), 160°C (X); and (c) Column 1 alone at 205°C ( ).

FIGURE 8.

Contour plot of net retention times in seconds on n-alkanes versus temperature and carbon number using data from Figure 7.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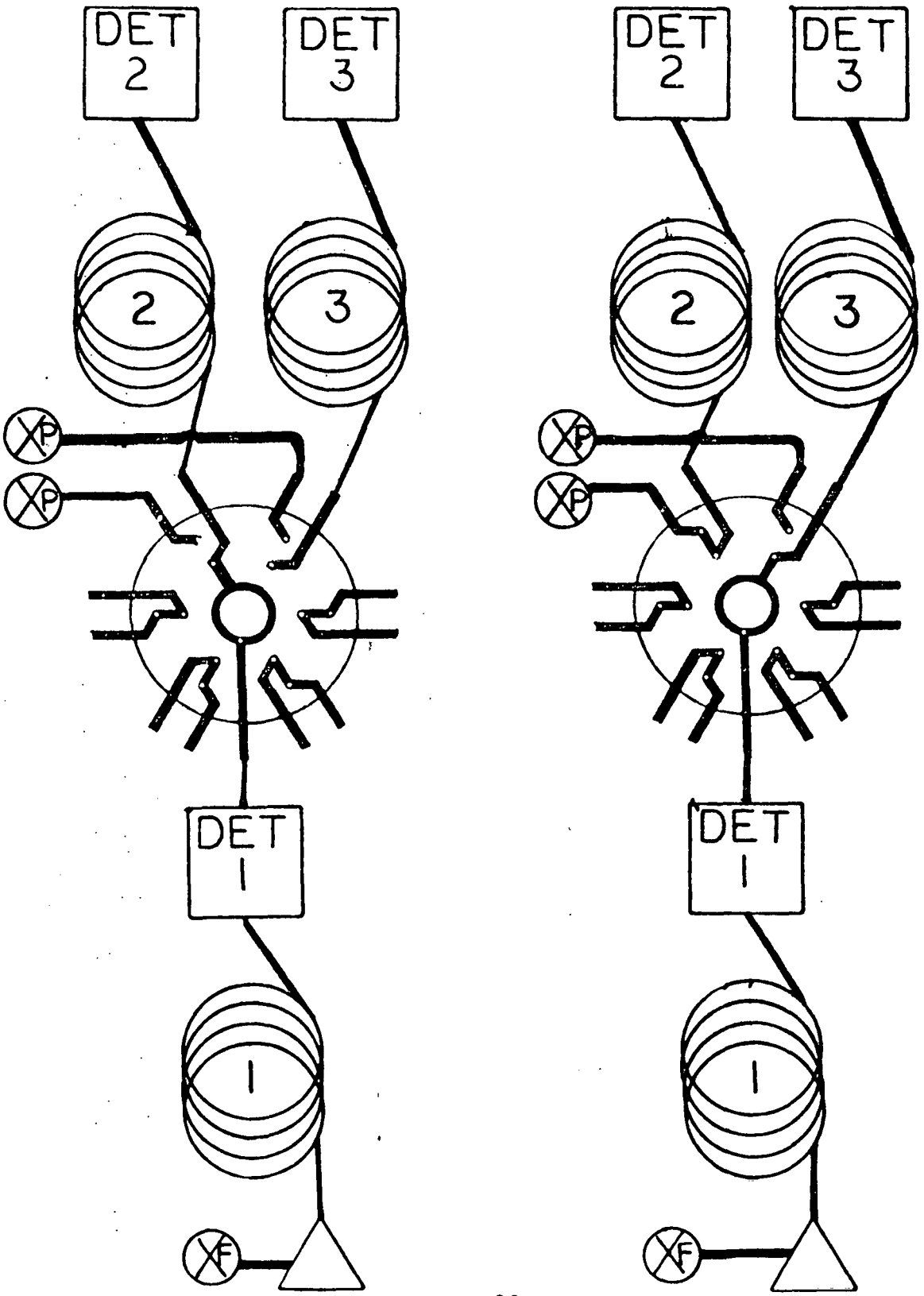
HIGH TEMP OVEN CONTROL
MID TEMP OVEN CONTROL
DET
DET
DET
DETECTOR BRIDGE
AMP-FILTER
A-D-C
AUTO INJ.
