

Purification, Purity, and Freezing Points of *n*-Decane, 4 Alkylcyclopentanes, 9 Alkylcyclohexanes, 2 Monoolefins, 1, 2-Butadiene, and 2-Butyne 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 18 hydrocarbons of the API-Standard and API-NBS series, including one paraffin, four alkylcyclopentanes, nine alkylcyclohexanes, two monoolefins, one diolefin, and one acetylene.

I. Introduction

Previous reports described the purification and determination of freezing points and purity of 66 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].³ This report describes the purification and determination of freezing points and purity of an additional 18 hydrocarbon compounds under this cooperative program, including *n*-decane, 4 alkylcyclopentanes, 9 alkylcyclohexanes, 2 monoolefins, 1,2-butadiene and 2-butyne.

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.

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.

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

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:

Isobutylcyclopentane.	1,1,3-Trimethylcyclohexane.
<i>cis</i> -1,3-Dimethylcyclohexane.	<i>n</i> -Butylcyclohexane.
<i>trans</i> -1,3-Dimethylcyclohexane.	Isobutylcyclohexane.
<i>n</i> -Propylcyclohexane.	<i>sec</i> -Butylcyclohexane.
Isopropylcyclohexane.	<i>tert</i> -Butylcyclohexane.
	2-Butyne.

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

1-Methyl-1-ethylcyclopentane.
<i>cis</i> -1-Methyl-2-ethylcyclopentane.
3,3-Dimethyl-1-butene.

By the Standard Oil Development Co., Esso Laboratories, Elizabeth, N. J., and Baton Rouge, La., through W. J. Sweeney:

1,2-Butadiene.

By the Tide Water Associated Oil Co., Associated, Calif., through H. Y. Hyde:

Cyclopentane (B) ⁴

By the API Research Project 6 at the National Bureau of Standards, through F. D. Rossini:

<i>n</i> -Decane.
1-Octene.

⁴ See footnote "a" of table 2.

TABLE 1.—Information on the purification of 18 API-Standard and API-NBS hydrocarbons

Compound ^a	Starting material provided by	Hydrocarbon charged for distillation		Distillation ^f								Volume of selected sample	
				Kind ^c	Azeo-trope-forming sub-stance ^d	Amount of hydrocarbon in the azeotropic distillate ^e	Distilling column number	Number of theo-retical plates (approx.)	Reflux ratio (approx.)	Rate of collection of distillate	Results plotted in figure		
		Volume	Purity									API-Standard	API-NBS
	laboratory ^b	liters	mole percent			percentage by volume				ml/hour		ml	ml
<u>Paraffins</u>													
n-Decane	APIRP 6 ^g	^h 5.80 ⁱ 5.80	98.55±0.06 99.84±0.02	Reg. Reg.			14 7	125 130	125/1 145/1	12.5 8.5	1 2	1400	415
<u>Alkylcyclopentanes</u>													
Cyclopentane (B)	Tide Water Assoc.	2.45	99.84±0.02	Reg.			2	100	150/1	2.0	3	1020	300
1-Methyl-1-ethylcyclopentane	Penn State	2.10		Azeo.	Ethanol	33	4	200	180/1	4.0	4	1240	370
cis-1-Methyl-2-ethylcyclopentane	Penn State	1.73	94.0±0.5	Azeo.	Isopropanol	14	4	200	180/1	4.0	5	716	158
Isobutylcyclopentane	APIRP 45	3.19 2.25	99.82±0.08	Reg. Azeo.	Cell.	59	13 10	130 135	155/1 165/1	8.0 4.5	6 7	1060	290
<u>Alkylcyclohexanes</u>													
cis-1,3-Dimethylcyclohexane ^j	APIRP 45	^l 5.90 ^m 5.98 2.60	99.89±0.05	Reg. Reg. Azeo.			15 7 15A	125 130 200	125/1 145/1 160/1	12.5 8.5 4.5	8 9 10	1200	360
trans-1,3-Dimethylcyclohexane ^k	APIRP 45	^l 5.90 ^m 3.00		Reg. Azeo.	Ethanol	35	15 9	125 135	125/1 165/1	12.5 4.5	8 11	1100	360
n-Propylcyclohexane	APIRP 45	1.48 ⁿ 1.05 ^o 2.25	99.48±0.08	Reg. Azeo. Azeo.	Cell. Cell.	40 40	8 9 9	130 135 135	145/1 165/1 165/1	8.5 4.5 4.5	12 13 14	365) 895) ^p	360
Isopropylcyclohexane	APIRP 45	3.90 1.79	98.8±0.1	Reg. Azeo.	Cell.	43	8 10	130 135	155/1 165/1	8.0 4.5	15 16	875	147
1,1,3-Trimethylcyclohexane	APIRP 45	2.72 1.80	99.64±0.08	Reg. Azeo.	Me. Cell.	63	7 10	130 135	155/1 165/1	8.0 4.5	17 18	905	199
n-Butylcyclohexane	APIRP 45	2.50 1.71	99.87±0.08	Reg. Azeo.	Butyl Cell.	52	13 10	130 135	155/1 165/1	8.0 4.5	19 20	870	215
Isobutylcyclohexane	APIRP 45	1.91 1.53	99.75±0.09	Reg. Azeo.	Butyl Cell.	63	2 11A	100 200	150/1 160/1	2.0 4.5	21 22	930	185
sec-Butylcyclohexane	APIRP 45	2.59 2.13		Reg. Azeo.	Butyl Cell.	54	12 11A	135 200	165/1 160/1	4.5 4.5	23 24	975	210
tert-Butylcyclohexane	APIRP 45	2.53 1.68	99.94±0.03	Reg. Azeo.	Cell.	26	12 8	135 130	165/1 145/1	4.5 8.5	25 26	1135	285
<u>Olefins</u>													
3,3-Dimethyl-1-butene	Penn State	3.80	98.86±0.15	Reg.			9	135	165/1	4.5	27	1300	385
1-Octene	APIRP 6 ^g	^q 6.00 ^r 6.15	99.70±0.13	Reg. Reg.			13 13	130 130	155/1 155/1	8.0 8.0	28 29	2000	600
<u>Diolefins</u>													
1,2-Butadiene	Std. Oil Dev.	1.40 1.00		Reg. Reg.			1 1	150 150	150/1 150/1	2.0 2.0	30 31	600	100
<u>Acetylenes</u>													
2-Butyne	APIRP 45	2.68		Reg.			1	150	150/1	2.0	32	1125	265

See footnotes on page 323.

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

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].

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].

Figures 1 to 32, inclusive, show graphically the results of the distillations listed in table 1. These figures give, as a function of volume of hydrocarbon distillate, 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 $\pm 0.01^\circ$ C), the freezing point of selected fractions of hydrocarbon distillate (in air at 1 atm usually with a precision near $\pm 0.003^\circ$ C), and the purity of the hydrocarbon distillate. The letters W, X, Y, Z indicate the disposition of the material, as follow: W, returned to the laboratory supplying the material; X, blended for redistillation; Y, used for the API-Standard material; Z, used for the API-NBS material.

Footnotes for Table 1, Page 322.

^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.

Std. Oil Dev.; Standard Oil Development Co. Esso Laboratories, Elizabeth, N. J. and Baton Rouge, La.

Tide Water Assoc.; Tide Water Associated Oil Co., Associated, Calif.

APIRP6; American Petroleum Institute Research Project 6 at the National Bureau of Standards, Washington 25, D. C.

^c The abbreviations are: Azco., azeotropic; Reg., regular.

^d The abbreviations are: Cell., Cellosolve (ethylene glycol monoethyl ether); me. Cell., methyl Cellosolve (ethylene glycol monomethyl ether); Butyl Cell., butyl Cellosolve (ethylene glycol monobutyl 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 [5] for further details.

^g Obtained by purchase of commercially available material from the Connecticut Hard Rubber Co., New Haven, Conn.

^h One of two similar charges.

ⁱ This charge consisted of material, having substantially the same composition, from each of the two previous distillations (see footnote "h").

^j See footnote "f" of table 2.

^k See footnote "g" of table 2.

^l One of three charges of similar material, two of which were 5.90 liters each, and the third of which was 2.50 liters. Both *cis* and *trans*-1,3-dimethylcyclohexane were obtained from this material (see fig. 8).

^m This charge consisted of material, having substantially the same composition, from each of the three previous distillations (see footnote "l").

ⁿ The 1.05 liters for this charge consisted of 0.93 liter from the first distillation in column 8 (see fig. 12), and 0.12 liter which was a second lot of *n*-propylcyclohexane supplied by the API Research Project 45.

^o This is a third lot of *n*-propylcyclohexane supplied by the API Research Project 45.

^p Total volume of the API-Standard sample was 1,260 ml.

^q One of two charges of similar material, the second of which was 6.28 liters.

^r This charge consisted of material, having substantially the same composition, from each of the two previous distillations (see footnote "q").

As demonstrated in the previous reports [2, 3], the blending of fractions of distillate for the preparation of material of the highest purity can be done safely only on the basis of freezing points of selected fractions.

IV. Freezing Points, Cryoscopic Constants, and Purity

Table 2 gives the following information for each of the 18 compounds, except as otherwise indicated: The kind of time-temperature curves, whether freezing or melting, used to determine the freezing point [6]; the freezing point of the actual sample, in air at 1 atm [6], for both the API-Standard and API-NBS lots; the calculated value of the freezing point for zero impurity [6]; 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 [6]; and the resulting calculated amount of impurity in the API-Standard and the API-NBS material.

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.

V. References

- [1] NBS Tech. News Bul. No. 350 (June 1946).
- [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. R. Glasgow, Jr., E. T. Murphy, C. B. Willingham, and F. D. Rossini, *J. Research NBS* **37**, 141 (1946) RP1734.
- [5] C. B. Willingham and F. D. Rossini, *J. Research NBS* **37**, 15 (1946) RP1724.
- [6] A. R. Glasgow, Jr., A. J. Streiff, and F. D. Rossini, *J. Research NBS* **35**, 355 (1945) RP1676.
- [7] 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.
- [8] American Petroleum Institute Research Project 44 at the National Bureau of Standards. Selected Values of Properties of Hydrocarbons. Table 7a.

TABLE 2.—Freezing points and purity of 18 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 ^c		Freezing point for zero impurity in air at 1 atm ^c	Cryoscopic constant ^b A	Calculated amount of impurity in the actual selected sample ^d	
		°C		°C	deg-1	mole percent	
		API-Standard	API-NBS			API-Standard	API-NBS
<u>Paraffins</u>							
n-Decane	F	29.680	-29.677	-29.673 ±0.005	^e (0.0585)	0.04±0.02	0.023±0.018
<u>Alkylcyclopentanes</u>							
Cyclopentane (B)	F	-93.902	-93.887	-93.77 ±0.04	^e (0.00227)	0.030±0.009	0.026±0.009
1-Methyl-1-ethylcyclopentane	F and M	-143.83	-143.82	-143.800 ±0.018	0.0444	0.13±0.08	0.09±0.08
cis-1-Methyl-2-ethylcyclopentane	F and M	-106.05(I)	-106.01(I)	-105.95 ±0.05(I) -106.62 ±0.05(III)(u)	0.0480(I)	0.48±0.24	0.29 ±0.24
Isobutylcyclopentane	M	-115.264	-115.254	-115.225 ±0.020	0.0405	0.16 ±0.08	0.12 ±0.08
<u>Alkylcyclohexanes</u>							
cis-1,3-Dimethylcyclohexane ^f	M	-75.588	-75.579	-75.560 ±0.015	^e (0.0333)	0.09±0.05	0.06±0.05
trans-1,3-Dimethylcyclohexane ^g	M	-90.145	-90.134	-90.100 ±0.020	^e (0.035)	0.16 ±0.07	0.12 ±0.07
n-Propylcyclohexane	M	-94.924	-94.918	-94.900 ±0.015	0.0324	0.08 ±0.05	0.06 ±0.05
Isopropylcyclohexane	F and M	-89.432	-89.416	-89.390 ±0.020	0.0373	0.16 ±0.07	0.10 ±0.07
1,1,3-Trimethylcyclohexane	F and M	-65.831	-65.809	-65.750 ±0.020	0.0260	0.21 ±0.05	0.15 ±0.05
n-Butylcyclohexane	M	-74.747	-74.736	-74.725 ±0.010	0.0385	0.08 ±0.04	0.042±0.039
Isobutylcyclohexane	M	-94.839(II)	-94.830(II)	-94.78 ±0.03(I) -95.72 ±0.04(III)(u)	0.0291(I)	0.17 ±0.09	0.15 ±0.09
sec-Butylcyclohexane						^h (0.30±0.20)	^h (0.25±0.20)
tert-Butylcyclohexane	M	-41.173	-41.170	-41.158 ±0.010	0.0303	0.05 ±0.03	0.04 ±0.03
<u>Olefins</u>							
3,3-Dimethyl-1-butene	F and M	-115.370	-115.365	-115.20 ±0.12	^e (0.00530)	0.09±0.06	0.09±0.06
1-Octene	F and M	-101.748	-101.746	-101.710 ±0.020	0.0637	0.24 ±0.13	0.23 ±0.13
<u>Diolefins</u>							
1,2-Butadiene	M	-136.209	-136.204	-136.190 ±0.012	^e (0.04462)	0.08±0.05	0.06 ±0.05
<u>Ace tylenes</u>							
2-Butyne	F and M	-32.296	-32.281	-32.260 ±0.020	^e (0.0191)	0.069±0.038	0.041±0.038

^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 is labeled (A). See reference [4].

