U. S. Department of Commerce
National Bureau of Standards

Research Paper RP2065
Volume 44, February 1950

Part of the Journal of Research of the National Bureau of Standards

Hydrocarbons in the 108° to 116° C Fraction of Petroleum[1]

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This report describes the analysis of the hydrocarbons in the 108° to 116° C aromatic-free fraction of petroleum, which is shown to be composed of eleven hydrocarbon compounds, of which three are present only in very small amount. For these eleven compounds, the normal boiling points (of high purity samples not from the present investigation) and the estimated amounts by volume in the original Ponca, Okla., crude petroleum are as follows: 2,5-dimethylhexane, 109.11° C, 0.055 percent; 1,*trans*-2,*cis*-4-trimethylcyclopentane, 109.28° C, 0.22 percent; 2,4-dimethylhexane, 109.43° C, 0.055 percent; 2,2,3-trimethylpentane, 109.84° C, 0.004 percent; 1, *trans*-2,*cis*-3-trimethylcyclopentane, 110.4° C, 0.26 percent; 3,3-dimethylhexane, 111.97° C, 0.03 percent; 2,3,4-trimethylpentane, 113.47° C, 0.005 percent; 1,1,2-trimethylcyclopentane, 113.72° C, 0.06 percent; 2,3,3-trimethylpentane, 114.76° C, 0.006 percent; 2,3-dimethylhexane, 115.61° C, 0.06 percent; and 2-methyl-3-ethylpentane 115.65° C, 0.04 percent.

I. Introduction

As part of the work of the American Petroleum Institute Research Project 6 at the National Bureau of Standards on the fractionation and analysis of hydrocarbons in petroleum [1, 2][2] the program has been completed on that part of petroleum normally boiling between 108° and 116° C. In addition to toluene, the separation of which was previously reported [12], this fraction of petroleum is shown to be composed of eleven hydrocarbon compounds, of which three are present only in very small amount. This report describes the foregoing work and gives the detailed results.

II. Material Analyzed

The American Petroleum Institute Research Project 6 at the National Bureau of Standards has had under investigation since 1928 a large quantity of a representative petroleum taken from a well at Ponca, Okla. [2, 10]. The material analyzed in the present investigation constituted that part of this original petroleum normally boiling between the 108° and 116° C, from which toluene had been removed [12]. The status of this material prior to the present investigation is described in reference [5]. The material was made aromatic-free by filtration through silica gel [16].

III. Method of Analysis and Results

The aromatic-free material of the original petroleum remaining after the previous treatment [4, 5, 11, 12] was "lined-up" by appropriate preliminary distillation and blending in order to obtain in one lot all of the remaining original petroleum normally boiling between 108° and 116° C. The results of this distillation are shown in figure 1. Further separation of this distillate into its constituents is described with respect to the components normally boiling within the ranges 108° to 111° C, 111° to 114° C, and 114° to 116° C, where more or less abrupt changes in the boiling point of the material occurred. The details of the distillations performed in this investigation are given in table 1. Reference [6] gives a description of the distilling columns used.

1. Material normally boiling from 108° to 111° C

The material normally boiling from 108° to 111° was separated by distillation and found to be composed of five compounds, 2,5-dimethylhexane at 109.11°C, 1,*trans*-2,*cis*-4-trimethylcyclopen-

[1] This investigation was performed as part of the work of the American Petroleum Institute Research Project 6 at the National Bureau of Standards on the "Analysis, Purification, and Properties of Hydrocarbons".

[2] Figures in brackets indicate the literature references at the end of this paper.

TABLE 1. *Information on the distillation for the analysis and separation of 11 hydrocarbons (3 trimethylcyclopentanes and 8 branched-chain-octanes) from the 108° to 116° C (aromatic-free) fraction of petroleum*

Material	Distillation								
	Distilling [a] column number	Number of equivalent theoretical plates at total reflux (approximately)	Reflux ratio (approximately)	Rate of collection of distillate	Kind of distillation	Azeotrope-forming substance, if used, and its volume	Volume of hydrocarbon charged	Volume of each fraction of distillate	Results plotted in figure
				ml/hr			*ml*	*ml*	
108° to 116° C (aromatic-free) fraction of petroleum (showing lower- and higher-boiling material).	2	100	125/1	2.4	Regular		3,880	7.4	1
108° to 111° C (aromatic-free) portion (part *A*, fig. 1).	11A	200	170/1	4.2	Azeotropic	Ethanol, 6,000 ml	1,950	17.6	2
1,*trans*-2,*cis*-3-Trimethylcyclopentane concentrate (part *B*, fig. 1).	1A	150	135/1	2.2	Regular		460	9.7	3
1, *trans*-2,*cis*-4-Trimethylcyclopentane concentrate (derived from part *A*, fig. 1).	1A	150	300/1	1.0	Azeotropic	Ethanol, 300 ml	138	9.8	4
2,5-Dimethylhexane concentrate (derived from part *A*, fig. 1).	1A	150	335/1	.9	do	Ethanol, 230 ml	110	9.9	5
2,4-Dimethylhexane concentrate (derived from part *A*, fig. 1).	1A	150	335/1	.9	do	Ethanol, 150 ml	58	5.2	6
108° to 110° C (aromatic-free) portion (part *A*, figs. 2 and 3).	1A	150	150/1	2.0	do	Methyl Cellosolve, 750 ml	850	14.3	8
3,3-dimethylhexane concentrate (part *B*, fig. 1).	1A	150	135/1	2.2	do	Methyl Cellosolve, 304 ml	177	17.9	9
1,1,2-Trimethylcyclopentane concentrate (part *C*, fig. 1; part *B*, fig. 9).	1A	150	115/1	2.6	Regular		305	10.1	10
1,1,2-Trimethylcyclopentane concentrate (part *A*, figs. 9 and 10).	4	200	400/1	1.8	Azeotropic	Methyl Cellosolve, 300 ml	83	11.7	11
2,3-Dimethylhexane and 2-methyl-3-ethylpentane concentrate (part *D*, fig. 1).	1A	150	165/1	1.8	Regular		515	11.5	12
2,3-Dimethylhexane and 2-methyl-3-ethylpentane concentrate (part *A*, fig. 12).	1A	150	175/1	1.7	Azeotropic	Methanol, 600 ml	112	11.8	13
2,3-Dimethylhexane and 2-methyl-3-ethylpentane concentrate (part *B*, fig. 12).	1A	150	175/1	1.7	do	Methyl Cellosolve, 400 ml	217	10.7	14

[a] For further details see reference [6].

tane [3] at 109.28° C, 2,4-dimethylhexane at 109.43° C, 2,2,3-trimethylpentane at 109.84° C, and 1,*trans*-2-*cis*-3-trimethylcyclopentane at 110.4° C.

Following the processing shown in figure 1, 1,*trans*-2,*cis*-3-trimethylcyclopentane was separated using azeotropic distillation with ethanol followed by a regular distillation. The results are shown in figures 2 and 3. In the azeotropic distillation, the ethanol which comprised about 53 percent by volume of the distillate, was extracted from the hydrocarbon portion of the distillate by three cold water extractions in a separatory funnel. A similar procedure was used for the removal of methyl Cellosolve, which comprised about 24 percent by volume of the distillate. The "best lot" of 1,*trans*-2,*cis*-3-trimethylcyclopentane selected from the final distillate (shown in fig. 3) had the following properties: boiling point at 760 mm Hg, 110.4 ±0.1 deg C; refractive index, n_D at 25° C, 1.4112; freezing point in air at 1 atmosphere, −113.08 ±0.02 deg C; amount of the main component, 98.60 ±0.15 mole percent.[4] The best lots of the hydrocarbons reported throughout this investigation were selected on the basis of the refractive index and the boiling point.

Following the distillations shown in figures 2 and 3, the part marked *A* in the figures was reprocessed by four azeotropic distillations, three with methyl Cellosolve (ethylene glycol mono-

[3] See reference [15] for nomenclature of substituted cycloparaffins.

