

## THE ISOLATION AND DETERMINATION OF AROMATICS IN GASOLINE BY GAS CHROMATOGRAPHY

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### INTRODUCTION

Evaluation and control of the aromatic hydrocarbon content of motor gasolines used in engine deposition and exhaust emission tests have necessitated the development of quantitative methods of analysis for the determination of aromatics. Gas liquid chromatographic analyses involving selective separation of saturates and olefins from aromatics through the use of polar liquid phases offer advantages in specificity when compared to other approaches, such as open tubular column GLC techniques<sup>(1)</sup>, due to the large variety of hydrocarbons present in broad boiling-point range fuels. However, the existing standard method using selective separation has an upper temperature limit of 300°F<sup>(2)</sup>, which excludes the analysis of motor gasolines. This upper temperature has been extended by the method described in this paper. This method combines selective isolation with a highly repeatable sampling technique and a quantitative analytical separation method. Benzene, toluene, ethylbenzene, m+p-xylene, o-xylene, isopropylbenzene, and C<sub>9</sub>+ aromatics containing components with boiling points as high as 486°F are quantitatively determined. An extended method provides for the analysis of fuels having boiling points as high as 550°F.

### EXPERIMENTAL

**Apparatus.** A high-pressure, high-temperature liquid sampling valve, obtained from Valco, Inc., Houston, Texas, was interfaced to an oven constructed in this laboratory. The oven was equipped with columns and switching valves as illustrated schematically in Figure 1. Isothermal heat control was obtained using a fan, heating coils, and a powerstat. This oven was interfaced to a Hewlett-Packard Model 7621 gas chromatograph equipped with a dual hydrogen-flame ionization detector (HFID), multilevel-oven temperature programmer, electrometer, and a 1-mV, 1/2-sec recorder. The electrometer output, coupled to a Vidar Autolab Model 6230 digital integrator equipped with an ASR-33 Teletype, provided retention times and peak areas as hard copy printout and punched paper tape records. Experiments to establish the separating characteristics of CEF columns were conducted in the Hewlett-Packard Model 7621 gas chromatograph. The detector inlet was modified to use flow restrictors made of 5-in. lengths of 0.010-in.-I.D. stainless steel tubing crimped to effect increased back pressure.

**Reagents.** Hydrocarbons used in calibration standards for composition and retention time determinations were 99+ mole percent pure and were obtained from the following sources:

- benzene, toluene, 3,5-di-isopropylbenzene—J. T. Baker Chemical Co., Phillipsburg, New York.
- ethylbenzene—Phillips Petroleum Company, Bartlesville, Oklahoma.
- o-xylene—Matheson Coleman & Bell, Norwood, Ohio.
- n-propylbenzene, 1,2,4-trimethylbenzene, n-pentylbenzene—Eastman Kodak Company, Rochester, New York.

The N,N-bis(2-cyanoethyl) formamide (CEF) used as the liquid phase in the separating column (C<sub>1</sub> in Figure 1) was obtained from Applied Science Laboratories, Inc., State College, Pennsylvania. The Dexsil 300 liquid phase used in the analytical column (C<sub>3</sub> in Figure 1) was obtained from Analabs, Inc., North Haven, Connecticut. The SE-30 liquid phase used in the restrictor column (C<sub>2</sub> in Figure 1) was obtained from Varian Aerograph, Walnut Creek, California, and the Chromosorb solid supports were also obtained from Varian Aerograph.

**Column Preparation.** All column packings were prepared by mixing dissolved liquid phase, using the solvents recommended by the manufacturer, with the column solid support on a weight-percent basis, and removing the solvent in a vacuum rotary evaporator at 115°F. After the columns were packed, they were conditioned at the maximum operating temperatures and carrier gas flows for 24 hr, except for the high-load CEF columns which were conditioned for a minimum of 3 days.

**Chromatographic Procedure.** The chromatographic system, schematically illustrated in Figure 1, was designed to analyze both the aromatic and the saturate-olefin fractions of gasoline. The chromatographic operating conditions common

to both of these analyses are given in Table I. For the aromatic fraction analysis, Valves  $V_2$  and  $V_3$  are initially in position A as in Figure 1; at 5 min,  $V_2$  is turned 90 deg CW to position B; at 7 min,  $V_3$  is turned 90 deg CW to position B. For the saturate-olefin fraction analysis,  $V_2$  and  $V_3$  are initially in positions A and B, respectively; at 4.5 min,  $V_2$  is turned to position B; at 6.5 min,  $V_3$  is turned to position A.

