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Composition of Gasoline from Coal Hydrogenation

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THE recent critical shortage of benzene (2) has focused attention on the necessity of providing additional supplies of this and related aromatic hydrocarbons. In the past, the chief source of benzene has been the light oil derived from coal carbonization. Although the United States possesses enormous reserves of coal, the quantity carbonized is determined principally by the steel industry, the major consumer of coke. Even a considerably greater expansion of the steel industry (and its coke requirements) than now contemplated would not provide the quantities of benzene necessary to satisfy the expanding chemical industry.

To meet the shortage of benzene on an immediate basis, the petroleum industry has provided new methods for processing gasoline, such as hydroforming and platforming. These result in a substantial conversion of nonaromatic components to benzene and other light aromatics (3). From the long-term point of view, however, it is possible that coal hydrogenation can continuously satisfy the constantly increasing demands for benzene and other aromatic compounds.

DESCRIPTION OF SAMPLES

The oils used in this investigation were produced at the 200-barrel-a-day plant of the Bureau of Mines at Louisiana, Mo. This plant utilizes the conventional two-stage German operation—a primary liquid-phase stage followed by vapor-phase processing of a portion of the liquid-phase product. In the first, or liquid-phase stage, a slurry of fresh coal suspended in a heavy recycle oil containing a catalyst—such as ferrous sulfate—is pumped with high-pressure hydrogen through a preheater into two hot vertical converters in series, where conversion of coal to liquid and gaseous products occurs. The product stream enters a "hot catchpot," which is held at a specified temperature. The material collected at this point contains the unconverted coal, other solids, and the heavy ends of the oil. This material, so-called "heavy-oil-let-down," or HOLD, is let down to atmospheric pressure. The vapors leaving the hot catchpot enter a "cold catchpot," where the bulk of the product oil and all of the water of reaction plus some injected water are condensed. The uncondensed gases leaving the cold catchpot are scrubbed to recover the light-boiling constituents, which are then combined with the cold-catchpot product. The total cold-catchpot oil is let down to atmospheric pressure and then fractionated into four fractions: gasoline, naphtha, middle oil, and bottoms. The bottoms are combined with the HOLD and, after a suitable solids purge, this material is used as the recycle oil for suspending fresh coal. The three distillate fractions constitute approximately 0.5 of the total

cold-catchpot oil. These combined fractions are hereafter referred to as "vapor-phase feed" because they constitute the fresh feed to the second or vapor-phase stage. When a coal hydrogenation plant is operated as a chemicals plant, the naphtha stream is extracted with acid and alkali to recover commercially valuable phenols and tar bases, and the dephenolized naphtha is employed as part of the vapor-phase feed. However, in the operation which produced the present samples, no tar acids or tar bases were removed from the vapor-phase feed.

The vapor-phase feed investigated in the present study was collected when the plant was processing a bituminous C coal from Rock Springs, Wyo. (analysis in Table I) under conditions outlined in Table II. The estimated yield of vapor-phase feed in this operation was approximately 4.3 barrels (~1000 pounds) per ton of moisture- and ash-free coal (7).

The vapor-phase feed was distilled into two fractions in the Bruceton pilot plant, one boiling to 145° C. and the other between 145° and 200° C. The second fraction was washed with acid and alkali and the neutral portion combined with the first fraction.

TABLE I. ANALYSIS OF ROCK SPRINGS, WYO., COAL
(Moisture-free basis)

Proximate	
Volatile matter	45.1
Fixed carbon	49.5
Ash	5.4
Total	100.0
Ultimate	
Hydrogen	5.3
Carbon	72.3
Nitrogen	1.5
Oxygen	14.4
Sulfur	1.1
Ash	5.4
Total	100.0
Heating value	12,970 B.t.u.