^b F indicates freezing and M indicates melting. See reference [6] for experimental details and the definition of the cryoscopic constant.

^c When a given hydrocarbon has more than one crystalline form, the several forms will be labeled I, II, and III, in order of decreasing temperature of fusion (or freezing point). Forms other than I will be, at their respective freezing points, in metastable equilibrium with the undercooled liquid, but will be unstable with respect to transition to some other solid form at the same temperature and pressure (1 atm). This is indicated by a letter u in parentheses following the Roman numeral.

^d The values in this column, except as otherwise indicated, were calculated as described in reference [6], using the values of the cryoscopic constants and freezing points for zero impurity given in the preceding columns.

^e Not determined in this investigation. From the "z" tables of the American Petroleum Institute Research Project 44 [7].

^f This isomer, formerly labeled "trans", has the following properties: Boiling point at 1 atm, 120.09° C; refractive index, n_D at 25° C, 1.4206; density at 25° C, 0.7620 g/ml [8].

^g This isomer, formerly labeled "cis", has the following properties: boiling point at 1 atm, 124.45° C; refractive index, n_D at 25° C, 1.4284; density at 25° C, 0.7806 g/ml [8].

^h Estimated by analogy with isomers subjected to similar purification.

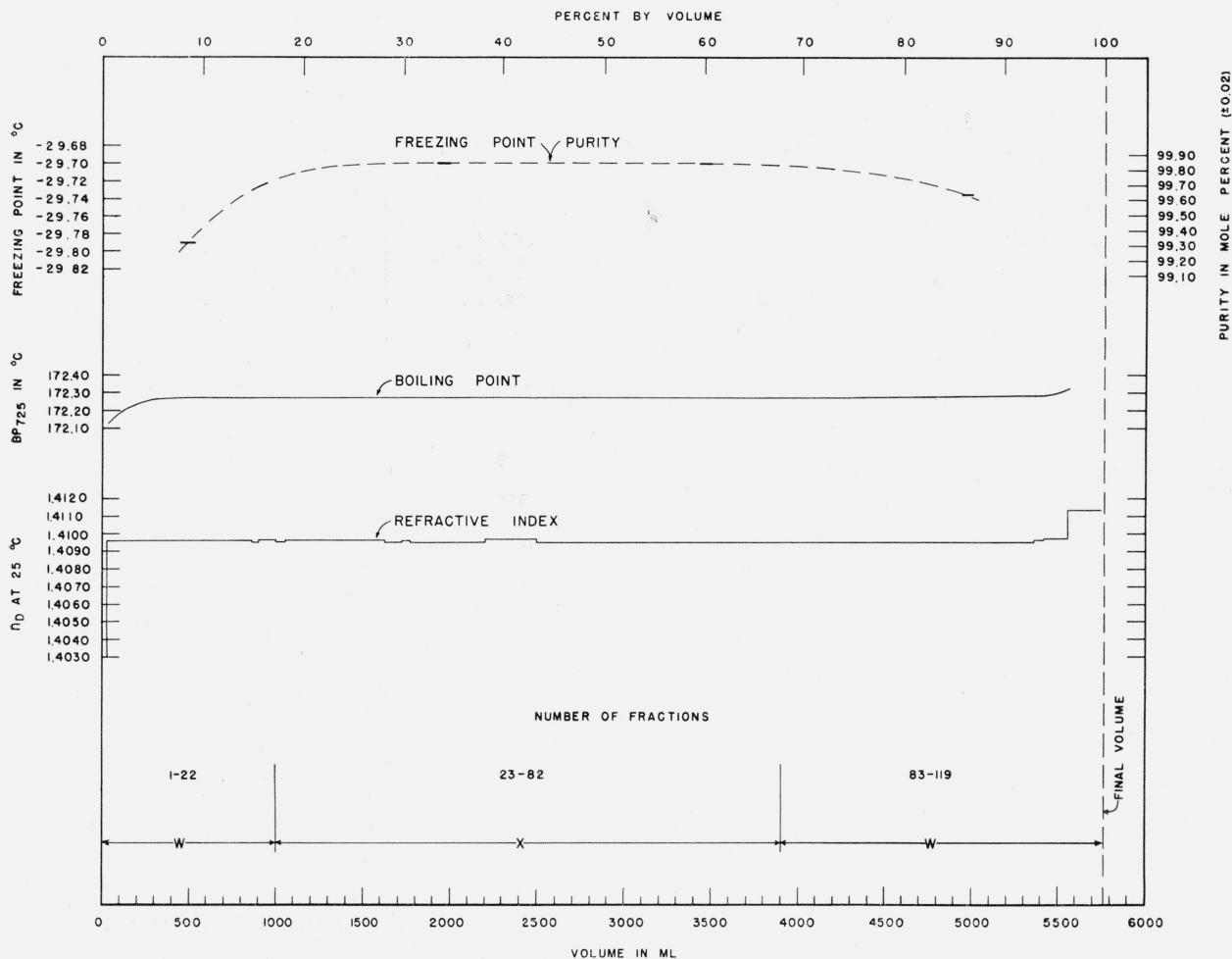


FIGURE 1.—Results of the first distillation of *n*-decane.

Regular distillation at 725 mm Hg in still 14 (7/27/45 to 8/18/45). This is one of two similar distillations.

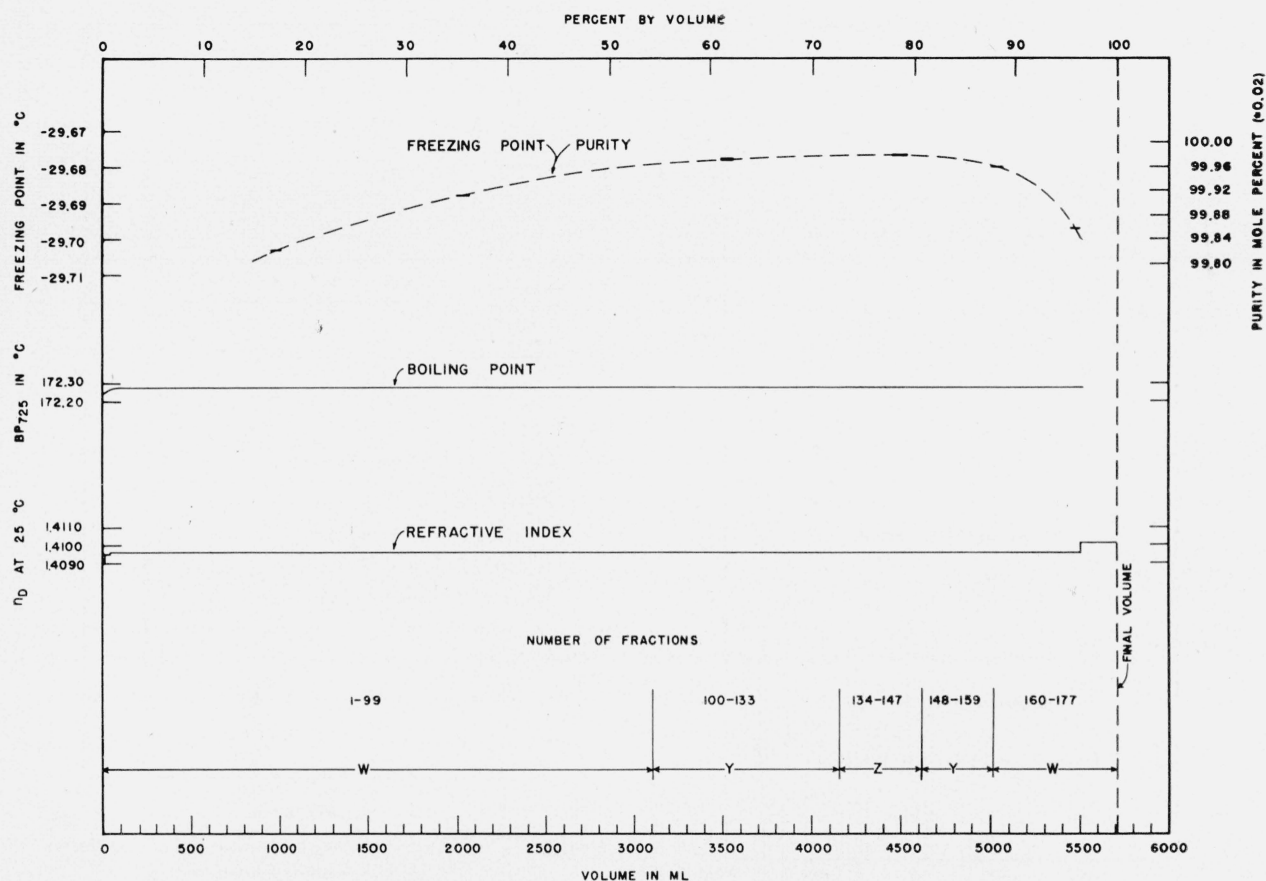


FIGURE 2.—Results of the second and final distillation of *n*-decane.

Regular distillation at 725 mm Hg in still 7 (9/18/45 to 10/22/45). See footnote "i" of table 1.

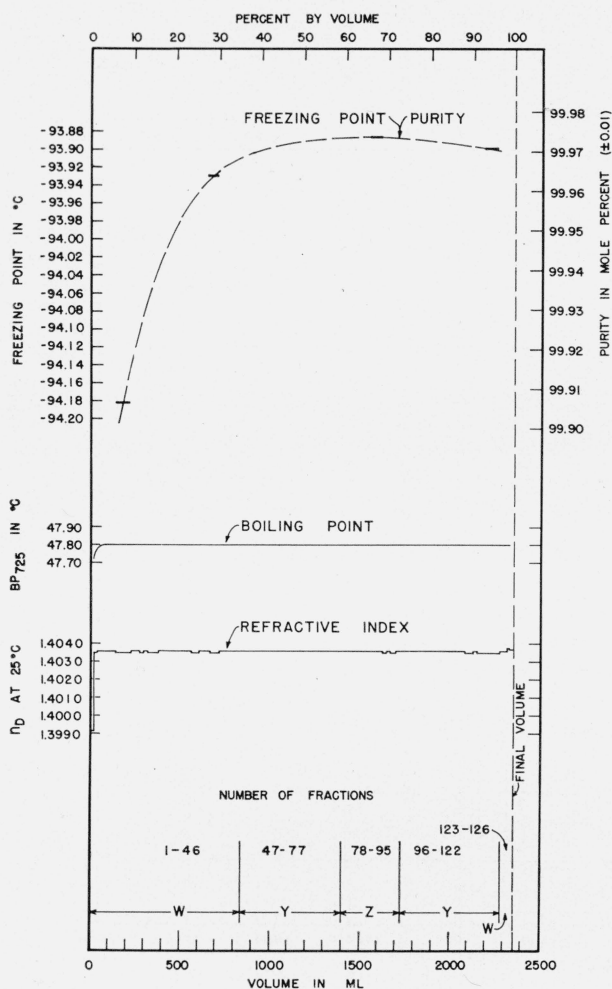


FIGURE 3.—Results of the first and only distillation of cyclopentane.

Regular distillation at 725 mm Hg in still 2 (1/29/46 to 3/25/46).