At the end of each test, the valves were returned to their initial positions for the next test and carrier gas flow allowed to stabilize for a minimum of 3 min.

**Quantitation of Aromatics.** Because of the many aromatic hydrocarbons present in gasolines, a direct or absolute calibration technique (as opposed to internal standardization) was employed using the calibration standard described in Table II.

**Sampling Procedure.** All gasoline samples were stored at 0°F in sealed metal containers or room temperature in 75-ml, nitrogen-pressurized stainless steel cylinders, double-ended with stainless steel valves. The cylinders were used to pressure introduce the sample into the GLC system via a 1- $\mu$ l heated liquid sampling valve. The sample was passed through a 7- $\mu$ m stainless steel filter before entering the liquid sampling valve, and other hydrocarbon samples were also introduced into the GLC system through the use of the liquid sampling valve, unless otherwise indicated. The cylinders were filled with 50 to 55 ml of test fluid after evacuation of the cylinder. Approximately 5 ml of the sample was used to purge the liquid sampling system during filling. The liquid sampling valve was held at 175°C, and the nitrogen driving pressure was 450 psig.

## RESULTS AND DISCUSSION

**Establishing the Separating Characteristics of N,N-bis(2-cyanoethyl) formamide.** The effects of N,N-bis(2-cyanoethyl) formamide (CEF) liquid phase loading and column length have been investigated at a column oven temperature of 120°C which is 5°C lower than the manufacturer's recommended maximum operating temperature. An n-heptane mixture containing 3% v tetradecane and 10% v benzene was injected by syringe into various columns both with and without a flow restrictor between the column and detector. The tetradecane could not be completely resolved from the benzene using 15, 25, and 35% wt CEF in columns varying in length up to 5 ft. Separation was effected using 35% wt CEF in columns at least 3 ft long when flow restrictors were employed between the column and detector. The data presented graphically in Figure 2 demonstrate the increased separating ability at a measured pressure of 37 psig between the restrictor and the column which was supplied carrier gas through a flow controller at a pressure of 70 psig. Also demonstrated in Figure 2 is the effect of CEF loading. At a loading of 15% wt CEF, benzene elutes before tetradecane; at a loading of 25% CEF, no separation is effected; and at 35% CEF, the desired separation was obtained with tetradecane being eluted before benzene. Under the latter condition, pentadecane was eluted before benzene but was not fully resolved. A 3-ft column containing 35% wt CEF was chosen for the GLC system in Figure 1 to allow ample time to effect column switching and assure quantitative isolation of benzene and higher boiling-point aromatics from tetradecane and lower boiling-point hydrocarbons. Most of the gasolines which were analyzed contained less than 0.25% v material having boiling points above that of tetradecane.

**Establishing Operating Procedure.** Because of CEF's upper temperature limit of 125°C and the need to hold the CEF column's temperature constant during separation of the aromatics, versatility in the analysis of the aromatics was not provided with the use of CEF alone. Through the use of appropriate valving, the CEF column can be used to isolate the aromatic fraction on another column which can then be temperature programmed to provide the desired separation of the aromatics present. The simplest system would involve the valving described in Figure 3, with the valve switching time determined and an appropriate temperature program chosen for the analytical column to effect the desired aromatics separation. A somewhat more complicated system, described schematically in Figure 1, was chosen for use in this laboratory. In this system, a packed column having SE-30 liquid phase was placed in the same oven as the CEF column to provide the required back pressure in the CEF column to obtain the desired separating efficiency. A needle valve or small bore tube could be used to provide the back pressure if no subsequent analysis of the effluent were made. The SE-30 column at 120°C provided long-term back pressure stability and a fair separation of hydrocarbons in the gasoline boiling-point range when the effluent was detected rather than vented. During the isolation process on the CEF column, it was important that the back pressure remained constant. The Dexsil 300 column in Figure 1 was used to analyze the CEF column effluent. Dexsil 300 has a lower bleed level than materials such as SE-30.