[4] Determined by N. C. Krouskop as described in reference [17], using the cryoscopic constants given in reference [18].

methyl ether) and one with ethanol to fractionate the material into concentrates of: (a) 2,5- and 2,4-dimethylhexane; (b) 1,*trans*-2,*cis*-4-trimethylcyclopentane; and (c) 1,*trans*-2,*cis*-3-trimethylcyclopentane. The first two concentrates were processed further by azeotropic distillation with ethanol to produce higher concentrations of 1,*trans*-2,*cis*-4-trimethylcyclopentane, 2,5-dimethylhexane, and 2,4-dimethylhexane. The results of these distillations are shown in figures 4, 5, and 6. The concentration of 2,5-dimethylhexane, 2,4-dimethylhexane, 2,2,3-trimethylpentane, and 1,*trans*-2,*cis*-4-trimethylcyclopentane in selected fractions of the distillate was determined by spectrographic infrared absorption measurements made by the Socony-Vacuum Laboratories, Paulsboro, N. J. The results are given in table 2. The fractions containing the highest concentration had the following amounts of the named component in mole percent: 2,5-dimethylhexane, 55 ±5; 2,4-dimethylhexane, 41 ±4; 2,2,3-trimethylpentane, 1.0 ±0.5; 1,*trans*-2,*cis*-4-trimethylcyclopentane, 84 ±5.

The following conclusions may be drawn from the results of the processing of the material normally boiling in the range 108° to 111° C: (a) The first azeotropic distillation (fig. 2) following the regular distillation (part *A* of fig. 1) produced an appreciable enhancement in the separation, as shown in figure 7; (b) ethanol was more effective than methyl Cellosolve in separating azeotropically the two dimethylhexanes from 1,*trans*-2,*cis*-4-trimethylcyclopentane, as shown in figure 8; whereas methyl Cellosolve was more effective than ethanol in separating the two trimethylcyclopentanes, one from the other, as shown in figure 8; (c) 1,*trans*-2,*cis*-3-trimethylcyclopentane is readily separated from the mixture of the five hydrocarbons by a combination of regular and azeotropic distillation, whereas 1,*trans*-2,*cis*-4-trimethylcyclopentane is much less readily separated by the same processes, requiring more distillation; (d) the two dimethylhexanes are only partially separated from each other and from 1,*trans*-2,*cis*-4-trimethylcyclopentane by regular and azeotropic distillation.

2. Material Normally Boiling from 111° to 114° C

The material normally boiling from 111° to 114° C was separated by distillation and found

TABLE 2. *Results of the spectrographic infrared absorption measurements,[a] of selected samples from the 108° to 116° C aromatic-free fraction of Ponca, Okla., petroleum*

Identification of Sample	2,2-Dimethylhexane	2,5-Dimethylhexane	1, *trans*-2, *cis*-4-Trimethylcyclopentane	2,4-Dimethylhexane	2,2,3-Trimethylpentane	1, *trans*-2, *cis*-3-Trimethylcyclopentane	3,3-Dimethylhexane	2,3,4-Trimethylpentane	1,1,2-Trimethylcyclopentane	2,3,3-Trimethylpentane	2,3-Dimethylhexane	2-Methyl-3-ethylpentane	Unknown
	PERCENTAGE BY WEIGHT IN THE SAMPLE												
Part *C*; figure 2	21 ±2	35 ±3	28 ±5	16 ±2	------	----	----	----	------	----	-------	-------	------
1, *trans*-2, *cis*-4-Trimethylcyclopentane; figure 4.	----	1 ±0.5	84 ±5	6 ±2	0.5 ±0.3	6 ±2	----	----	------	----	-------	-------	2.5 ±1.0
2,5-Dimethylhexane; figure 5.	2 ±1	55 ±5	21 ±2	22 ±2	------	----	----	----	------	----	-------	-------	------
Part *A*; figure 5	----	20 ±2	47 ±5	32 ±3	1.0 ±0.5	----	----	----	------	----	-------	-------	------
Part *A*; figure 6	----	18 ±2	40 ±4	41 ±4	1.0 ±.5	----	----	----	------	----	-------	-------	------
Part *B*; figure 6	----	29 ±3	35 ±4	35 ±4	1.0 ±.5	----	----	----	------	----	-------	-------	------
3,3-Dimethylhexane; figure 9.	----	-----	----	----	------	10 ±2	86 ±5	----	1.0 ±0.5	----	-------	-------	[b]3 ±1
Part *C*; figure 9	----	-----	----	----	------	11 ±2	79 ±5	----	2 ±1	----	-------	-------	8 ±2
Part *A*; figure 11	----	-----	----	----	------	----	31 ±3	17 ±2	52 ±4	----	-------	-------	------
Part *B*; figure 11	----	-----	----	----	------	----	16 ±2	17 ±2	67 ±5	----	-------	-------	------
Part *A*; figure 13	----	-----	----	----	------	----	----	5 ±2	63 ±5	10 ±2	12 ±2	10 ±2	------
Part *B*; figure 13	----	-----	----	----	------	----	----	----	19 ±2	4 ±2	47 ±5	30 ±3	------
Part *D*; figure 13	----	-----	----	----	------	----	----	----	------	----	66 ±5	34 ±3	------
Part *B*; figure 14	----	-----	----	----	------	----	----	----	------	----	60 ±5	40 ±5	------
Original of experiment No. 4; figure 15.	----	-----	----	----	------	----	----	----	------	----	54.5 ±5.0	45.5 ±5.0	------
Part *B* of experiment No. 4; figure 15.	----	-----	----	----	------	----	----	----	4 ±2	3 ±1	48 ±5	45 ±5	------

[a] Made by the Socony-Vacuum Laboratories, Paulsboro, N. J. A description of the apparatus is given in reference [3].

to be composed of three compounds, 3,3-dimethylhexane at 111.97° C, 2,3,4-trimethylpentane at 113.47° C, and 1,1,2-trimethylcyclopentane at 113.72° C.

Following the distillation shown in figure 1, the parts marked *B* and *C* in that figure were processed to separate 3,3-dimethylhexane and 1,1,2-trimethylcyclopentane. 3,3-Dimethylhexane was separated by azeotropic distillation with methyl Cellosolve as shown in figure 9, and 1,1,2-trimethylcyclopentane was separated by regular distillation followed by azeotropic distillation with methyl Cellosolve as shown in figures 10 and 11, respectively. The concentration of 3,3-dimethylhexane and 2,3,4-trimethylpentane in selected fractions of the distillate was determined by spectrographic infrared absorption measurements made by the Socony-Vacuum Laboratories. The results are given in table 2. The fractions containing the highest concentration had the following amounts of the named component in mole percent: 3,3-dimethylhexane, 86 ±5; 2,3,4-trimethylpentane, 17 ±2. The "best lot" of 1,1,2-trimethylcyclopentane selected from the final distillate shown in figure 11 had the following properties: Boiling point at 760 mm Hg, 113.7 ±0.1° C; refractive index, n_D, at 25° C, 1.4192; freezing point in air at 1 atmosphere, −30.21 ±0.05° C; amount of the main component, 97.50 ±0.06 mole percent (see footnote 4).

From the results of the processing of the material normally boiling in the range 111° to 114° C, it can be concluded that 3,3-dimethylhexane and 1,1,2-trimethylcyclopentane are both readily separable from this fraction of the Ponca, Okla., petroleum by high efficiency distillation.

3. Material Normally Boiling from 114° to 116° C

The material normally boiling from 114° to 116° C was processed by distillation, supplemented by adsorption, and was found to be composed of three compounds, 2,3,3-trimethylpentane at 114.76° C, 2,3-dimethylhexane at 115.61° C, and 2-methyl-3-ethylpentane at 115.65° C.

Following the distillation shown in figure 1, the portion of this distillate (part *D*) containing the concentrate of the above three hydrocarbons was distilled regularly, followed by azeotropic distillation with methanol and methyl Cellosolve as shown in figures 12, 13, and 14, respectively. Distillate from the above azeotropic distillations, containing mainly a mixture of 2,3-dimethylhexane and 2-methyl-3-ethylpentane, was processed further by adsorption [5] [16, 19] through glass columns (790 cm in length and 1 cm in diameter) with silica gel [6] as the adsorbent and ethanol as the desorbing liquid. The results of the separation of 2-methyl-3-ethylpentane from 2,3-dimethylhexane by adsorption are given in figure 15. The charges for these separations by adsorption were as follows: 1, part *A* of figure 14; 2, part *B* of 1 above; 3, part *C* of figure 13 and part B of figure 14; and 4, part *A* from 1, part *A* from 2, and part *B* from 3 above. The "best lot" from petroleum of the two paraffins was selected from the adsorption separations shown in figure 15 as follows: 2-methyl-3-ethylpentane, part *B* from experiment 4; 2,3-dimethylhexane, parts *C*, *C*, and *D* from experiments 1, 2, and 3, respectively.