VALVE POSITION CONTROL
PDP 11/20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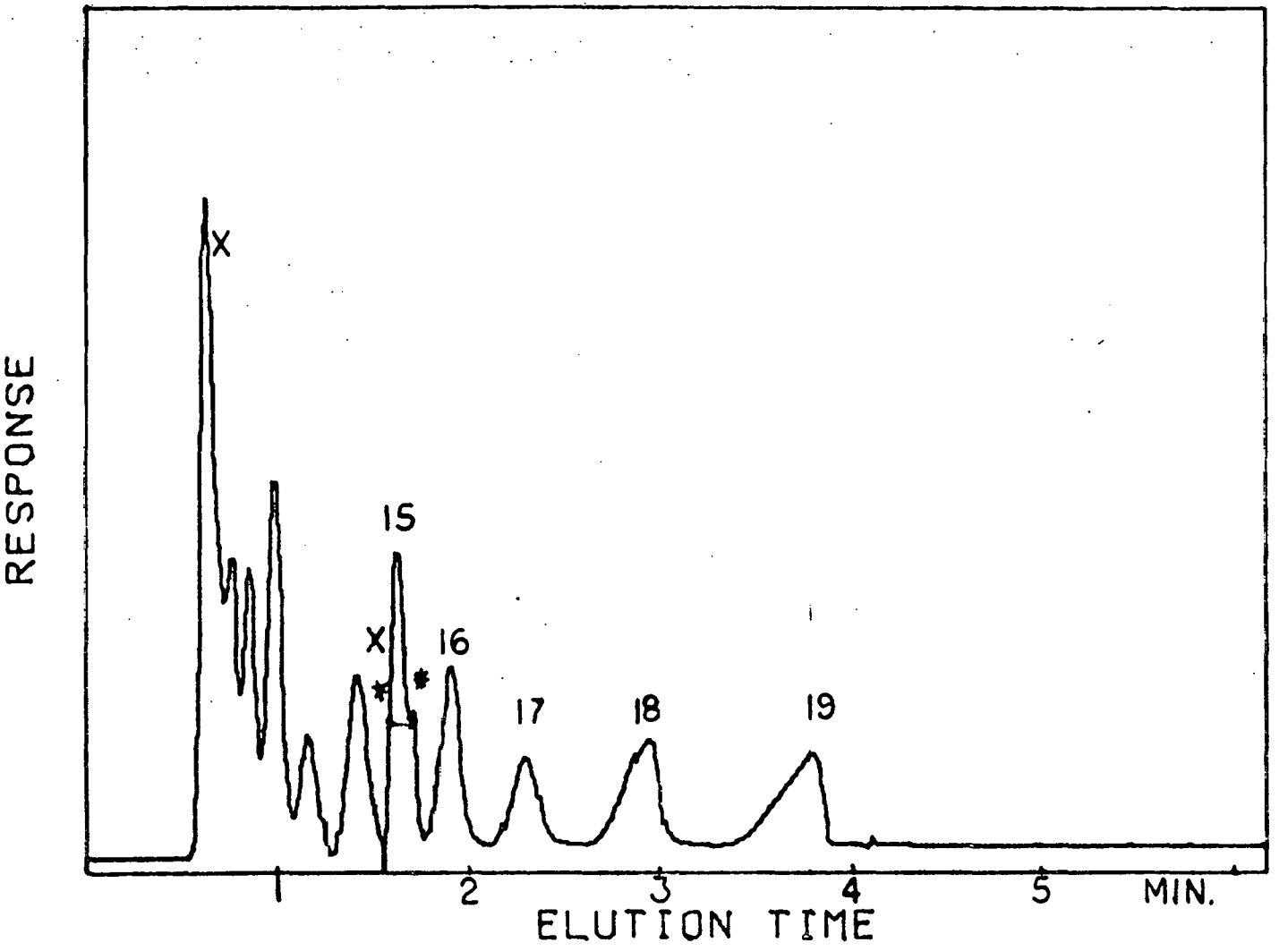
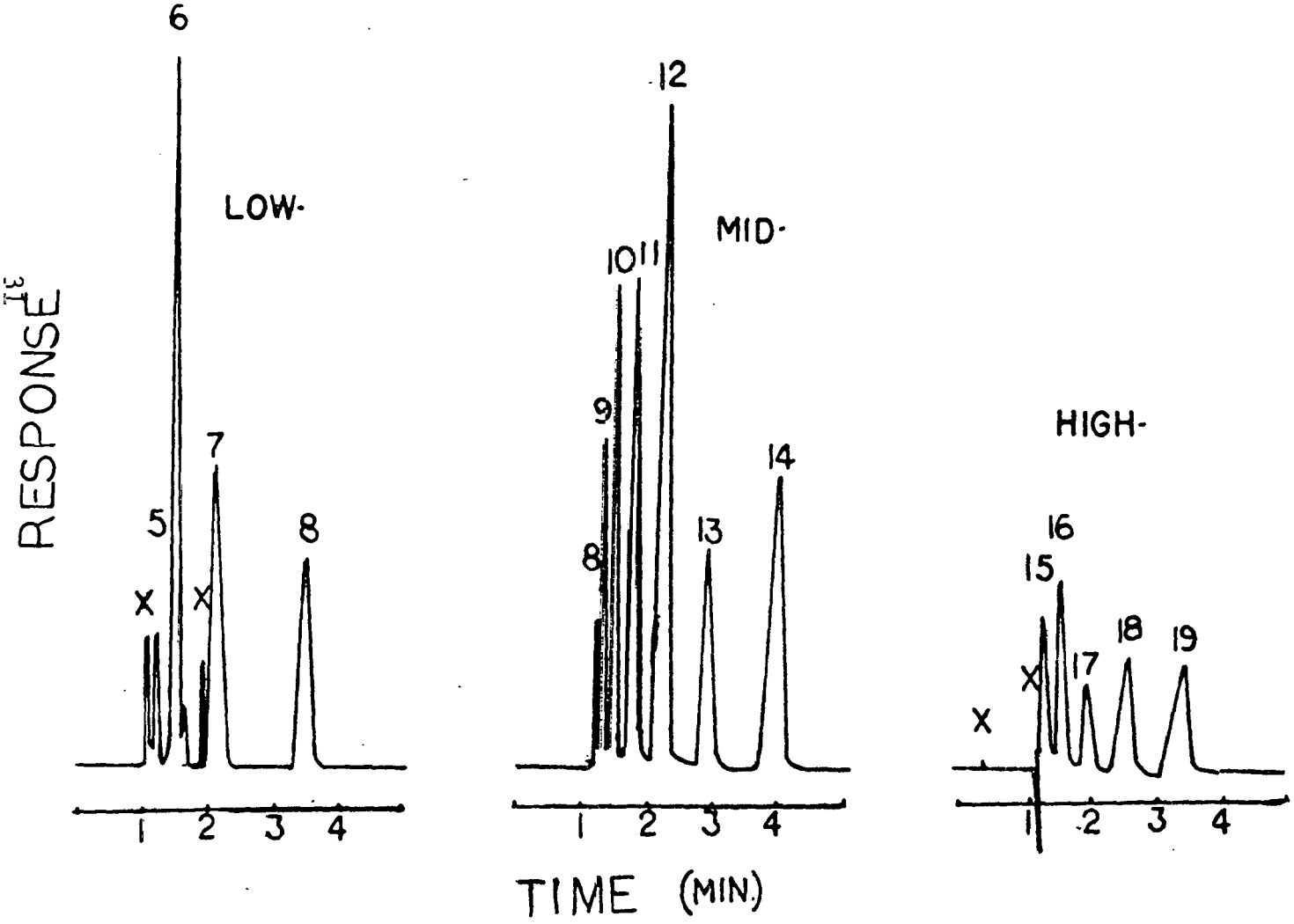