This system allowed for the analysis of both the aromatic and the saturate-olefin fractions, depending upon the valve switching sequence chosen. Preliminary separations had to be made to optimize the operating parameters involving both oven temperature programming and valve switching times. A calibration standard was specifically formulated to provide the following:

- (1) Checks of the system's isolating efficiency by comparing the analytical ratios of benzene (10% v) to toluene (10% v) with the ideal ratio (1:1) and by determining the presence or absence of tetradecane, which should be

absent; the same criteria were used to establish the time at which the backflush valve,  $V_2$  in Figure 1, and the column switching valve,  $V_3$ , were turned. The column switching valve was turned 1 to 2 min after the turning of the backflush valve.

- (2) A calibration of the system in terms of aromatic concentration vs digital integrator area over a nominal concentration range of 1 to 10% v through the use of response factors.
- (3) A calibration of the system in terms of boiling-point temperature vs retention time.

By varying the time before backflushing the CEF column, it was possible to separate higher boiling-point saturates from the aromatics, as the data in Figure 4 illustrate. Figure 4a shows the relative recovery with no valve switching. All of the saturate and aromatic components of the sample were recovered, and, except in Figure 4a, the effluent from the CEF column was switched to the Dexsil 300 column 2 min after the backflush time. As the backflush time was varied from 5 to a maximum of 9 min, increasing amounts of saturates as well as aromatics were vented, and, as a result, they were not a part of the analyzed effluent from the CEF column. In order to raise the applicable boiling point (548°F at a backflush time of 9 min), analysis of the lower boiling-point aromatics had to be sacrificed. This higher boiling-point procedure had to be used in addition to the standard procedure in order to analyze fuels for the lower boiling aromatics as well as for aromatics boiling above 486°F.

In order to analyze the saturate-olefin fraction of the fuel, the aromatics were vented through the SE-30 column and the saturate-olefin fraction passed into the Dexsil 300 column. The valve switching schedule for this analysis was not the same as that for the aromatics' analysis. The Dexsil 300 column at 0°C did not create as much back pressure on the CEF column as did the SE-30 column at 120°C. The result of the lower back pressure was twofold and predictable from Figure 2. The lower back pressure resulted in shorter retention time and lower isolation efficiency, but the effects were slight.

A 1- $\mu$ l liquid sampling valve was used to eliminate sample injection errors due to the wide volatility ranges of gasolines. Neither peak areas from calibration standards nor premium or regular grade gasolines varied outside of the repeatability established for triplicate analyses, when the valve temperature or nitrogen pressure on the sample cylinder was varied over the ranges of 160° to 190°C and 400 to 500 psig, respectively. Without having been disassembled, the valve was operated at 175°C and 450 psig nitrogen in the analysis of several hundred samples over the past year.

**Analyses of Full-Range Motor Gasolines.** Analytical results for two motor gasolines, one a premium grade and the other a regular grade, were chosen to demonstrate the type of results routinely obtainable through the use of this GLC system. Figure 5 contains reproductions of chromatograms obtained from these two gasolines and the calibration standard described in Table II. A chromatogram was obtained for both the aromatic fraction and the saturate-olefin fraction of each of the three samples. The calibration standard provided a linear volumetric relationship based on integrated peak area and a linear boiling-point relationship based on peak retention time for the aromatics. The aromatic volumetric data in Tables III and IV were computed on the basis of the summed weighted areas for the aromatics contained in the calibration standard and were not rounded off so as to reflect precision for the triplicate analyses of each of the gasolines. Only the areas for benzene, toluene, and ethylbenzene were weighted by multiplying by the response factors, 0.904, 0.939, and 0.973, respectively. These response factors were experimentally determined during detector optimization which also included evaluation of o-xylene, n-propylbenzene, 1,2,4-trimethylbenzene, and n-pentylbenzene for linear concentration response. These response factors also compare favorably with values of 0.893, 0.935, and 0.971, respectively, which are the reciprocals of published response factors.<sup>(3)</sup> Response factors for other aromatics vary  $\pm 3$  percent from unity, which is a deviation directly translatable as a relative error for many of the unresolved aromatics. The use of carrier gas, makeup carrier gas, hydrogen, and purge airflow rates for optimized detector response and sensitivity in this system revealed negligible deviations from linearity for the aromatics investigated except for benzene and toluene. Relative deviations of approximately  $\pm 2$  percent of the concentration in the range 3 to 15 percent by volume were found for both benzene and toluene.

The repeatability for triplicate analyses was found to be particularly good as indicated by the data contained in Tables III and IV for the two gasolines. Similarly, good repeatability was obtained for the calibration standard. For an externally calibrated system, reproducibility within these repeatability limits was therefore a function of the repeatability with which the calibration standards could be prepared and analyzed.