The concentration of 2,3,3-trimethylpentane, 2,3-dimethylhexane, and 2-methyl-3-ethylpentane in selected fractions of the distillate and the filtrate from the adsorptions was determined by spectrographic infrared absorption measurements made by the Socony-Vacuum Laboratories. The results are given in table 2. The fractions containing the highest concentration had the following amounts of the named component in mole percent: 2,3,3-trimethylpentane, 10 ±2; 2,3-dimethylhexane, 66 ±5; 2-methyl-3-ethylpentane, 45 ±5.

The following conclusions may be drawn from the results of the processing of the material normally boiling in the range 114° to 116° C: (a) Regular and azeotropic distillation produced essentially a binary mixture of the two close-boiling paraffins, 2,3-dimethylhexane and 2-methyl-3-ethylpentane; (b) fractionation by adsorption resulted in a further slight separation of these two paraffins and a further separation of the small amount of 1,1,2-trimethylcyclopentane associated with them.

IV. Amounts of the Components in Petroleum

The calculation of the amounts by volume of the 11 compounds in the aromatic-free fraction of petroleum normally boiling between 108° and 116° C was made by appropriate reduction, as

[5] Performed under the supervision of B. J. Mair, in charge of Fractionation and Analysis, Section on Thermochemistry and Hydrocarbons.

[6] Silica gel No. 22-08, Davison Chemical Company, Baltimore, Md.

described in reference [13], of the data on boiling point and refractive index as a function of the volume of distillate, as shown in figure 1, supplemented by the results of the spectrographic infrared absorption measurements on selected fractions as listed in table 2. The spectrographic data served to establish the relative amounts of 2,5-dimethylhexane and 2,4-dimethylhexane, of 2,3-dimethylhexane and 2-methyl-3-ethylpentane, and of the three trimethylpentanes. The data on boiling point and refractive index as a function of the volume of distillate were utilized to the fullest extent of their precision by plotting the data on expanded scales of temperature, refractive index, and volume.

The relative amounts by volume in the aromatic-free fraction 108° to 116° C of the Ponca, Okla., petroleum was translated into amounts based on the original crude petroleum using unpublished data (percentage of total crude this fraction comprised) obtained in the investigation of the American Petroleum Institute Research Project 6 on the gasoline fraction of seven representative crudes, which includes the Ponca, Okla., petroleum [8, 9, 14]. Table 3 gives these amounts, together with the amount of toluene [9], in the original crude petroleum.

TABLE 3. *Amounts of the 12 hydrocarbons constituting the 108° to 116° C fraction of the petroleum*

Component	Boiling point at 1 atm[a]	Relative amount by volume in the aromatic-free portion	Amount in the original crude petroleum[b]
	° C		*Percentage by volume*
2,5-Dimethylhexane	109.11	7.	0.055
1,*trans*-2,*cis*-4-Trimethylcyclopentane	109.28	28.	.22
2,4-Dimethylhexane	109.43	7	.055
2,2,3-Trimethylpentane	109.84	0.5	.004
1,*trans*-2,*cis*-3-Trimethylcyclopentane	110.4	33.	.26
Toluene	110.62		*.51
3,3-Dimethylhexane	111.97	3.	.03
2,3,4-Trimethylpentane	113.47	0.6	.005
1,1,2-Trimethylcyclopentane	113.72	7.	.06
2,2,3-Trimethylpentane	114.76	0.8	.006
2,3-Dimethylhexane	115.61	8.	.06
2-Methyl-3-ethylpentane	115.65	5.	.04
Total		100.	1.30

[a] From reference [7].

[b] For each component, the percentage by volume of the gasoline fraction, 40° to 180° C, is about 3 times the value in this column.

[c] From reference [9].

From the data given in table 3, the following points may be made regarding the composition with respect to volume of the 108° to 116° C fraction of petroleum:

(a) The material is comprised entirely of three types of hydrocarbons with the following relative amounts: isoparaffins, 20; cyclopentanes, 41; and an alkyl benzene, 39.

(b) The isoparaffins with dialkyl and trialkyl substituents are present in the relative amounts of 15 and 1, respectively.

(c) The relative amounts of the isoparaffins and the alkyl cyclopentanes are approximately 1 and 2, respectively.

(d) The alkyl benzene, toluene, constitutes about two-fifths of the entire fraction 108° to 116° C.

(e) Two of the alkyl cyclopentanes (1,*trans*-2, *cis*-4-trimethylcyclopentane and 1,*trans*-2, *cis*-3-trimethylcyclopentane) together constitute about 60 percent of the aromatic-free fraction 108° to 116° C.

Acknowledgment is made to F. P. Hochgesang, Socony-Vacuum Laboratories, Paulsboro, N. J., for the spectrographic infrared absorption measurements reported in table 2 of this paper.

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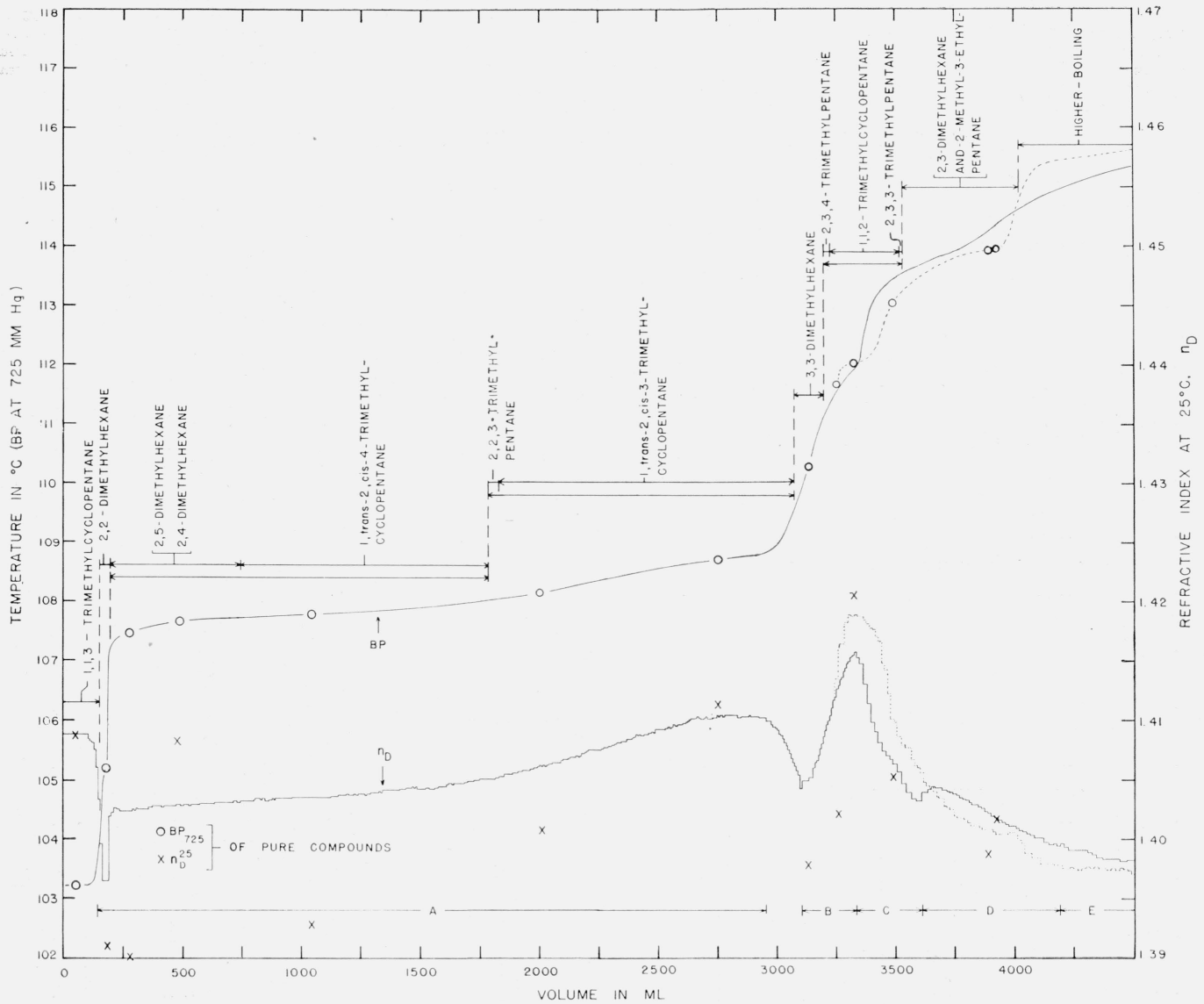

FIGURE 1. *Distillation of the 108° to 116° C (aromatic-free) fraction of Ponca, Okla., petroleum.*

The relative amounts by volume of the various components are indicated in the upper portion of the figure. The dashed curves for the boiling point and the refractive index are those obtained after redistillation of this portion of the distillate. The portions A to E, enclosed with arrows, were redistilled.