TABLE II. OPERATING CONDITIONS—LIQUID-PHASE
HYDROGENATION OF ROCK SPRINGS COAL

System pressure	7700 p.s.i.g.
Temperature No. 1 converter	878° F.
Temperature No. 2 converter	892° F.
Hot-catchpot liquid	577° F.
Hot-catch vapor space	750° F.
Gas to heater	280,000 cu. ft./hour
Paste rate	1215 gal./hour
Coal rate	57 tons/day, dry

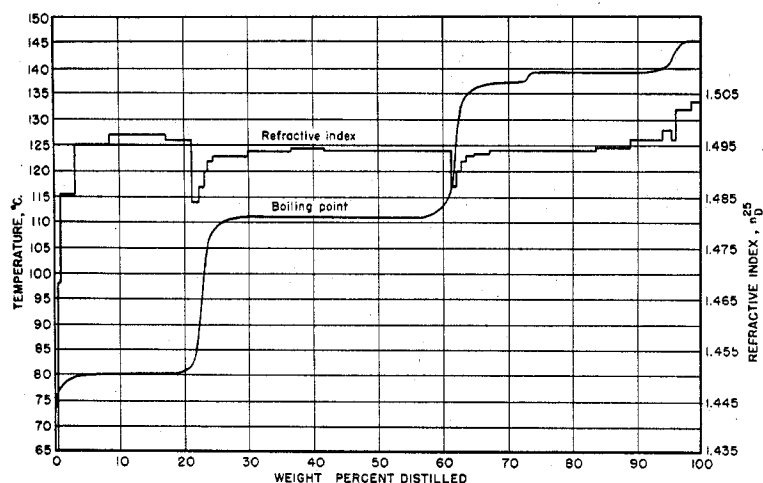


Figure 1. Distillation of Aromatic Portion from Vapor-Phase Gasoline Boiling between 50° and 150° C.

These combined fractions were 19.1% by weight of the vapor-phase feed and this was the portion used for subsequent analysis.

The second sample studied consisted of the stabilized gasoline (boiling point 50° to 200° C.) produced by a recycle vapor-phase hydrogenation operation conducted on the vapor-phase feed at a total pressure of 10,000 pounds per square inch and at 490° C. over a catalyst prepared from molybdenum on fuller's earth. This material is hereafter described as the "vapor-phase product." The yield of vapor-phase product from vapor-phase feed is usually approximately quantitative on a volume basis.

SCOPE OF INVESTIGATION

A detailed analysis is presented of the aromatics through C₈ in the vapor-phase feed and the vapor-phase product. The cycloparaffins that are potential sources of these aromatics are also characterized.

EXPERIMENTAL

In the separations and analyses described in the following paragraphs, both vapor-phase feed and vapor-phase product were treated identically. For the sake of convenience and clarity, reference is made to only one sample, although the treatment was the same for the other sample, except where otherwise indicated.

1. Isolation of the C₆ to C₈ Hydrocarbon Portion. A 40-plate, 1-inch-diameter Oldershaw column was used for distilling an 8-liter sample of the dry oil. The fraction boiling between 50° and 150° C. at 740 mm. of mercury was used for subsequent characterization.

2. Separation of Hydrocarbon Types. A 2-liter portion of the distillate was passed through two jacketed, stainless steel columns connected in series and containing silica gel (Davison) of 250 to 350 mesh; coolant at 10° F. was cir-

culated through the jackets. The first column was 2 inches in diameter and 6 feet long, the second was 1 inch in diameter and 9 feet long. Ethyl alcohol was used as a displacing agent. The desired flow rates were secured by subjecting the top of the column to air pressure of about 49 pounds per square inch. This treatment effected separation of the oil into a paraffin-naphthene portion, an olefin portion, an aromatic portion, and a strongly adsorbed portion containing oxygen, nitrogen, or sulfur compounds. The results of this separation are shown in Table III.

3. Distillation of Hydrocarbon Types. The aromatic portion was distilled in a Heligrad column 2 feet long, and 8 mm. in diameter, operating at an efficiency of approximately 15 theoretical plates. These distillations are plotted in Figures 1 and 3. The intermediate C₆ to C₇ portion and the intermediate C₇ to C₈ and all the C₈ fractions were examined by ultraviolet spectroscopy for the individual components.

