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Purification, Purity, and Freezing Points of 30 Hydrocarbons of the API-Standard and API-NBS Series[1]

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This report describes the purification and determination of freezing points and purity of 30 hydrocarbons of the API-Standard and API-NBS series, including eight paraffins, six cycloparaffins, three aromatics, twelve olefins, and one acetylene.

I. Introduction

Previous reports described the purification and determination of freezing points and purity of 84 hydrocarbon compounds of the API-Standard and API-NBS series, which were produced as part of the cooperative program on Standard Samples of hydrocarbons of the National Bureau of Standards and the American Petroleum Institute [1, 2, 3, 4].[3]

This report describes the purification and determination of freezing points and purity of an additional 30 hydrocarbon compounds under this cooperative program, including 8 paraffin hydrocarbons, 5 alkylcyclopentanes, 11 monoolefins, 3 diethylbenzenes, ethylcyclobutane, 1-butyne and cyclohexene. Four of these additional 30 compounds are second and purer lots of compounds, the first lots of which were described in the earlier reports.

The final lots of the material labeled API-Standard are sealed "in vacuum" in glass ampoules and made available as NBS Standard Samples of hydrocarbons, by the American Petroleum Institute and the National Bureau of Standards. The material labeled API-NBS is made available in appropriate small lots, through the American Petroleum Institute Research Project 44 at the National Bureau of Standards, on loan to qualified investigators for the measurement of needed properties.

II. Materials

The starting materials were supplied as follows:

By the API Research Project 45 on the synthesis and properties of hydrocarbons of low molecular weight at the Ohio State University, Columbus, Ohio, under the supervision of C. E. Boord:

Ethylcyclobutane.
cis-1,3-Dimethylcyclopentane.
trans-1,3-Dimethylcyclopentane (B).[4]
n-Butylcyclopentane.
1-Hexene (one-half).
trans-2-Hexene.
trans-3-Hexene.
3-Methyl-1-pentene.
2-Methyl-2-pentene.
2-Ethyl-1-butene (one-half).
1-Heptene.
1,3-Diethylbenzene (one-third).
1-Butyne (Ethylacetylene).

By the Hydrocarbon Laboratory at the Pennsylvania State College, State College, Pa., under the supervision of F. C. Whitmore:

2,3,3-Trimethylhexane.
2,2,4-Trimethylhexane.
2,3,3-Trimethylhexane.
2,3,5-Trimethylhexane.
3,3,4-Trimethylhexane.
cis,cis,cis-1,2,3-Trimethylcyclopentane.
cis,trans,cis-1,2,3-Trimethylcyclopentane.

[1] This investigation was performed at the National Bureau of Standards as part of the work of the American Petroleum Institute Research Project 6 on the Analysis, purification, and properties of hydrocarbons.

[2] Research associate on the American Petroleum Institute Research Project 6 at the National Bureau of Standards.

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

[4] In this paper (B) following the name of a compound indicates that for the API-NBS series, it is a second (and usually slightly purer) sample of the given compound, the first sample of which will be labeled (A).

TABLE 1. *Information on the purification of 30 API-Standard and API-NBS hydrocarbons*

Compound [a]	Laboratory [b] providing starting material	Hydrocarbon charged for distillation		Distillation [f]								Volume of selected sample	
		Volume	Purity	Kind [c]	Azeotrope-forming substance [d]	Amount of hydrocarbon in the azeotrope distillate [e]	Distilling column number [f]	Number of theoretical plates [f] (approx.)	Reflux ratio [f] (approx.)	Rate of collection of distillate	Results plotted in figure	API-Standard	API-NBS
Paraffins													
		liters	mole %			% by volume				ml/hour		ml	ml
2,2-Dimethylpropane [g] (Neopentane)	Univ. Oil Prod.	2.00		Reg.			I	150	150/1	2.0	1	475	
2,2-Dimethylpropane (Neopentane)	Calif. Res. Corp.	0.98		Reg.			I	150	150/1	2.0	2	[h]500	250
2,2,3-Trimethylbutane (B)	General Motors	0.40	99.80±0.05				h				3		250
2,2,4-Trimethylpentane (B)	APIRP6 [i]	0.30	99.83±0.05				h				4		180
2,2,3-Trimethylpentane	Penn State	2.71		Reg.			12	135	165/1	4.5	5		
		1.81		Azeo.	Calf.	66	5A	200	160/1	4.5	6	990	256
2,3,4-Trimethylpentane	Penn State	3.00		Azeo.	Calf.	72	15	150	155/1	8.0	7		
		2.44		Reg.			12	135	155/1	4.5	8		
		1.83		Azeo.	Calf.	71	15A	200	160/1	4.5	9	985	195
2,3,3-Trimethylpentane	Penn State	2.24		Reg.			12	135	165/1	4.5	10		
		1.81		Azeo.	Calf.	60	4	200	160/1	4.5	11	785	156
2,3,5-Trimethylhexane	Penn State	2.12		Reg.			12	135	165/1	4.5	12		
		1.67		Azeo.	Me. Cell.	62	11A	200	160/1	4.5	13	1015	225
2,3,4-Trimethylhexane	Penn State	2.44		Reg.			12	135	165/1	4.5	14		
		1.54		Azeo.	Calf.	50	15A	200	160/1	4.5	15	1040	215
Cycloparaffins													
Ethylcyclobutane	APIRP45	2.48	99.62±0.04	Reg.			12	135	165/1	4.5	16		294
cis-1,3-Dimethylcyclopentane	APIRP45	[j] 6.3 [a]		Reg.			4	200	160/1	4.5	17		
		[k] 5.49		Reg.			15A	200	160/1	4.5	18		
		2.61	96.42±0.30	Azeo.	Ethanol	52	4	200	160/1	4.5	19		
		1.66	98.72±0.27	Azeo.	Ethanol	60	2A	200	160/1	4.5	20	585	160
trans-1,3-Dimethylcyclopentane (B)	APIRP45	[j] 5.3 [a]		Reg.			4	200	160/1	4.5	17		
		[l] 5.66		Reg.			15A	200	160/1	4.5	21		
		2.67	92.5±0.4	Azeo.	Ethanol	43	3A	200	160/1	4.5	22		
		1.73	99.21±0.10	Azeo.	Ethanol	62	2A	200	160/1	4.5	23	[q]4050	270
cis,cis,cis-1,2,3-Trimethylcyclopentane	Penn State	1.24	96.90±0.10	Reg.			4	200	160/1	4.5	24		
		0.90	98.22±0.08	Azeo.	Me. Cell.	56	15A	200	160/1	4.5	25	460	100
cis,trans,cis-1,2,3-Trimethylcyclopentane	Penn State	0.14	98.46±0.12	Azeo.	Me. Cell.	75	1A	150	150/1	2.0	26		
		0.06	99.79±0.04	Azeo.	Me. Cell.	77	4	200	160/1	4.5	27	175	50
		[m] 0.29	99.86±0.04										
n-Butylcyclopentane	APIRP45	6.05	99.85±0.03	Reg.			14	125	125/1	12.5	28		
		1.90	99.92±0.03	Azeo.	Cell.	40	11A	200	160/1	4.5	29	1158	345

[a] See footnote a of table 2.

[b] The abbreviations represent the following laboratories: APIRP45; American Petroleum Institute Research Project 45 at the Ohio State University, Columbus, Ohio. Penn State; Hydrocarbon Laboratory at the Pennsylvania State College, State College, Pa. NACA; National Advisory Committee for Aeronautics, Flight Propulsion Research Laboratory, Cleveland, Ohio. General Motors; General Motors Corporation, Detroit, Mich. NBS Auto Sec.; Automotive Section, National Bureau of Standards, Washington, D. C. Calif. Res. Corp.; California Research Corporation, Richmond, Calif. Phillips; Phillips Petroleum Company, Bartlesville, Okla. Univ. Oil Prod.; Universal Oil Products Co., Riverside, Ill. APIRP6; American Petroleum Institute Research Project 6 at the National Bureau of Standards, Washington, D. C.

[c] The abbreviations are: Azeo., azeotropic; Reg., regular.

[d] The abbreviations are: Cell., Cellosolve (ethylene glycol monoethyl ether); Me. Cell., methyl Cellosolve (ethylene glycol monomethyl ether); Me. Carb., methyl Carbitol (diethylene glycol monomethyl ether).

[e] Approximate value obtained from the actual volume of hydrocarbon recovered by extracting the azeotrope-forming substance with water in separatory funnels.

[f] See reference [6] for further details.

[g] This is a second and improved API-Standard sample of this hydrocarbon.

[h] This material was given its final purification by B. J. Mair by the method of adsorption. See references [6, 7].

[i] Obtained by purchase of commercially available material from the Rohm & Haas Co., Philadelphia, Pa.

[j] One of two similar charges. Both *cis* and *trans*-1,3-dimethylcyclopentane were obtained from this material (see fig. 17).

[k] This charge consisted of material having substantially the same composition, from each of the two previous distillations (see footnote j), together with material from the first distillation of the concentrate of the *trans* isomer (see footnote z and fig. 21).

[l] This charge consisted of material having substantially the same composition from each of the two previous distillations (see footnote j).

[m] This is a second lot of *cis, trans, cis*-1,2,3-trimethylcyclopentane supplied by the Pennsylvania State College.

[n] This material was divided into seven charges for purification by adsorption (see footnote h and fig. 31).

[q] This is a second lot of 1,3-diethylbenzene supplied by the API Research Project 45.

Compound [a]	Laboratory providing starting material	Hydrocarbon charged for distillation: Volume	Hydrocarbon charged for distillation: Purity	Distillation: Kind [c]	Distillation: Azeotrope-forming substance [d]	Distillation: Amount of hydrocarbon in the azeotropic distillate [e]	Distillation: Distilling column number [f]	Distillation: Number of theoretical plates [f] (approx.)	Distillation: Reflux ratio [f] (approx.)	Distillation: Rate of collection of distillate	Distillation: Percent plotted in figure	Volume of sample selected: API-Standard	Volume of sample selected: API-NBS
Aromatics													
		liters	mole %			% by volume				ml/hour		mL	mL
1,2-Diethylbenzene	NACA	5.60 [n]3.29	97.69±0.06	Reg.			4 h	200	160/1	4.5	30 31	750	170
1,3-Diethylbenzene (B)	NACA	2.40	98.78±0.10	Azeo.	Me. Cyclo.	73	8	135	145/1	8.5	32		
		1.08	99.15±0.08	Reg.			12	135	165/1	4.5	33	1080	175
	APIRP45	[o]0.93	99.15±0.06										
1,4-Diethylbenzene	NACA	3.56	99.40±0.05	Reg.			9	135	150/1	4.5	34		
		1.88		Azeo.	Me. Cyclo.	66	11A	200	160/1	4.5	35		
		[p]2.42					h				36	1110	245
Olefins													
1-Pentene (B)	Phillips	4.65		Reg.			3A	200	160/1	4.5	37	[q]240	470
1-Hexene	APIRP45	3.19	98.4±0.2	Reg.			9	135	165/1	4.5	38		
		1.81	99.80±0.08	Reg.			4	200	160/1	4.5	39	[r]695	545
	APIRP5 [s]	5.13	80.6±0.5	Reg.			13	130	155/1	8.0	40		
		3.15	99.44±0.12	Azeo.	Ethanol	77	9	135	165/1	4.5	41	[r]780	
trans-2-Hexene	APIRP45	[t]3.45	97.5±0.2	Reg.			11A	200	160/1	4.5	42		
		[u]2.44	99.6±0.1	Reg.			2A	200	160/1	4.5	43	1030	350
trans-3-Hexene	APIRP45	6.50	98.7±0.1	Reg.			13	130	155/1	8.0	44		
		1.71	99.57±0.03	Reg.			11A	200	160/1	4.5	45	1020	200
3-Methyl-1-pentene	APIRP45	[v]5.00		Reg.			9	135	165/1	4.5	46		
		[w]2.44		Reg.			11A	200	160/1	4.5	47	1000	175
2-Methyl-2-pentene	APIRP45	3.13	99.80±0.05	Reg.			4	200	160/1	4.5	48	1120	335
3-Methyl-cis-2-pentene	General Metals	[x]6.00		Reg.			8	130	145/1	8.5	49		
		[y]3.80		Reg.			2A	200	160/1	4.5	50		
		2.18	99.62±0.08	Azeo.	Ethanol	76	12A	200	160/1	4.5	51	1045	250
2-Ethyl-1-butene	APIRP45	2.85	97.2±0.2	Reg.			12	135	165/1	4.5	52		
		1.77	99.4±0.06	Azeo.	Ethanol	78	3A	200	160/1	4.5	53	[bb]820	[bb]140
	APIRP5 [s]	6.47	70.8 b.	Reg.			10	135	165/1	4.5	54		
		1.59	99.52± 0.06	Azeo.	Ethanol	79	4	200	160/1	4.5	55	[cc]515	[bb]140
1-Heptene	APIRP45	2.89	97.8±0.3	Reg.			12	135	165/1	4.5	56		
		1.96	99.47±0.10	Reg.			4	200	160/1	4.5	57	965	220
2,4,4-Trimethyl-1-pentene	NBS Auto Sec.	[dd]6.00		Reg.			14	125	125/1	12.5	58		
		[ee]6.04	99.26±0.08	Reg.			13	150	150/1	8.0	59		
		2.80	99.67±0.06	Azeo.	Me. Cell.	79	15A	200	160/1	4.5	60	1160	280
2,4,4-Trimethyl-2-pentene	NBS Auto Sec.	[dd]6.00		Reg.			14	125	125/1	12.5	58		
		[ee]4.54		Reg.			10	135	165/1	4.5	61		
		2.75		Azeo.	Ethanol	60	11A	200	160/1	4.5	62	1140	320
Cyclohexene	APIRP5 [z]	5.20	99.13±0.06	Reg.			10	155	165/1	4.5	63	2250	735
Acetylenes													
1-Butyne	APIRP45	2.95		Reg.			1	150	150/1	2.0	64	1125	265

[a] This material was divided into five charges for purification by adsorption (see footnote h and fig. 36). It consisted of 0.80 l. from the first distillation in column 9 (see fig. 34) and 1.62 l. from the second distillation in column 11A (see fig. 35).

[b] Obtained by purchase of commercially available material from the Connecticut Hard Rubber Co., New Haven, Conn.

[c] The total volume of the API-Standard sample was 1,475 ml.

[d] One of two similar charges.

[e] This charge consisted of material having substantially the same composition, from each of the two previous distillations (see footnote s).

[f] One of two charges of similar material, one of which was 5.00 l. and the other 2.80 l.

[g] This charge consisted of material, having substantially the same composition, from each of the two previous distillations (see footnote u).

[h] One of three charges of similar material, two of which were 6.00 l. each, and the third of which was 5.30 l.

[i] This charge consisted of material, having substantially the same composition, from each of the three previous distillations (see footnote w).

[j] Obtained by purchase of commercially available material from the Eastman Kodak Co., Rochester, N. Y.

[bb] The total volume of the API-Standard sample was 1,135 ml.

[cc] The total volume of the API-NBS sample was 280 ml.

[dd] One of four similar charges. Both 2,4,4-trimethyl-1-pentene and 2,4,4-trimethyl-2-pentene were obtained from this material (see fig. 58).

[ee] This charge consisted of material, having substantially the same composition, from each of the four previous distillations (see footnote cc).

By the National Advisory Committee for Aeronautics, through its Flight Propulsion Research Laboratory at Cleveland, Ohio:

1,2-Diethylbenzene.
1,3-Diethylbenzene (B) (two-thirds).
1,4-Diethylbenzene.

By the General Motors Corporation Research Laboratories, Detroit, Mich., through T. A. Boyd and W. G. Lovell:

2,2,3-Trimethylbutane (B).
3-Methyl-*cis*-2-pentene.

By the Hydrocarbons Research Laboratory, Automotive Section, National Bureau of Standards, through F. L. Howard and D. B. Brooks:

"Diisobutylene" (2,4,4-Trimethyl-1-pentene+2,4,4-Trimethyl-2-pentene)

By the California Research Corporation, Richmond, Calif., through A. Kremser:

2,2-Dimethylpropane (Neopentane).

By the Phillips Petroleum Co., Bartlesville, Okla., through F. E. Frey:

1-Pentene (B).

By the Universal Oil Products Co., Riverside, Ill., through V. Haensel:

2,2-Dimethylpropane (Neopentene)

By the API Research Project 6 at the National Bureau of Standards, by purchase:

2,2,4-Trimethylpentane (B).	2-Ethyl-1-butene (one-half).
1-Hexene (one-half).	Cyclohexene.

Table 1 summarizes the amounts of the starting materials and gives some additional information as to source and purity.

[a] See footnote 4.

III. Purification

The procedure followed in the process of purification and determination of purity was the same as that described in the previous reports [2, 3, 4], except that in the purification of 2,2,3-trimethylbutane, 2,2,4-trimethylpentane, 1,2-diethylbenzene, and 1,4-diethylbenzene, use was made of the process of adsorption.

In addition to the name of the laboratory supplying the starting materials, table 1 and its footnotes give complete information for each distillation for each of the compounds.

Details of the distillation apparatus and operations are described in reference [5], and details of the adsorption apparatus and operations are given in references [6] and [7].

Figures 1 to 64, inclusive, show graphically the results of the distillation and adsorption operations listed in table 1. These figures give, as appropriate, as a function of volume of hydrocarbon in the distillate or filtrate, the refractive index (n_d at 25° C, to ± 0.0001), the boiling point of the distillate (at the controlled pressure of 724.5 mm Hg, to ± 0.01 deg C), the freezing point of selected fractions of hydrocarbon distillate (in air at 1 atm, usually, with a precision near ± 0.003 deg C), and the purity of the hydrocarbon distillate. The letters *W*, *X*, *Y*, *Z*, indicate the disposition of the material as follows: *W*, returned to the laboratory suppling the material; *X*, blended for redistillation; *Y*, used for the API-Standard material; *Z*, used for the API-NBS material.

As demonstrated in the previous reports [2, 3, 4], the blending of fractions of distillate for the preparation of material of the highest purity can be done safely only on the basis of the freezing points of selected fractions. This is similarly true for the blending of fractions of filtrate from the adsorption process.

IV. Freezing Points, Cryoscopic Constants, and Purity

Table 2 gives the following information for each of the 30 compounds, except as otherwise indi-

Footnotes for table 2.

[a] (B) following the name of a compound indicates that for the API-NBS series, it is a second (and usually slightly purer) sample of the given compound, the first sample of which will be labeled (A).

[b] F indicates freezing, and M indicates melting. See reference [8] for experimental details and the definition of the cryoscopic constant.

[c] The values in this column, except as otherwise indicated, were calculated as described in reference [8], using the values of the cryoscopic constants and freezing points for zero impurity given in the preceding columns.

