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Purification, Purity, and Freezing Points of 8 Nonanes, 11 Alkylcyclopentanes, 6 Alkylcyclohexanes, and 4 Butylbenzenes of the API-Standard and API-NBS Series*[1]

By Anton J. Streiff,[2] Evelyn T. Murphy,[2] Janice C. Cahill,[2] Helen F. Flanagan,[2] Vincent A. Sedlak,[2] Charles B. Willingham, and Frederick D. Rossini

This report describes the purification and determination of freezing points and purity of 29 hydrocarbons of the API-Standard and API–NBS series, including 8 nonanes, 11 alkylcyclopentanes, 6 alkylcyclohexanes, and 4 butylbenzenes.

I. Introduction

A previous report described the purification, and determination of freezing points and purity of 37 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. This report describes the purification and determination of freezing points and purity of an additional 29 hydrocarbon compounds under this cooperative program, including 8 nonanes, 6 alkylcyclohexanes, 11 alkylcyclopentanes, and 4 butylbenzenes.[3]

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 [4].** The material labeled API–NBS is made available in appropriate small lots on loan to qualified investigators for the measurement of needed properties.[4]

II. Materials

The starting materials were supplied as follows:[5]

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:

3,3-Diethylpentane.
2,2,3,3-Tetramethylpentane.
Ethylcyclohexane (*B*).
1,1-Dimethylcyclohexane.
cis-1,2-Dimethylcyclohexane.
trans-1,2-Dimethylcyclohexane.
Ethylcyclopentane (one-half) (*B*).
trans-1,3-Dimethylcyclopentane.
n-Propylcyclopentane.
Isopropylcyclopentane.
1,1,3-Trimethylcyclopentane (three-fourths).

*Presented before the Division of Petroleum Chemistry of the American Chemical Society, Chicago, Ill., September 1946.

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

[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] The cooperative program was carried on under the API Research Project 46 Committee on Hydrocarbons for Spectrometer Calibration (W. J. Sweeney, chairman). Further details are given in references [1, 2, 3].

[4] The allocation of the API–NBS samples of hydrocarbons is handled by the Advisory Committee for the API Research Project 44 on the "Collection, Analysis, and Calculation of Data on the Properties of Hydrocarbons" (W. E. Kuhn, chairman).

[5] (*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 [5]).

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

2,4,4-Trimethylhexane.
Ethylcyclopentane (one-half) (*B*).
1,1-Dimethylcyclopentane.
cis-1,2-Dimethylcyclopentane.
trans-1,2-Dimethylcyclopentane.
1,1,2-Trimethylcyclopentane.
cis, cis, trans-1,2,4-Trimethylcyclopentane.
cis, trans, cis-1,2,4-Trimethylcyclopentane.

By the National Advisory Committee for Aeronautics, through its Aeronautical Engine Research Laboratory at Cleveland, Ohio, and the Automotive Section at the National Bureau of Standards:

n-Butylbenzene (*B*).
Isobutylbenzene (*B*).
sec-Butylbenzene (*B*).
tert-Butylbenzene (*B*).
2,2,3,4-Tetramethylpentane.
2,2,4,4-Tetramethylpentane.
2,3,3,4-Tetramethylpentane.

By the Standard Oil Development Co., Elizabeth, N. J., through W. J. Sweeney:

cis-1,4-Dimethylcyclohexane. *trans*-1,4-Dimethylcyclohexane.

By the Gulf Oil Co. Fellowship at the Mellon Institute of Industrial Research, Pittsburgh, Pa., through W. A. Gruse:

1,1,3-Trimethylcyclopentane (one-fourth).

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

n-Nonane. 2,2,5-Trimethylhexane.