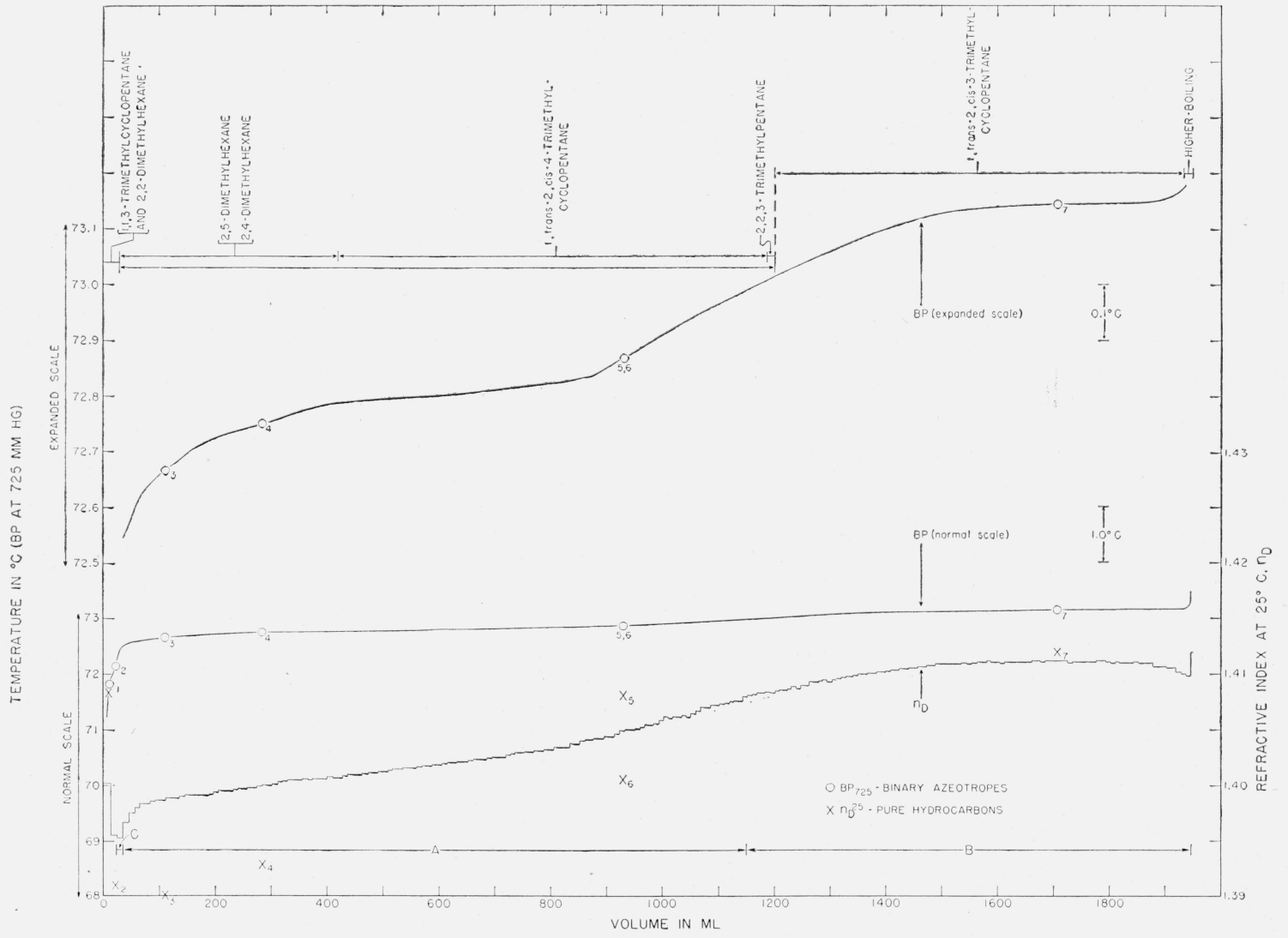

FIGURE 2. *Distillation of the 108° to 111° C (aromatic-free) portion (part A, fig. 1).*

The ordinate scale on the right gives the refractive indices of the hydro carbon portion of the azeotropic distillate. The scale of ordinates on the left gives a normal and an expanded scale of boiling point for the azeotropic distillate. The scale of abscissas gives the volume of the hydrocarbon portion of the distillate. The relative amounts by volume of the various components are indicated in the upper portion of the figure. The portions A and B, enclosed with arrows, were redistilled. The fraction marked C was analyzed by spectrographic infrared-absorption measurements. The numbers accompanying the circles, which indicate the azeotropic boiling point of the pure hydrocarbon with ethanol, refer to the following: (1) to 2,2-dimethylhexane; (2) to 1,1,3-trimethylcyclopentane; (3) to 2,5-dimethylhexane; (4) to 2,4-dimethylhexane; (5) to 1,*trans*-2,*cis*-4-trimethylcyclopentane; (6) to 2,2,3-trimethylpentane; and (7) to 1,*trans*-2,*cis*-3-trimethylcyclopentane.

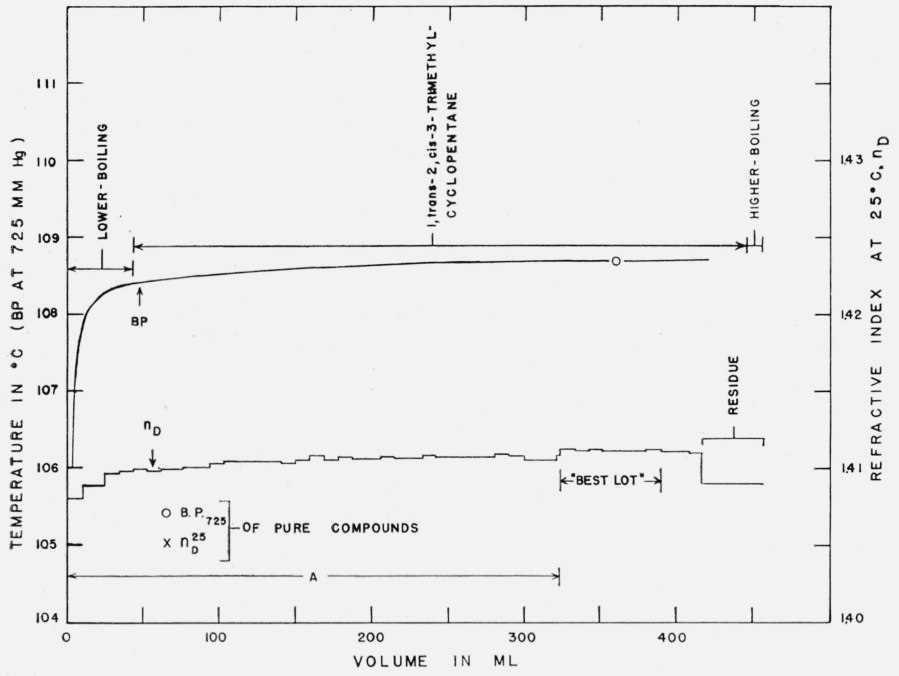

FIGURE 3. *Distillation of the 1,trans-2,cis-3-trimethlylcyclopentane concentrate (part B, fig. 2).*

The relative amounts by volume of the various components are indicated in the upper portion of the figure. The portion A, enclosed with arrows, was redistilled.