[d] Not determined in this investigation. From the z tables of the American Petroleum Institute Research Project 44, [9].

[e] This is a second and improved API-Standard sample of this hydrocarbon.

[f] Estimated by analogy with isomers subjected to similar purification.

[g] This cryoscopic constant was determined by the procedure given on page 371 of reference [8].

TABLE 2. *Freezing points and purity of 50 API–Standard and API–NBS hydrocarbons*

Compound [a]	Kind of time-temperature observations used to determine the freezing point [b]	Freezing point of the actual selected sample in air at 1 atm.		Freezing point for zero impurity in air at 1 atm.	Cryoscopic constant [b]	Calculated amount of impurity in the actual selected sample [c]	
		API–Standard	API–NBS			API–Standard	API–NBS
Paraffins							
		°C	°C	°C	deg^{-1}	mole %	mole %
2,2-Dimethylpropane (Neopentane)	F	−16.65	—	−16.550±0.020	[d](0.00595)	0.06±0.02	—
2,2-Dimethylpropane (Neopentane)	F	−16.587	−16.580	−16.550±0.020	[d](0.00595)	[e]0.022±0.002	0.018±0.012
2,2,3-Trimethylbutane (B)	F	—	−24.960	−24.930±0.020	[d](0.00430)	—	0.09±0.008
2,2,4-Trimethylpentane (B)	M	—	−107.393	−107.368±0.013	[d](0.04031)	—	0.11±0.05
2,2,3-Trimethylhexane	—	—	—	—	—	[f]0.30±0.20	[f]0.25±0.20
2,2,4-Trimethylhexane	M	—	—	−120.0±0.3	0.06	0.30±0.20	0.25±0.20
2,3,3-Trimethylhexane	M	−116.829	−116.815	−116.800±0.020	0.0447	0.13±0.06	0.07±0.06
2,3,5-Trimethylhexane	—	—	—	—	—	[f]0.30±0.20	[f]0.25±0.20
3,3,4-Trimethylhexane	M	−101.27	−101.24	−101.20±0.03	0.033	0.23±0.10	0.13±0.10
Cycloparaffins							
Ethylcyclobutane	M	—	−142.770	−142.750±0.015	0.0378	—	0.06±0.06
cis-1,3-Dimethylcyclopentane	M	−134.04	−133.990	−133.90±0.05	0.0461	0.65±0.23	0.41±0.23
trans-1,3-Dimethylcyclopentane (B)	M	−133.714	−133.706	−133.690±0.015	[d](0.02596)	[e]0.11±0.07	0.07±0.06
cis cis, cis-1,2,3-Trimethylcyclopentane	F and M	−116.471	−116.458	−116.430±0.025	0.0245	0.10±0.06	0.07±0.06
cis, trans, cis-1,2,3-Trimethylcyclopentane	M	−112.756	−112.727	−112.705±0.012	0.0365	0.11±0.04	0.08±0.04
n-Butylcyclopentane	M	−107.993	−107.992	−107.985±0.010	0.0424	0.034±0.025	0.030±0.025
Aromatics							
1,2-Diethylbenzene	F	−31.257	−31.250	−31.240±0.010	0.0239	0.05±0.03	0.03±0.03
1,3-Diethylbenzene (B)	M	−83.938	−83.933	−83.920±0.010	0.0369	0.07±0.04	0.05±0.04
1,4-Diethylbenzene	F	−42.878	−42.860	−42.850±0.010	0.0240	0.07±0.02	0.023±0.020
Olefins							
1-Pentene (B)	M	−165.264	−165.250	−165.220±0.020	[d](0.06000)	[e]0.26±0.12	0.18±0.12
1-Hexene	M	−139.628	−139.626	−139.600±0.015	0.0509	0.14±0.08	0.13±0.08
trans-2-Hexene	M	−133.001	−132.998	−132.970±0.020	0.0564	0.17±0.11	0.16±0.11
trans-3-Hexene	F and M	−115.441	−115.439	−115.430±0.015	0.055	0.06±0.03	0.05±0.03
3-Methyl-1-pentene	F and M	−153.1	−153.1	−153.0±0.1	[g]0.03	0.30±0.20	0.25±0.20
2-Methyl-2-pentene	F and M	−135.088	−135.078	−135.070±0.015	0.0508	0.09±0.05	0.04±0.05
3-Methyl-cis-2-pentene	M	−138.474	−138.466	−138.445±0.015	0.0509	0.15±0.08	0.11±0.08
2-Ethyl-1-butene	F and M	−131.551	−131.540	−131.530±0.010	0.0454	0.10±0.04	0.06±0.04
1-Heptene	M	−119.075	−119.067	−119.035±0.020	0.0509	0.20±0.10	0.16±0.10
2,4,4-Trimethyl-1-pentene	F and M	−93.508	−93.496	−93.480±0.010	[d](0.0330)	0.09±0.03	0.05±0.03
2,4,4-Trimethyl-2-pentene	F and M	−106.356	−106.353	−106.330±0.015	[d](0.0300)	0.08±0.05	0.07±0.05
Cyclohexene	M	−103.517	−103.515	−103.500±0.015	[d](0.01376)	0.052±0.020	0.052±0.020
Acetylenes							
1-Butyne (Ethylacetylene)	F and M	−125.756	−125.749	−125.720±0.020	[g]0.056	0.13±0.07	0.10±0.07

cated: the kind of time-temperature curves, whether freezing or melting, used to determine the freezing point [8]; the freezing point of the actual sample, in air at 1 atm [8], for both the API-Standard and API-NBS lots; the calculated value of the freezing point for zero impurity [8]; the value of the cryoscopic constant, determined from the lowering of the freezing point on the addition of a known amount of an appropriate impurity [8]; and the resulting calculated amount of impurity in the API-Standard and the API-NBS materials.

Grateful acknowledgment is made to the other organizations and individuals listed in section II of this report for their contributions of materials for use in this work.

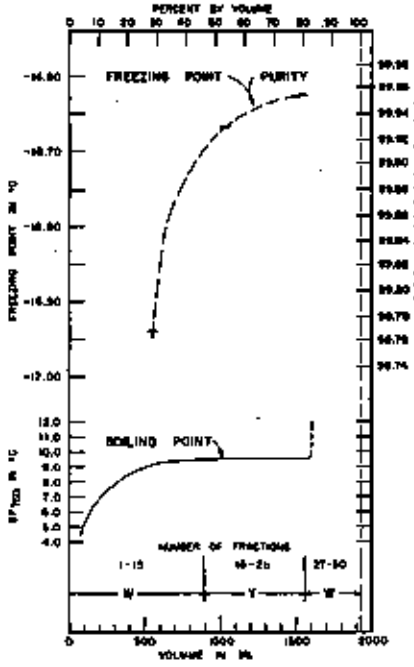


FIGURE 1. *Results of the first and only distillation of the first lot of 2,2-dimethylpropane (neopentane).*

This material had not been fractionated by the supplier. Regular distillation at atmospheric pressure in Still 1 (5/2/45 to 5/25/45).

V. References

[1] NBS Tech. News Bul., No. 350 (June, 1945).
[2] A. J. Streiff, E. T. Murphy, V. A. Sedlak, C. B. Willingham, and F. D. Rossini, J. Research NBS **37**, 331 (1946) RP1752.
[3] A. J. Streiff, E. T. Murphy, J. C. Cahill, H. F. Flanagan, V. A. Sedlak, C. B. Willingham, and F. D. Rossini, J. Research NBS **38**, 53 (1947) RP1760.
[4] A. J. Streiff, E. T. Murphy, J. C. Zimmerman, L. F. Soule, V. A. Sedlak, C. B. Willingham, and F. D. Rossini, J. Research NBS **39**, 321 (1947) RP1833.
[5] C. B. Willingham and F. D. Rossini, J. Research NBS **37**, 15 (1946) RP1724.
[6] B. J. Mair, A. L. Gaboriault, and F. D. Rossini, Ind. Eng. Chem. **39**, 1072 (1947).
[7] Purification of five hydrocarbons by adsorption. API Research Project 6. National Bureau of Standards. A. J. Streiff, B. J. Mair, and F. D. Rossini. Unpublished.
[8] A. R. Glasgow, Jr., A. J. Streiff, and F. D. Rossini, J. Research NBS **35**, 355 (1945) RP1676.
[9] American Petroleum Institute Research Project 44 at the National Bureau of Standards. Selected values of properties of hydrocarbons. Tables 1z, 2z, 3z, 5z, 6z, 7z, and 8z.

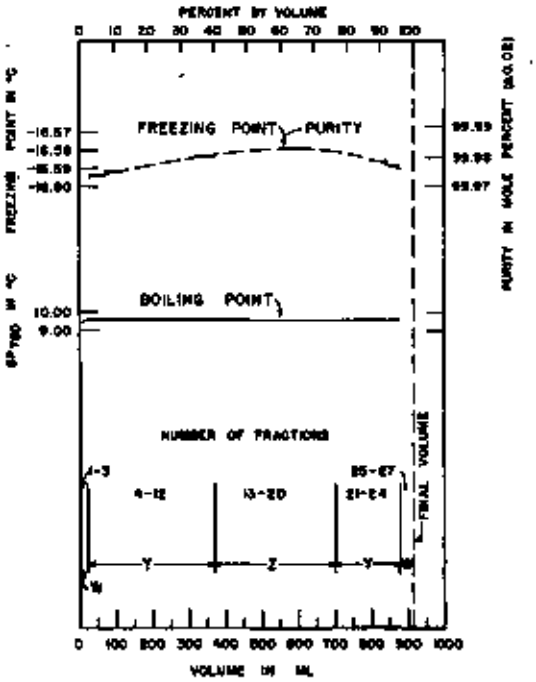


FIGURE 2. *Results of the first and only distillation of a second lot of 2,2-dimethylpropane (neopentane).*

This material had been fractionated by the supplier. Regular distillation at atmospheric pressure in Still 1 (1/22/46 to 2/28/46).

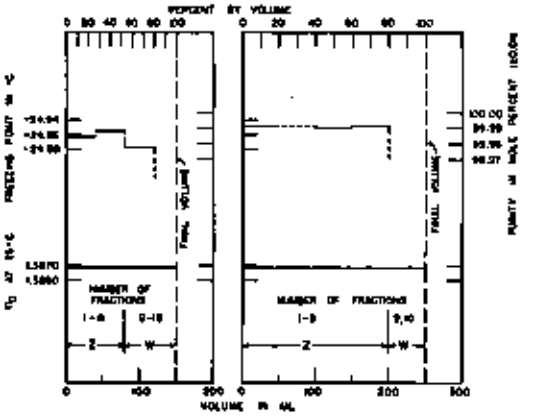


FIGURE 3. *Results of the purification by adsorption of 2,2,3-trimethylbutane.*

Adsorption in Column 10 (4/16/47); column 10 (5/8/47).

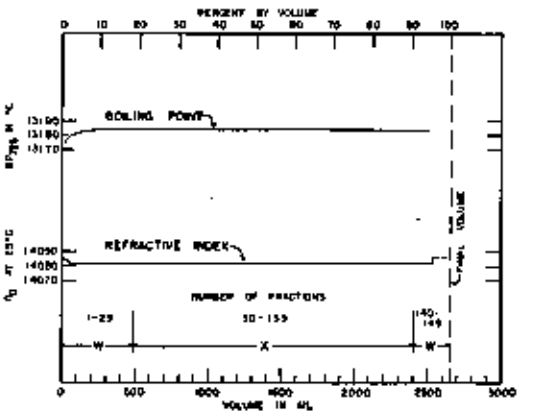


FIGURE 5. *Results of the first distillation of 2,2,3-trimethylhexane.*

Regular distillation at 725 mm Hg in Still 12 (10/7/46 to 11/4/46).

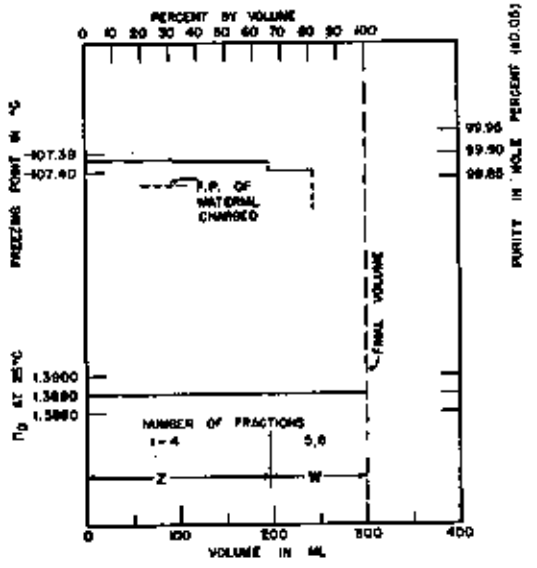


FIGURE 4. *Results of the purification by adsorption of 2,2,4-trimethylpentane.*

Adsorption in Column 10 (4/8/47).

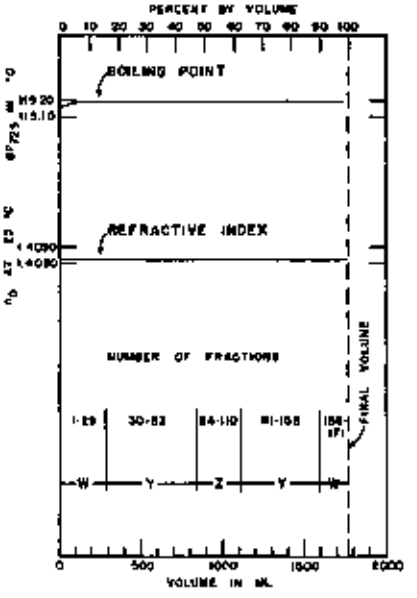


FIGURE 6. *Results of the second and final distillation of 2,2,3-trimethylhexane.*

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in Still 3A (7/24/47 to 8/25/47).

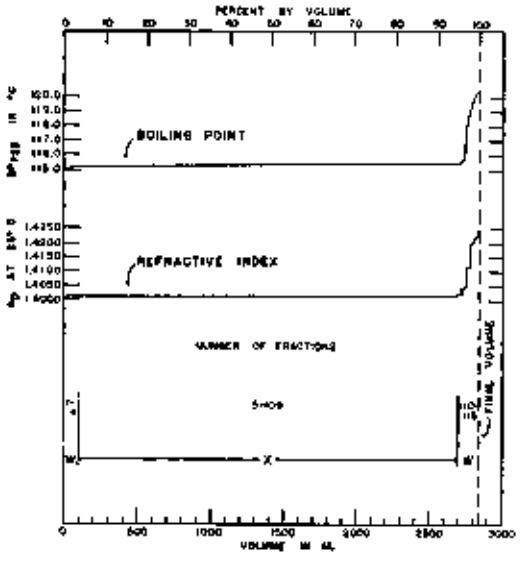


FIGURE 7. *Results of the first distillation of 2,2,4-trimethylhexane.*

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in Still 13 (10/21/44 to 11/15/44).

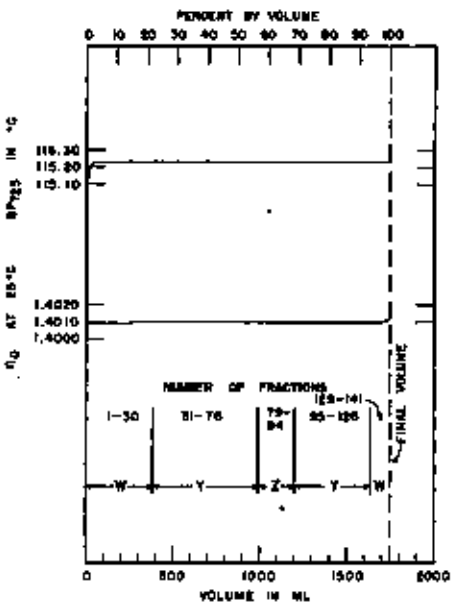


FIGURE 9. *Results of the third and final distillation of 2,2,4-trimethylhexane.*

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in Still 15A (1/8/47 to 2/3/47).

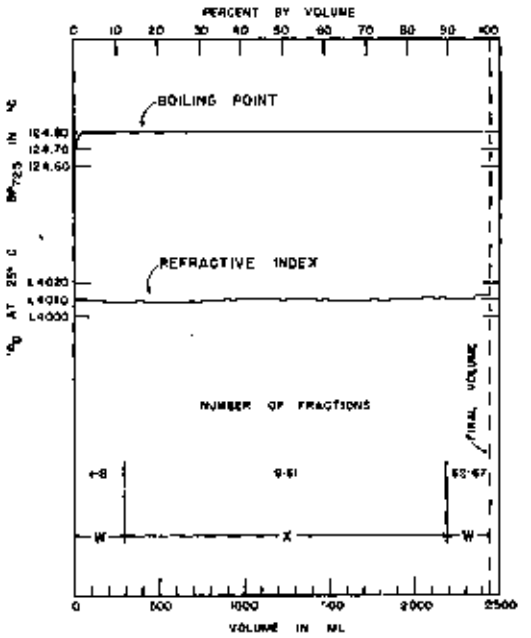


FIGURE 8. *Results of the second distillation of 2,2,4-trimethylhexane.*

Regular distillation at 725 mm Hg in Still 13 (9/3/45 to 9/29/45).

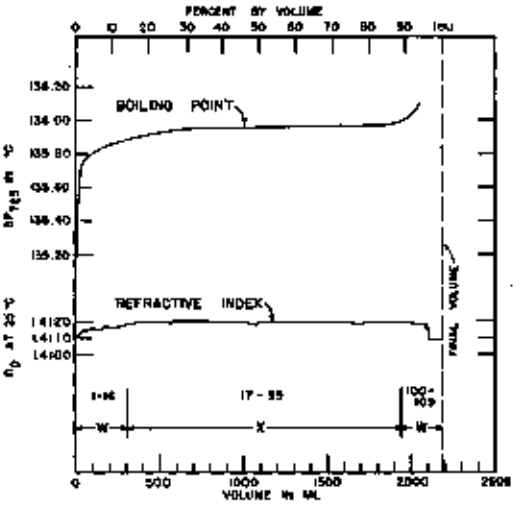


FIGURE 10. *Results of the first distillation of 2,3,3-trimethylhexane.*

Regular distillation at 725 mm Hg in Still 13 (8/21/46 to 9/11/46).