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

TABLE 1.—*Information on the purification of 29 API-Standard and API-NBS hydrocarbons*

Compound [a]	Starting material [b] provided by—	Hydrocarbon charged for distillation		Distillation [f]								Volume of selected sample	
		Volume	Purity	Kind [c]	Azeotrope-forming substance [d]	Amount of hydrocarbon in the azeotropic 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/hr*		*ml*	*ml*
n-Nonane	APIRP6 [g]	5.20	99.05	Reg			14	125	130/1	12.0	1		
		2.75	99.75	Azeo	Cell	49	13	130	145/1	8.5	2	1350	305
2,2,5-Trimethylhexane	APIRP6 [h]	2.40	99.50	Reg			3	100	120/1	2.5	3	1550	250
2,4,4-Trimethylhexane	Penn State	2.62	99.26	Azeo	Cell	70	13	130	145/1	8.5	4		
		2.08	99.54	Reg			12	135	185/1	4.0	5	1065	248
2,2,3,3-Tetramethylpentane	APIRP45	2.00		Azeo	Cell	61	10	135	165/1	4.5	6		
		1.38	99.90	Reg			11A	200	180/1	4.0	7	818	150
2,2,3,4-Tetramethylpentane	NACA	11.89	([i])	do			6	125	125/1	12.5	8		
		2.85		Azeo	Me. Cell	58	10	135	165/1	4.5	9	998	295
2,2,4,4-Tetramethylpentane	do	1.32	98.62	do	Cell	76	4	200	145/1	5.0	10	855	165
2,3,3,4-Tetramethylpentane	do	4.83	94.61	Reg			8	130	155/1	8.0	11		
		2.35	99.86	Azeo	Cell	58	8	130	155/1	8.0	12	1100	385
3,3-Diethylpentane	APIRP45	3.05	[j] 99.31	Reg			13	130	155/1	8.0	13		
		1.75	99.89	Azeo	Cell	55	13	130	145/1	8.5	14	895	152
ALKYLBENZENES													
n-Butylbenzene (*B*)	NACA	3.75		Reg			9	135	165/1	4.5	15	1415	375
Isobutylbenzene (*B*)	do	3.69	99.75	do			8	130	155/1	8.0	16	1190	337
sec-Butylbenzene (*B*)	do	2.47	99.09	Azeo	Me Carb	83	8	130	145/1	8.5	17		
		1.53	99.73	do	do	83	13	130	150/1	8.25	18		
		1.32	99.73	Reg			12	135	165/1	4.5	19	1000	132
tert-Butylbenzene (*B*)	do	3.23	99.87	Azeo	Me Carb	86	8	130	145/1	8.5	20	1225	365

Footnotes on page 56.

TABLE 1.—*Information on the purification of 29 API-Standard and API-NBS hydrocarbons*—Continued