An aliquot of the paraffin-naphthene portion was distilled at a reflux ratio of 120 to 1 in a Heligrad column 6 feet long and 13 mm. in diameter, operating at an efficiency of approximately 50 theoretical plates. Fractions boiling over 2° C. intervals were collected. These fractions are hereafter referred to

TABLE IV. COMPARISON OF ANALYTICAL METHODS

(C₆ to C₈ hydrocarbons as weight per cent of paraffin-naphthene portion of 50° to 150° C. cut of vapor-phase feed)

	R. ^a	Ult. ^b	Ms. ^c	Ms. ^d
Paraffins	24.6	24.8 ^e	23.13 ^e	22.87
Hexanes	6.7	4.32 ^f
Heptanes	9.1	10.0 ^g	9.26	10.76
Octanes	8.8	10.6 ^h	9.55	7.79
Naphthenes	57.12	56.1 ^e	59.58 ^e	61.39
C ₆	14.54	15.26
C ₇	22.23	21.6 ^g	23.40	24.54
C ₈	20.35	19.2 ^h	20.92	21.59
Methylcyclopentane	3.63	4.91 ^f
Dimethylcyclopentanes	4.55	6.21
Ethylcyclopentane	4.83	3.38
Propylcyclopentanes	2.73
Cyclohexane	10.71	10.35 ^f
Methylcyclohexane	12.85	14.95
Dimethylcyclohexanes	6.48
Trimethyl, methylethylcyclopentanes and ethylcyclohexane	12.38

^a Refractivity intercept method on individual fractions of narrow-boiling range.

^b Ultimate analysis of blends used in column Ms.^c.

^c Mass spectrographic analysis for total paraffins and total naphthenes in blends of narrow-boiling fractions above 82° C.

^d Mass spectrographic analysis for individual compounds in blends used in column Ms.^c.

^e Totals obtained by using values from column Ms.^d where necessary.

^f On individual narrow-boiling fractions below 82° C.

^g C, 83.30% ± 0.06; H, 14.90% ± 0.01.

^h C, 85.16% ± 0.04; H, 14.89% ± 0.03.

TABLE III. DISTRIBUTION OF HYDROCARBON TYPES IN OILS FROM COAL HYDROGENATION

Component	Vapor-Phase Feed	Vapor-Phase Product
50° to 150° C. cut in gasoline portion (200° C. end point)	62.7	66.3
50° to 150° C. cut in total oil sample	12.0	66.3
Paraffin-naphthene portion of 50° to 150° C. cut	62.3	69.9
Aromatic portion of 50° to 150° C. cut	21.1	27.6
Olefin portion of 50° to 150° C. cut	9.6	1.1
O, N, and/or S-containing compounds in 50° to 150° C. cut	7.1	1.4
C ₆ to C ₈ paraffins, naphthenes, aromatics in 50° to 150° C. cut	75.2	88.5
C ₆ to C ₈ paraffins, naphthenes, aromatics in total oil	9.02 ^a	58.7 ^a

^a Used as conversion factor in Table VI.

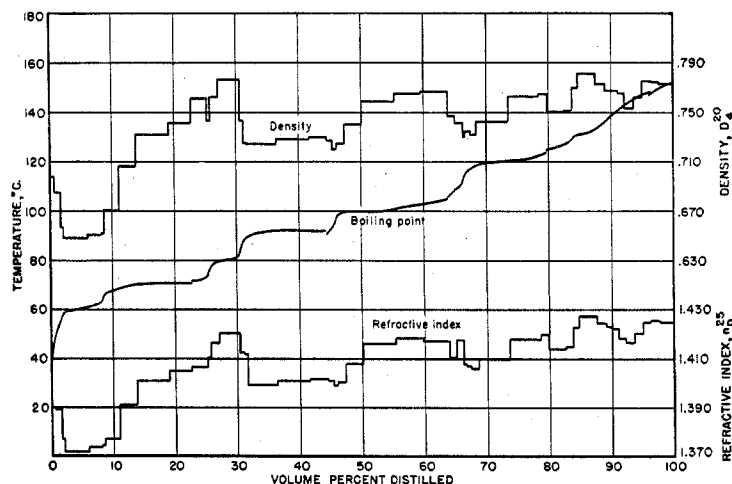


Figure 2. Distillation of Paraffin-Naphthene Portion from Vapor-Phase Gasoline Boiling between 50° and 150° C.