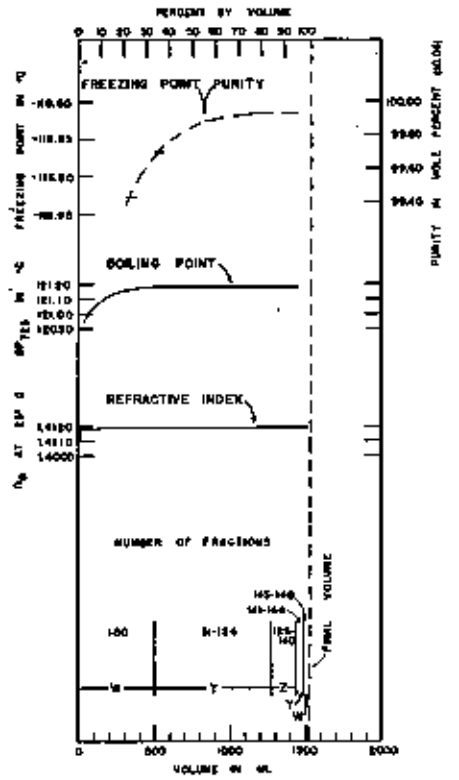


FIGURE 11. *Results of the second and final distillation of 2,3,5-trimethylhexane.*

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in Still 4 (12/27/46 to 1/23/47).

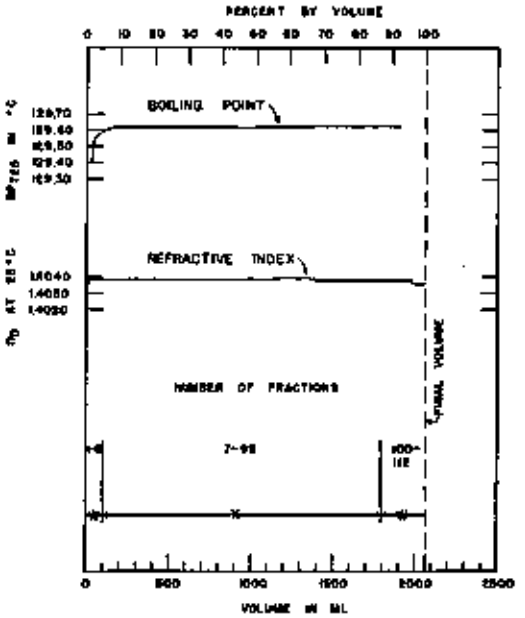


FIGURE 12. *Results of the first distillation of 2,3,5-trimethylhexane.*

Regular distillation at 725 mm Hg in Still 12 (7/29/46 to 8/19/46).

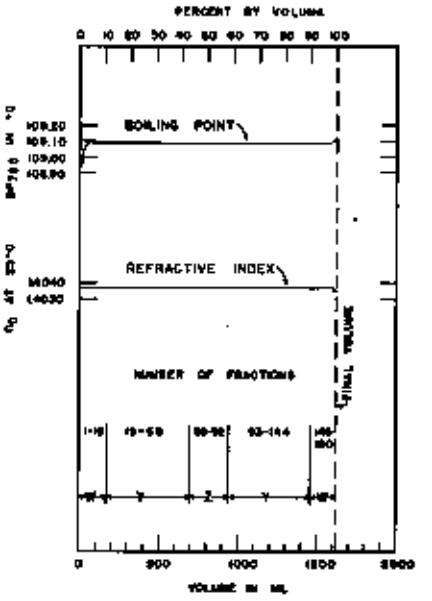


FIGURE 13. *Results of the second and final distillation of 2,3,5-trimethylhexane.*

Azeotropic distillation with ethylene glycol monomethyl ether at 725 mm Hg in Still 11A (10/24/46 to 11/24/46).

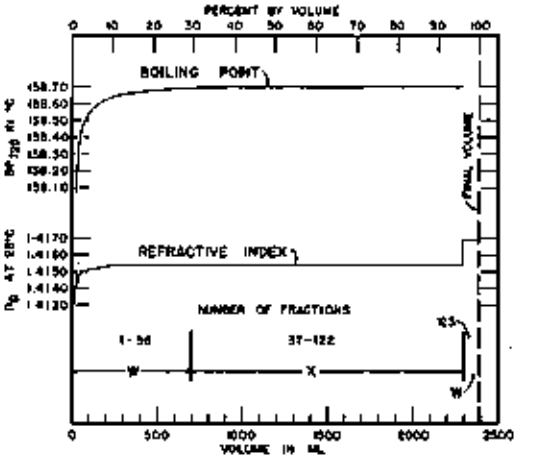


FIGURE 14. *Results of the first distillation of 3,3,4-trimethylhexane.*

Regular distillation at 725 mm Hg in Still 12 (9/12/46 to 10/5/46).

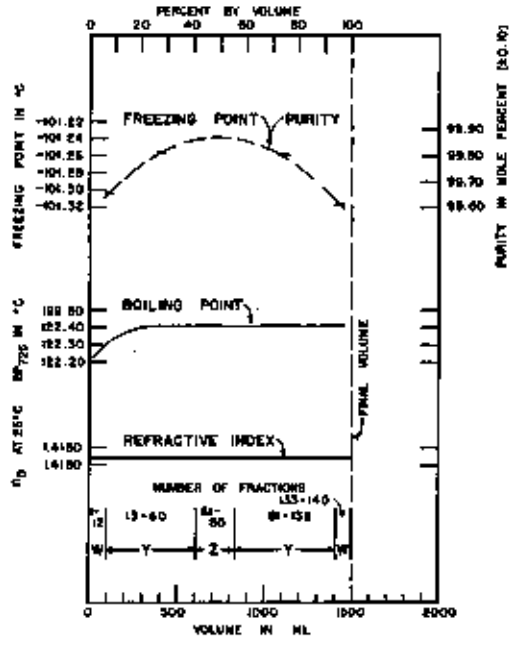


FIGURE 15. *Results of the second and final distillation of 3,3,4-trimethylhexane.*

Azeotropic distillation with ethylene glycol monoethyl ether at 735 mm Hg in Still 15A (8/19/47 to 9/8/47).

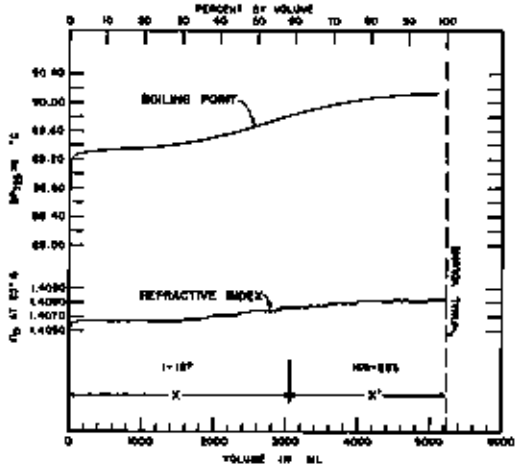


FIGURE 17. *Results of the first distillation of cis and trans-1,3-dimethylcyclopentane.*

Regular distillation at 725 mm Hg in Still 4 (10/1/46 to 11/20/46). One or two distillations of similar material. See footnote j of table 1. Fractions 2 to 167 (marked "x") were redistilled to obtain *trans*-1,3-dimethylcyclopentane (see fig. 21 and footnote x of table 1). Fractions 168 to 253 (marked "x'") were redistilled to obtain *cis*-1,3-dimethylcyclopentane (see fig. 18 and footnote k of table 1).

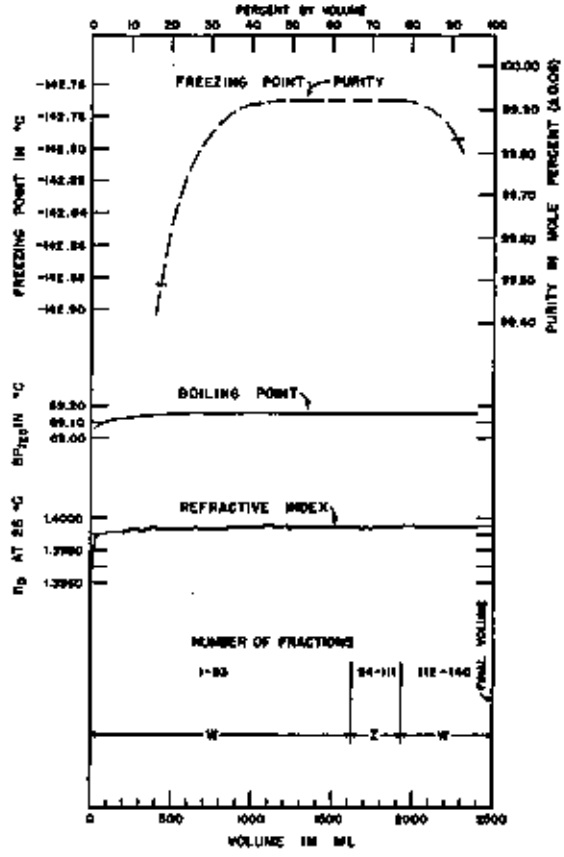


FIGURE 16. *Results of the first and only distillation of ethylcyclobutane.*

Regular distillation at 735 mm Hg in Still 12 (7/3/46 to 7/27/46).

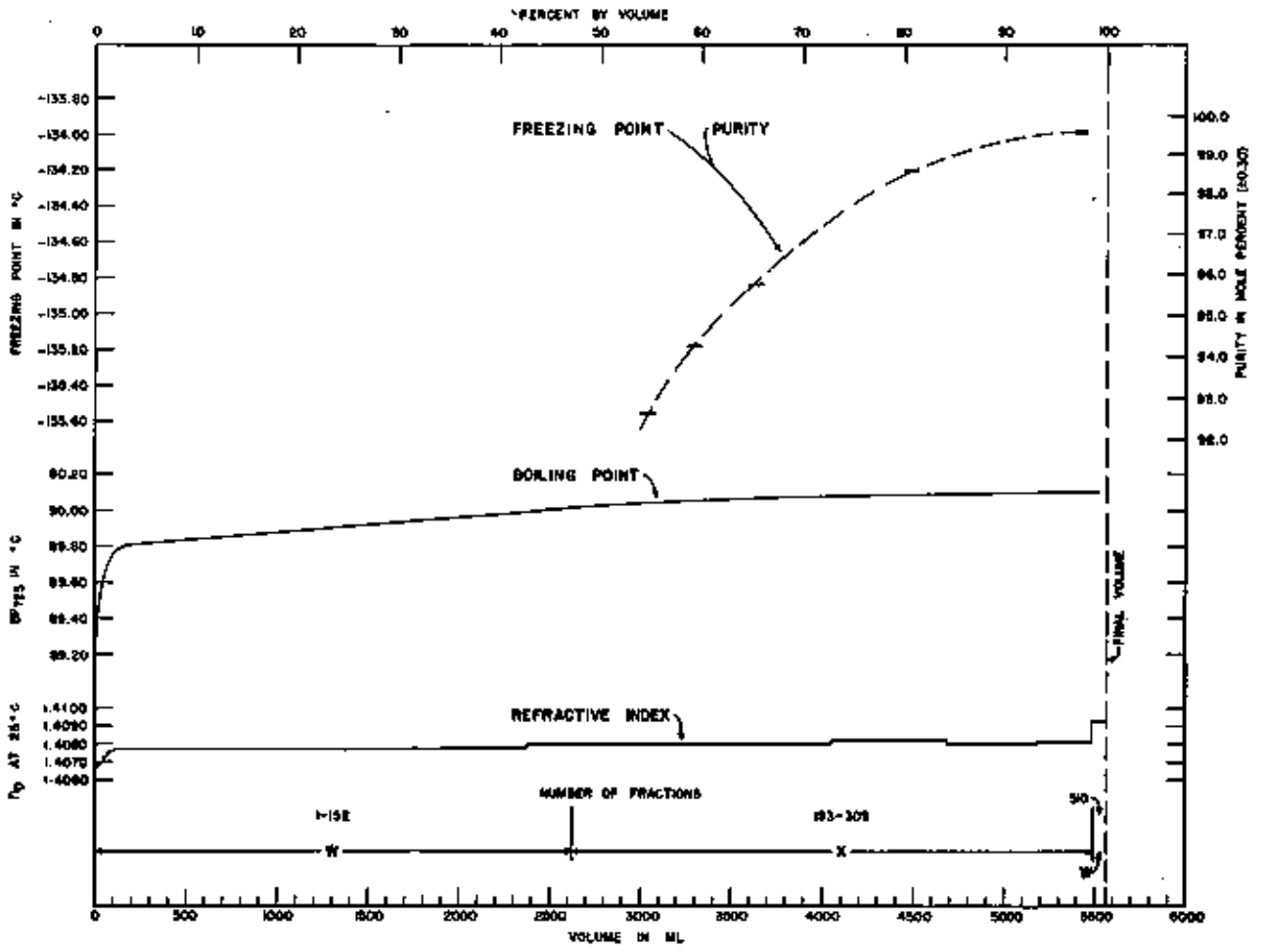


FIGURE 18. *Results of the second distillation of cis -1,3-dimethylcyclopentane.*
Regular distillation at 725 mm Hg in Still 15A (4/25/47 to 5/17/47). See footnote k of table 1.

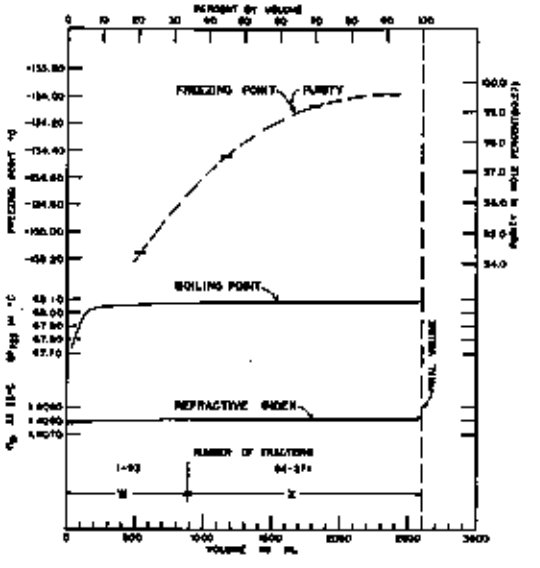


FIGURE 19. *Results of the third distillation of cis -1,3-dimethylcyclopentane.*

Azeotropic distillation with ethanol at 725 mm Hg in Still 4 (6/26/47 to 8/13/47).

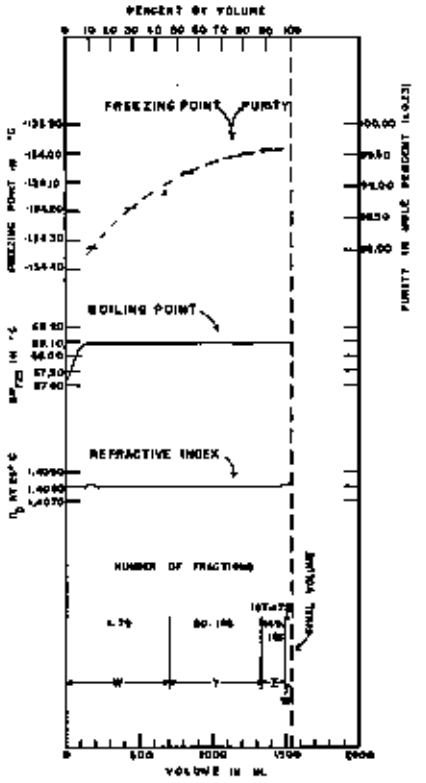


FIGURE 20. *Results of the fourth and final distillation of cis-1,3-dimethylcyclopentane.*

Azeotropic distillation with ethanol at 735 mm Hg in Still 2A (8/14/47 to 9/22/47).

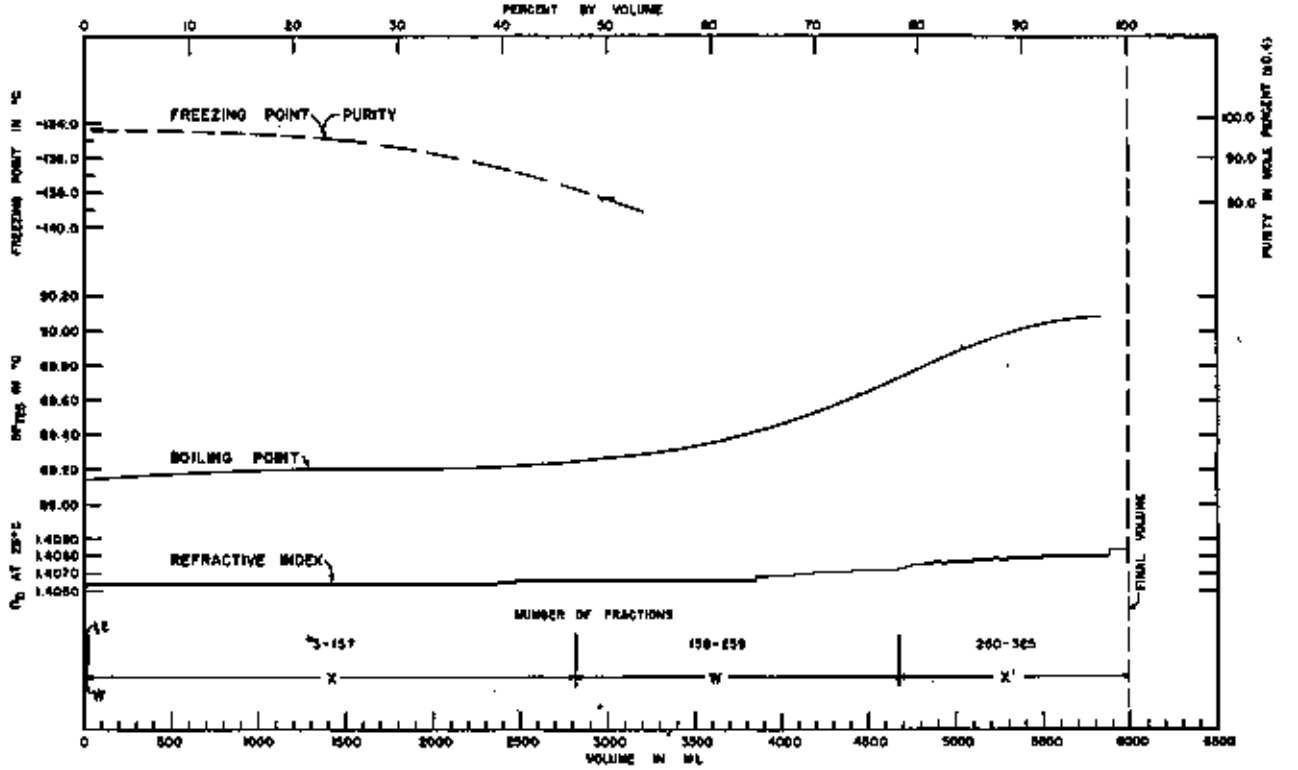


FIGURE 21. *Results of the second distillation of trans-1,3-dimethylcyclopentane.*

Regular distillation at 735 mm Hg in Still 15A (2/4/47 to 4/3/47). See footnote s of table 1. The part marked "x'" was used as part of the charge for the concentrate of cis isomer. (See footnote k of table 1, and fig. 18.)