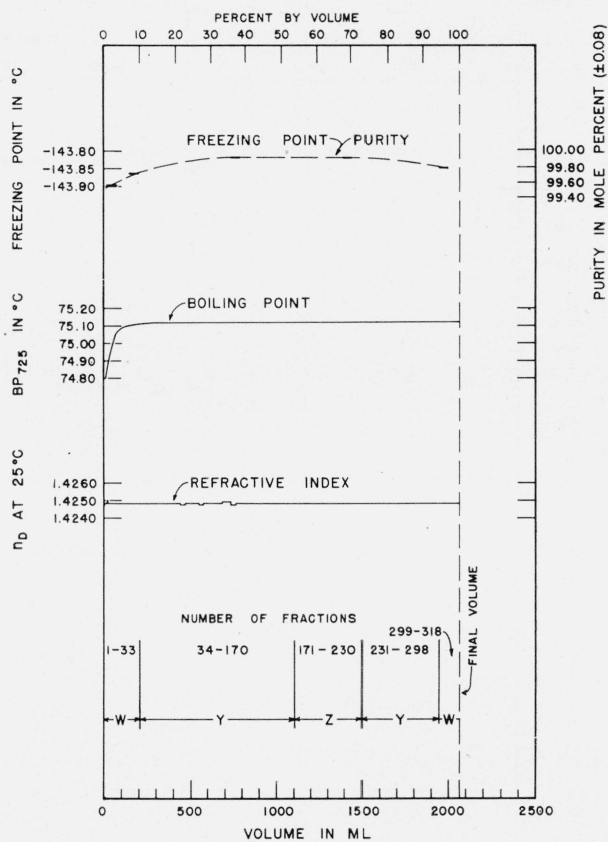


FIGURE 4.—Results of the first and only distillation of 1-methyl-1-ethylcyclopentane.

Azeotropic distillation with ethanol at 725 mm Hg in still 4 (2/25/46 to 4/22/46).

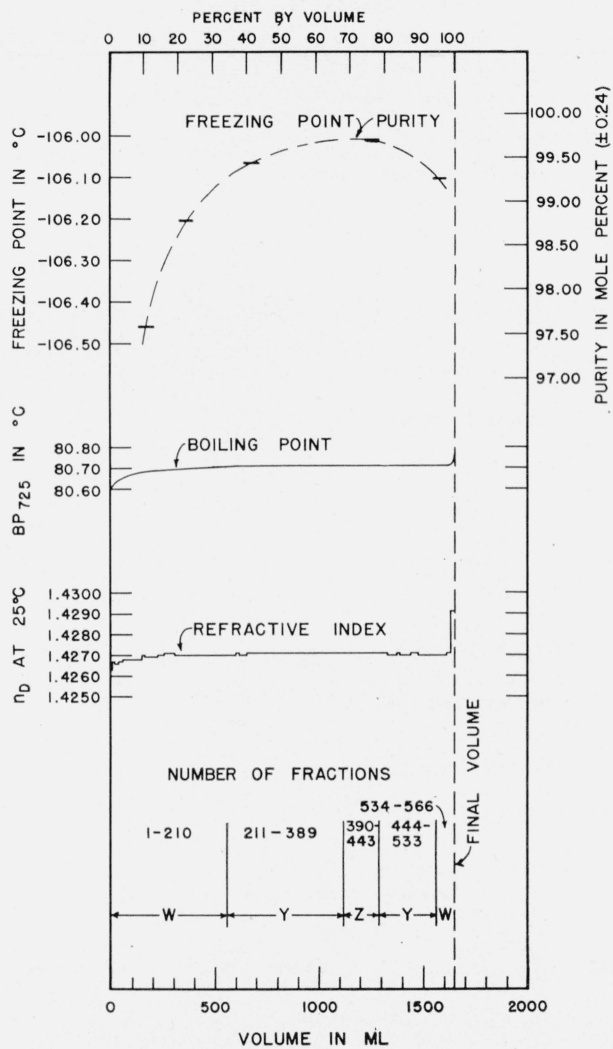


FIGURE 5.—Results of the first and only distillation of *cis*-1-methyl-2-ethylcyclopentane.

Azeotropic distillation with isopropanol at 725 mm Hg in still 4 (5/22/46 to 8/28/46).

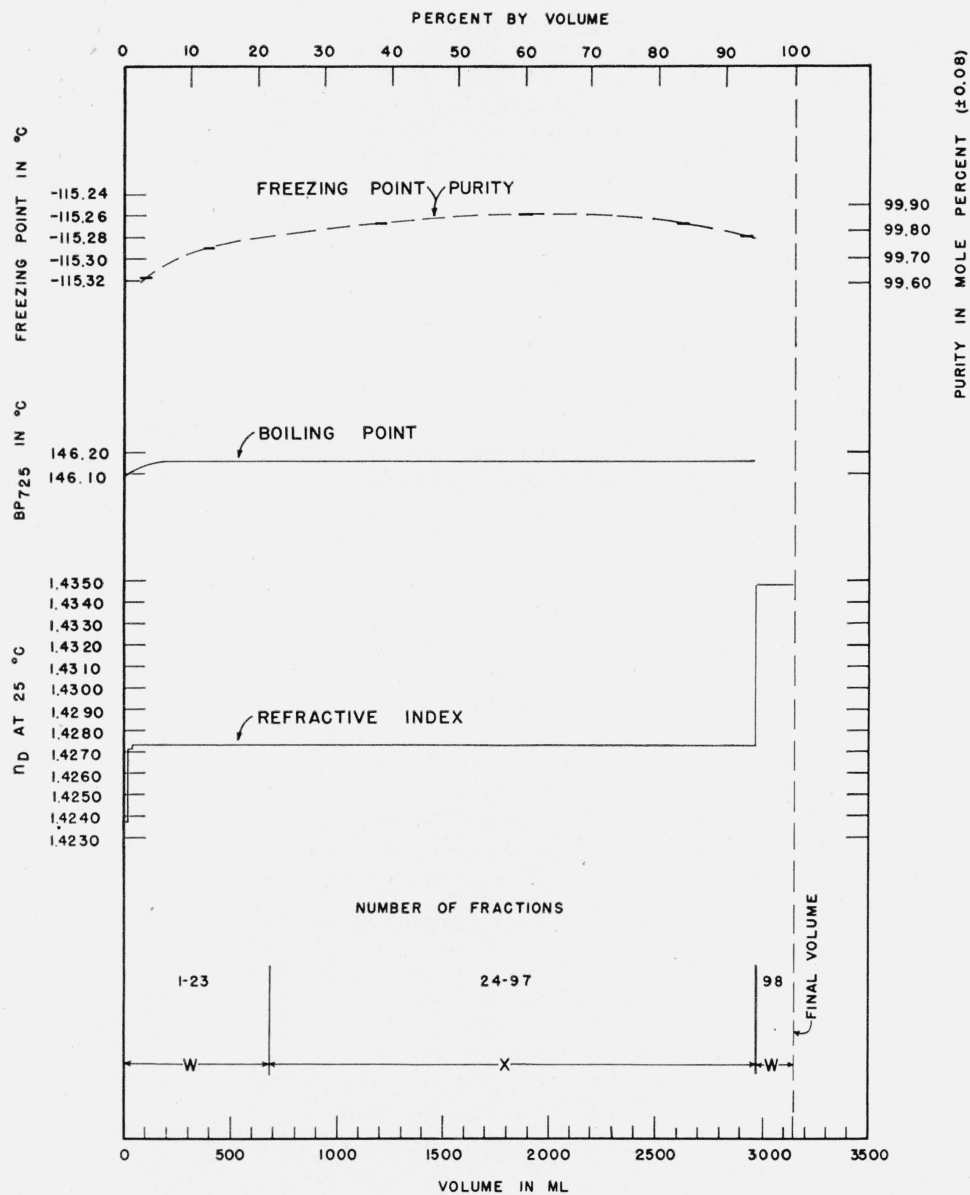


FIGURE 6.—Results of the first distillation of isobutylcyclopentane.

Regular distillation at 725 mm Hg in still 13 (12/7/45 to 12/28/45).

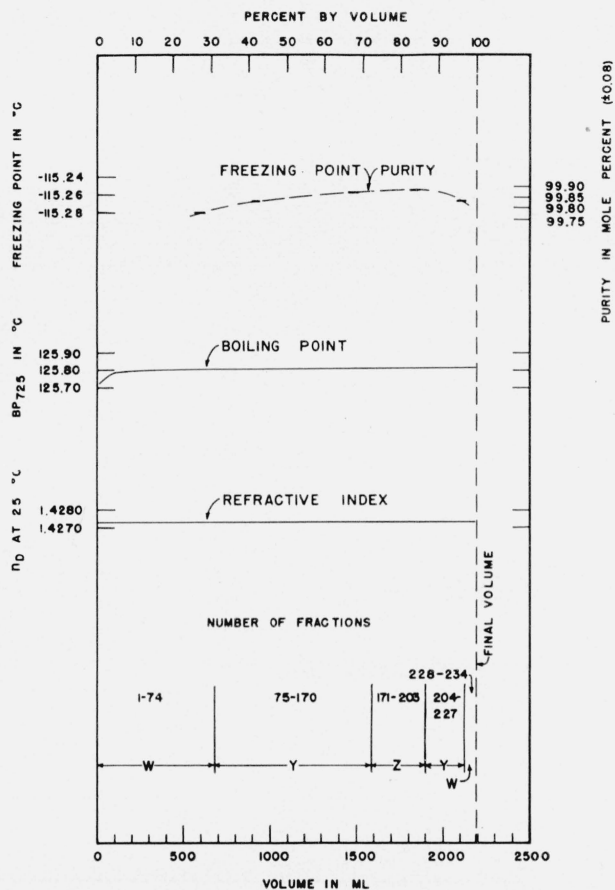


FIGURE 7.—Results of the second and final distillation of isobutylcyclopentane.

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in still 10 (9/3/46 to 10/14/46).

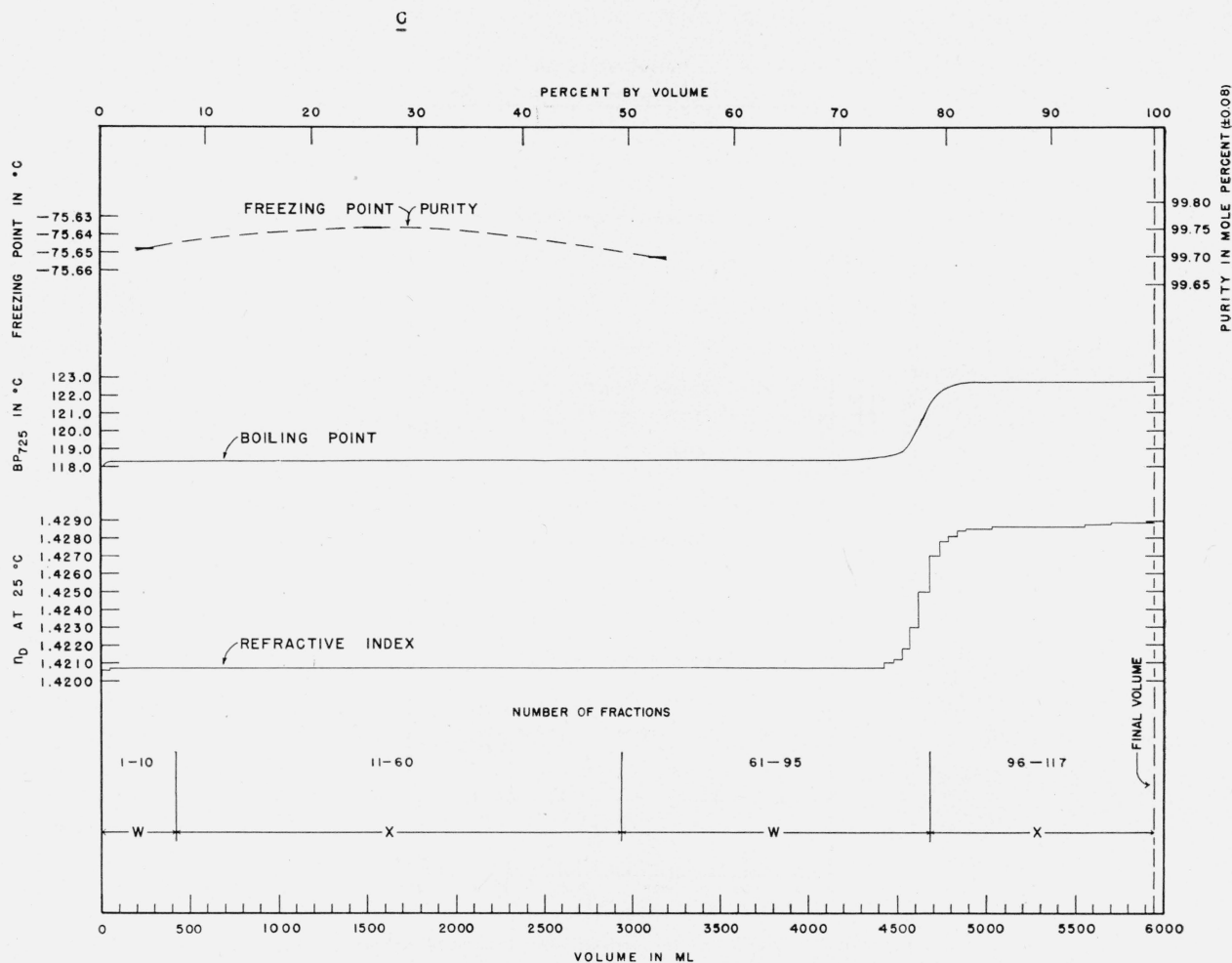


FIGURE 8.—Results of the first distillation of *cis* and *trans*-1,3-dimethylcyclohexane.