The interpretation of variations in aromatic concentrations between gasoline samples or batches of reference fuels is directly dependent upon the accuracy with which they are determined. The approach used to establish relative errors was to evaluate the accuracy of analyses of blends prepared from pure hydrocarbons. By use of a single batch of the calibration standard described in Table II as an absolute calibrant, an analysis was made of blends of benzene, toluene, ethylbenzene, o-xylene, 1,2,4-trimethylbenzene, n-propylbenzene, n-pentylbenzene, and 3,5-di-isopropyltoluene in n-heptane. The highest relative errors observed for benzene, toluene, and ethylbenzene at concentrations of 15, 10, 5, 2.5, 1.25, and 0.625 percent

by volume were +4, +4, +2, +4, +4, and +8 percent, respectively. Essentially, the same maximum values were observed for the other aromatics except that concentrations above 5% v were not evaluated. Since additional errors could exist in the analysis of a gasoline sample, as opposed to these blends, the relative errors observed were increased for the concentration ranges of 5 to 20, 2 to 5, and 0.5 to 2 percent by volume to  $\pm 4$ ,  $\pm 6$ , and  $\pm 10$  percent, respectively. These were the more liberal values used as guides in evaluating data obtained by the aromatics procedure in this laboratory. Obviously, much more accurate analyses were possible for the resolved aromatics through the use of multiple standards and repetitive analyses. For the purposes for which these data are presently used, accuracy achievable without the use of multiple standards and repetitive analyses has been adequate, since new batches of calibration standards are analyzed to maintain reproducibility. In this way, fuels were analyzed with better month-to-month reproducibility than would be indicated from the relative error.

The boiling points reported for the aromatics in Tables III and IV were obtained through a linear best fit of boiling point versus retention time data given in Figure 6 for the aromatic fraction analysis. Retention time repeatability for the peaks was found to vary no more than  $\pm 3$  sec from the mean retention times over the entire range for triplicate analyses. Some of the  $C_9+$  aromatic peaks given in Tables III and IV have boiling points near the 486°F upper temperature limit and are therefore not considered to be definitely aromatic.

Generally, the saturate-olefin fractions of fuels were not analyzed in detail except for determining their boiling-point distributions, data which have been found useful in the evaluation of other analytical methods both in use and under development. The boiling-point data in Figure 7 for the boiling-point distribution of the saturate-olefin fractions of the two gasolines were obtained through a fourth order polynomial best fit of the boiling point vs retention time data in Figure 6. Areas for the individual peaks in the chromatograms shown in Figure 5 for the saturate-olefin fractions were divided by the total of the peak areas and normalized to obtain a distribution in terms of weight percent.

While the ability of CEF to isolate the aromatics from the saturates plus olefins in gasolines has been demonstrated and taken advantage of in the analytical procedures discussed in this paper, these procedures do not provide for the specific determination of the aromatic hydrocarbons which have been identified simply as  $C_9+$ . Methods for greatly improving resolution of the  $C_9+$  aromatics are being considered for future evaluation.

#### CREDIT

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#### REFERENCES

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2. 1970 Annual Book of ASTM Standards, Part 17, p. 789, American Society for Testing and Materials, Philadelphia, Pa.
3. W. A. Dietz, *J. of G. C.*, **5**, 68 (1967).

TABLE I. CHROMATOGRAPHIC OPERATING CONDITIONS FOR THE ISOLATION AND DETERMINATION OF AROMATIC AND SATURATE-OLEFIN FRACTIONS OF MOTOR GASOLINES

Instrument	Hewlett-Packard Model 7621 GC
Detector	Hydrogen-Flame Ionization
Liquid Sampling Valve:	
Temperature	175°C
Pressure	450 psig N <sub>2</sub>
Volume	1 $\mu$ l
Detector Temperature	250°C
CEF Column	35% wt N,N-bis(2-cyanoethyl) formamide on 60/80 Chromosorb P, AW-DMCS, 3' X 0.19" I.D. X 0.25"-O.D. aluminum
SE-30 Column	5% wt SE-30 on 100/120 Chromosorb G, AW-DMCS, 15' X 0.09" I.D. X 0.13"-O.D. stainless steel (316)
Dexsil 300 Column	4% wt Dexsil 300 on 80/100 Chromosorb G, AW-DMCS, 15' X 0.09" I.D. X 0.13"-O.D. stainless steel (316)
CEF Column Oven Temperature	120°C
Dexsil 300 Column Oven	12 min at 0°C, linear program of 8°C/min. to 225°C, 10 min at 225°C, recover to 0°C
Carrier Gas Flow	25 ml helium/min
Auxiliary Carrier Gas Flow	60 ml helium/min
Hydrogen Flow	35 ml/min
Airflow	375 ml/min
Recorder Attenuation	32