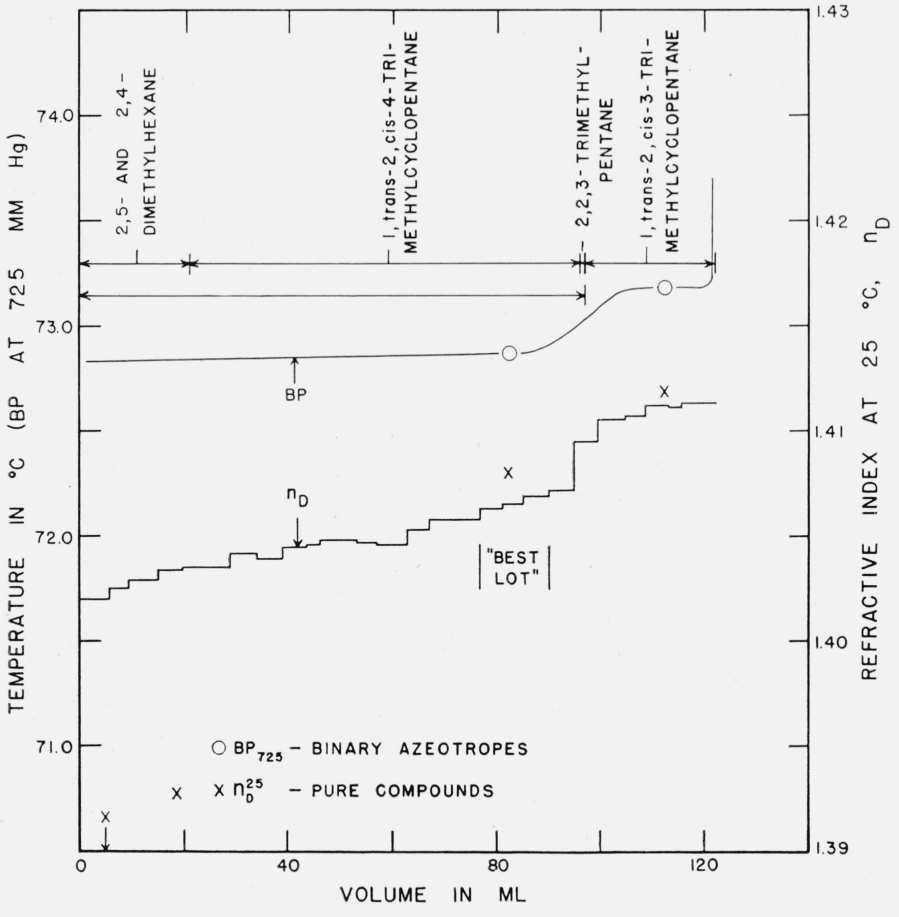

FIGURE 4. *Distillation of the concentrate of 1,trans-2,cis-4-trimethylcyclopentane (derived from part A, fig. 1).*

The ordinate scale on the right gives the refractive indices of the hodrocarbon portion of the azeotropic distillate, and the ordinate scale on the left gives the boiling point of the azeotropic distillate. The scale of abscissas gives the volume of the hydrocarbon portion of the distillate.

The relative amounts by volume of the various components are indicated in the upper portion of the figure. The portion labeled "best lot" was analyzed by spectrographic infrared absorption measurements, the results of which are shown in table 2.

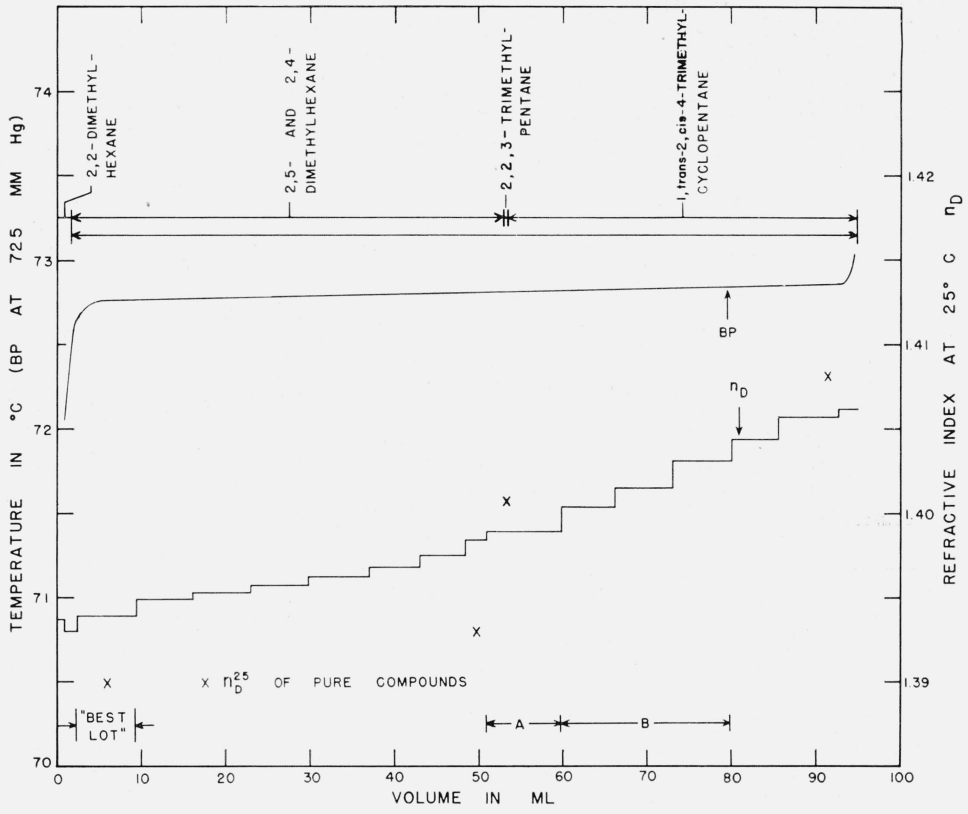

FIGURE 5. *Distillation of the 2,5-dimethylhexane concentrate (derived from part A, fig. 1).*

The ordinate scale on the right gives the refractive indices of the hydrocarbon portion of the azeotropic distillate, and the ordinate scale on the left gives the boiling point of the azeotropic distillate. The scale of abscissas gives the volume of the hydrocarbon portion of the distillate. The relative amounts by volume of the various components are indicated in the upper portion of the figure. The portions A and B, enclosed with arrows, were redistilled. The portion labeled "best lot" was analyzed by spectrographic infrared absorption measurements, the results of which are shown in table 2.

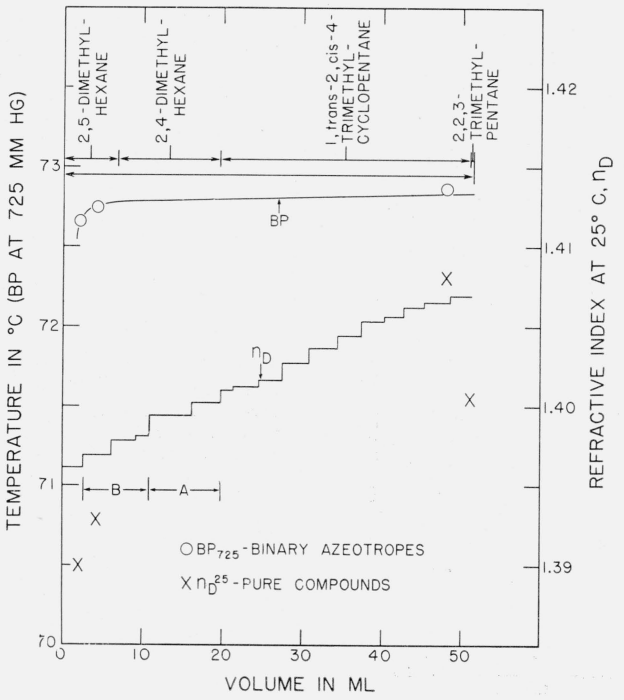

FIGURE 6. *Distillation of the 2,4-dimethylhexane concentrate (derived from part A, fig. 1).*

The ordinate scale on the right gives the refractive indices of the hydrocarbon portion of the azeotropic distillate and the ordinate scale on the left gives the boiling point of the azeotropic distillate. The scale of abscissas gives the volume of the hydrocarbon portion of the distillate. The relative amounts by volume of the various components are indicated in the upper portion of the figure. The portions A and B, enclosed by arrows, were analyzed by spectrographic infrared absorption measurements, the results of which are shown in table 2.