Regular distillation at 725 mm Hg in still 15 (3/27/46 to 4/17/46). One of three distillations of similar material. See footnote "l" of table 1. Fractions 11 to 60 were redistilled to obtain *cis*-1,3-dimethylcyclohexane (see fig. 9 and footnote "f" of table 2). Fractions 96 to 117 were redistilled to obtain *trans*-1,3-dimethylcyclohexane (see fig. 11 and footnote "g" of table 2).

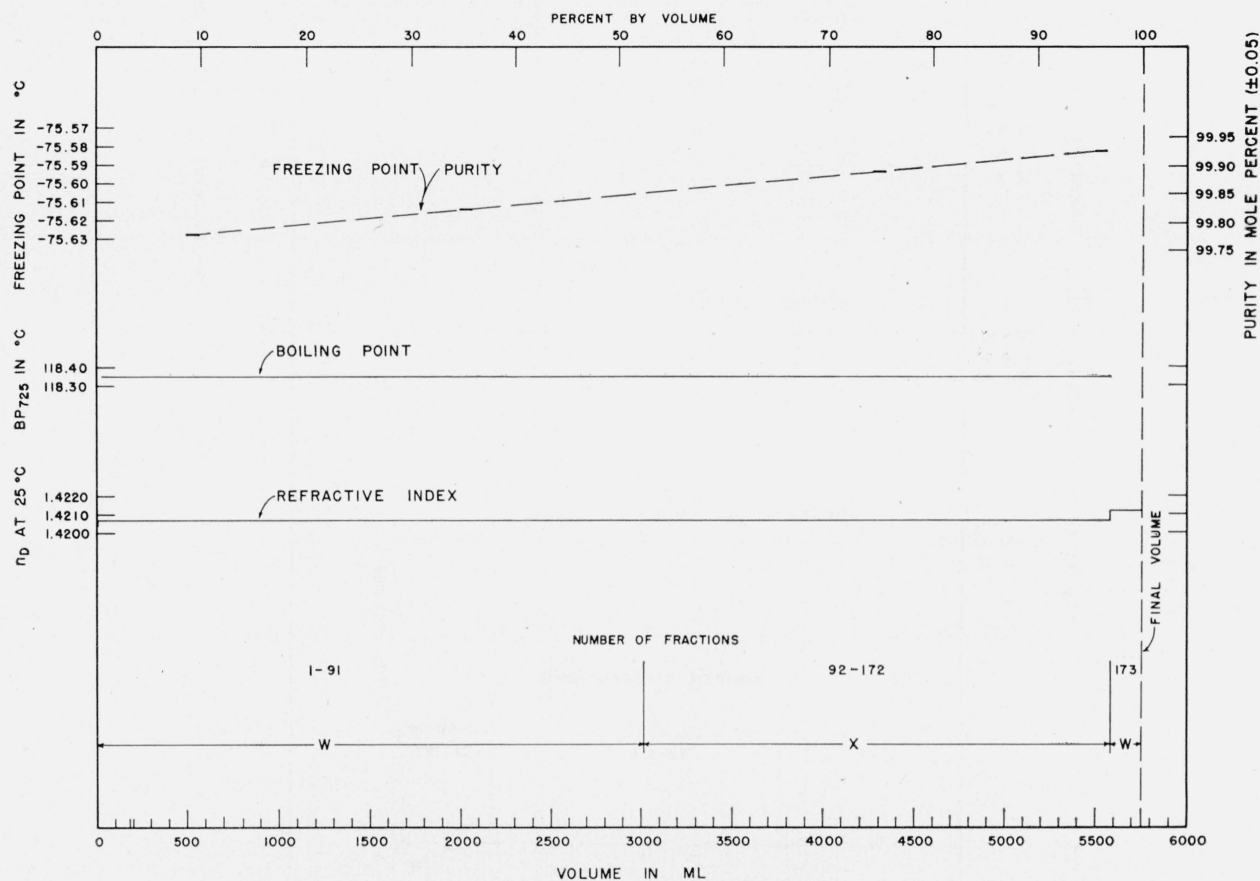


FIGURE 9.—Results of the second distillation of *cis*-1,3-dimethylcyclohexane (see footnote "f" of table 2).

Regular distillation at 725 mm Hg in still 7 (5/3/46 to 6/3/46).

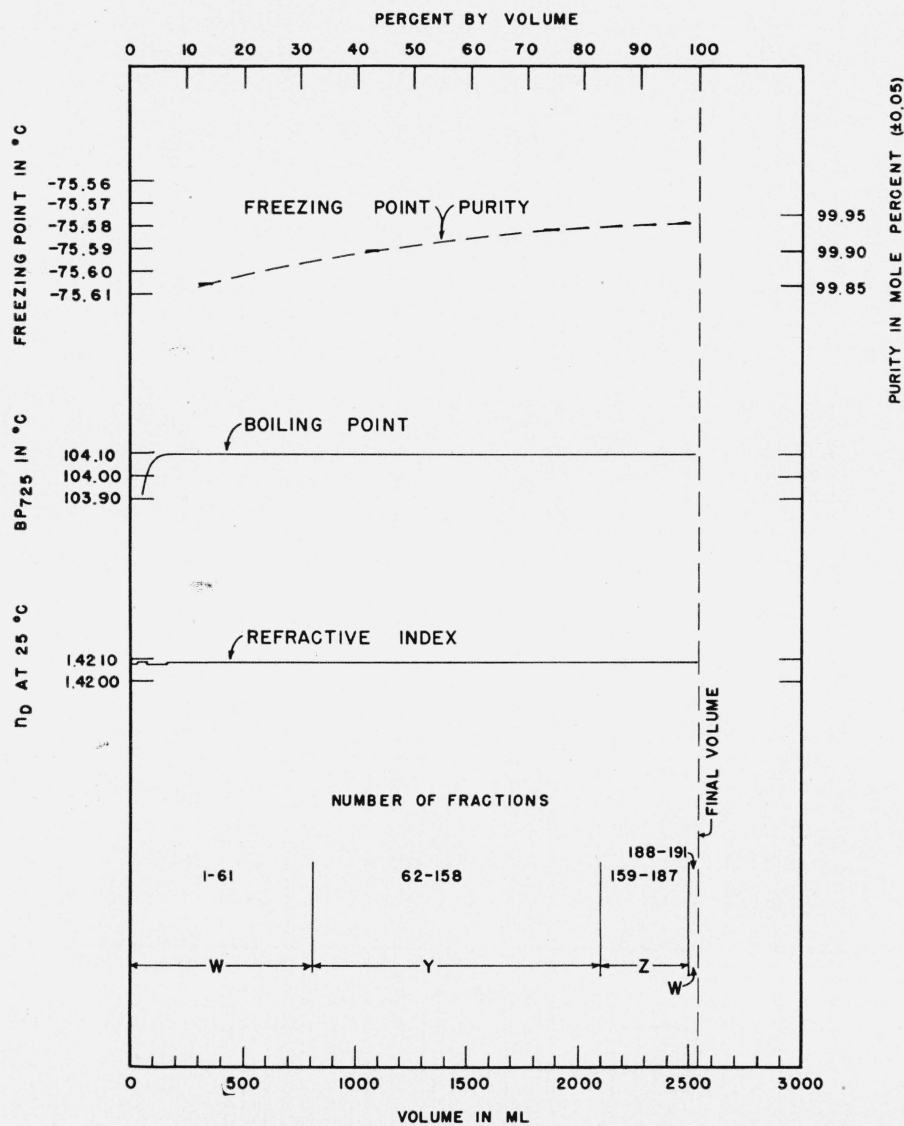


FIGURE 10.—Results of the third and final distillation of *cis*-1,3-dimethylcyclohexane (see footnote "f" of table 2).

Azeotropic distillation with ethylene glycol monomethyl ether at 725 mm Hg in still 15A (6/29/46 to 8/5/46).

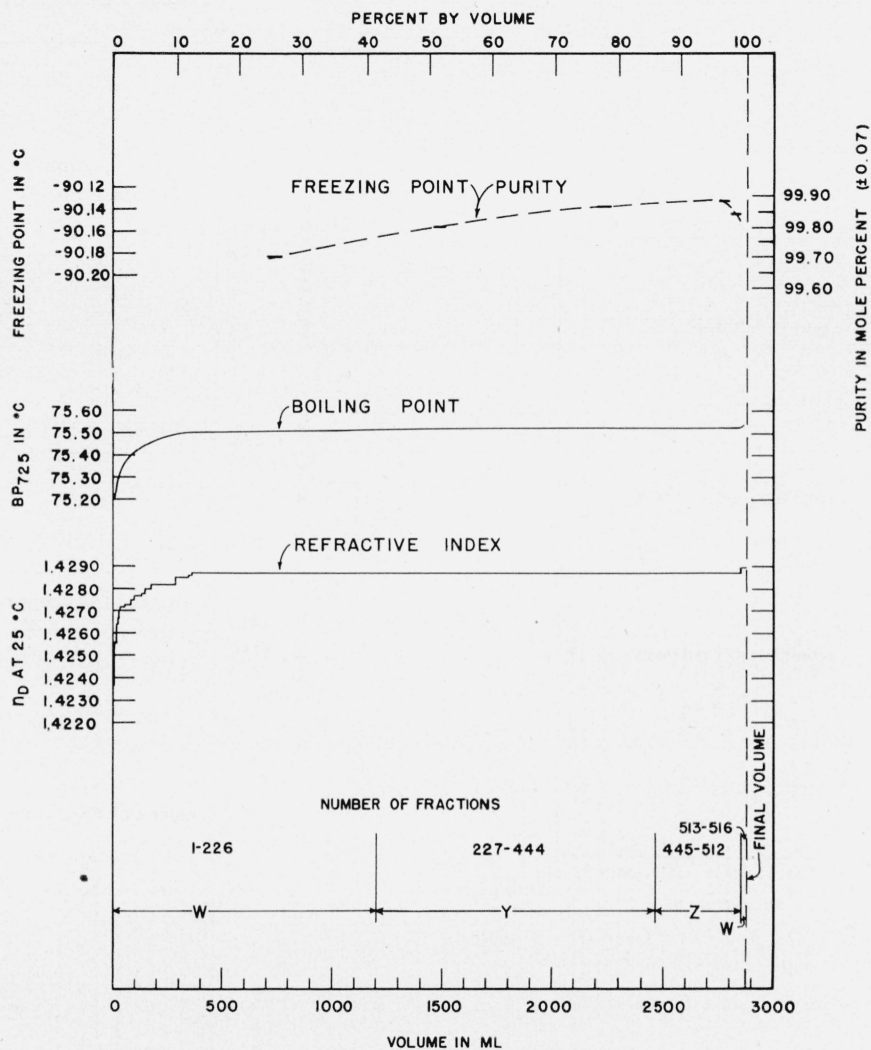


FIGURE 11.—Results of the second and final distillation of *trans*-1,3-dimethylcyclohexane (see footnote "g" of table 2).

Azeotropic distillation with ethanol at 725 mm Hg in still 9 (5/4/46 to 8/5/46).