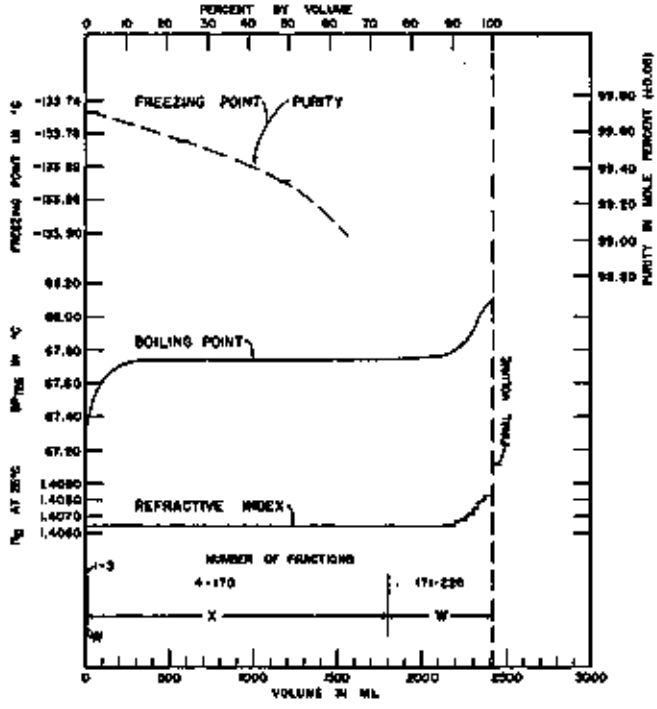


FIGURE 22. *Results of the third distillation of trans-1,3-dimethylcyclopentane.*

Azeotropic distillation with ethanol at 725 mm Hg in Still 3A (4/17/47 to 5/28/47).

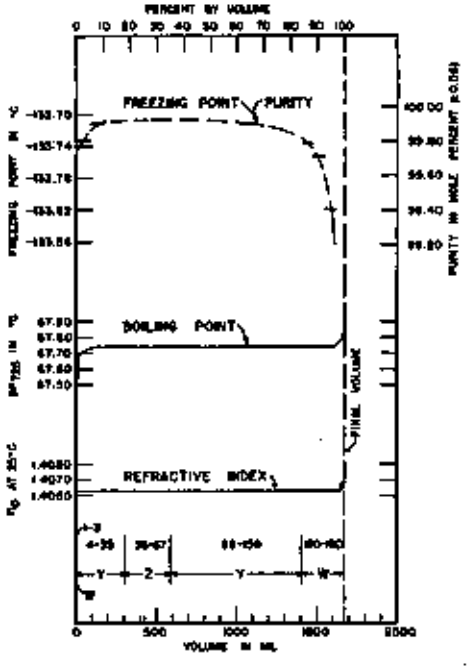


FIGURE 23. *Results of the fourth and final distillation of trans-1,3-dimethylcyclopentane.*

Azeotropic distillation with ethanol at 725 mm Hg in Still 2A (7/9/47 to 8/13/47).

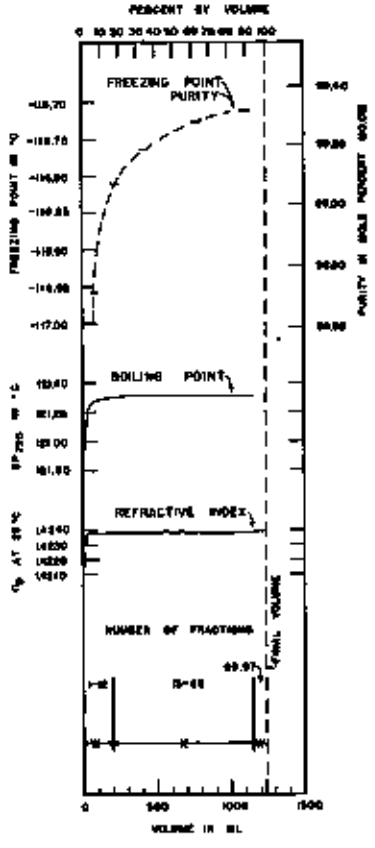


FIGURE 24. *Results of the first distillation of cis,cis,cis-1,2,3-trimethylcyclopentane.*

Regular distillation at 725 mm Hg in Still 4 (11/22/46 to 12/5/46).

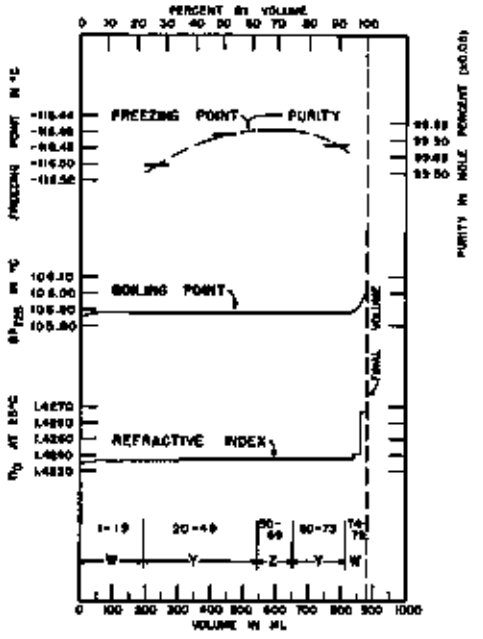


FIGURE 25. *Results of the second and final distillation of cis,cis,cis-1,2,3-trimethylcyclopentane.*

Azeotropic distillation with ethylene glycol monomethyl ether at 725 mm Hg in Still 15A (12/26/46 to 1/8/47).

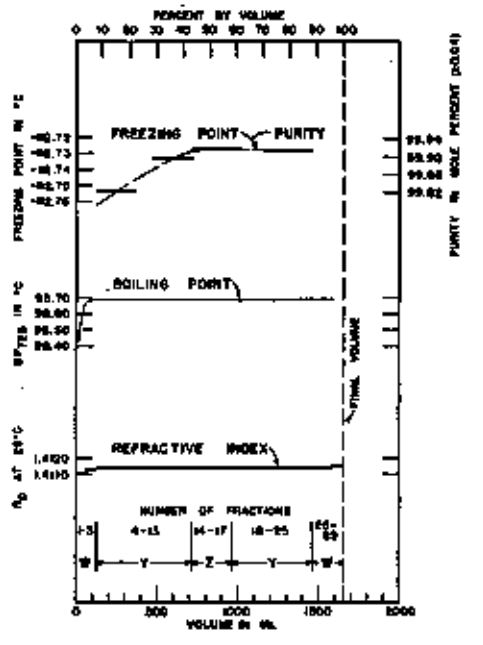


FIGURE 27. *Results of the second and final distillation of cis,trans,cis-1,2,3-trimethylcyclopentane.*

Azeotropic distillation with ethylene glycol monomethyl ether at 725 mm Hg in Still 4 (2/4/47 to 2/11/47). (See footnote m of table 1.)

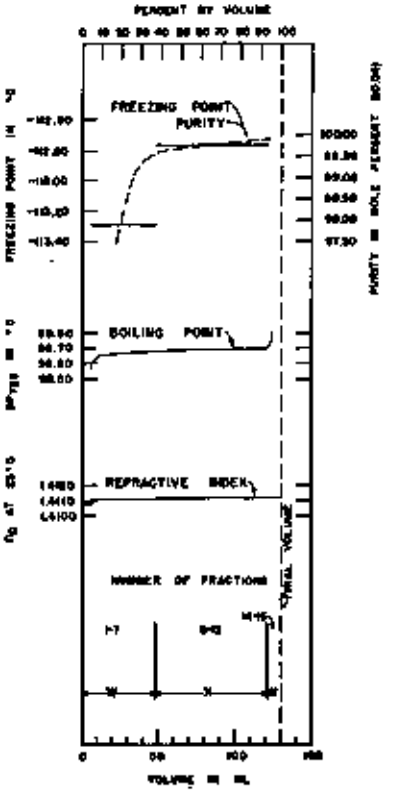


FIGURE 26. *Results of the first distillation of cis,trans,cis-1,2,3-trimethylcyclopentane.*

Azeotropic distillation with ethylene glycol monomethyl ether at 725 mm Hg in Still 1A (12/26/46 to 1/3/47).

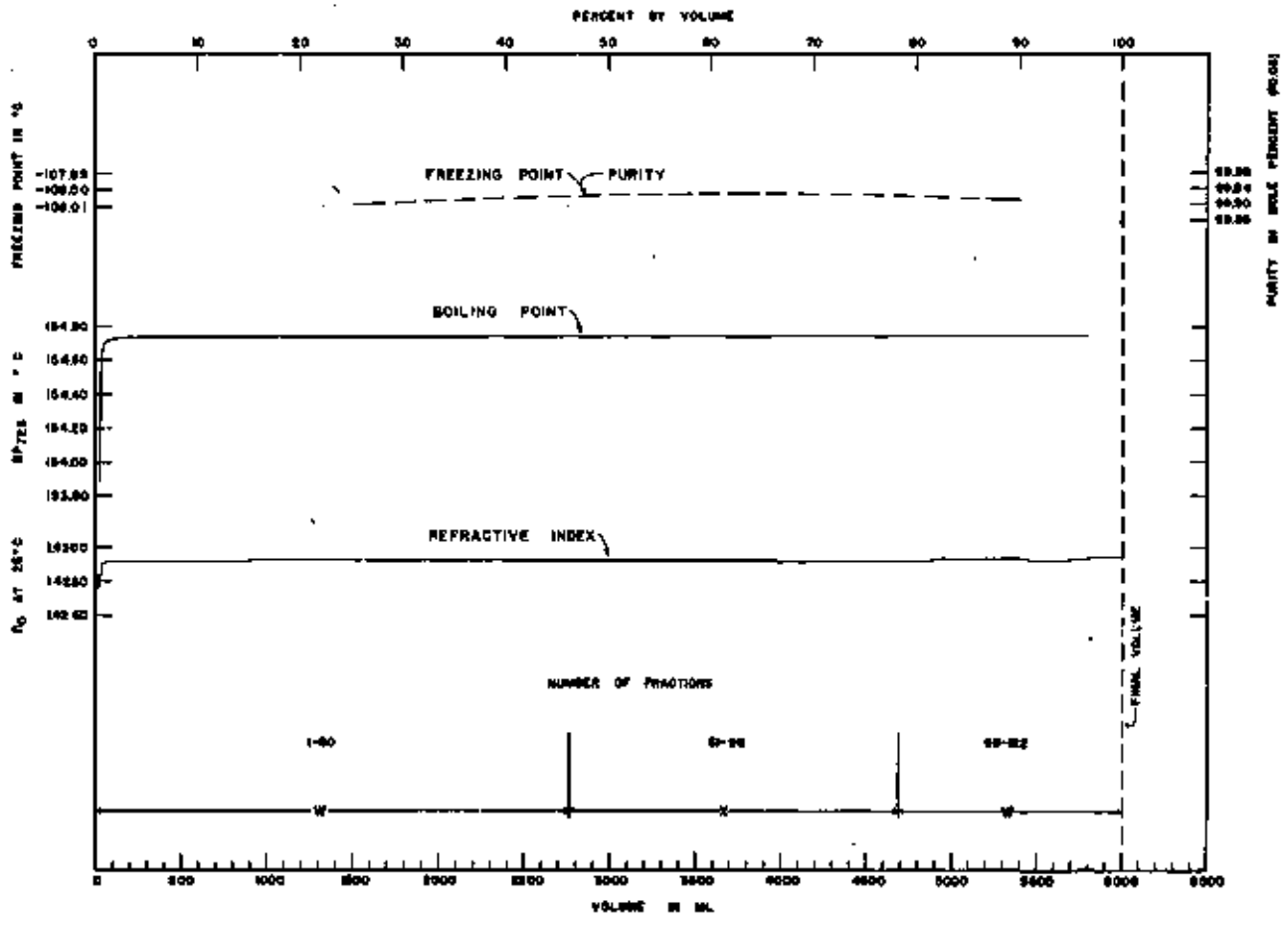


FIGURE 28. *Results of the first distillation of n-butylcyclopentane.*

Regular distillation at 725 mm Hg in Still 14 (12/16/46 to 1/7/47).

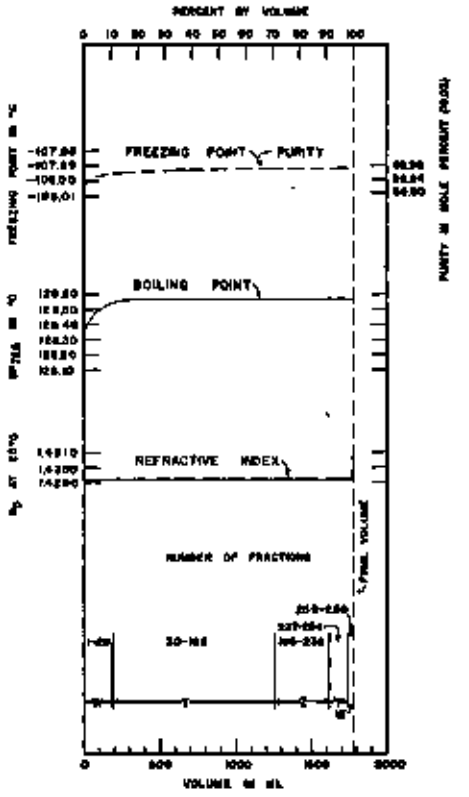


FIGURE 29. *Results of the second and final distillation of n-butylcyclopentane.*

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in Still 11A (2/25/47 to 4/14/47).

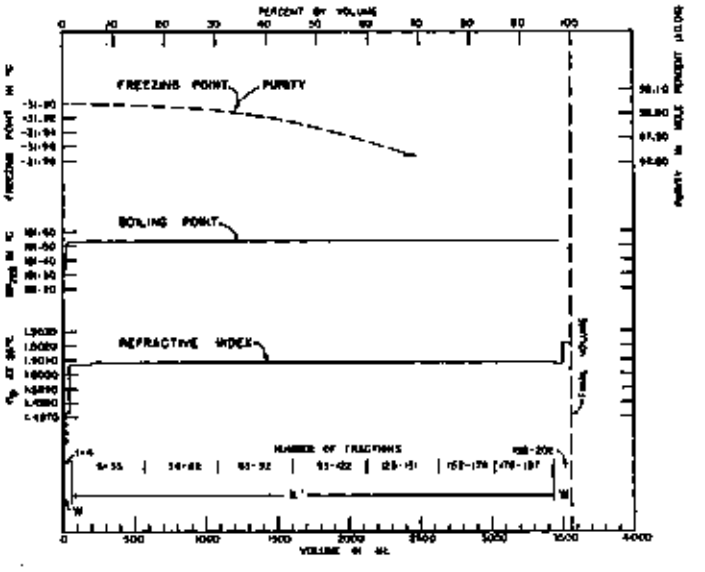


FIGURE 30. *Results of the first distillation of 1,3-diethylbenzene.*

Regular distillation at 725 mm Hg in Still 4 (4/2/47 to 5/8/47). The part of the distillate marked "X" received its final purification by adsorption. (See fig. 31 and footnote n of table 1.)

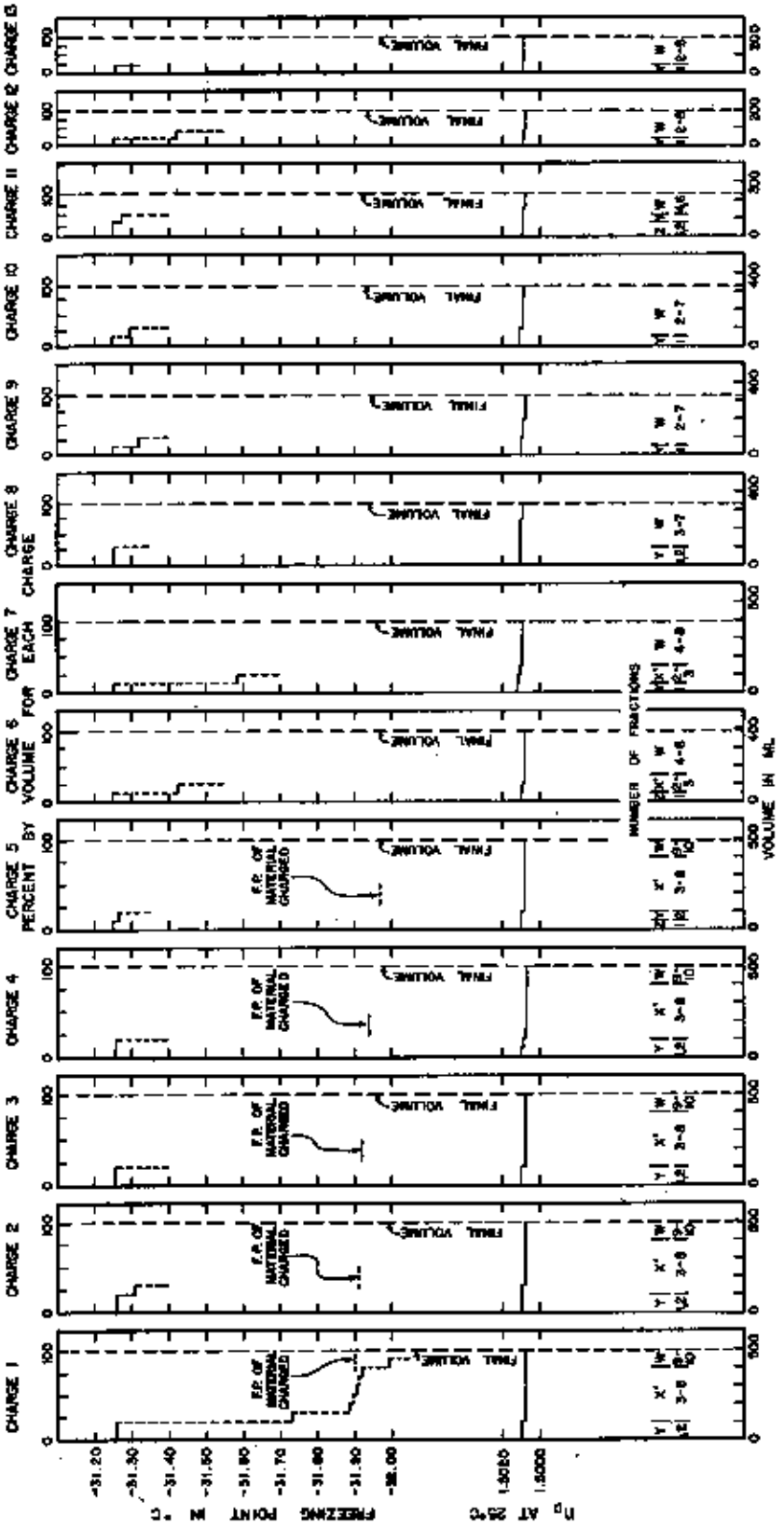
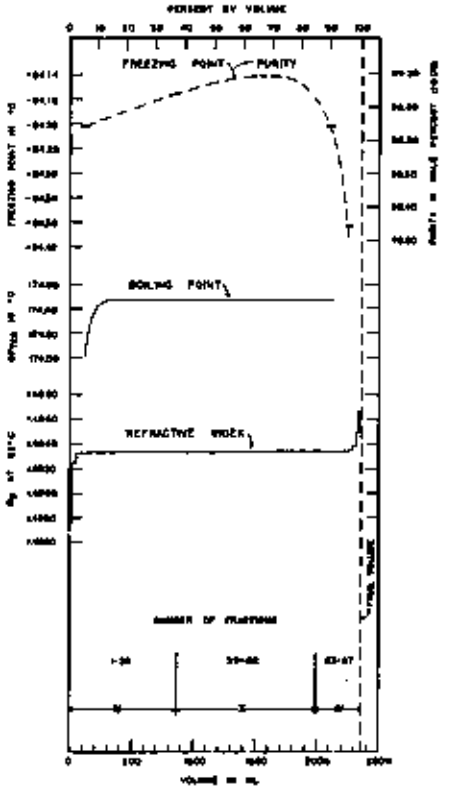


FIGURE 31. *Results of the final purification of 1,3-diethylbenzene by adsorption.*

See legend for figure 30 and footnote n of table 1. The part of the filtrate from each of the first 7 charges marked "X" was blended together for subsequent charges 8 to 13.