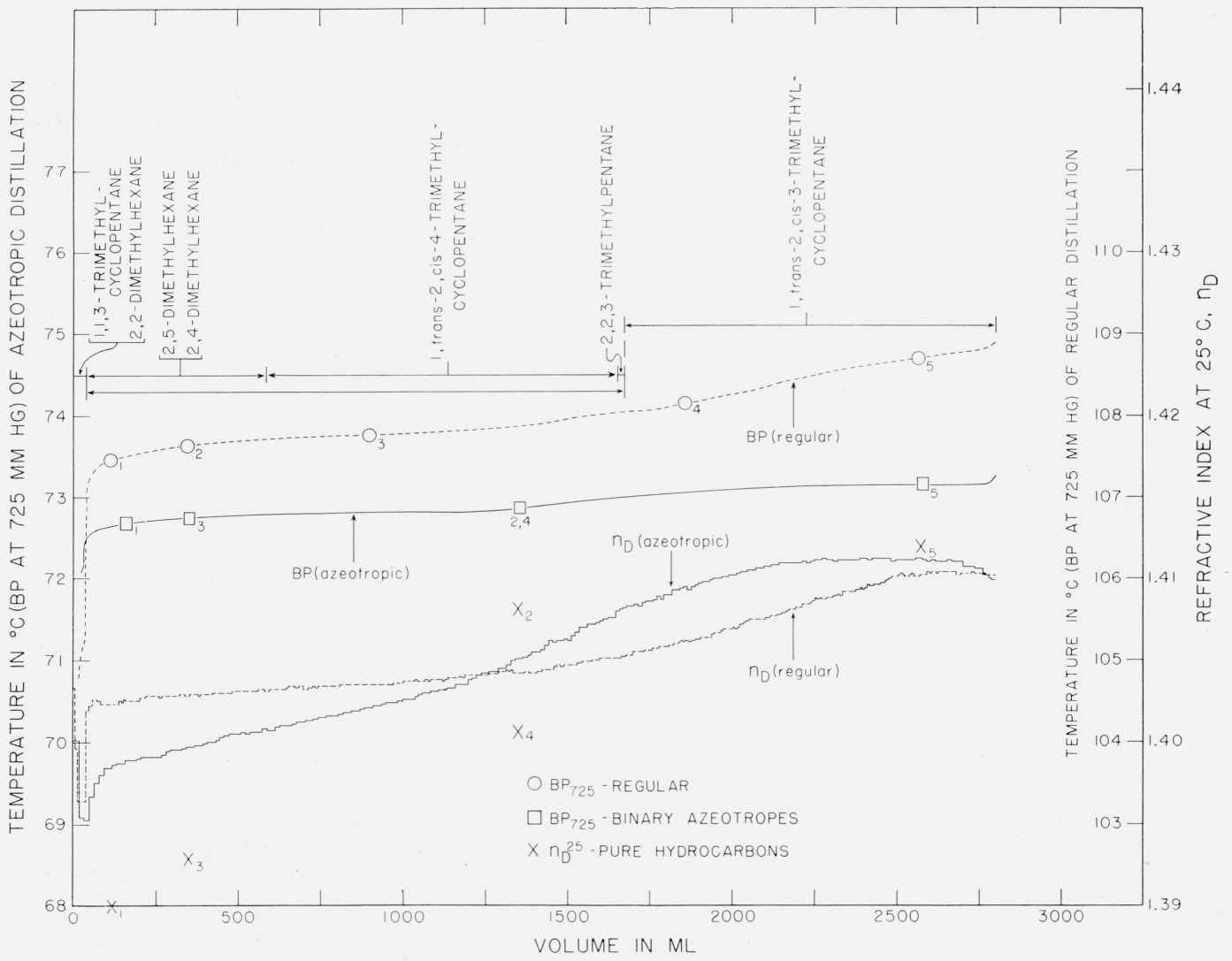

FIGURE 7. *Comparison of separation of 108° to 111° C material by regular and azeotropic distillation with ethanol.*

The outer ordinate scale on the right gives the refractive indices of the hydrocarbon portion of the azeotropic distillate, and the refractive index of the distillate from the regular distillation. The inner ordinate scale on the right gives the boiling point of the distillate from the regular distillation. The scale of abscissas gives the volume of the hydrocarbon portion of the distillate. The numbers accompanying the boiling point and refractive index values shown in the figure refer to the following: (1) to 2,5-dimethylhexane; (2) to 1,*trans*-2,*cis*-4-trimethylcyclopentane; (3) to 2,4-dimethylhexane; (4) to 2,2,3-trimethylpentane; and (5) to 1,*trans*-2,*cis*-3-trimethylcyclopentane. The relative amounts by volume of the various components are indicated in the upper portion of the figure

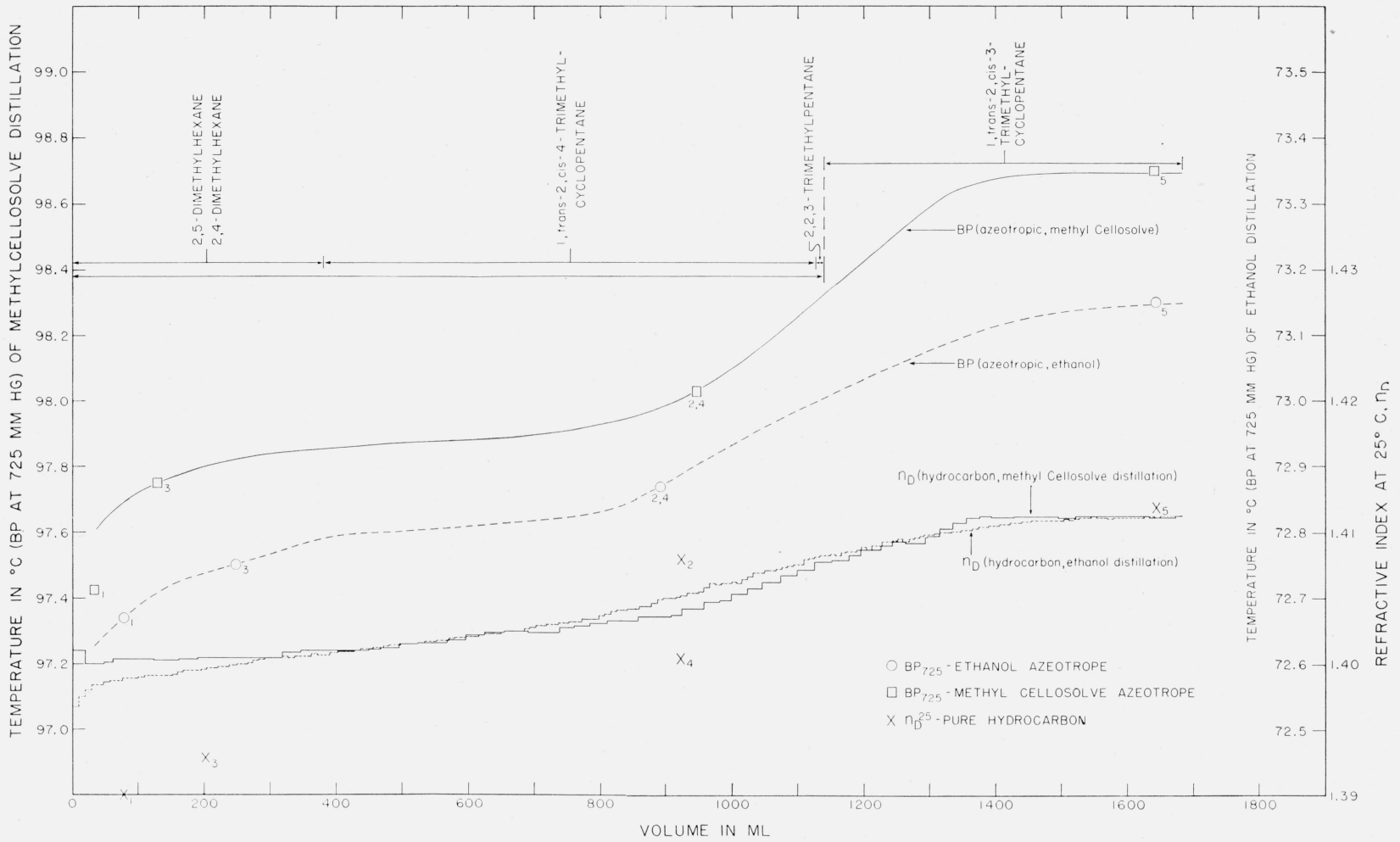

FIGURE 8. *Comparison of the separation of 108° to 111° C material by azeotropic distillation with ethanol and methyl Cellosolve.*