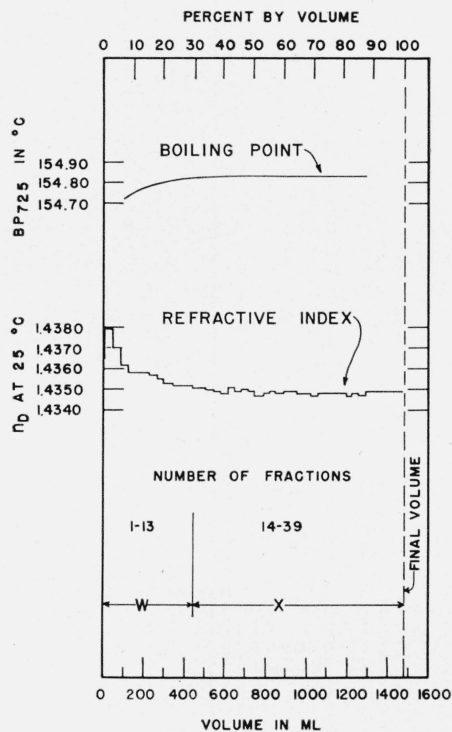


FIGURE 12.—Results of the first distillation of *n*-propylcyclohexane.

Regular distillation at 725 mm Hg in still 8 (5/5/44 to 5/11/44).

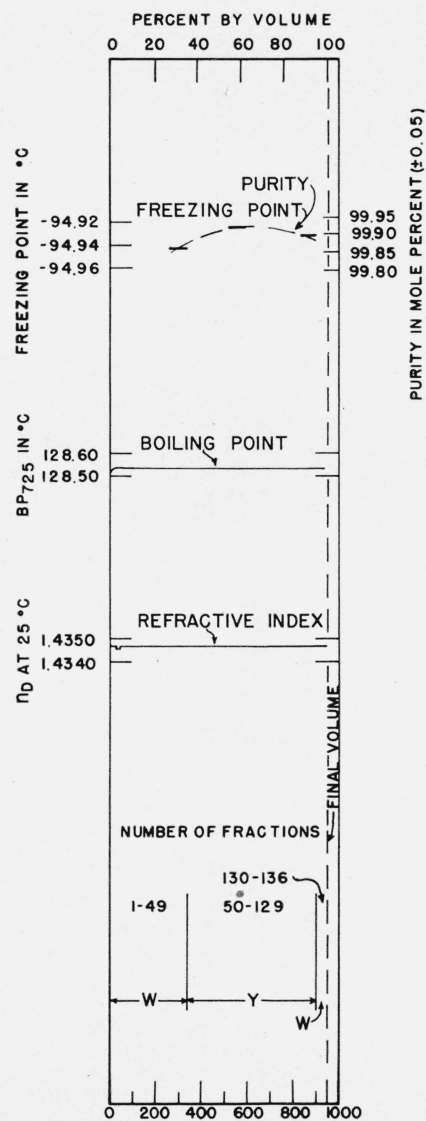


FIGURE 13.—Results of the second distillation of *n*-propylcyclohexane.

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in still 9 (6/14/44 to 7/10/44).

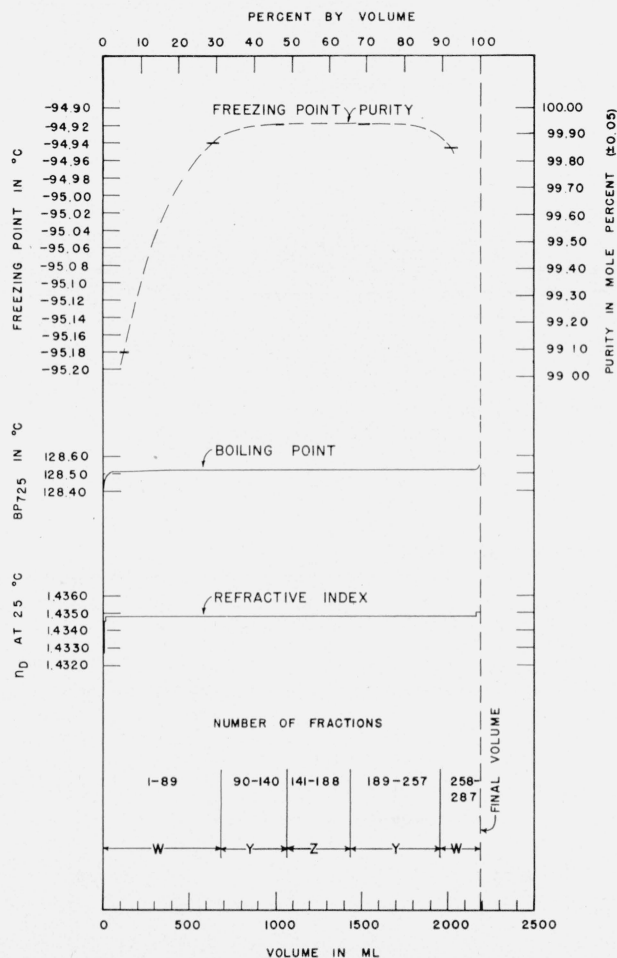


FIGURE 14.—Results of the third distillation of *n*-propylcyclohexane.

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in still 9 (3/13/46 to 5/3/46). See footnote "c" of table 2.

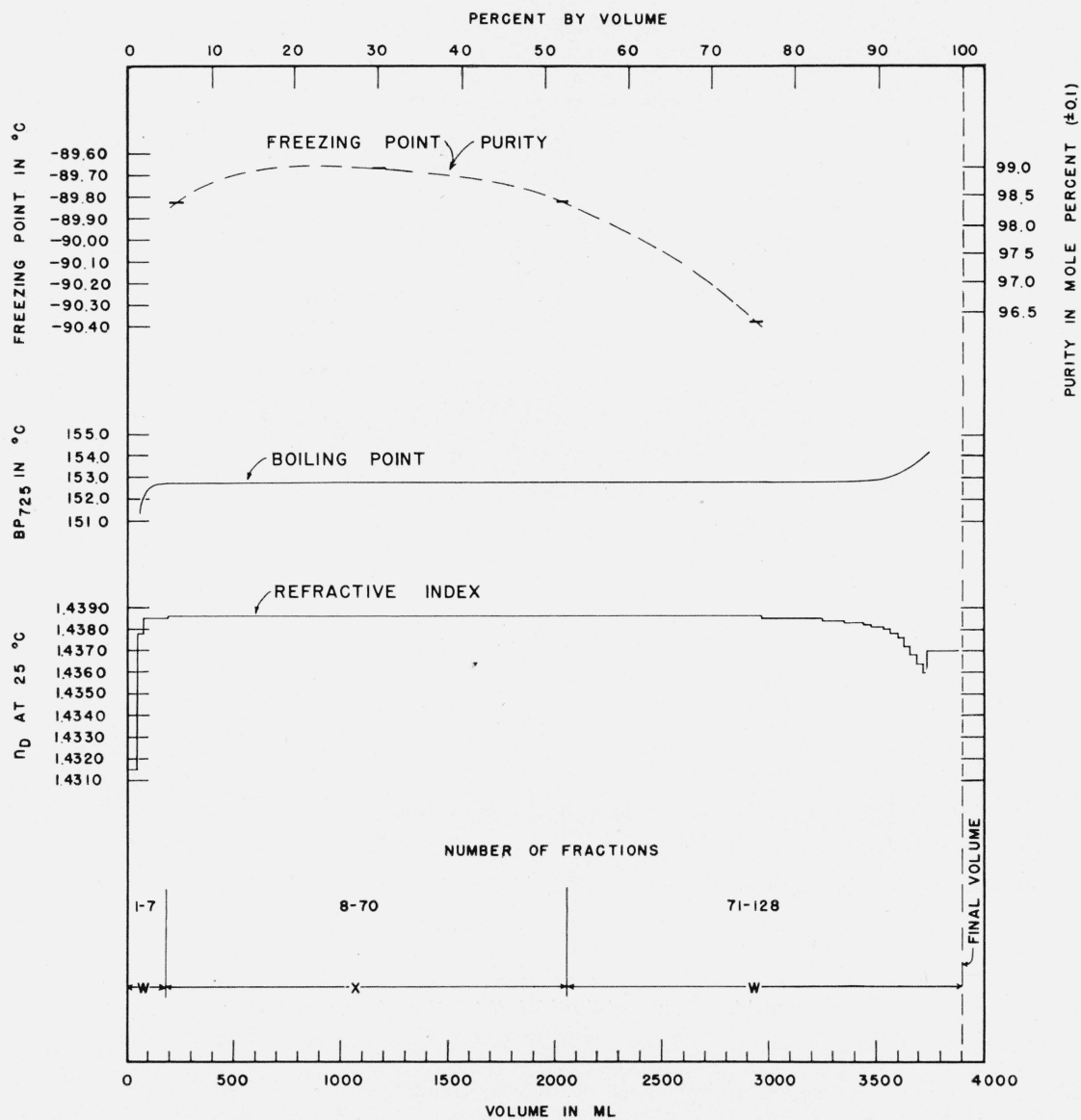


FIGURE 15.—Results of the first distillation of isopropylcyclohexane.

Regular distillation at 725 mm Hg in still 8 (10/18/45 to 11/12/45).

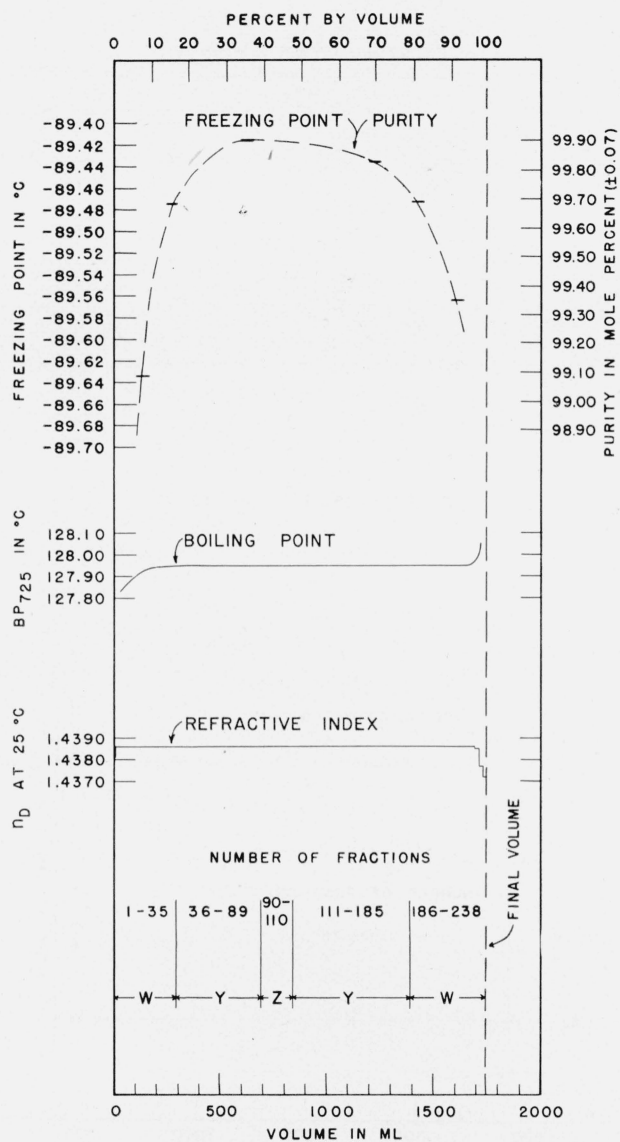


FIGURE 16.—Results of the second and final distillation of isopropylcyclohexane.

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in still 10 (3/2/46 to 4/12/46).