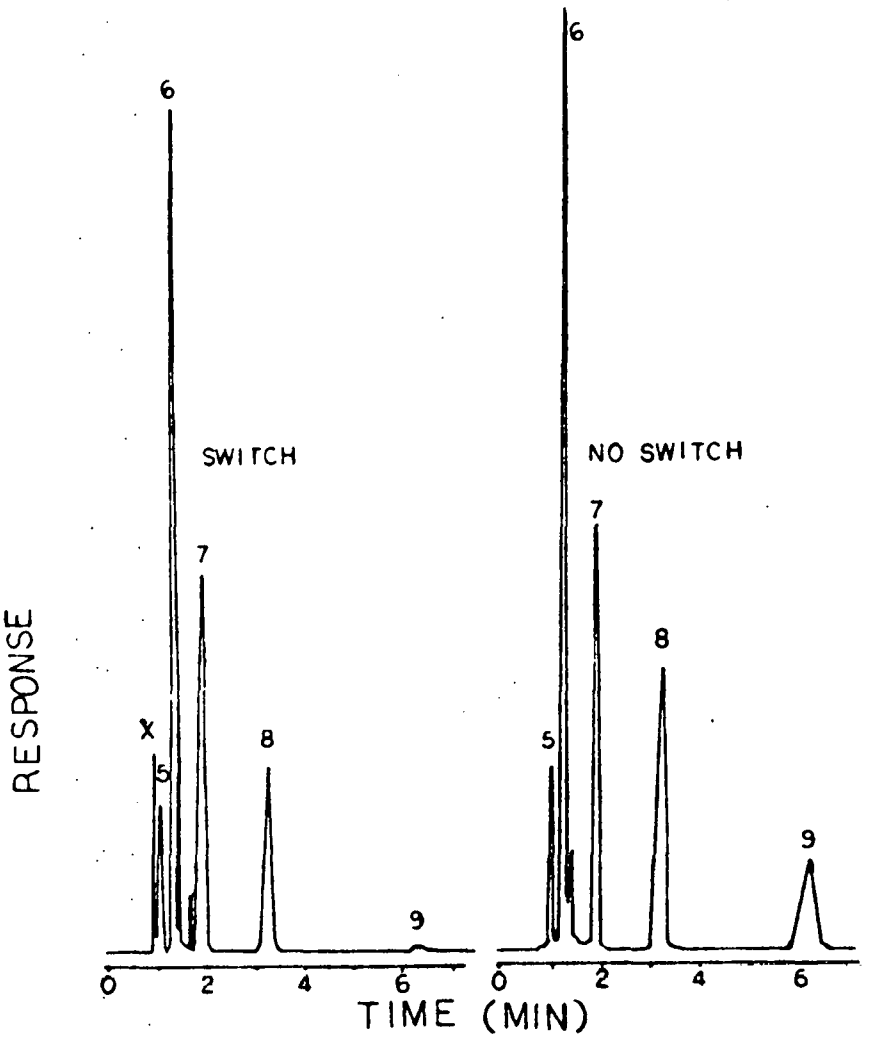
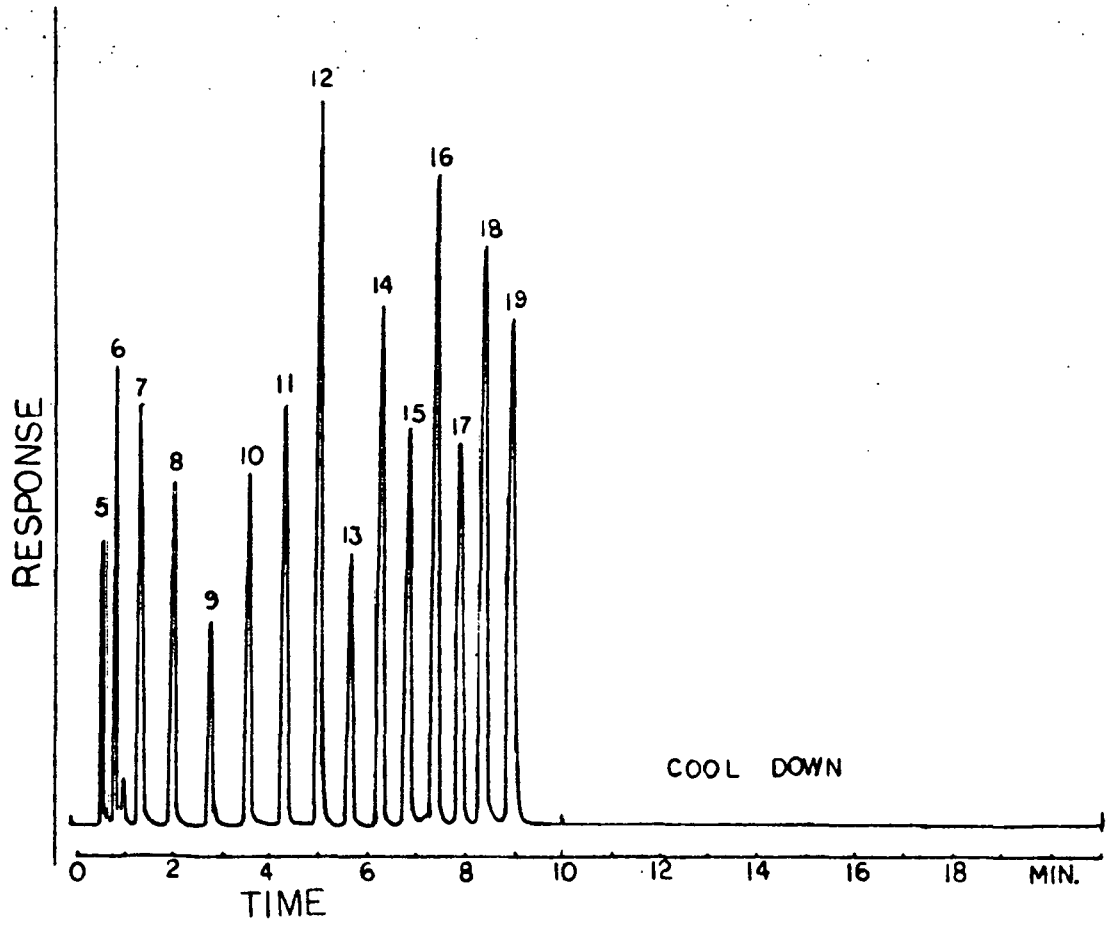
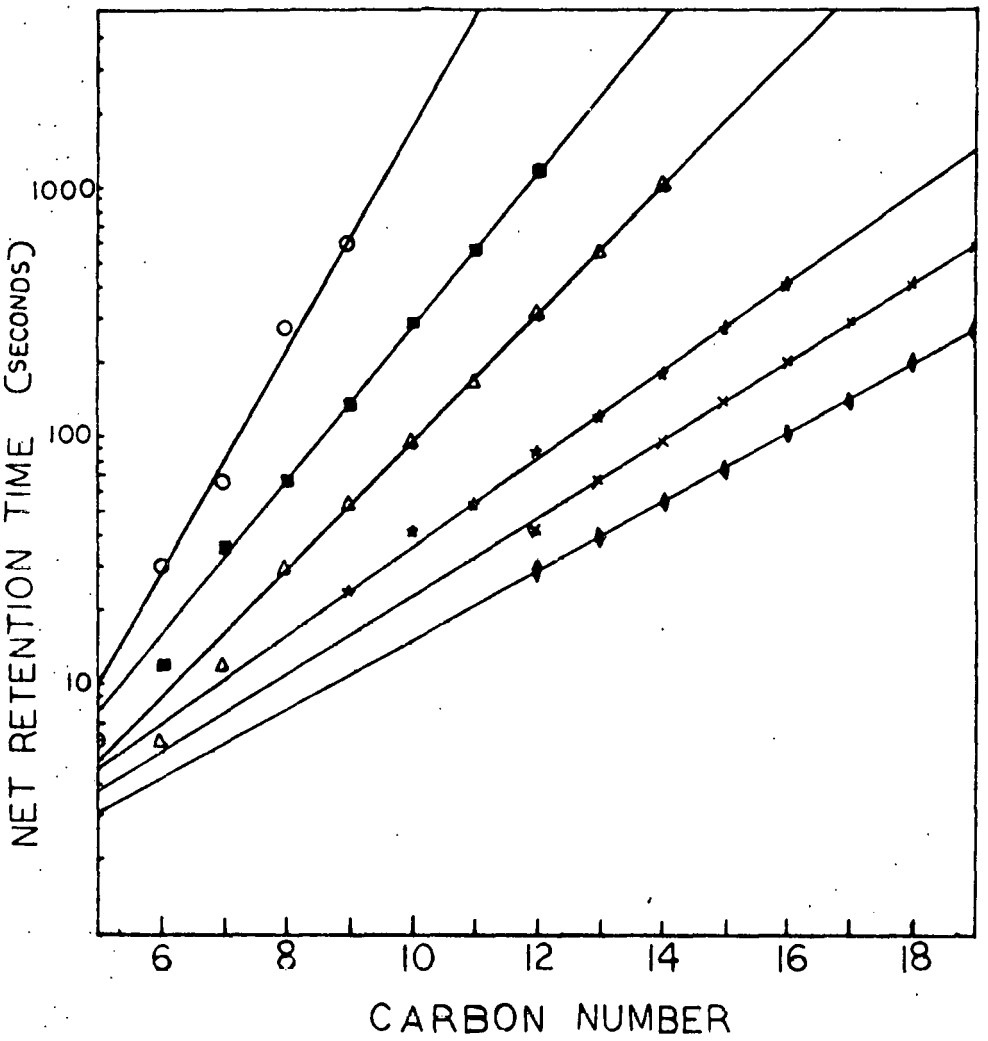
DET 3
2
3
XP
XP
DET 1
1
XF
DET 2
DET 3
2
3
XP
XP
DET 1
1
XF