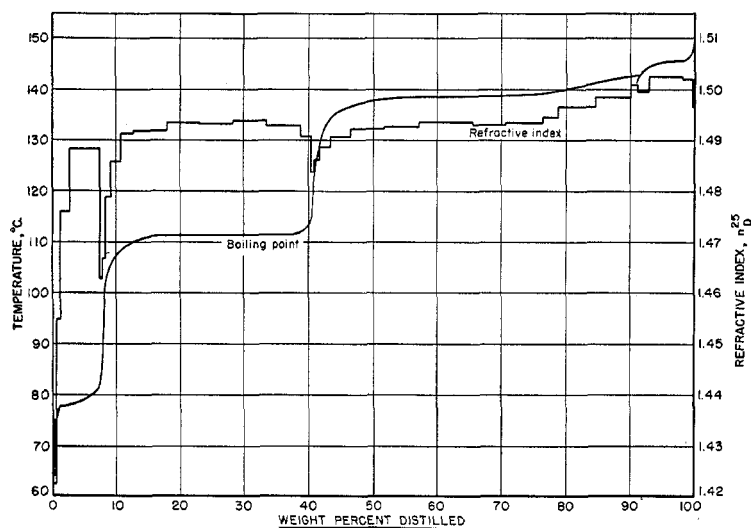


Figure 3. Distillation of Aromatic Portion from Vapor-Phase Feed Boiling between 50° and 150° C.

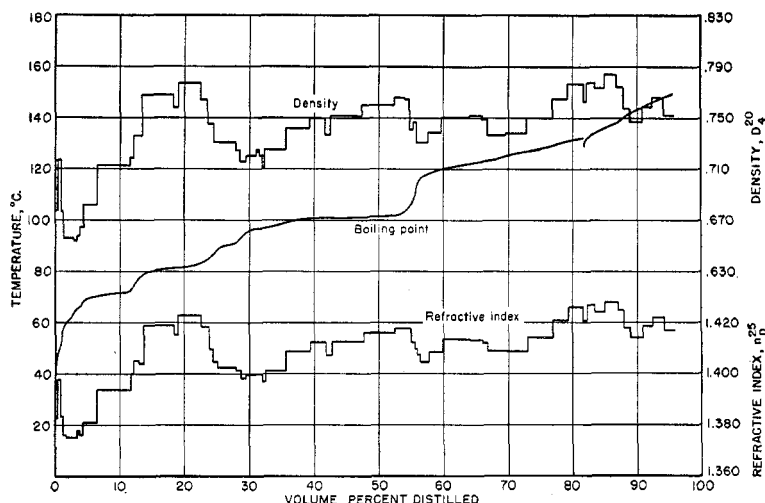


Figure 4. Distillation of Paraffin-Naphthene Portion from Vapor-Phase Feed Boiling between 50° and 150° C.

as narrow-boiling fractions. The refractive index and density of each fraction were measured, with the results shown in Figures 2 and 4. The width of each horizontal mark indicates the relative size of the fraction.

4. Analysis of Paraffin-Naphthene Portion. This part of the oil was analyzed by several methods.

REFRACTIVITY-INTERCEPT METHOD. The concentration of naphthenes in a paraffin-naphthene fraction of a particular boiling range can be estimated from charts on which the refractivity intercept is plotted against the density. The refractivity intercept, R , is defined as $R = n_D - 0.5d$, where n_D is the refractive index of the sodium D line and d , the density; both measurements are made at the same temperature (5). In carefully fractionated material, estimates of the various C_6 naphthenes and some of the C_7 naphthenes may be made. Of the C_8 naphthenes only the total can be determined by this method. The results are shown in Tables IV and V.