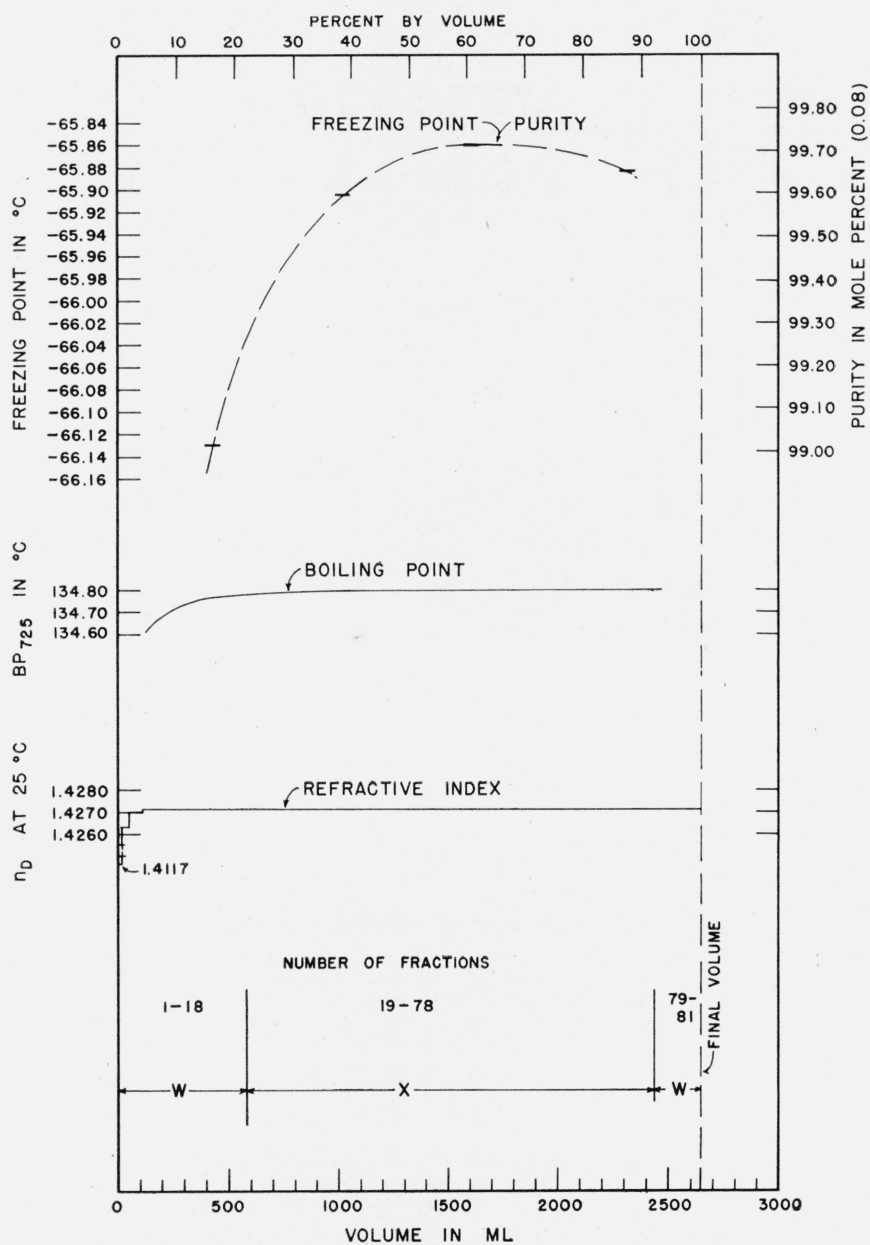


FIGURE 17.—Results of the first distillation of 1,1,3-trimethylcyclohexane.

Regular distillation at 725 mm Hg in still 7 (10/25/45 to 11/9/45).

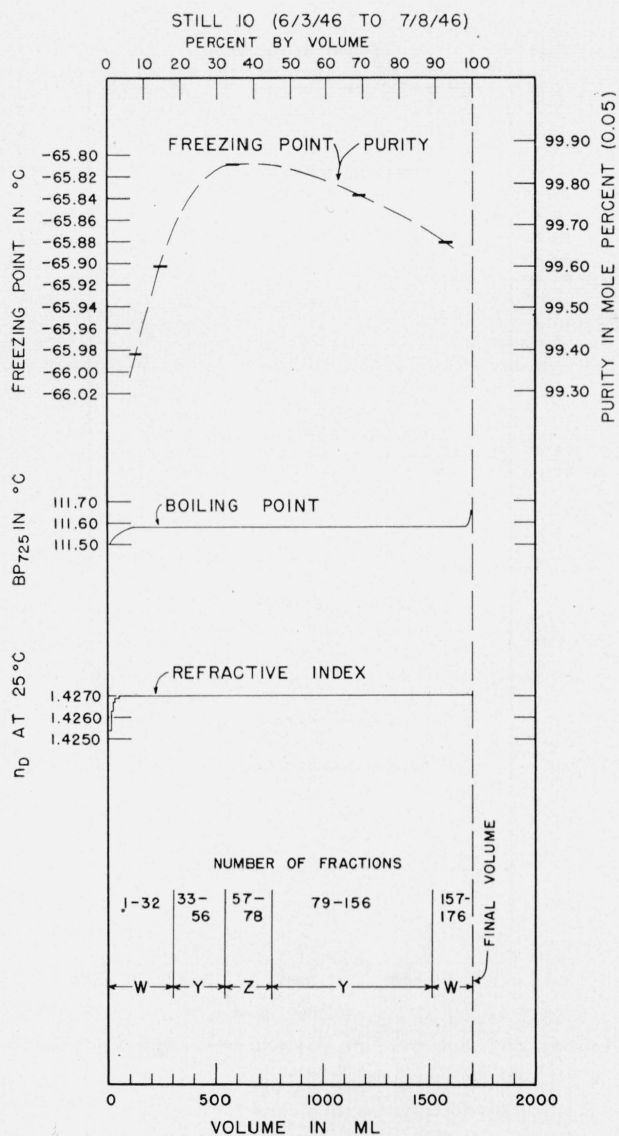


FIGURE 18.—Results of the second and final distillation of 1,1,3-trimethylcyclohexane.

Azeotropic distillation with ethylene glycol monomethyl ether at 725 mm Hg in still 10 (6/3/46 to 7/8/46).

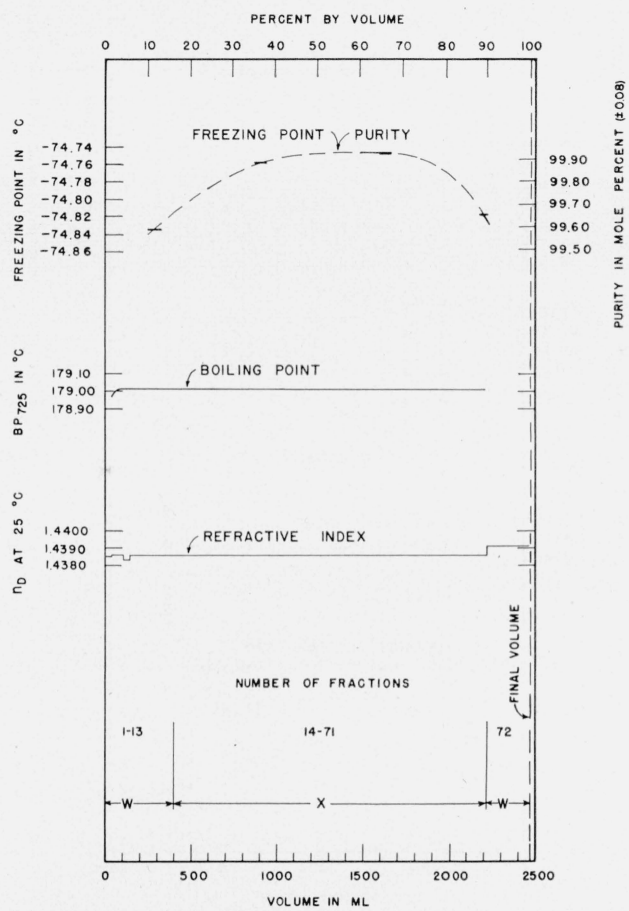


FIGURE 19.—Results of the first distillation of *n*-butylcyclohexane.

Regular distillation at 725 mm Hg in still 13 (5/15/45 to 5/31/45).

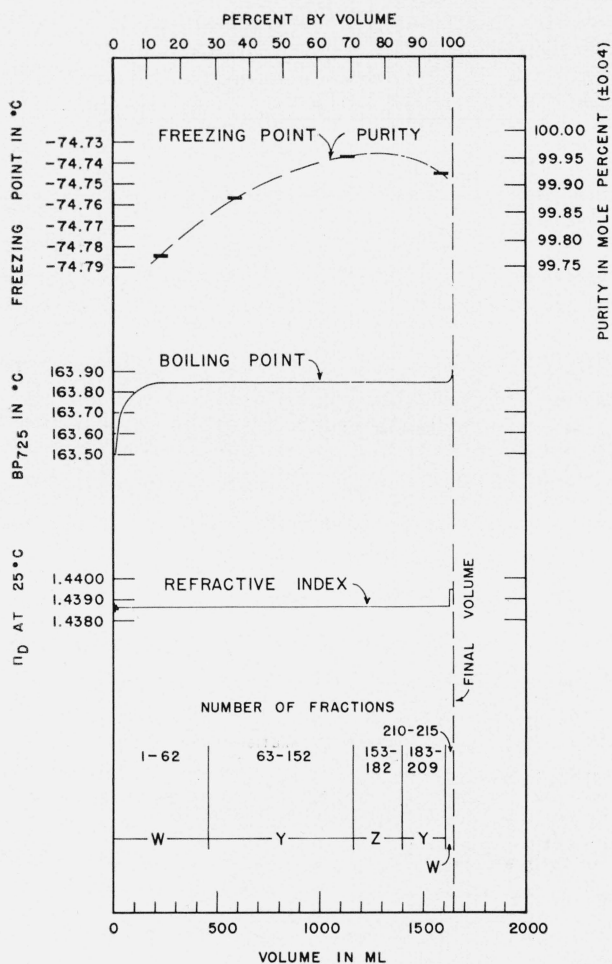


FIGURE 20.—Results of the second and final distillation of *n*-butylcyclohexane.

Azeotropic distillation with ethylene glycol monobutyl ether at 725 mm Hg in still 10 (7/23/46 to 8/31/46).

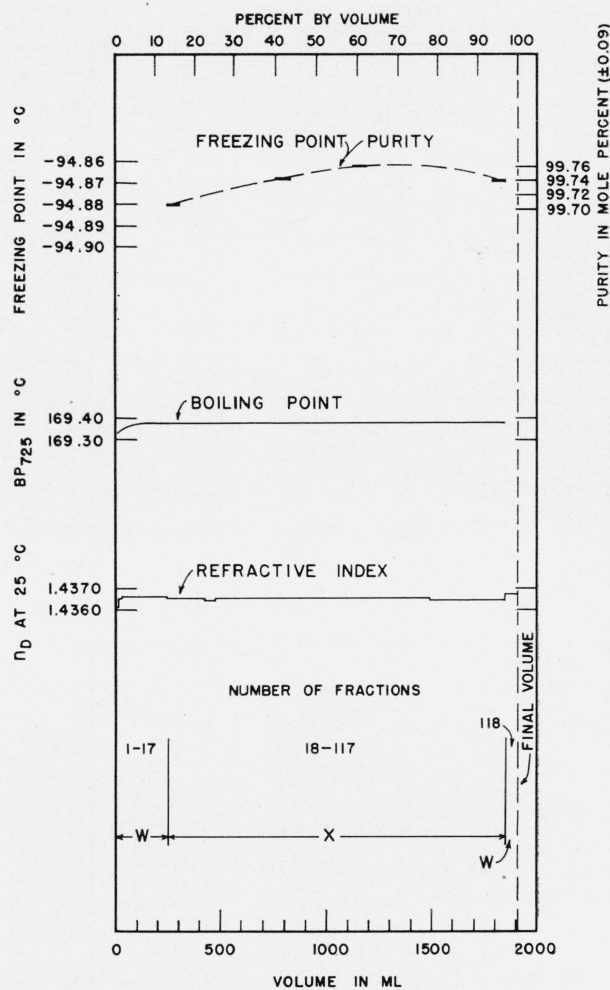


FIGURE 21.—Results of the first distillation of isobutylcyclohexane.

Regular distillation at 725 mm Hg in still 2 (12/7/45 to 1/21/46).