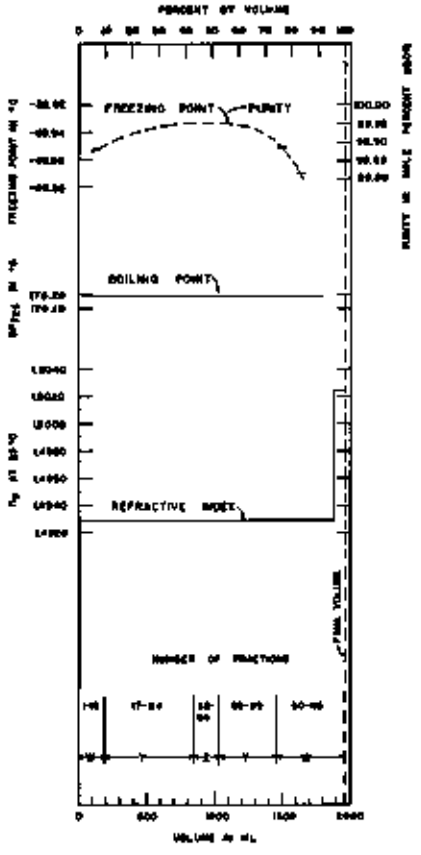


FIGURE 32. *Results of the first distillation of 1,3-diethyl-benzene.*

Azeotropic distillation with diethylene glycol monomethyl ether at 725 mm Hg in Still 8 (11/9/44 to 11/29/44).

FIGURE 33. *Results of the second and final distillation of 1,3-diethylbenzene.*

Regular distillation at 725 mm Hg in Still 12 (10/26/45 to 11/16/45). See footnote a of table 1.

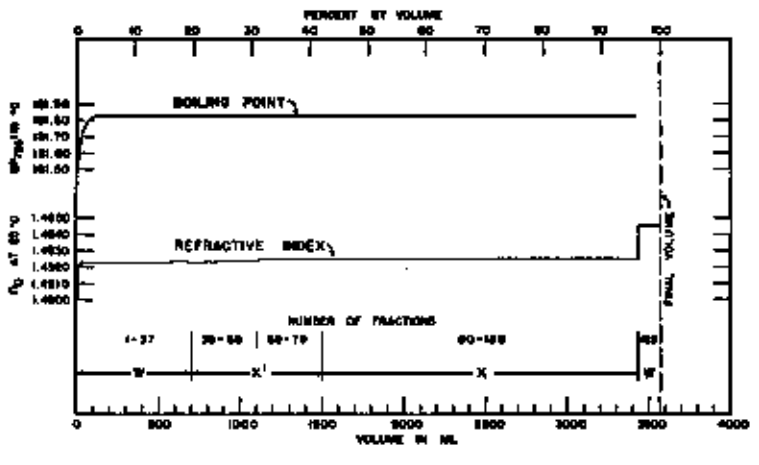


FIGURE 34. *Results of the first distillation of 1,4-diethylbenzene.*

Regular distillation at 725 mm Hg in Still 9 (1/8/47 to 2/10/47). The distillate marked "X" was given its final purification by adsorption. (See results in columns 13 and 11 in fig. 36.)

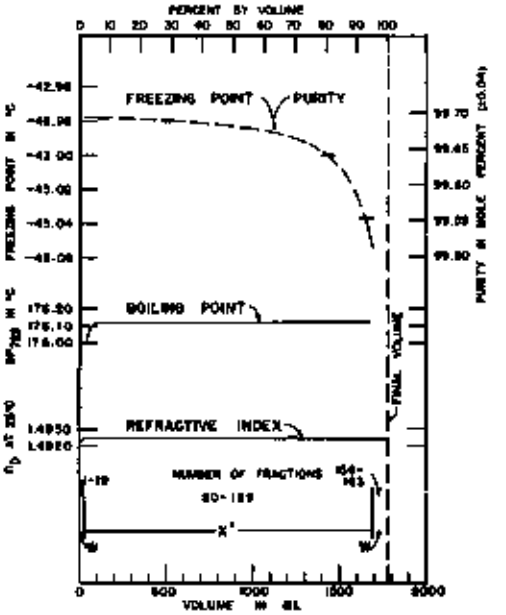


FIGURE 35. *Results of the second distillation of 1,4-diethylbenzene.*

Azeotropic distillation with diethylene glycol monomethyl ether at 725 mm Hg in Still 11A (4/17/47 to 5/19/47). The part of the distillate marked "X" was blended and divided into three similar charges for final purification by adsorption. (See results in columns 1, 2, and 3 in fig. 36.)

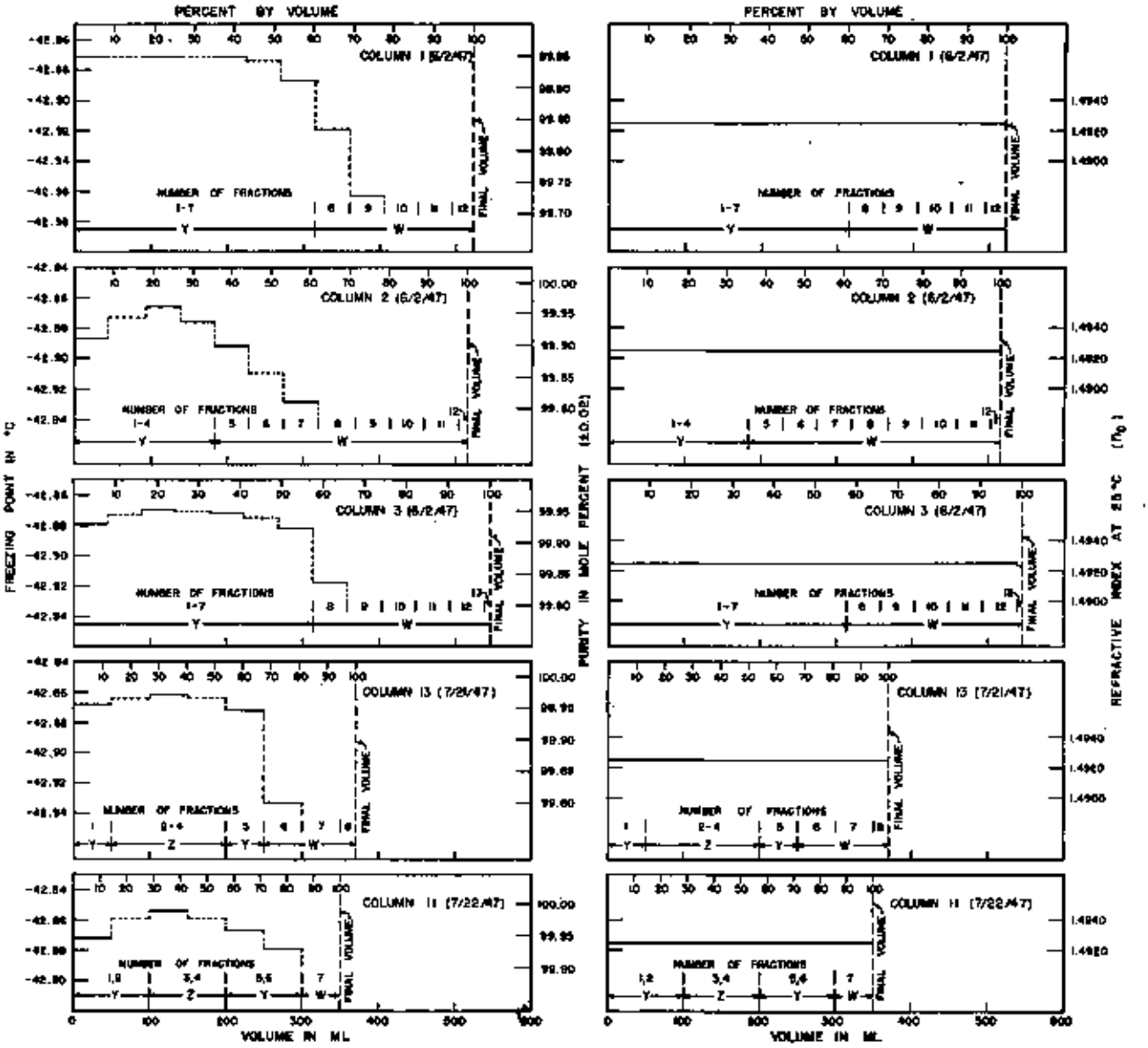


FIGURE 36. *Results of the final purification of 1,4-diethylbenzene by adsorption.*

See legends for figures 34 and 35 and footnote p of table 1.

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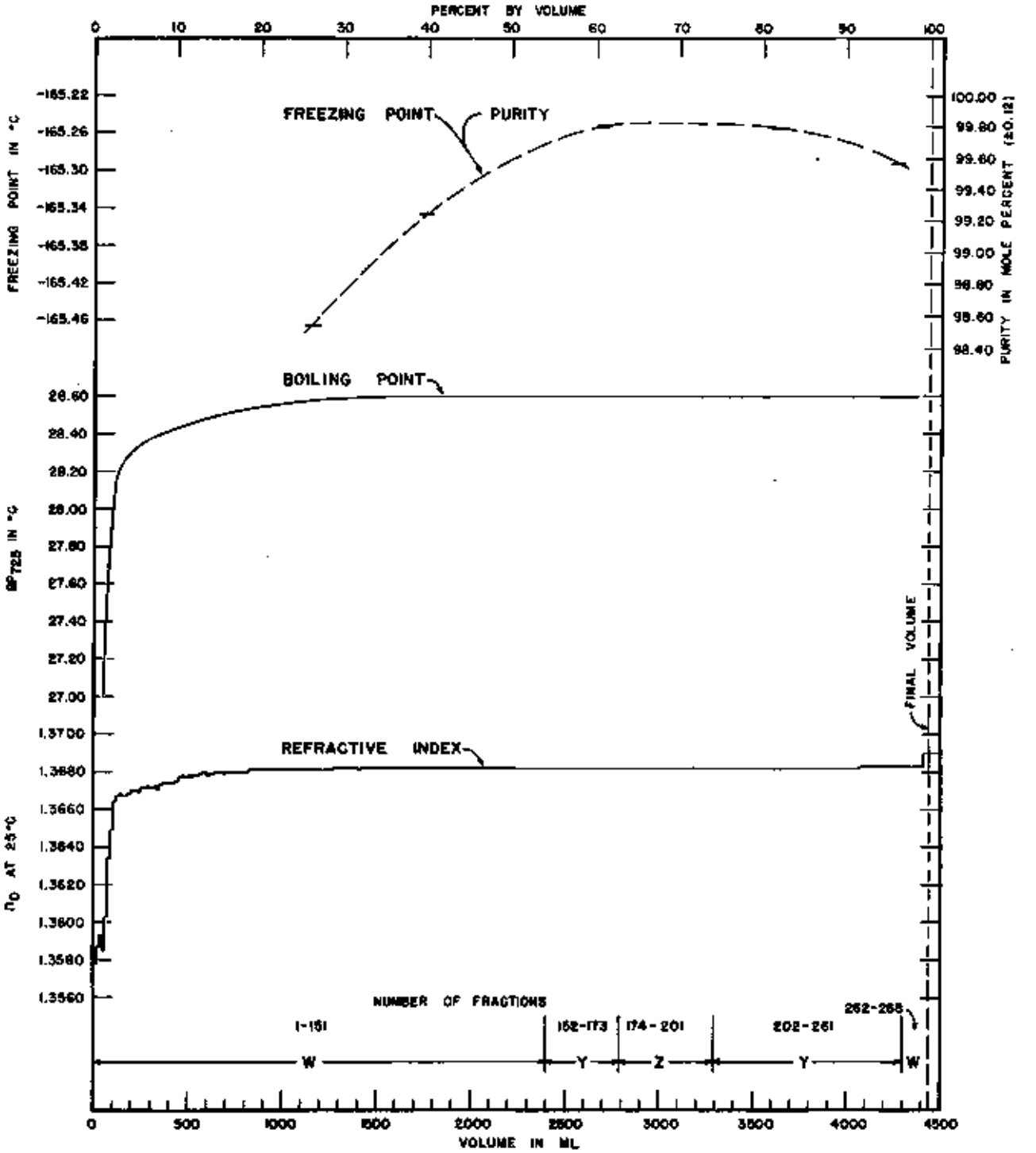

FIGURE 37. *Results of the first and only distillation of 1-pentene.*

Regular distillation at 725 mm Hg in Still 3A (1/21/47 to 3/24/47).

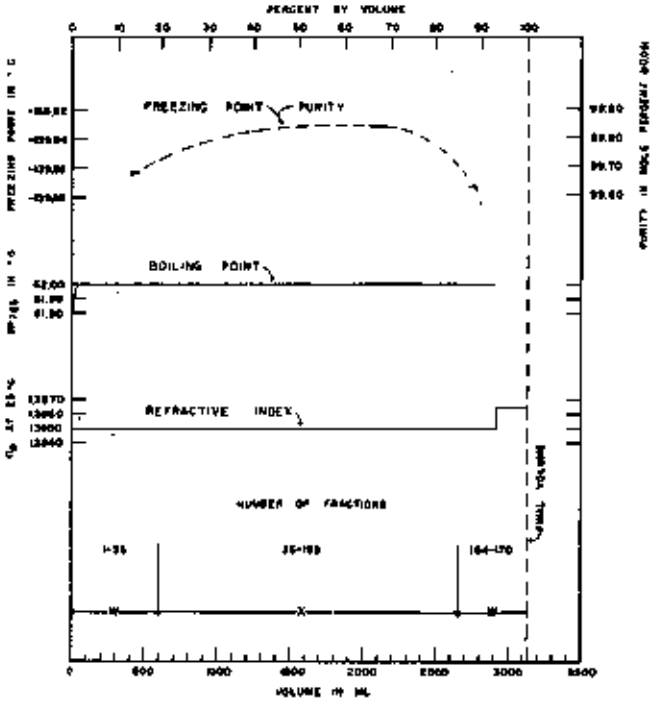

FIGURE 38. *Results of the first distillation of the first lot of 1-hexene.*

Regular distillation at 725 mm Hg in Still 9 (9/30/46 to 11/1/46).

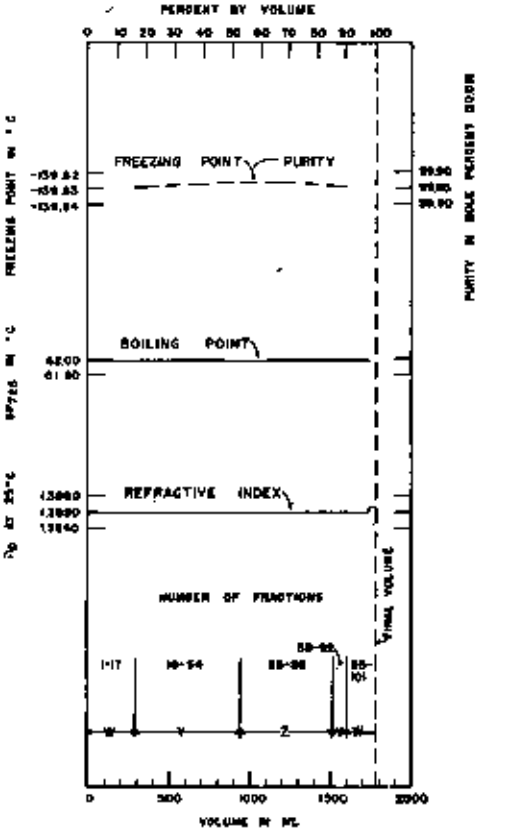

FIGURE 39. *Results of the second and final distillation of the first lot of 1-hexene.*

Regular distillation at 725 mm Hg in Still 4 (2/13/47 to 3/4/47). See footnote r of table I.