CARBON TO HYDROGEN RATIOS. A relatively simple method for estimating the distribution of paraffins and naphthenes in an olefin aromatic-free mixture of hydrocarbons of known boiling range is by analysis for carbon and hydrogen. Paraffins have the composition C_nH_{2n+2} , while monocyclic naphthenes have the general formula C_nH_{2n} , where n is the number of carbon atoms in the molecule. When the carbon to hydrogen ratio is known for a sample containing only saturated hydrocarbons of

the same number of carbon atoms, the proportion of cyclic to noncyclic molecules can be calculated.

A 100-ml. sample of the paraffin-naphthene portion was distilled in the 2-foot column. Three wide-boiling fractions boiling between 60° and 72° C., 72° and 110° C., and 110° and 135° C. were collected to segregate the isomeric paraffins into fractions containing the same number of carbon atoms. The first and lowest-boiling cut contained practically all the C_6 paraffins and some methylcyclopentane. The fraction boiling between 72° and 110° C. contained all the C_7 paraffins and C_8 and C_7 naphthenes. The mixture of naphthenes does not affect the paraffin-naphthene analysis, because the carbon to hydrogen ratio is constant for naphthenes. If it is desirable to separate C_8 from C_7 naphthenes an additional cut point at 83° C. can be used. The fraction boiling between 110° and 135° contained C_8 hydrocarbons. Results of the analysis of the paraffin-naphthene portion by ultimate composition of the three distillate fractions are shown in Table V. Two blends of the narrow-boiling fractions, boiling between 83° and 110° and 110° and 136° C., were analyzed for paraffin-naphthene distribution by ultimate analysis. The results appear in Table IV.

MASS SPECTROSCOPIC ANALYSIS. Friedel (4) has described a mass spectroscopic method for analyzing mixtures of paraffins and cycloparaffins. The method distinguishes among paraffins, cyclopentanes, monosubstituted cyclopentanes, disubstituted cyclopentanes, cyclohexane, monosubstituted cyclohexanes, and disubstituted cyclohexanes.

The analysis of the wide-boiling fractions for total paraffins and total naphthenes is given in Table V, while the analysis of the two blends of the narrow-boiling fractions is given in Tables IV and V. Spectroscopic examination of these blends for all components is also given. Probably the most accurate data were obtained by spectrographic analysis of each of the narrow-boiling fractions obtained by distillation of the paraffin-naphthene portion, Table V. This time-consuming operation did not give results very different from those secured by mass spectrographic analysis of the blends, Table V, except that it was thus possible to distinguish ethylcyclohexane from its mixture with some unknown methylethylcyclopentane which gave the same mass peak. If mass spectrographic analysis is to be employed, it is possible to secure an almost complete breakdown of composition by examination of wide-boiling fractions. Such analyses are described in detail by Friedel (4).

COMPARISON OF METHODS. The most accurate and complete analysis for individual paraffins and naphthenes in the C_6 to C_8 range can be achieved by mass spectrographic analysis. If information on the proportions of paraffins and naphthenes in the C_6 to C_8 range is all that is desired, carbon-hydrogen analysis of

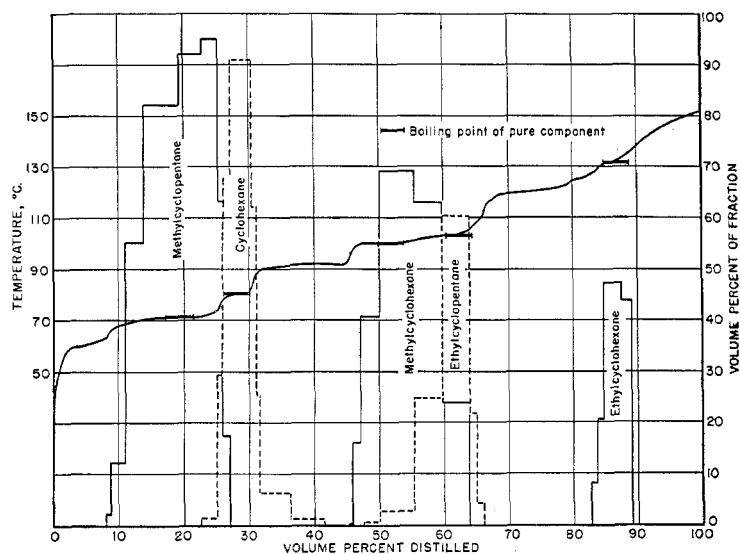


Figure 5. Distribution of Some Naphthenes in Distillate Fractions of Paraffin-Naphthene Portion from Vapor-Phase Gasoline