TABLE II. CALIBRATION STANDARD

Component Identification Number*	Component	Concentration, % v
1	Benzene	10.0
2	Toluene	10.0
3	Ethylbenzene	8.0
4	m+p-Xylene	0.0
5	o-Xylene	0.0
6	Isopropylbenzene	0.0
7	n-Propylbenzene	4.0
8	1,2,4-Trimethylbenzene	2.0
9	n-Pentylbenzene	1.0
10	n-Butane	0.5
11	n-Pentane	0.8
12	n-Hexane	28.2
13	n-Heptane	28.2
14	n-Octane	0.8
15	n-Nonane	0.8
16	n-Decane	0.8
17	n-Undecane	0.8
18	n-Dodecane	0.8
19	n-Tridecane	0.8
20	n-Tetradecane	0.8
21	n-Pentadecane	0.8
22	n-Hexadecane	0.8

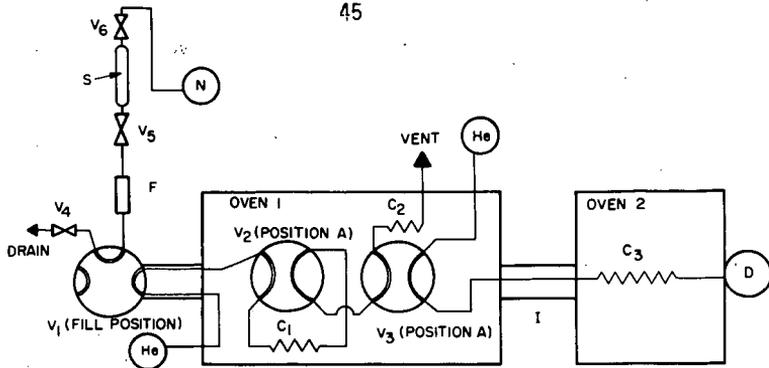
\*Same component identification numbers used throughout this paper.

TABLE III. THE AROMATIC COMPOSITION OF A PREMIUM GRADE GASOLINE

Component	Average Retention Time, sec	B.P., °F	Volumetric Concentration, %		
			Run 1	Run 2	Run 3
Benzene	1255	176	0.770	0.765	0.759
Toluene	1451	231	16.990	16.952	16.987
Ethylbenzene	1593	277	3.134	3.129	3.122
m+p-Xylene	1617	285	11.201	11.187	11.229
o-Xylene	1651	296	3.566	3.553	3.563
Isopropylbenzene	1682	306	0.133	0.136	0.136
C <sub>9</sub> +	1725	319	0.535	0.531	0.528
C <sub>9</sub> +	1743	325	2.626	2.617	2.620
C <sub>9</sub> +	1765	332	1.517	1.517	1.516
C <sub>9</sub> +	1800	342	3.147	3.144	3.143
C <sub>9</sub> +	1839	355	0.616	0.611	0.612
C <sub>9</sub> +	1873	366	1.384	1.381	1.379
C <sub>9</sub> +	1913	378	0.966	0.962	0.957
C <sub>9</sub> +	1945	388	0.093	0.091	0.089
C <sub>9</sub> +	1972	396	0.599	0.599	0.595
C <sub>9</sub> +	1994	403	0.109	0.107	0.104
C <sub>9</sub> +	2013	410	0.302	0.304	0.300
C <sub>9</sub> +	2066	426	0.441	0.446	0.452
C <sub>9</sub> +	2110	440	0.015	0.016	0.012
C <sub>9</sub> +	2137	448	0.014	0.024	0.012
C <sub>9</sub> +	2203	468	0.032	0.037	0.033
C <sub>9</sub> +	2224	475	0.006	0.010	0.006
Total			48.190	48.119	48.154