503370—48——8

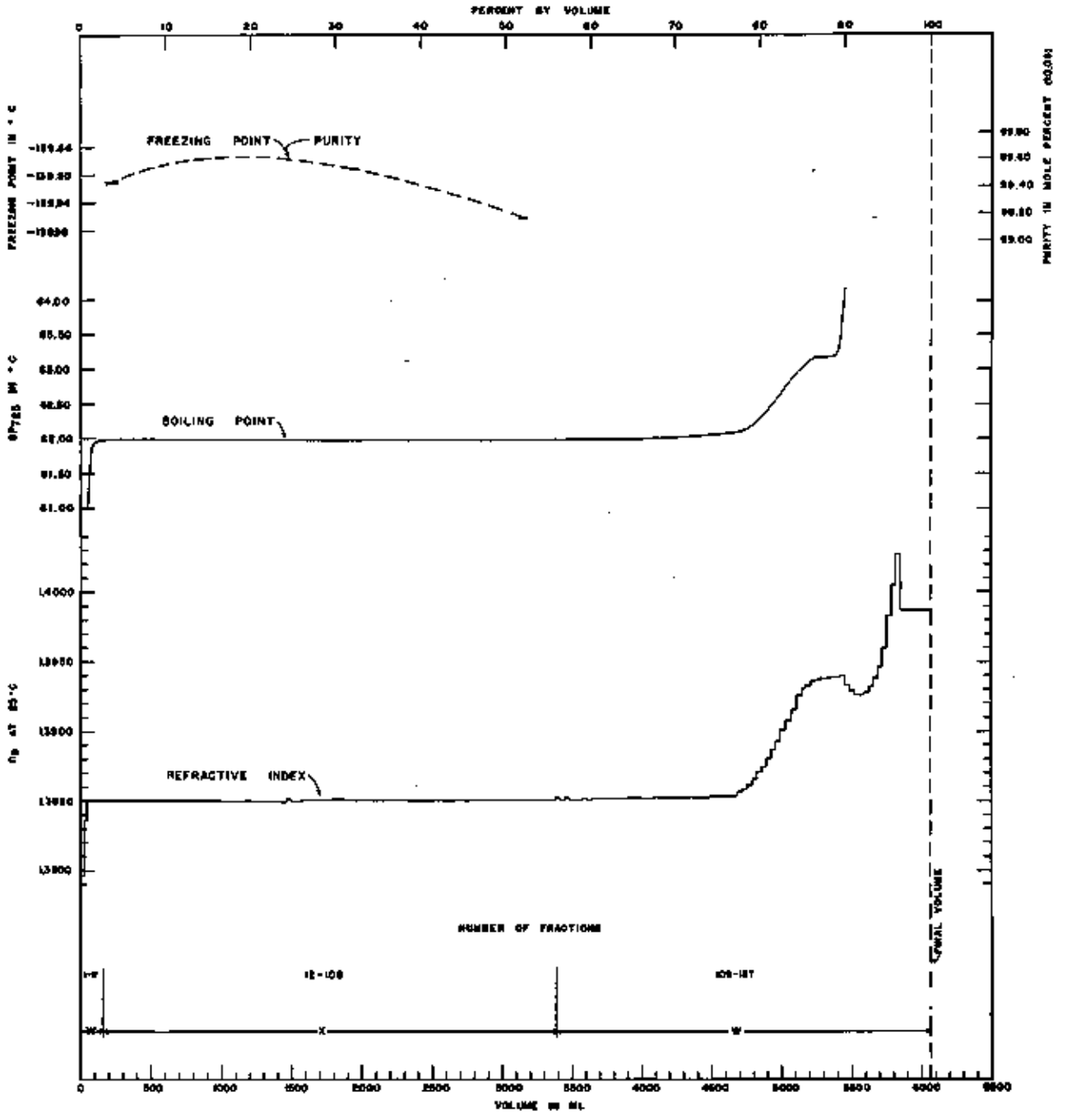

FIGURE 40. *Results of the first distillation of a second lot of 1-hexene.*

Regular distillation at 725 mm Hg in Still 13 (12/9/46 to 1/20/47).

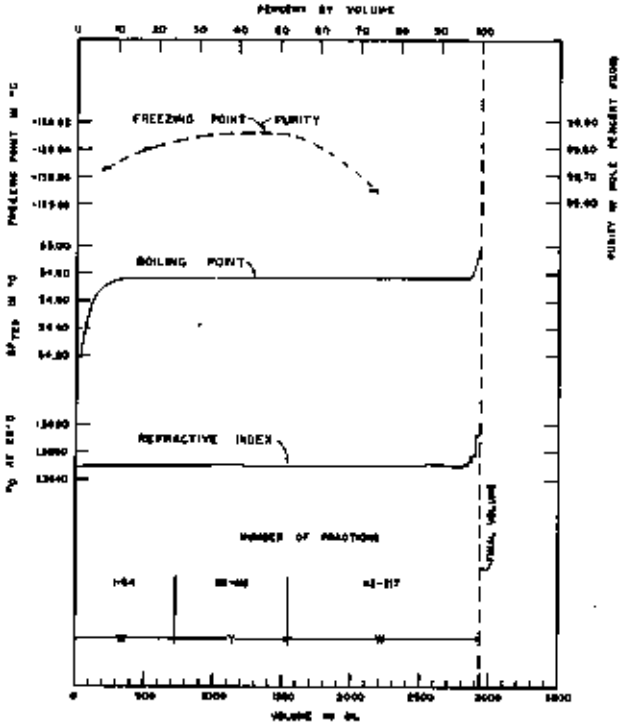

FIGURE 41. *Results of the second and final distillation of the second lot of 1-hexene.*

Azeotropic distillation with ethanol at 725 mm Hg in Still 9 (2/11/47 to 3/28/47). See footnote r of table 1.

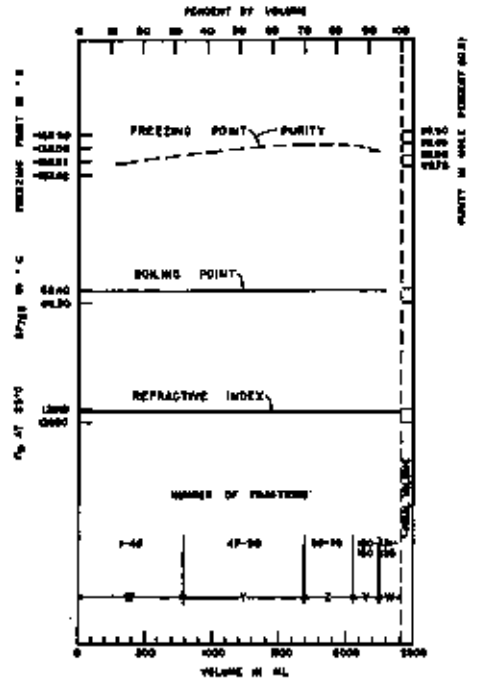

FIGURE 43. *Results of the second and final distillation of trans-2-hexene.*

Regular distillation at 725 mm Hg in Still 2A (3/13/47 to 4/7/47). See footnotes s and t of table 1.

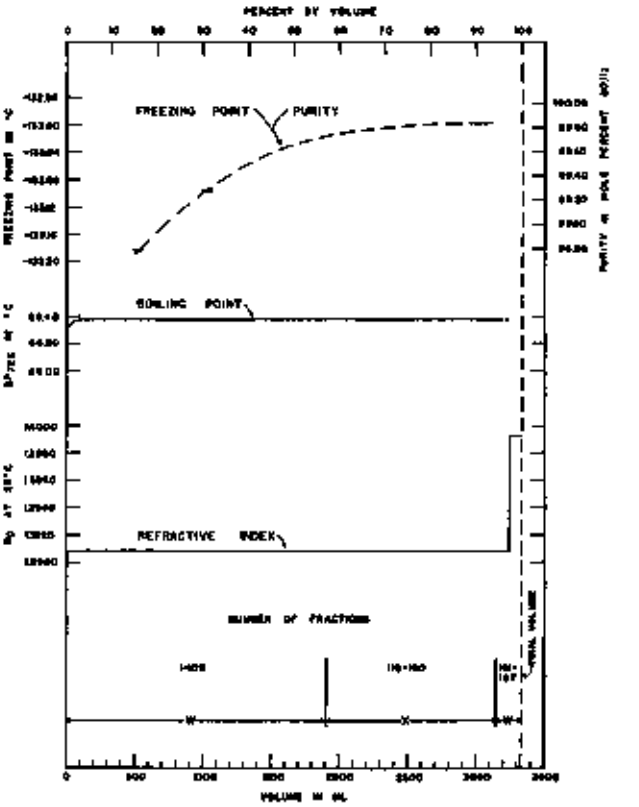

FIGURE 42. *Results of the first distillation of trans-2-hexene.*

Regular distillation at 725 mm Hg in Still 11A (12/17/46 to 1/20/47). One of two distillations of similar material.

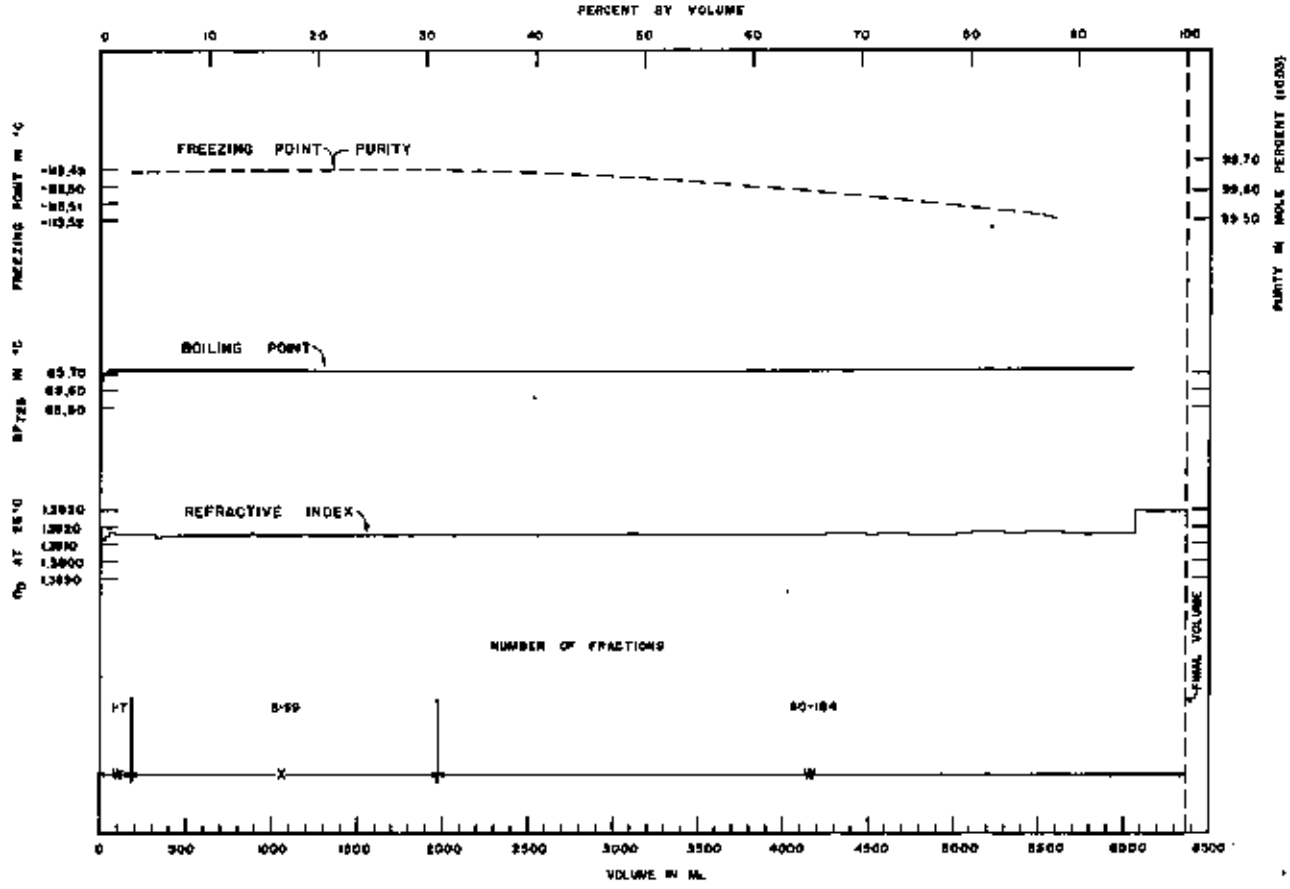

FIGURE 44. *Results of the first distillation of trans-3-hexene.*
Regular distillation at 726 mm Hg in Still 13 (7/29/46 to 9/3/46).

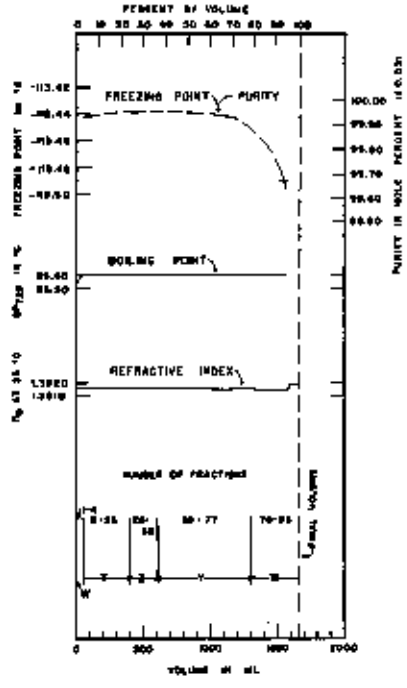

FIGURE 45. *Results of the second and final distillation of trans-3-hexene.*

Regular distillation at 725 mm Hg in Still 11A (11/27/46 to 12/16/46).

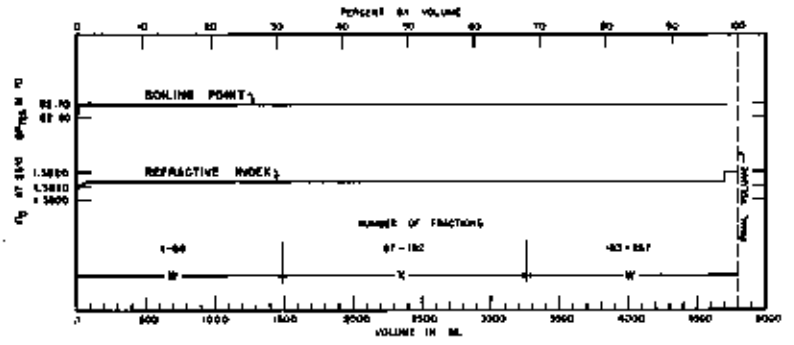

FIGURE 46. *Results of the first distillation of 3-methyl-1-pentene.*

Regular distillation at 725 mm Hg in Still 9 (11/4/46 to 12/30/46). One of two distillations of similar material. See footnote u of table I.

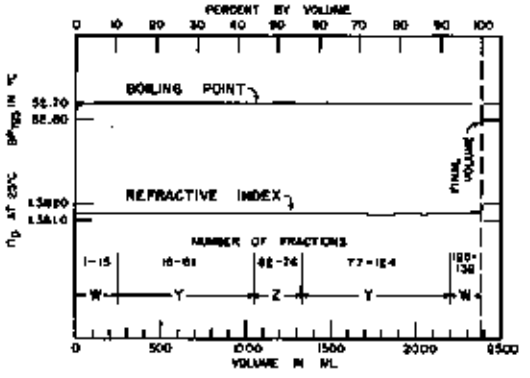

FIGURE 47. *Results of the second and final distillation of 3-methyl-1-pentene.*

Regular distillation at 725 mm Hg in Still 11A (5/20/47 to 6/16/47).

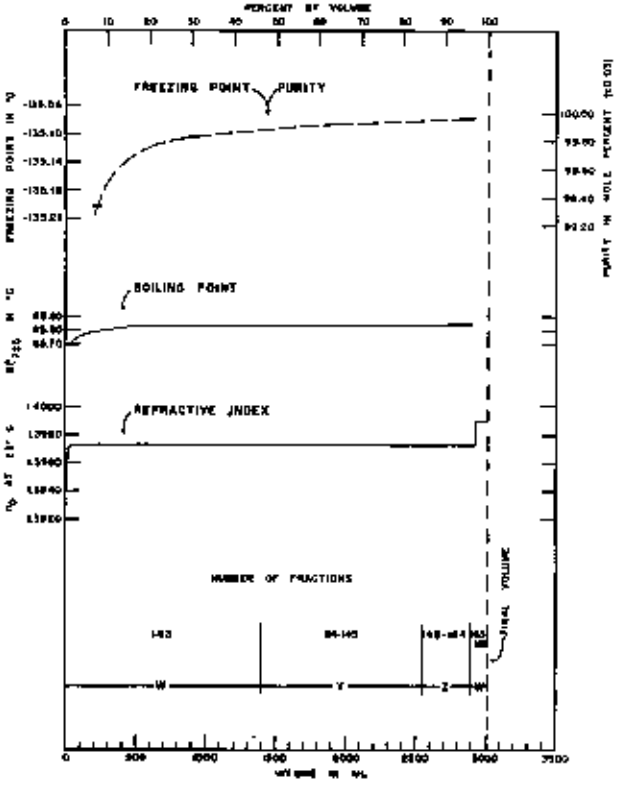

FIGURE 48. *Results of the first and only distillation of 2-methyl-2-pentene.*

Regular distillation at 725 mm Hg in Still 4 (5/28/47 to 6/25/47).

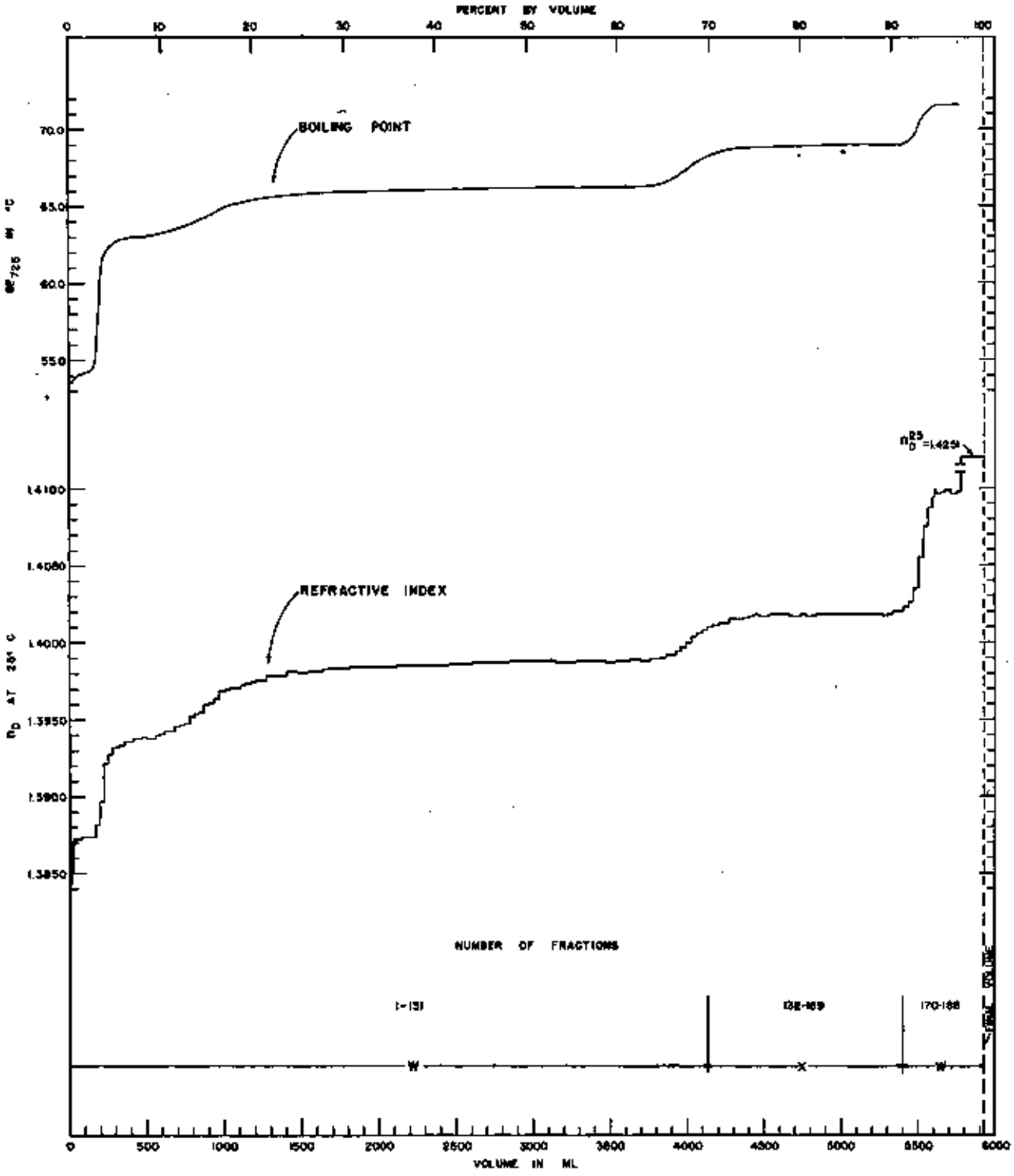

FIGURE 49. *Results of the first distillation of 3-methyl-cis-2-pentene.*

Regular distillation at 725 mm Hg in Still S (12/12/45 to 1/21/46). One of three distillations of similar material. See footnote w of table 1.