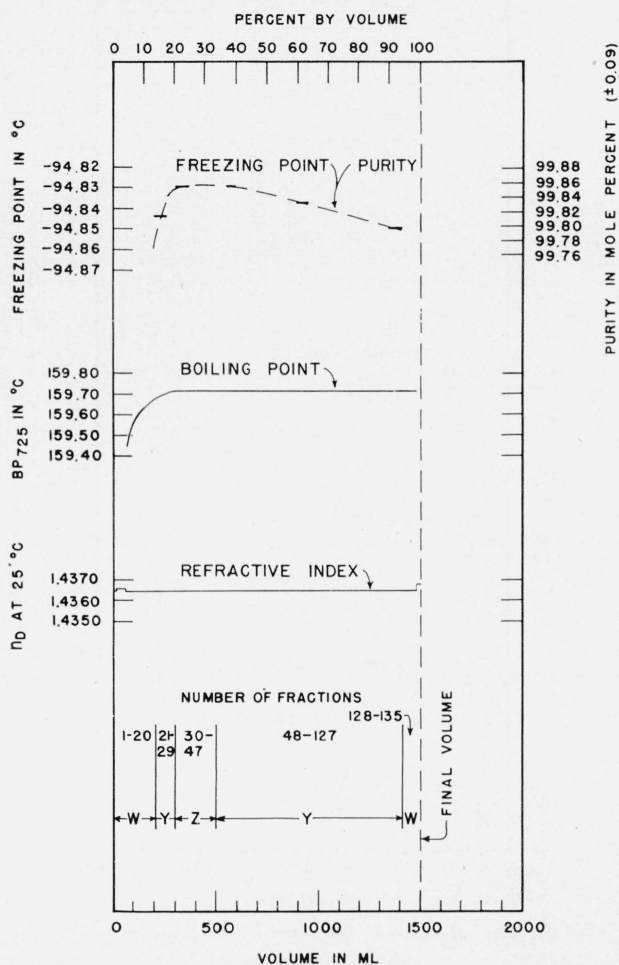


FIGURE 22.—Results of the second and final distillation of isobutylcyclohexane.

Azeotropic distillation with ethylene glycol monobutyl ether at 725 mm Hg in still 11A (7/25/46 to 8/20/46).

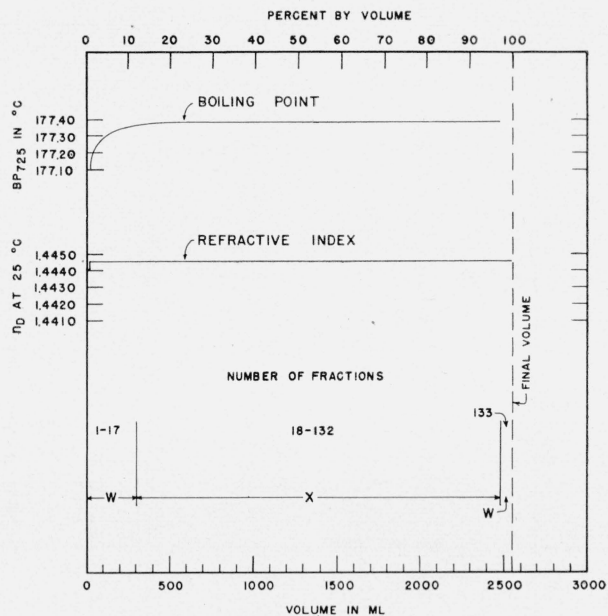


FIGURE 23.—Results of the first distillation of sec-butylcyclohexane.

Regular distillation at 725 mm Hg in still 12 (6/3/46 to 6/27/46).

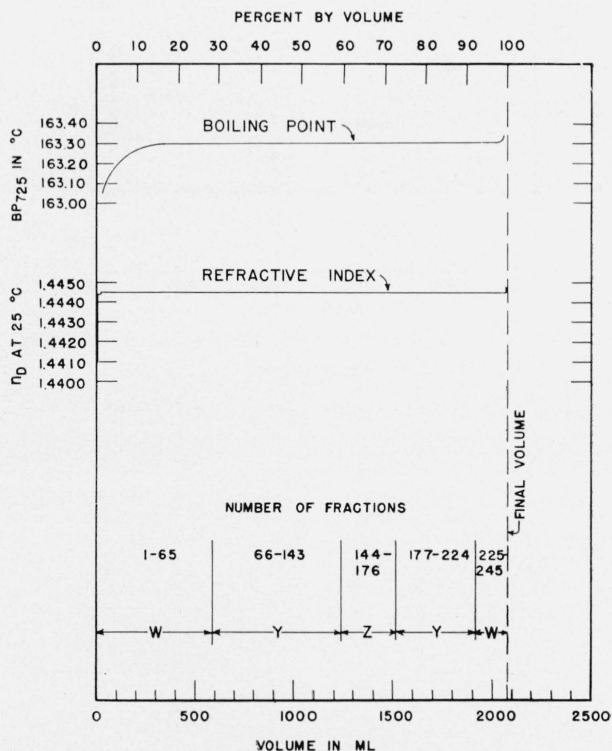


FIGURE 24.—Results of the second and final distillation of sec-butylcyclohexane.

Azeotropic distillation with ethylene glycol monobutyl ether at 725 mm Hg in still 11A (8/21/46 to 10/4/46).

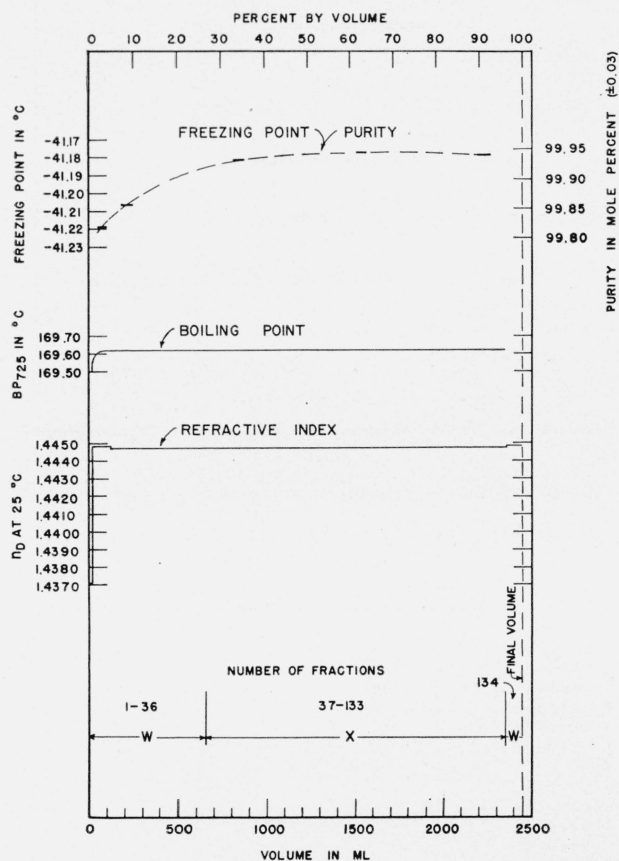


FIGURE 25.—Results of the first distillation of *tert*-butylcyclohexane.

Regular distillation at 725 mm Hg in still 12 (1/2/46 to 1/29/46).

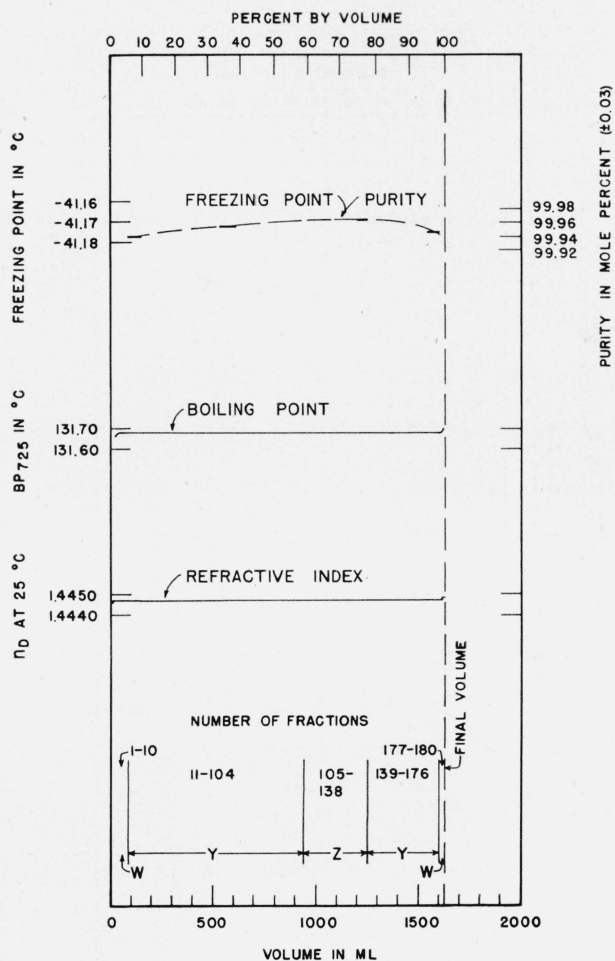


FIGURE 26.—Results of the second and final distillation of *tert*-butylcyclohexane.

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in still 8 (3/15/46 to 4/18/46).

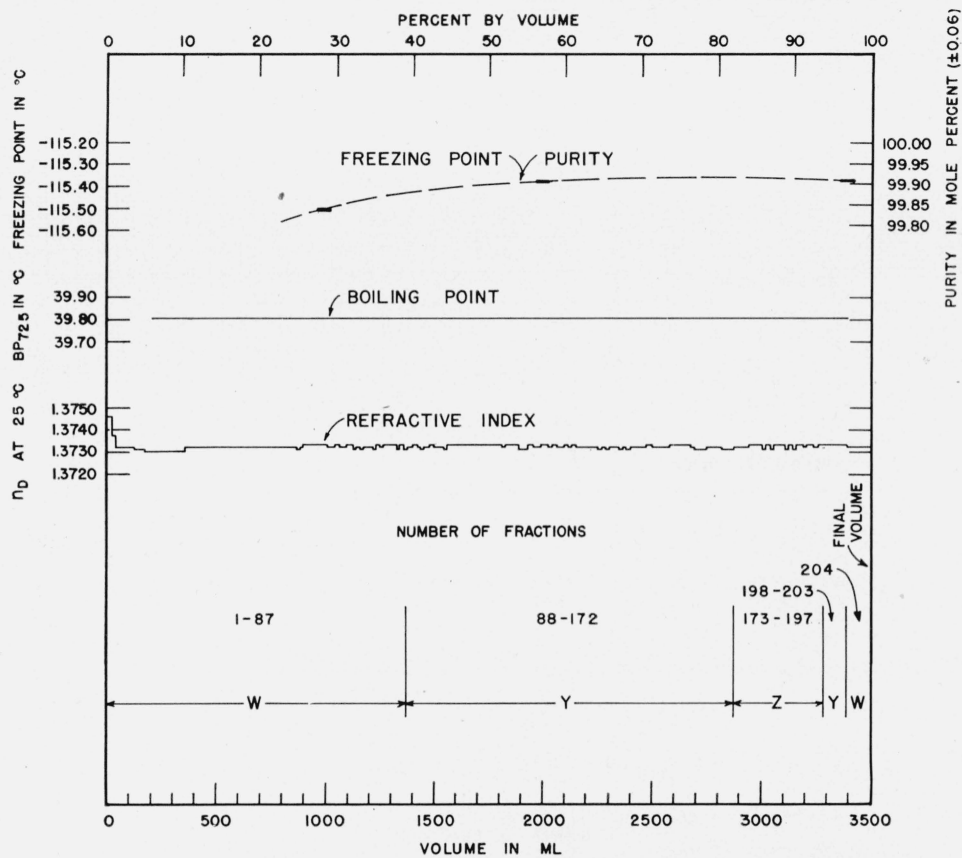


FIGURE 27.—Results of the first and only distillation of 3,3-dimethyl-1-butene.

Regular distillation at 725 mm Hg in still 9 (12/27/44 to 2/15/45).