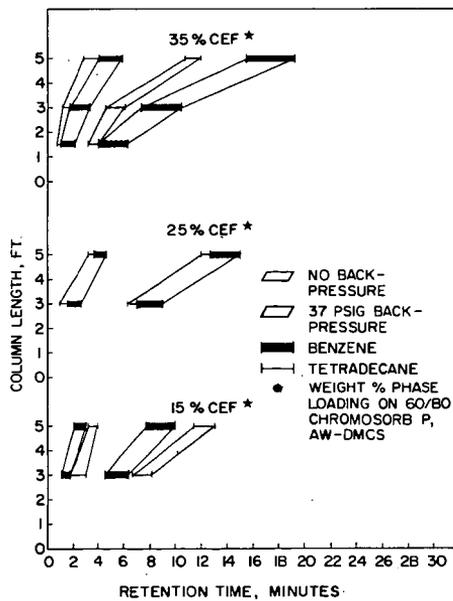
TABLE IV. THE AROMATIC COMPOSITION OF A REGULAR GRADE GASOLINE

Component	Average Retention Time, sec	B.P., °F	Volumetric Concentration, %		
			Run 1	Run 2	Run 3
Benzene	1257	176	0.419	0.403	0.405
Toluene	1447	231	2.713	2.651	2.682
Ethylbenzene	1595	277	1.965	1.911	1.933
m+p-Xylene	1617	284	7.798	7.726	7.774
o-Xylene	1652	295	2.880	2.846	2.875
Isopropylbenzene	1684	305	0.149	0.146	0.148
C <sub>9</sub> +	1728	319	0.657	0.619	0.634
C <sub>9</sub> +	1746	325	3.365	3.323	3.351
C <sub>9</sub> +	1768	332	1.958	1.934	1.953
C <sub>9</sub> +	1802	343	4.346	4.305	4.333
C <sub>9</sub> +	1842	355	0.911	0.907	0.908
C <sub>9</sub> +	1875	365	2.079	2.056	2.068
C <sub>9</sub> +	1916	379	1.463	1.440	1.451
C <sub>9</sub> +	1947	388	0.143	0.138	0.142
C <sub>9</sub> +	1974	397	0.915	0.903	0.909
C <sub>9</sub> +	1996	404	0.198	0.180	0.185
C <sub>9</sub> +	2015	409	0.497	0.494	0.498
C <sub>9</sub> +	2068	426	0.724	0.714	0.722
C <sub>9</sub> +	2110	440	0.030	0.026	0.028
C <sub>9</sub> +	2139	448	0.026	0.024	0.024
C <sub>9</sub> +	2156	454	0.015	0.013	0.014
C <sub>9</sub> +	2205	468	0.064	0.061	0.061
C <sub>9</sub> +	2225	475	0.012	0.011	0.011
Total			33.327	32.831	33.109



- OVEN 1 ISOTHERMAL CEF COLUMN OVEN
- OVEN 2 TEMPERATURE PROGRAMMED DEXSIL 300 COLUMN OVEN
- V<sub>1</sub> HEATED SAMPLING VALVE IN FILL POSITION; TURN 90° CW TO INJECT POSITION.
- V<sub>2</sub> BACKFLUSH VALVE IN POSITION A; TURN 90° CW TO POSITION B.
- V<sub>3</sub> COLUMN SWITCHING VALVE IN POSITION A; TURN 90° CW TO POSITION B.
- V<sub>4</sub>, V<sub>5</sub>, V<sub>6</sub> SHUT-OFF VALVES
- F 7 MICROINCH IN LINE SAMPLE FILTER
- C<sub>1</sub> CEF COLUMN
- C<sub>2</sub> SE-30 COLUMN
- C<sub>3</sub> DEXSIL 300 COLUMN
- N NITROGEN PRESSURE REGULATOR
- S HIGH PRESSURE CYLINDER CONTAINING SAMPLE
- He HELIUM CARRIER GAS
- D FLAME IONIZATION DETECTOR
- I HEATED INTERFACE, SAME TEMPERATURE AS OVEN 1

FIGURE 1. SCHEMATIC DIAGRAM OF GAS CHROMATOGRAPHIC SYSTEM FOR THE ISOLATION AND DETERMINATION OF AROMATICS IN GASOLINE



Note: Conditions—All columns were 0.19 in. I.D. aluminum; oven temperature was 120°C; helium carrier gas flow rate was 25 ml/min.