TABLE V. COMPARISON OF ANALYTICAL METHODS

C₆ to C₈ hydrocarbons as weight per cent of paraffin-naphthene portion of 50° to 150° C. cut of vapor-phase product

	R. ^a	Ult. ^b	Ms. ^c	Ms. ^d	Ms. ^e	Ms. ^f
Paraffins	23.90	26.3	22.44 ^g	23.13 ^g	23.6	24.27
Hexanes	11.31	9.4 ^h	9.2	11.02
Heptanes	7.40	9.5 ⁱ	6.60	7.36	9.9	8.40
Octanes	5.19	7.4 ^j	4.82	4.75	4.5	4.85
Naphthenes	62.80	58.7	66.12 ^g	65.35 ^g	64.0	63.73
C ₆	17.19	43.3 ^{h,i}	18.17	...
C ₇	26.22	...	27.60	26.73	45.5	25.41
C ₈	19.39	15.4 ^j	20.35	20.45	18.5	20.15
Methylcyclopentane	12.17	12.80
Dimethylcyclopentanes	9.88	12.83	...	11.99
Ethylcyclopentane	6.92	4.40	...	4.25
Propylcyclopentanes	2.01	...	1.83
Cyclohexane	5.02	5.37
Methylcyclohexane	9.42	9.50	...	9.17
Dimethylcyclohexanes	6.50	...	6.25
Ethylcyclohexane	2.48
Trimethyl and methyl-ethylcyclopentanes	11.94	...	9.59

^a, ^c, and ^d same as in Table IV.

^b Ultimate analysis of fractions of wide-boiling-range combustion-train method.

^e Mass spectrographic analysis for total paraffins and total naphthenes of fractions used in Ult.^b.

^f Mass spectrographic analysis for all components in fractions used in R.^a.

^g Totals obtained by using values from column Ms.^b where necessary.

^h C, 84.61% ± 0.08; H, 15.51% ± 0.05.

ⁱ C, 85.41% ± 0.11; H, 14.80% ± 0.04.

^j C, 85.31% ± 0.04; H, 14.78% ± 0.01.

TABLE VI. COMPOSITION OF VAPOR-PHASE PRODUCT

Component	Weight, %
C ₆ to C ₈ Paraffins	11.15
C ₆ to C ₈ Naphthenes	29.29
C ₆ to C ₈ Aromatics	18.30
C ₉ + Aromatics (b.p. <185° C.)	6.10 ^a
C ₉ + Paraffins (b.p. <176° C.)	2.92
C ₉ + Cyclopentanes (b.p. <176° C.)	2.61
C ₉ + Cyclohexanes (b.p. <176° C.)	6.04
Hydrindane	0.55
Other bicyclic naphthene (b.p. <176° C.)	0.30
Paraffins and naphthenes (b.p. 176–185° C.)	2.05
Olefins (b.p. 50–185° C.)	1.28
O, N, and/or S compounds (b.p. 50–185° C.)	2.22
Material boiling below 50° C.	9.64
Material boiling above 185° C.	2.83
Losses and unaccounted for	4.72
Total	100.00

^a Includes approximately 0.6% indan.

three wide-boiling samples is adequate. If a mass spectrometer is unavailable and information regarding individual compounds in the C₆ to C₇ range is desired, the determination of the refractivity intercept on narrow-boiling cuts is satisfactory.