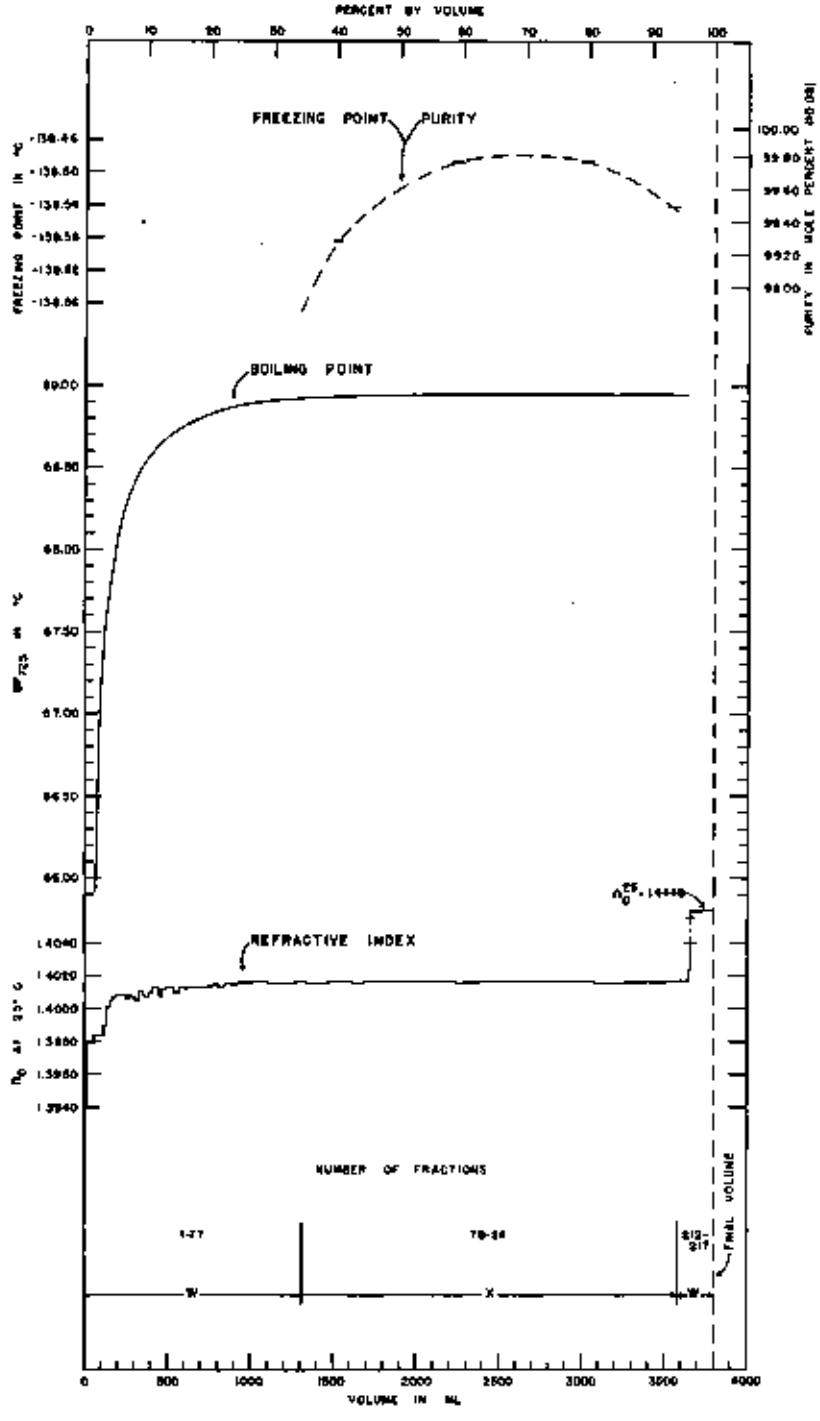

FIGURE 50. *Results of the second distillation of 3-methyl-cis-2-pentene.*

Regular distillation at 725 mm Hg in Still 2A (4/8/47 to 5/19/47). See footnotes w and x to table 1.

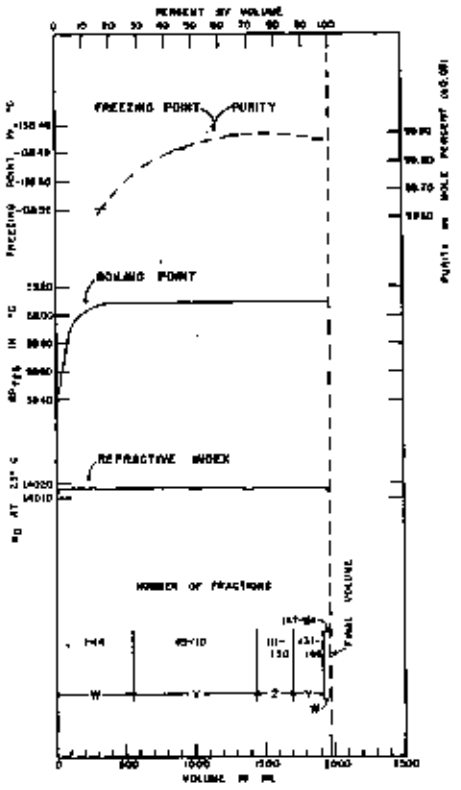

FIGURE 51. *Results of the third and final distillation of 3-methyl-cis-2-pentene.*

Azeotropic distillation with ethanol at 726 mm Hg in Still 12A (6/20/47 to 7/17/47).

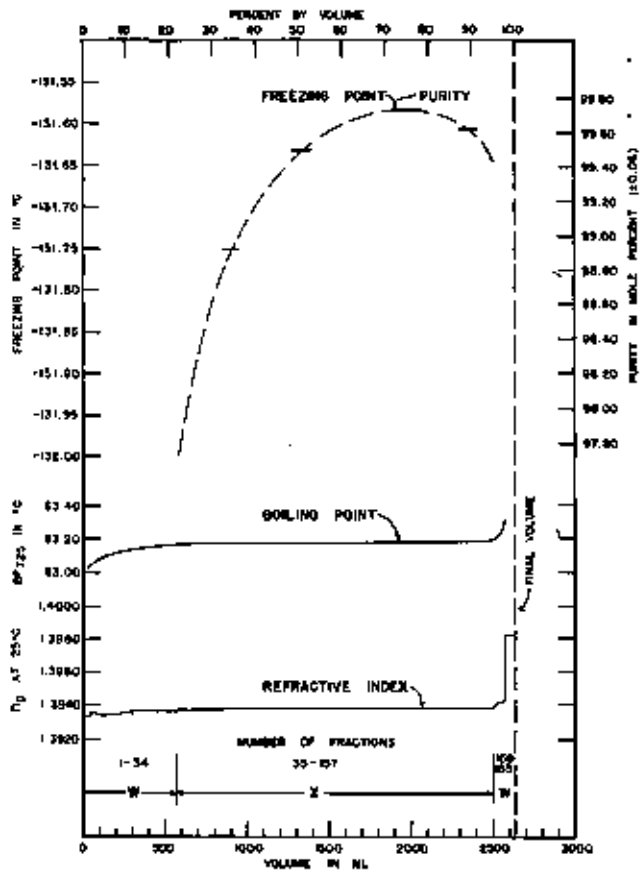

FIGURE 52. *Results of the first distillation of the first lot of 2-ethyl-1-butene.*

Regular distillation at 725 mm Hg in Still 12 (2/7/46 to 3/11/46).

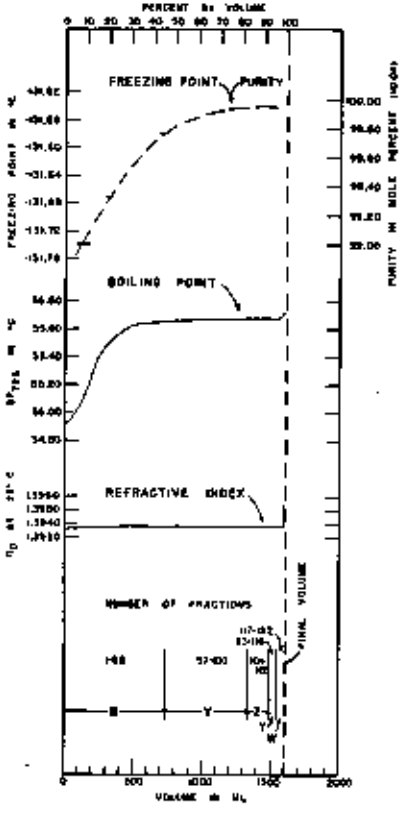

FIGURE 53. *Results of the second and final distillation of the first lot of 2-ethyl-1-butene.*

Azeotropic distillation with ethanol at 725 mm Hg in Still 3A (3/25/47 to 4/17/47). See footnotes aa and bb to table 1.

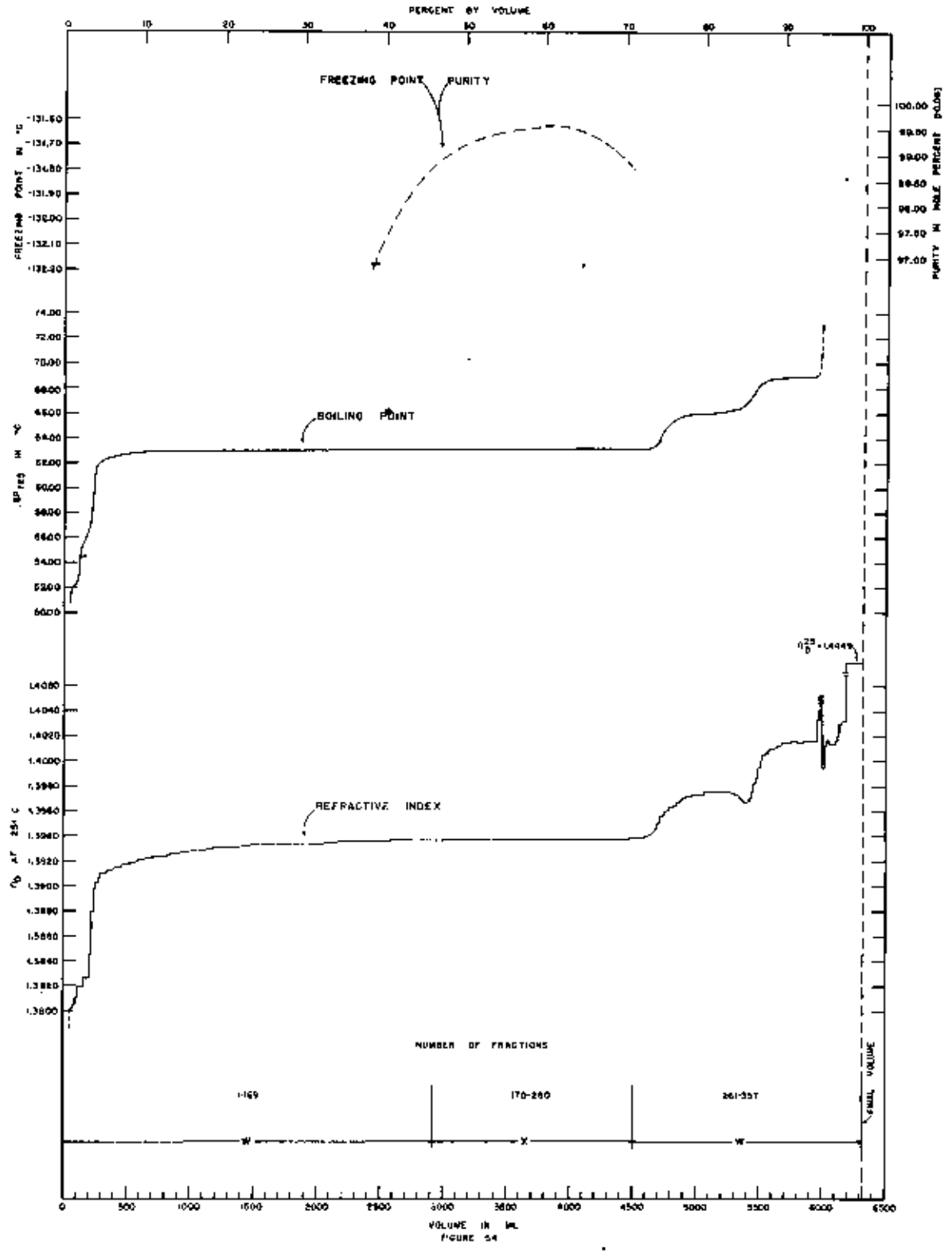

FIGURE 54. *Results of the first distillation of the second lot of 2-ethyl-1-butene.*

Regular distillation at 725 mm Hg in Still 10 (12/20/46 to 2/27/47).

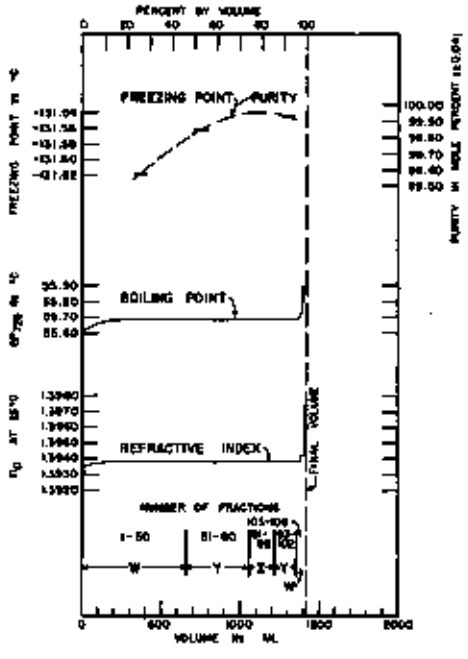

FIGURE 55. *Results of the second and final distillation of the second lot of 2-ethyl-1-butene.*

Azeotropic distillation with ethanol at 725 mm Hg in Still 4 (5/5/47 to 5/25/47.) See footnotes aa and bb to table 1.

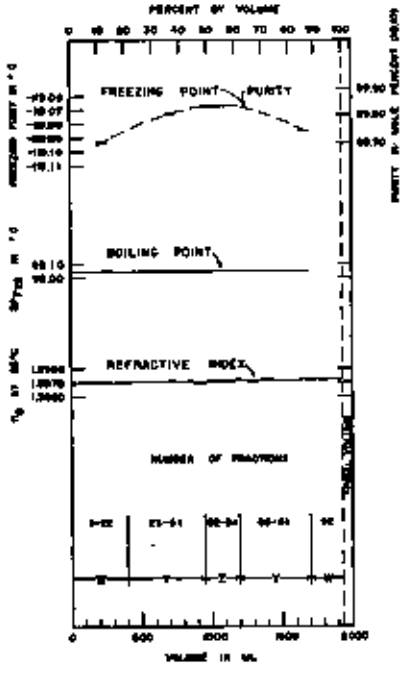

FIGURE 57. *Results of the second and final distillation of 1-heptene.*

Regular distillation at 725 mm Hg in Still 4 (12/6/46 to 12/26/46).

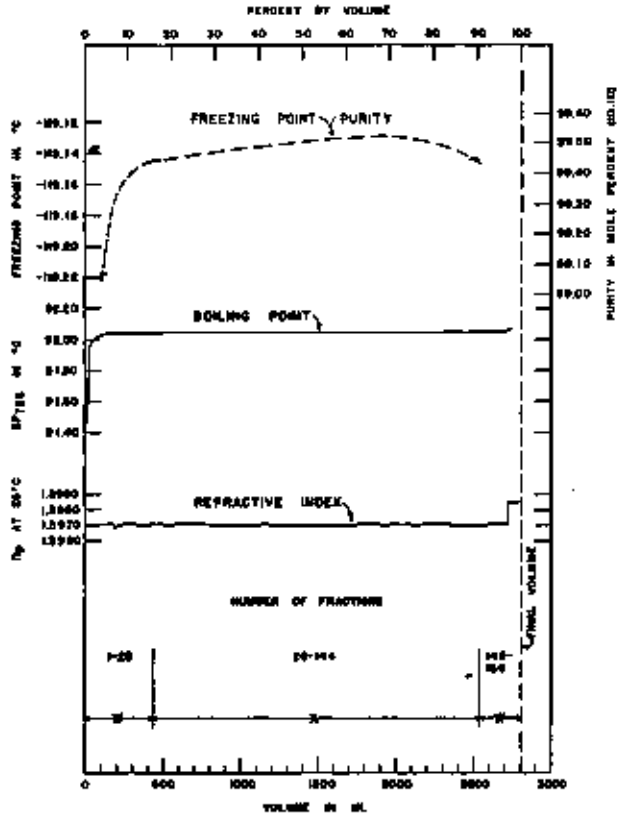

FIGURE 56. *Results of the first distillation of 1-heptene.*

Regular distillation at 725 mm Hg in Still 12 (4/30/46 to 5/31/46).