FIGURE 2. EFFECT OF COLUMN PARAMETERS ON THE SEPARATION OF TETRADECANE FROM BENZENE

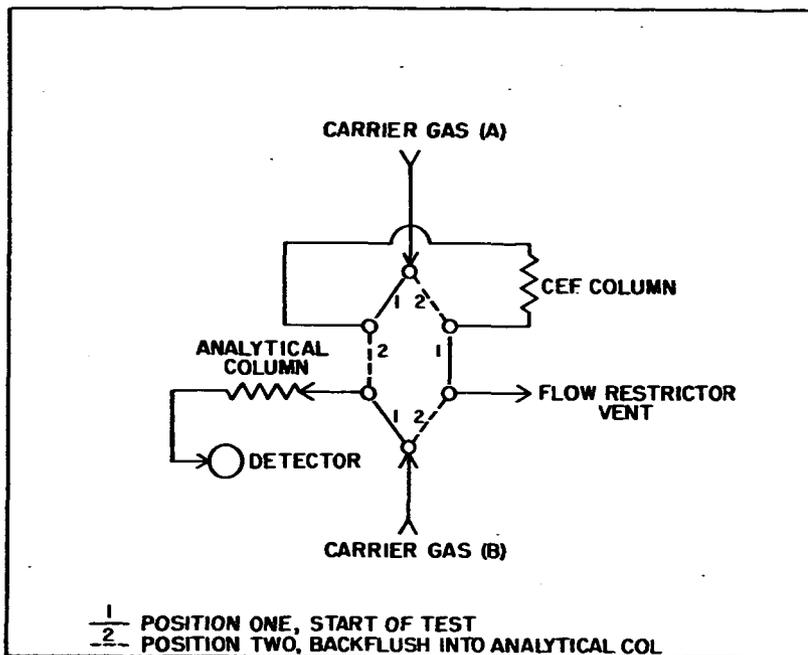


FIGURE 3. SIMPLIFIED SYSTEM FOR AROMATICS ANALYSIS USING TWO-POSITION VALVE

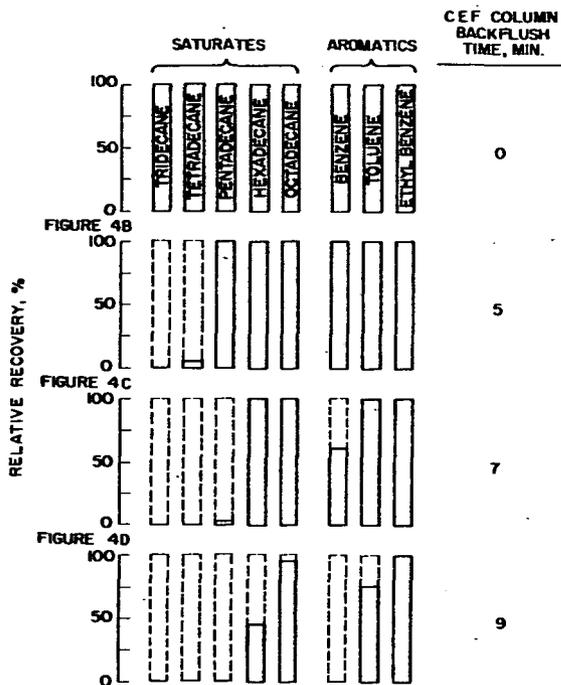
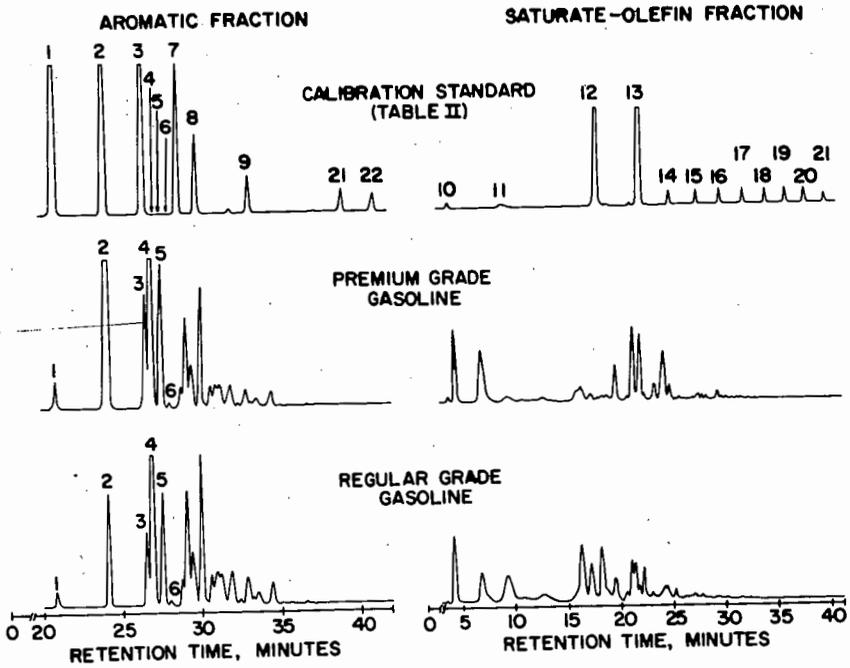