5. Analysis of Vapor-Phase Product Boiling between 150° and 185° C. A 530-gram aliquot of this fraction obtained from the initial distillation (described in 1) was chromatographed on the 1-inch stainless steel column packed with silica gel. After rechromatographing on a small column, this sample was shown to contain 57.2% paraffin-naphthenes, 2.9% olefins, 33.5% aromatics, and 6.5% oxygen, nitrogen, and/or sulfur-containing compounds. The paraffin-naphthene portion was blended with the residue from the paraffin-naphthene portion of the material boiling between 50° and 150° C. (described in 3) and the mixture was distilled. Individual narrow-boiling fractions up to 176° C. were analyzed by mass spectrometry. The aromatic portion was distilled and a portion of the material with high refractive index was analyzed for indan by sulfonation. The results of the analyses of this portion of the gasoline are incorporated in Table VI which shows the detailed composition of the vapor-phase product.

DISCUSSION OF RESULTS

The various methods of analysis for the paraffin-naphthene portion give roughly comparable results. The choice of a particular method depends on the precision and the amount of information required as well as on the equipment and manpower available.

TABLE VII. DISTRIBUTION BY WEIGHT-PERCENTAGE OF C₆ to C₈ NONOLEFINIC HYDROCARBONS

Component	In Vapor-Phase Feed ^a	In Vapor-Phase Product ^b
Paraffins	21.92	19.01
Hexanes	4.14	8.63
Heptanes	10.31	6.58
Octanes	7.47	3.80
Naphthenes	58.83	49.90
Methylcyclopentane	4.71	10.02
Ethylcyclopentane	3.24	3.33
Dimethylcyclopentanes	5.95	9.39
Propylcyclopentanes	2.62	1.43
Trimethyl and methylethylcyclopentanes	4.31	7.51
Cyclohexane	9.92	4.21
Methylcyclohexane	14.33	7.18
Ethylcyclohexane	7.55	1.94
Dimethylcyclohexanes	6.21	4.89
Aromatics	19.25	31.09
Benzene	0.80	6.68
Toluene	6.60	12.65
Ethylbenzene	4.12	5.00
<i>o</i> -Xylene	2.89	1.48
<i>m</i> -Xylene	3.37	3.55
<i>p</i> -Xylene	1.46	1.73
Total	100.00	100.00

See Table III:

^a Multiply figures in this column by 0.0902 to secure concentration on basis of total oil.

^b Multiply figures in this column by 0.587 to secure concentration on basis of total oil.

Caution must be exercised in drawing conclusions based on distillation data. In Figure 5 the distribution of some of the individual cycloparaffins is plotted against their location on the boiling point curve obtained in the 50-plate distillation of the paraffin-naphthene portion of the vapor-phase gasoline. In the figure, broken lines have been used where necessary to indicate overlap of compounds. Although the bulk of each component was present in the portion of the distillate boiling within 2° C. of the boiling point of the pure compound, small but significant quantities appeared in fractions as much as 8° or 10° C. above and below that boiling point. Distillation alone is not capable of giving completely sharp separations, even when nearly ideal solutions are distilled in columns of relatively high efficiency. The accuracy of an analysis (such as that by refractivity intercept) which assumes the presence or absence of any particular compound or compounds in a narrow-boiling fraction is therefore limited by the distillation technique.