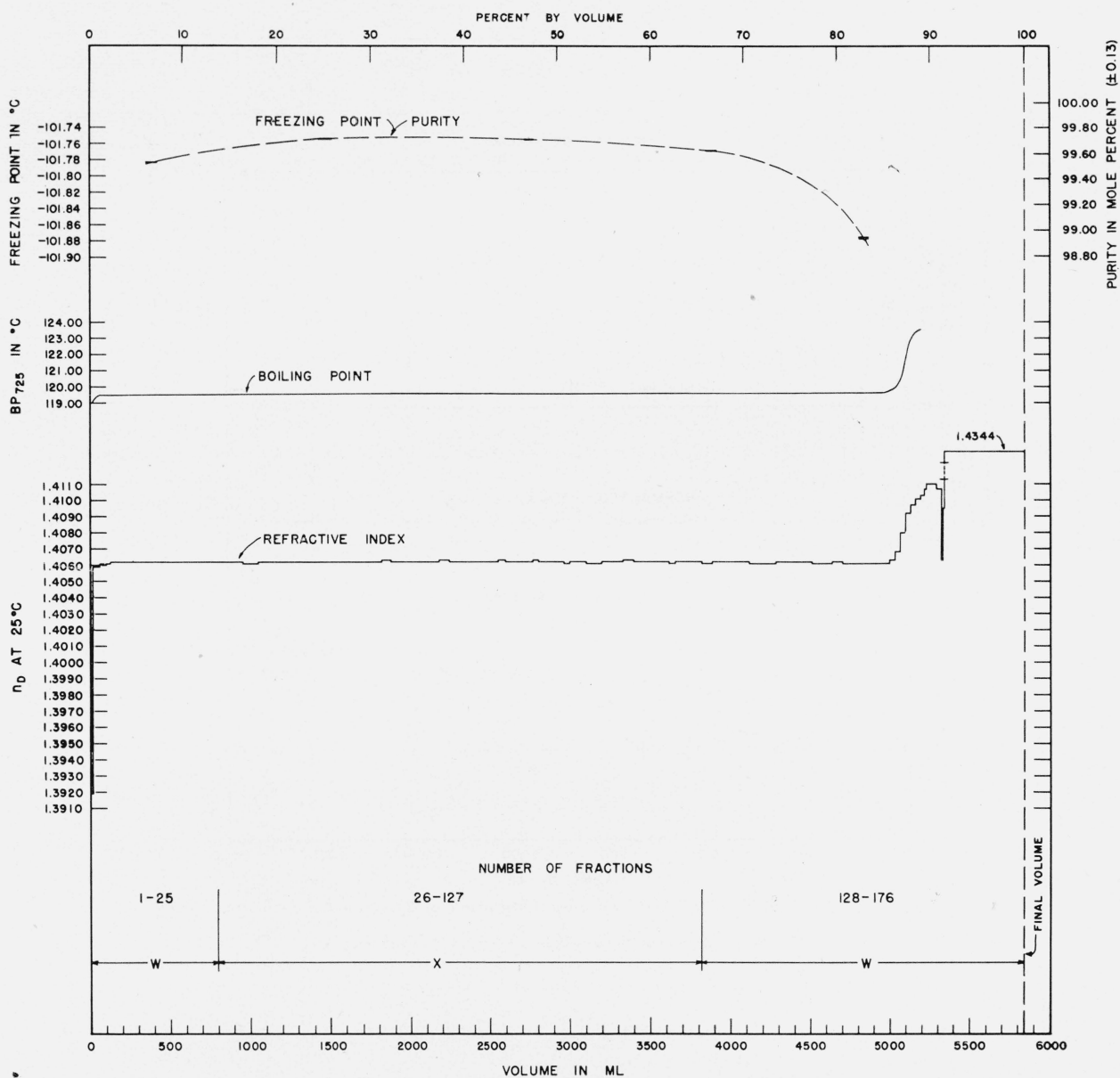


FIGURE 28.—Results of the first distillation of 1-octene.

Regular distillation at 725 mm Hg in still 13 (1/3/46 to 2/4/46). This is one of two distillations of similar material.

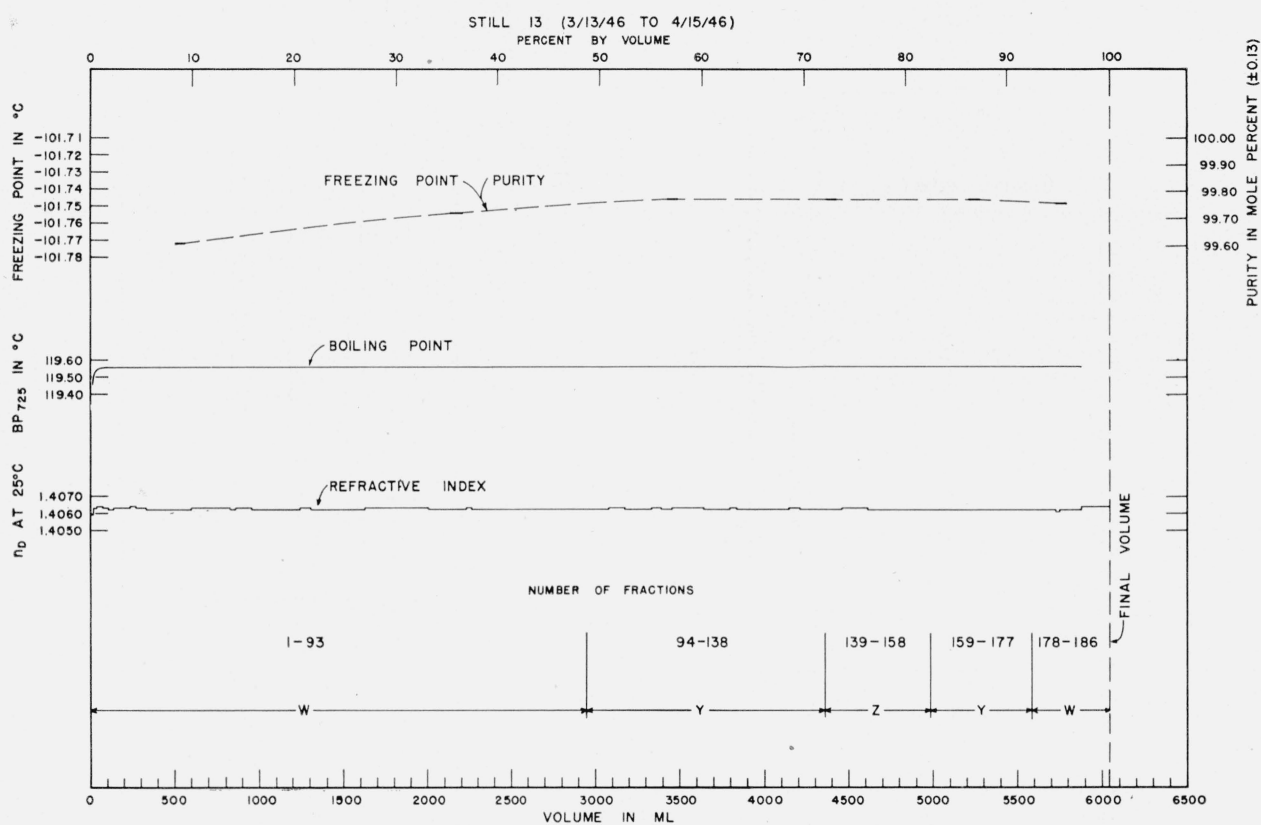


FIGURE 29.—Results of the second and final distillation of 1-octene.

Regular distillation at 725 mm Hg in still 13 (3/13/46 to 4/15/46).

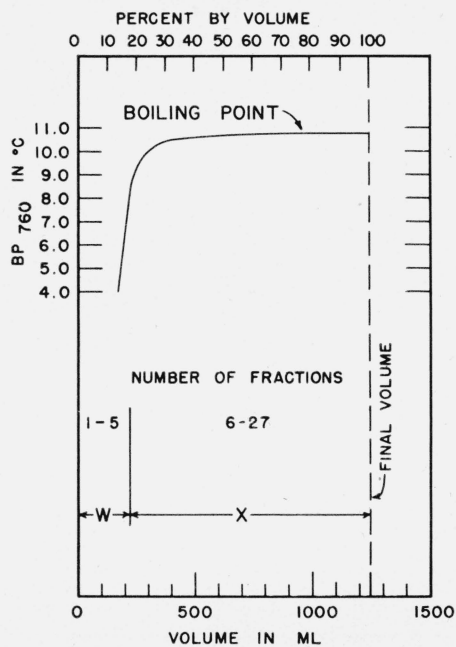


FIGURE 30.—Results of the first distillation of 1,2-butadiene.

Regular distillation at atmospheric pressure in still 1 (6/21/45 to 7/24/45).

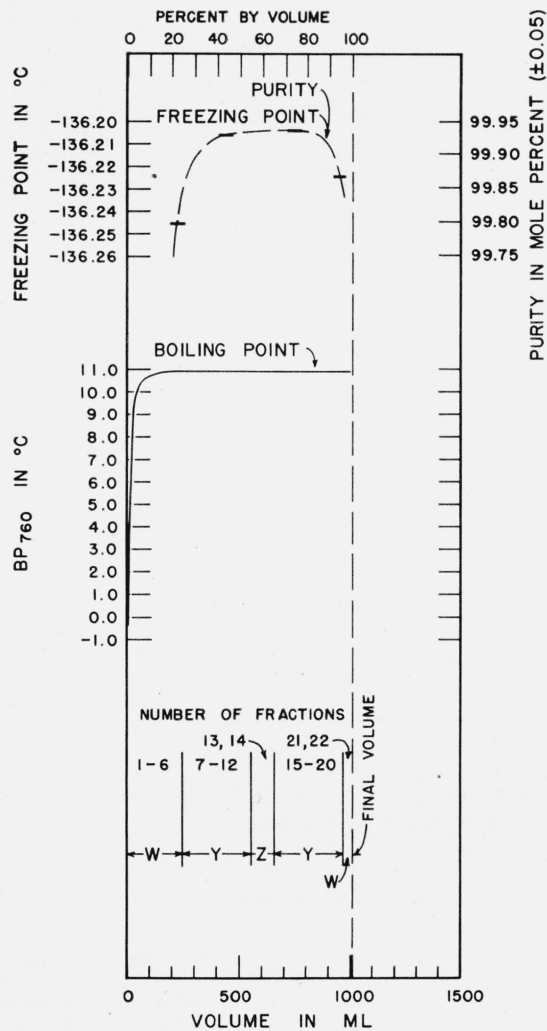


FIGURE 31.—Results of the second and final distillation of 1,2-butadiene.

Regular distillation at atmospheric pressure in still 1 (2/28/46 to 3/27/46).

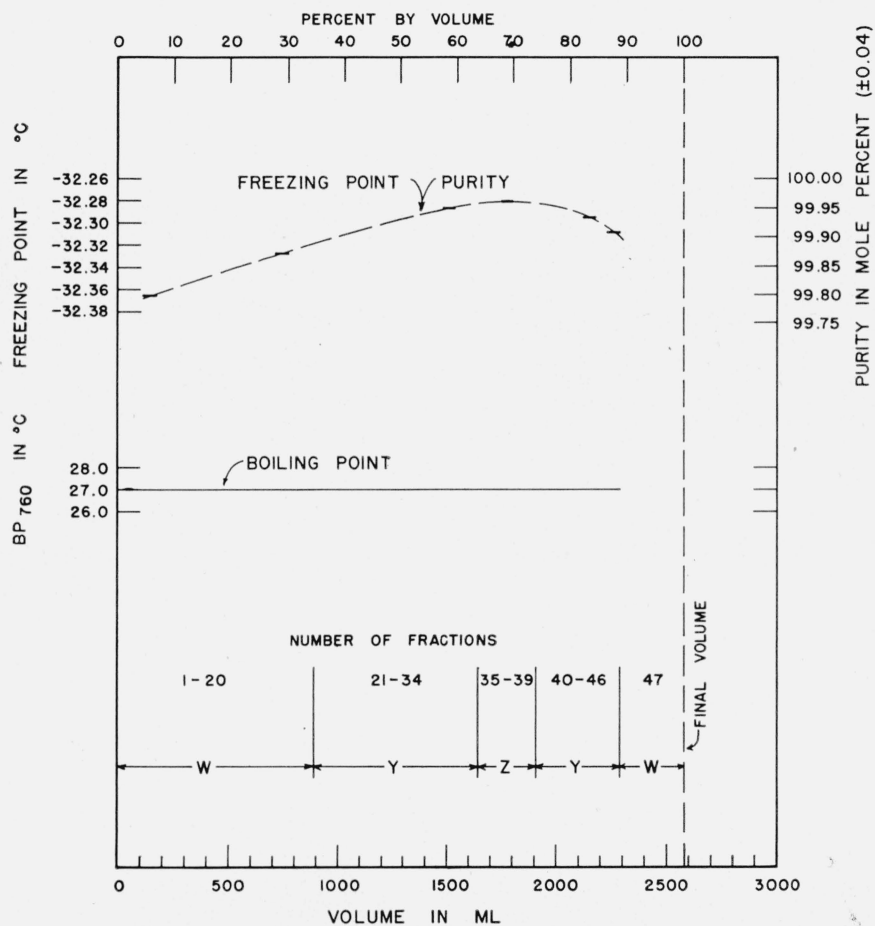


FIGURE 32.—Results of the first and only distillation of 2-butyne.

Regular distillation at atmospheric pressure in still 1 (11/8/45 to 1/7/46).

WASHINGTON, June 11, 1947.