FIGURE 4. EFFECT OF CEF COLUMN BACKFLUSH TIME ON ANALYTICAL RECOVERY



Note: Component identification numbers are given in Table II.

FIGURE 5. REPRODUCTION OF CHROMATOGRAMS FOR A CALIBRATION STANDARD, A PREMIUM GRADE GASOLINE, AND A REGULAR GRADE GASOLINE

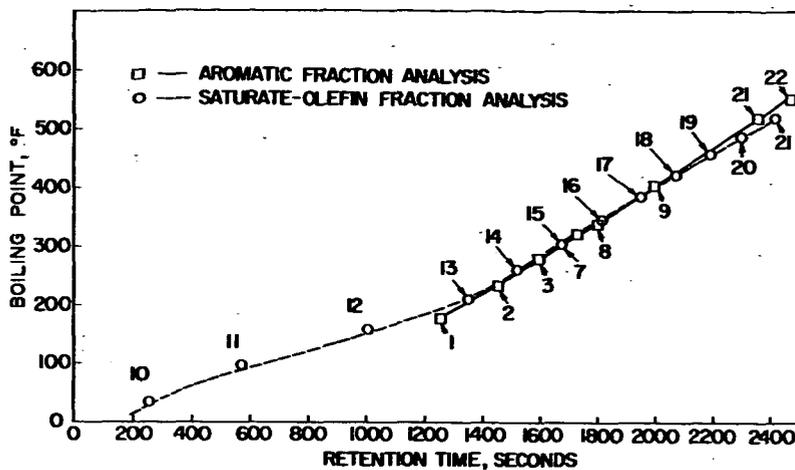


FIGURE 6. CALIBRATION CURVES FOR BOILING POINT VERSUS CHROMATOGRAPHIC PEAK RETENTION TIME

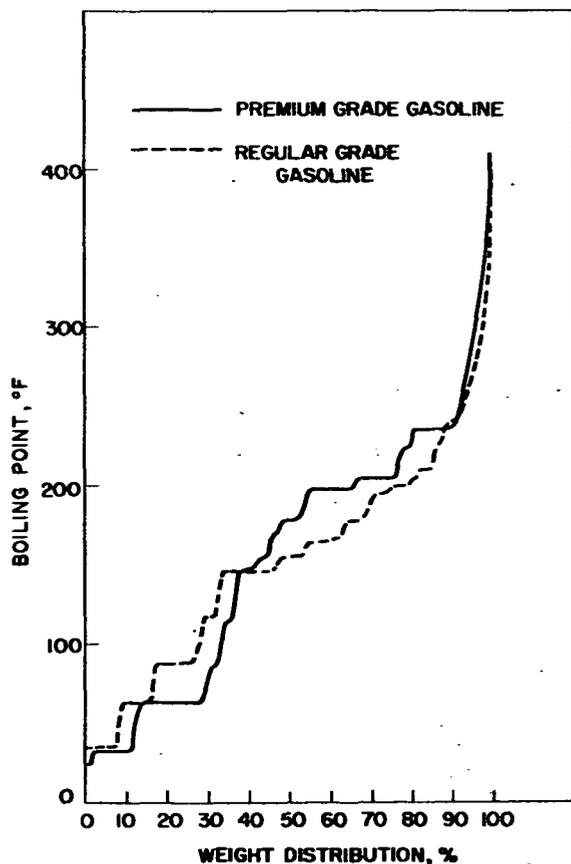


FIGURE 7. BOILING-POINT DISTRIBUTION OF THE SATURATE-OLEFIN FRACTIONS OF TWO GASOLINES