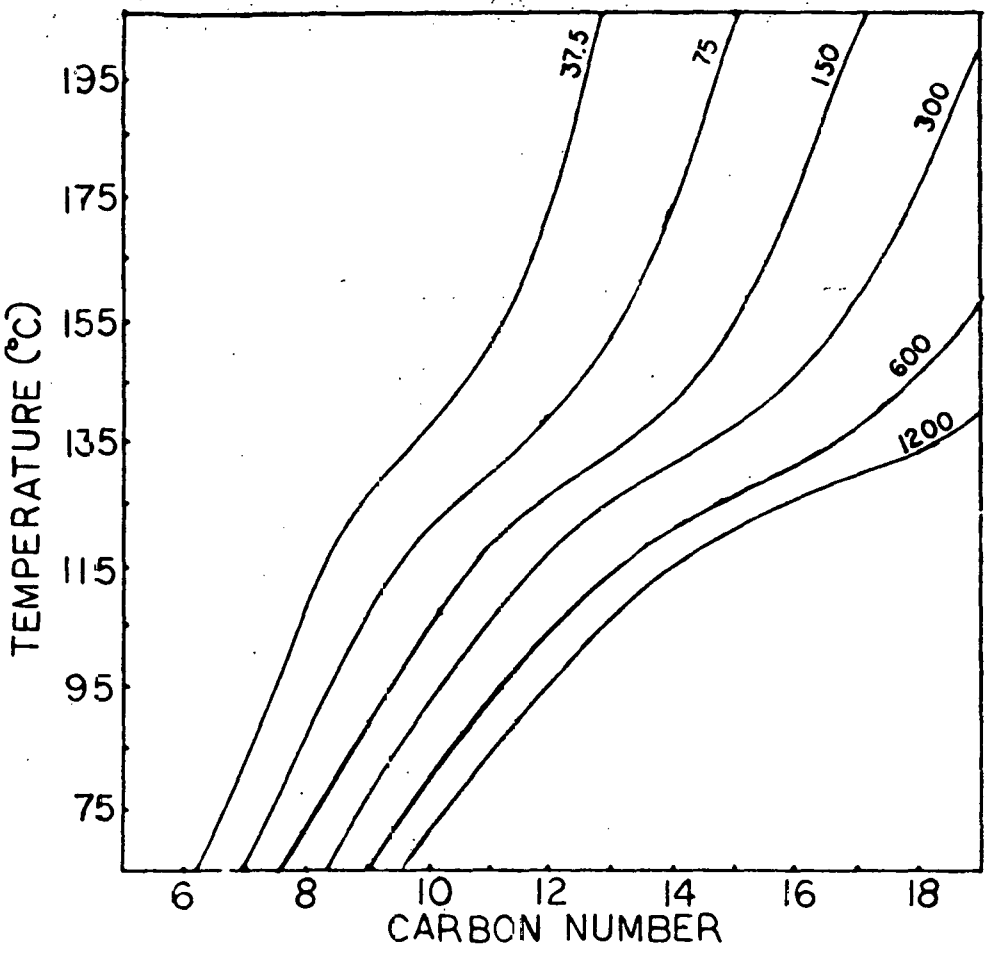

RESPONSE
X
15
X
16
17
18
19
1
2
3
4
5
MIN.
ELUTION TIME


RESPONSE
LOW-
MID-
HIGH-
TIME (MIN.)
1
2
3
4
X
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19


RESPONSE
SWITCH
NO SWITCH
X
5
6
7
8
9
0
2
4
6
TIME (MIN)


RESPONSE
5
6
7
8
9
10
11
12
13
14
15
16
17
18
19
COOL DOWN
0
2
4
6
8
10
12
14
16
18
MIN.
TIME


NET RETENTION TIME (SECONDS)
1000
100
10
6
8
10
12
14
16
18
CARBON NUMBER


37.5
75
150
300
600
1200
195
175
155
135
115
95
75
TEMPERATURE (°C)
6
8
10
12
14
16
18
CARBON NUMBER