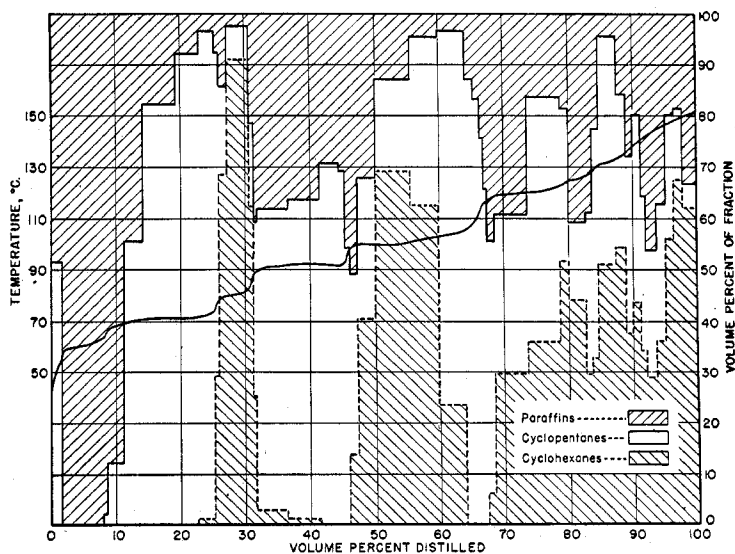


Figure 6. Paraffin-Naphthene Distribution in Vapor-Phase Gasoline

The distribution of the C_6 to C_8 paraffins and naphthenes in the vapor-phase product as determined by mass spectrographic analysis of narrow-boiling cuts in the 50-plate distillation is given in Figure 6. Examination of this figure in conjunction

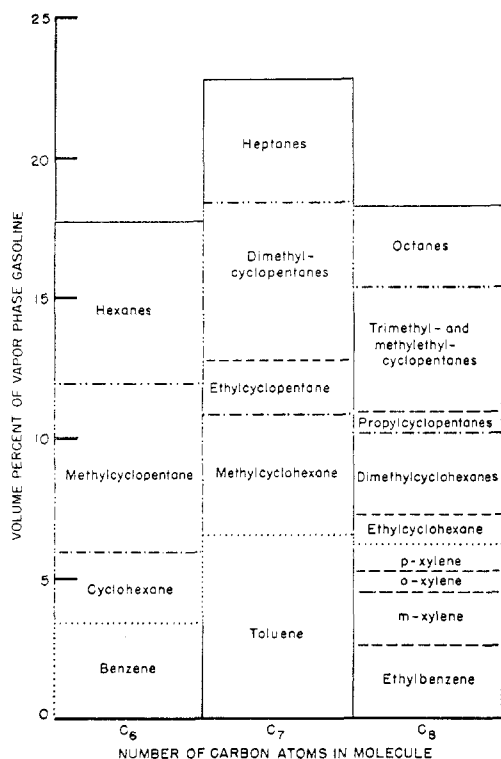


Figure 7. C_6 to C_8 Hydrocarbons in Vapor-Phase Gasoline

Area under.....represents aromatic content of vapor phase gasoline
 Area under-----represents aromatics available by dehydrogenation
 Area under-----represents aromatics available by isomerization and dehydrogenation

with Figure 5 shows that temperature cut points of 72° and 110° C. segregate the molecular weight species required for ultimate analysis. Furthermore the spectral data similarly indicate that these were the points of molecular weight change for the paraffins.

In Figure 7 the hydrocarbons are arranged in the order of their importance as sources of light aromatics. It is assumed that the conversion of naphthenes to aromatics can be made quantitative on a volume basis (6).

The compositions of the vapor-phase feed and the vapor-phase product with respect to the C_6 to C_8 nonolefinic hydrocarbons are given in Table VII. Examination of this table indicates that vapor-phase processing, in addition to effecting molecular weight reduction and aromatization, also results in isomerization and yields an isomer distribution more nearly corresponding to that expected from thermodynamic considerations (1, 8). This is shown by the shift from cyclohexanes to cyclopentanes, by the decrease in the ratios of ethylcyclohexane to dimethylcyclohexanes, and of ethylcyclopentane to dimethylcyclopentanes, and by the increase in the ratio of *m*-xylene to *o*-xylene.

The gasoline produced by coal hydrogenation is extremely rich in naphthenes and is, therefore, an ideal stock for conversion to aromatics by processes such as hydroforming and platforming. If, for example, one assumes that the naphthenes are converted to the corresponding aromatics in yields of 80%, then the aromatic content of such processed gasoline would be that indicated in the table below.

	Wt. % of Total Product
Benzene	10.6
Toluene	16.7
C_8 aromatics	14.9

On this basis, the yield of benzene from coal hydrogenation can be 18.0 gallons per ton of dry, ash-free coal compared to 2 gallons per ton secured by coal carbonization.

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