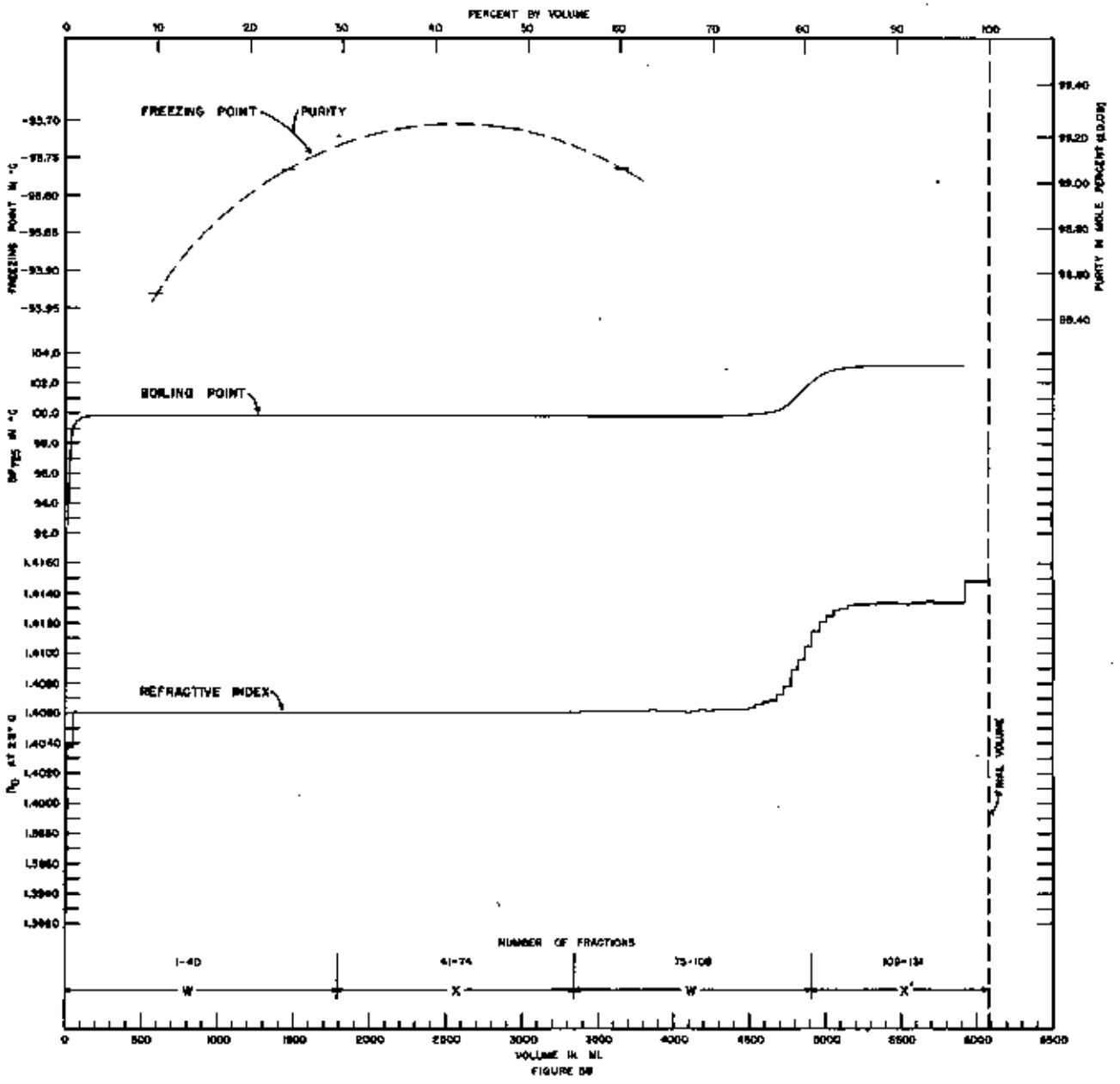

FIGURE 58. *Results of the first distribution of 2,4,4-trimethyl-1-pentene and 2,4,4-trimethyl-2-pentene.*

Regular distillation at 725 mm Hg in Still 14 (12/19/45 to 1/12/46). One of four distillations of similar material. See footnote cc of table 1. Fractions 41 to 74 (marked "x") were redistilled to obtain 2,4,4-trimethyl-1-pentene (see fig. 60). Fractions 109 to 131 (marked "x'") were redistilled to obtain 2,4,4-trimethyl-2 pentene (see fig. 61).

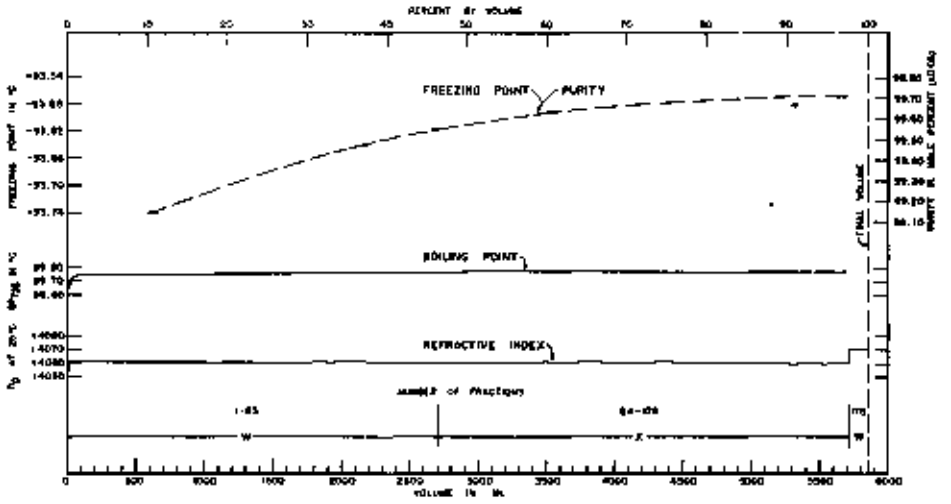

FIGURE 59. *Results of the second distillation of 2,4,4-trimethyl-1-pentene.*
Regular distillation at 725 mm Hg in Still 13 (4/16/46 to 5/20/46). See footnote dd to table 1

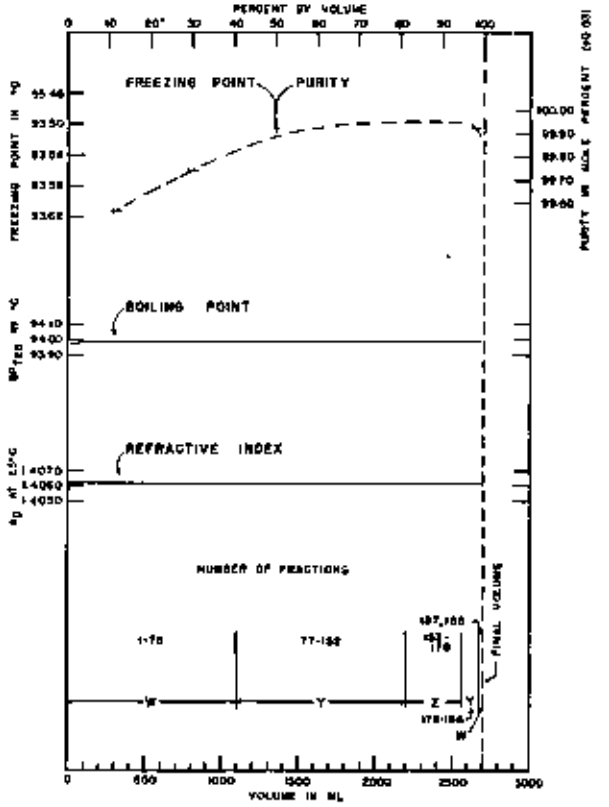

FIGURE 60. *Results of the third and final distillation of 2,4,4-trimethyl-1-pentene.*
Azeotropic distillation with ethylene glycol monomethyl ether at 725 mm Hg in Still 15A (6/26/47 to 7/30/47).

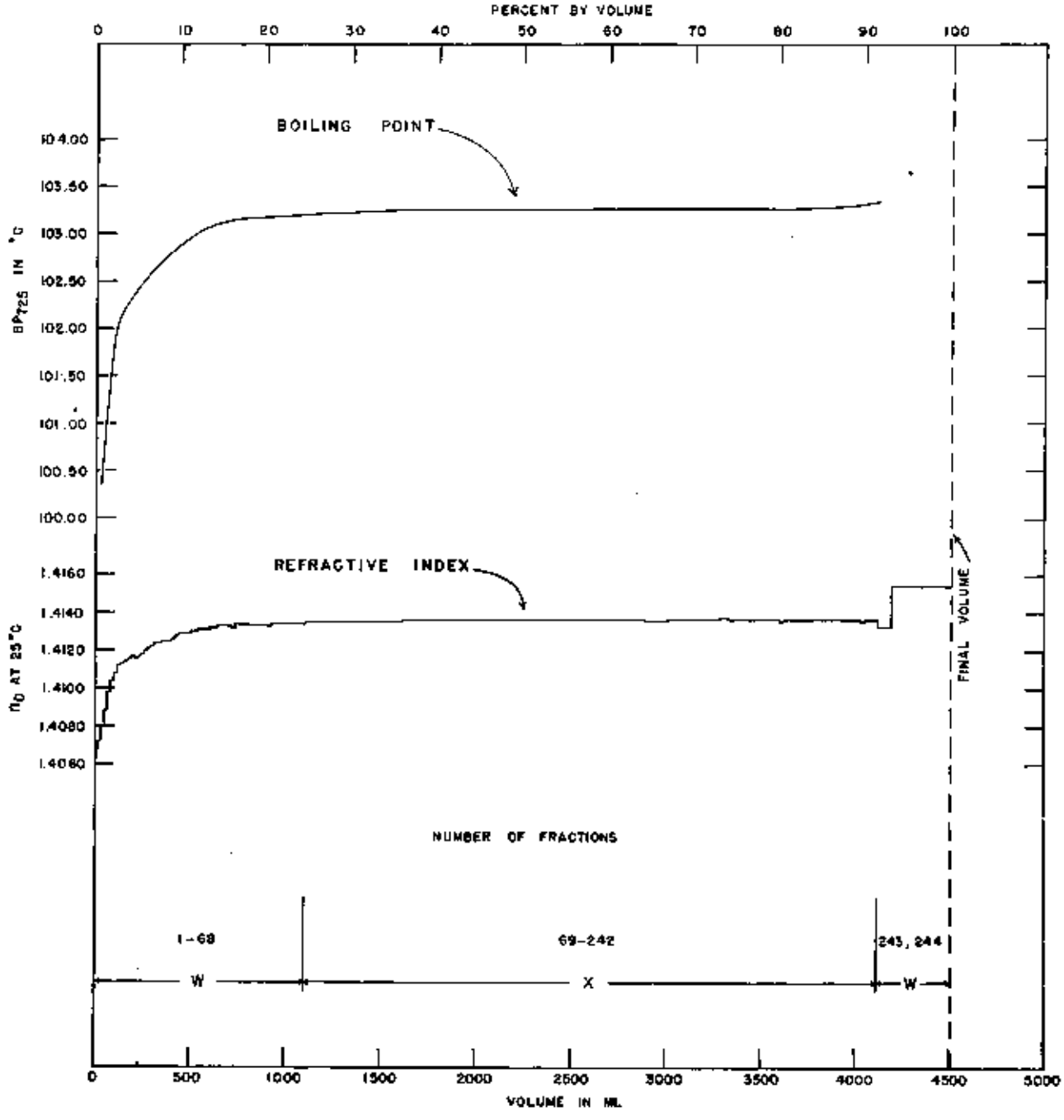

FIGURE 61. *Results of the second distillation of 2,4,4-trimethyl-2-pentene.*

Regular distillation at 725 mm Hg in Still 10 (4/16/46 to 5/31/46). See footnote dd to table 1.

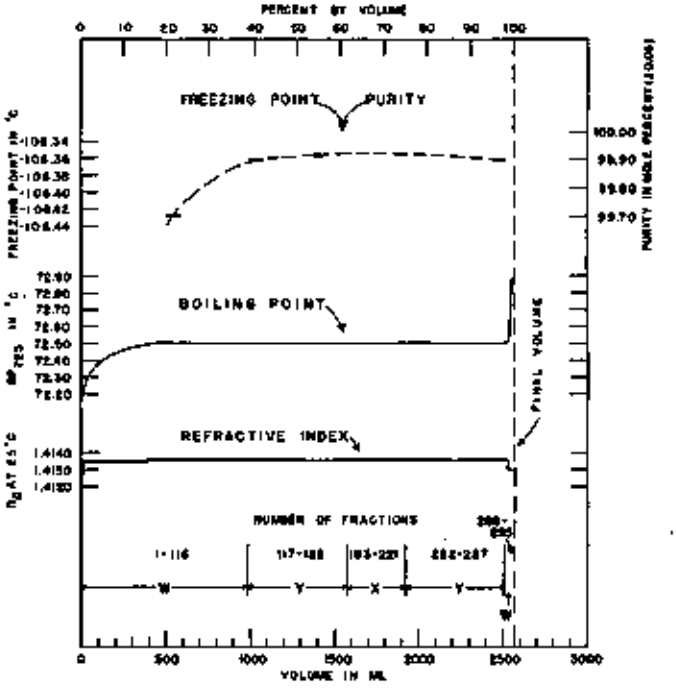

FIGURE 62. *Results of the third and final distillation of 2,4,4-trimethyl-2-pentene.*

Azeotropic distillation with ethanol at 725 mm Hg in Still 11A (6/17/47 to 8/8/47).

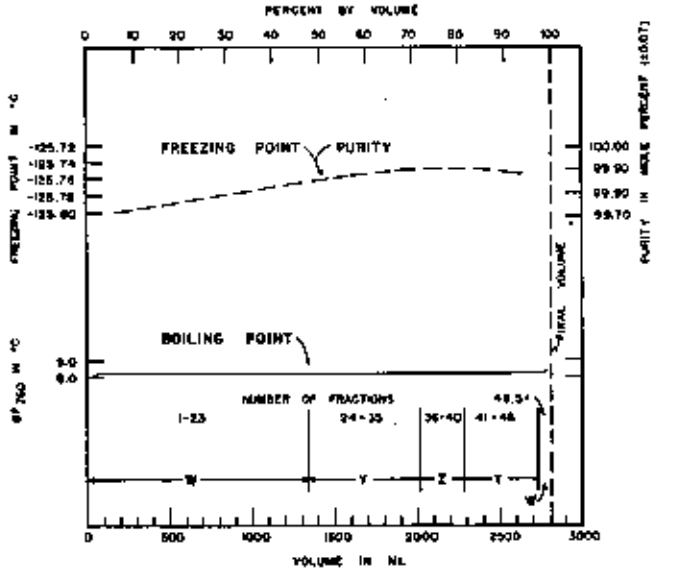

FIGURE 64. *Results of the first and only distillation of 1-butyne.*

Regular distillation at atmospheric pressure in Still I (6/6/45 to 11/5/45).

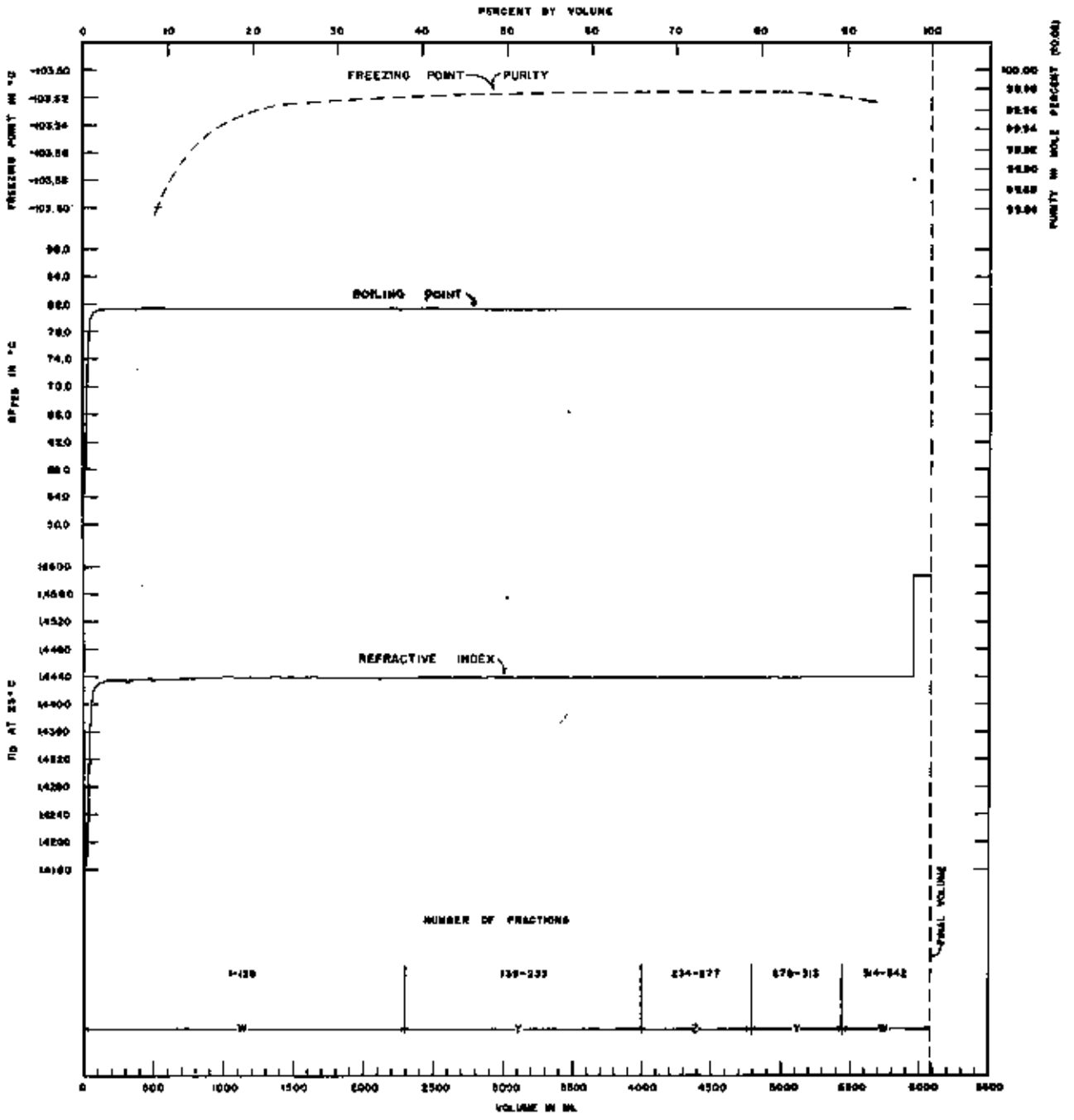

FIGURE 63. *Results of the first and only distillation of cyclohexene.*

Regular distillation at 725 mm Hg in Still 10 (10/15/46 to 12/19/46).

WASHINGTON, May 21, 1948.