The outer ordinate scale on the right gives the refractive indices of the hydrocarbon portion of the azeotropic distillate. The scale of abscissas gives the volume of the hydrocarbon portion of the distillate. The numbers accompanying the boiling point and refractive index values shown in the figure refer to the following: (1) to 2,5-dimethylhexane; (2) to 1,*trans*-2,*cis*-4-tri-methylcyclopentane; (3) to 2,4-dimethylhexane; (4) to 2,2,3-trimethylpentane and (5) to 1,*trans*-2,*cis*-3-trimethylcyclopentane. The relative amounts by volume of the various components are indicated in the upper portion of the figure.

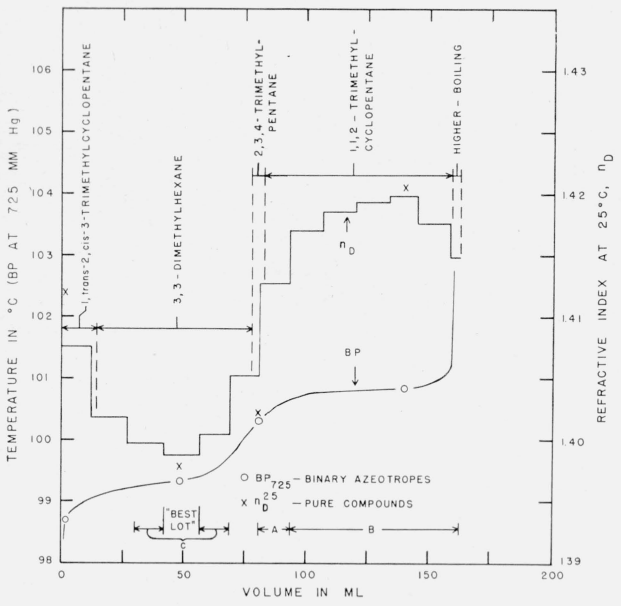

FIGURE 9. *Distillation of the 3,3-dimethylhexane concentrate (part B, fig. 1).*

The ordinate scale on the right gives the refractive indices of the hydrocarbon portion of the azeotropic distillate, and the ordinate scale on the left gives the boiling point of the azeotropic distillate at 725 mm Hg. The scale of abscissas gives the volume of the hydrocarbon portion of the distillate. The relative amounts by volume of the various components are indicated in the upper portion of the figure. The portions A and B, enclosed with arrows, were redistilled. The portions labeled "best lot" and C were analyzed by spectrographic infrared absorption measurements, the results of which are shown in table 2.

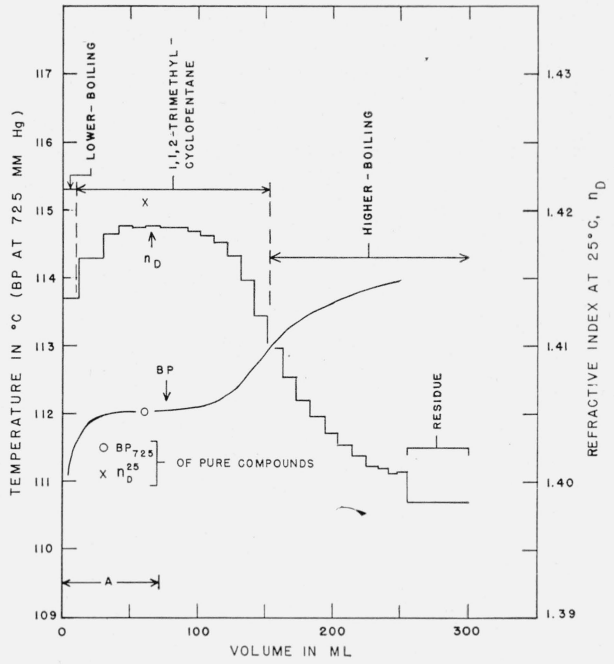

FIGURE 10. *Distillation of the 1,1,2-trimethylcyclopentane concentrate (part C, fig. 1; part B, fig. 9).*

The relative amounts by volume of the various components are indicated in the upper portion of the figure. The portion A, enclosed with arrows, was redistilled.

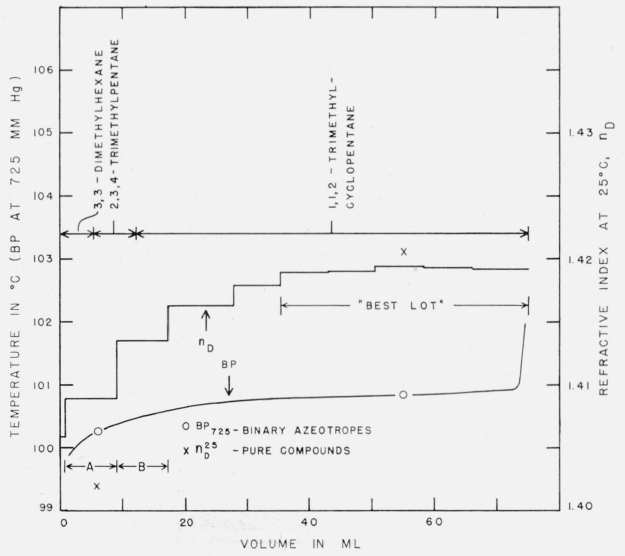

FIGURE 11. *Distillation of the 1,1,2-trimethylcyclopentane concentrate (part A, figs. 9 and 10).*

The ordinate scale on the right gives the refractive indices of the hydro carbon portion of the azeotropic distillate, and the ordinate scale on the left gives the boiling point of the azeotropic distillate. The scale of abscissas gives the volume of the hydrocarbon portion of the distillate. The relative amounts by volume of the various components are indicated in the upper portion of the figure. The lettered fractions A and B were analyzed by spectrographic infrared absorption measurements, the results of which are given in table 2.

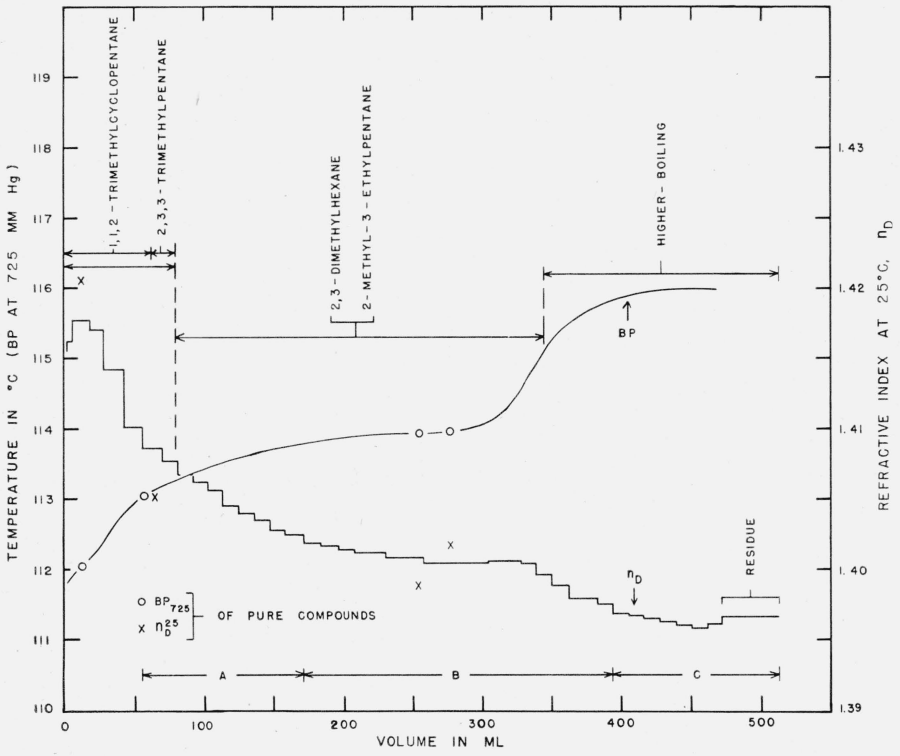

FIGURE 12. *Distillation of the 2,3-dimethylhexane and 2-methyl-3-ethylpentane concentrate (part D, fig. 1).*

The relative amounts by volume of the various components are indicated in the upper portion of the figure. The portions A, B, and C, enclosed with arrows, were redistilled.

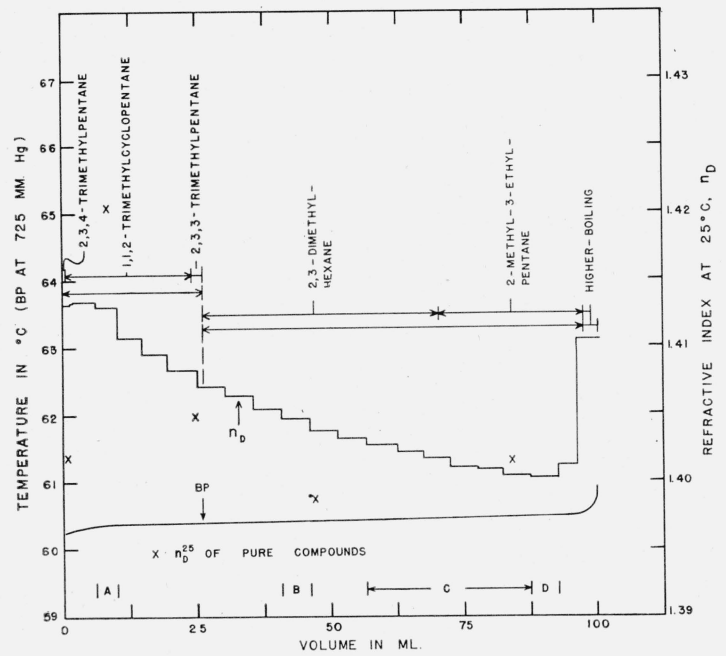

FIGURE 13. *Distillation of the 2,3-dimethylhexane and 2-methyl-3-ethylpentane concentrate (part A, fig. 12).*

The ordinate scale on the right gives the refractive indices of the hydrocarbon portion of the azeotropic distillate, and the ordinate scale on the left gives the boiling point of the azeotropic distillate. The scale of abscissas gives the volume of the hydrocarbon portion of the distillate. The relative amounts by volume of the various components are indicated in the upper portion of the figure. The portion C, enclosed with arrows, was reprocessed by adsorption. The lettered fractions A, B, and D were analyzed by spectrographic infrared absorption measurements, the results of which are shown in table 2.

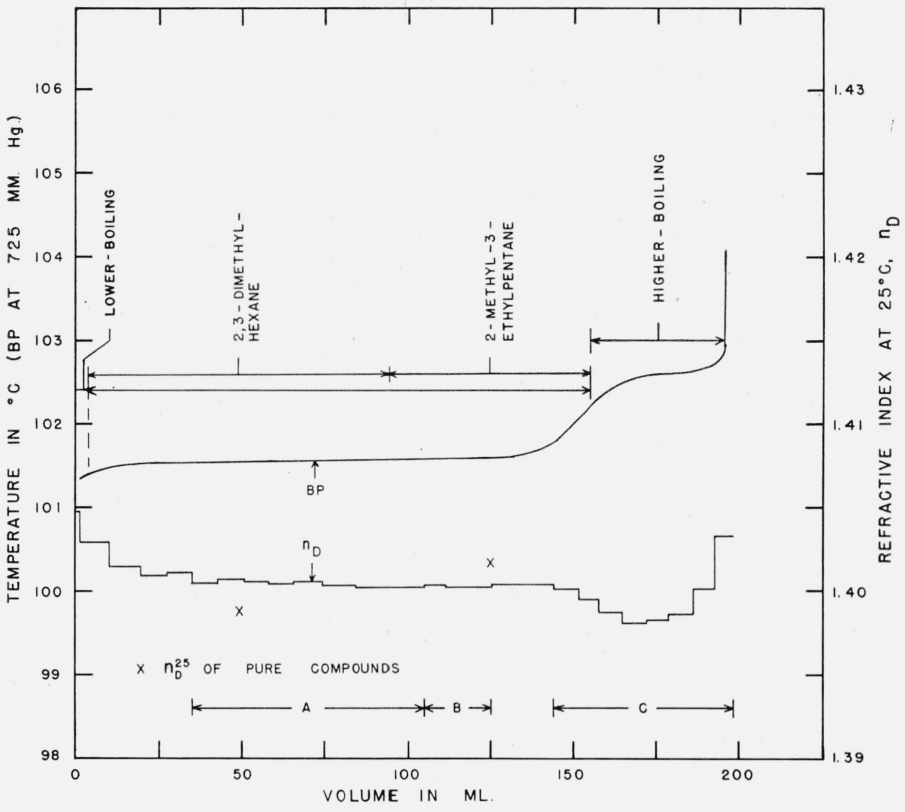

FIGURE 14. *Distillation of the 2,3-dimethylhexane and 2-methyl-3-ethylpentane concentrate (part B, fig. 12).*

The ordinate scale on the right gives the refractive indices of the hydrocarbon portion of the azeotropic distillate, and the ordinate scale on the left gives the boiling point of the azeotropic distillate. The scale of abscissas gives the volume of the hydrocarbon portion of the distillate. The relative amounts by volume of the various components are indicated in the upper portion of the figure. The portions A and B, enclosed with arrows, were processed further by adsorption, the results of which are given in figure 15. Portion A was analyzed by spectrographic infrared absorption measurements the results of which are given in table 2. Portion C was processed with higher-boiling material.

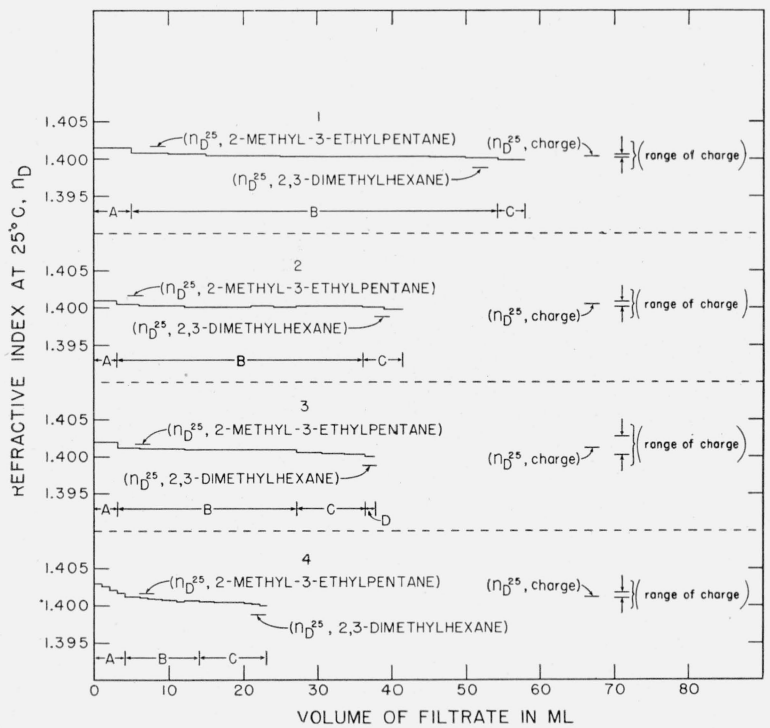

FIGURE 15. *Results of adsorption experiments on 2,3-di-methylhexane and 2-methyl-3-ethylpentane.*

The portions A, B, C, and D, enclosed with arrows, were either reprocessed by adsorption or selected as concentrates of the individual components; see text for further discussion.

WASHINGTON, September 8, 1949.