Compound [a]	Starting material [b] provided by—	Hydrocarbon charged for distillation: Volume	Hydrocarbon charged for distillation: Purity	Distillation [f]: Kind [c]	Distillation [f]: Azeotrope-forming substance [d]	Distillation [f]: Amount of hydrocarbon in the azeotropic distillate [e]	Distillation [f]: Distilling column number [f]	Distillation [f]: Number of theoretical plates [f] (approx.)	Distillation [f]: Reflux ratio [f] (approx.)	Distillation [f]: Rate of collection of distillate	Distillation [f]: Results plotted in figure	Volume of selected sample: API-Standard	Volume of selected sample: API-NBS
ALKYLCYCLOPENTANES													
		Liters	*Mole %*			*% by volume*				*ml/hr*		*ml*	*ml*
Ethylcyclopentane (B)	APIRP45	3.47	99.47	Reg.			15	125	110/1	14.0	21		
	Penn State	2.90	96.29										
		2.80	99.86	Azeo	Ethanol	52	13	130	155/1	8.0	22	1400	375
1,1-Dimethylcyclopentane	Penn State	1.82	99.958	do	do	64	9	135	165/1	4.5	23	1126	260
cis-1,2-Dimethylcyclopentane.	do	1.36	99.90	do	do	53	9	135	165/1	4.5	24	780	120
trans-1,2-Dimethylcyclopentane.	do	1.46	99.68	do	do	61	4	200	160/1	4.5	25	906	138
trans-1,3-Dimethylcyclopentane.	APIRP45	3.00	73.6	Reg.			4	200	145/1	5.0	26		
		1.83	90.0	Azeo	Methanol	55	10	135	165/1	4.5	27		
		1.13	94.6	do	Ethanol	63	4	200	180/1	4.0	28	570	102
n-Propylcyclopentane	do	1.30		Reg.			4	200	145/1	5.0	29	[k] 333	[l] 75
		0.30	95.1										
	do	[m] 2.70	97.06	do			13	130	155/1	8.0	30	972	275
	do	[n] 0.44	98.67										
Isopropylcyclopentane	do	5.71		do			10	135	145/1	5.0	31	1180	370
1,1,2-Trimethylcyclopentane.	Penn State	1.32		Azeo	Isoprop	33	9	135	165/1	4.5	32		
		1.07		Reg.			3	100	150/1	2.0	33	680	115
1,1,3-Trimethylcyclopentane.	Gulf-Mellon	0.60		do			3	100	120/1	2.5	34		
	APIRP45	0.50											
		[o] 1.80		do			12	135	185/1	4.0	35		
		1.57		Azeo	Me. Cell	80	12	135	165/1	4.5	36		
		0.80		do	Isoprop	46	7	130	145/1	8.5	37		
		0.51											
		[p] 0.52		do	do	46	4	200	180/1	4.0	38	460	65
cis, cis, trans-1,2,4-Trimethylcyclopentane.	Penn State	3.00		Reg.			4	200	180/1	4.0	39		
		1.86	98.5	Azeo	Isoprop	30	11	([u])	160/1	4.5	40		
		1.28	98.9	do	do	30	4	200	145/1	5.0	41	515	81
cis, trans, cis-1,2,4-Trimethylcyclopentane.	do	1.80	98.5	Reg.			2	100	150/1	2.0	42		
		1.42	99.66	Azeo	Ethanol	48	9	135	165/1	4.5	43	790	125
ALKYLCYCLOHEXANES													
Ethylcyclohexane	APIRP45	5.06		Reg.			8	130	145/1	8.5	44		
		2.37	99.78										
	do	[q] 0.29	99.81	Azeo	Cell	63	15	125	125/1	12.5	45	2360	
	do	[r] 0.27	99.50										
1,1-Dimethylcyclohexane	do	1.50	98.95	Reg.			12	135	185/1	4.0	46		
		1.36		Azeo	Ethanol	64	9	135	165/1	4.5	47	732	131
cis-1,2-Dimethylcyclohexane	do	[s] 9.16		Reg.			5	125	125/1	12.5	48		
		2.84		do			9	135	165/1	4.5	49	1090	243
trans-1,2-Dimethylcyclohexane.	do	[s] 9.16		do			5	125	125/1	12.5	48		
		5.21		do			7	130	155/1	8.0	50		
		2.39	99.84	Azeo	[t] Isoprop Cell	21 73	13	130	145/1	8.5	51	1034	330
cis-1,4-Dimethylcyclohexane	Std. Oil Dev	1.70	99.90	do	Ethanol	30	9	135	165/1	4.5	52	1000	215
trans-1,4-Dimethylcyclohexane.	do	1.90	99.66	do	do	36	10	135	165/1	4.5	53	1160	260

Footnotes on page 56.

[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 [6].

[b] The abbreviations represent the following laboratories: APIRP45; American Petroleum Institute Research Project 45 (formerly the American Petroleum Institute Hydrocarbon Research Project) 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, Aeronautical Engine Research Laboratory, Cleveland, Ohio, and the Automotive Section, National Bureau of Standards Washington, D. C. Std. Oil Dev.; Standard Oil Development Co., Elizabeth, New Jersey. Gulf-Mellon; Gulf Oil Co. Fellowship at the Mellon Institute of Industrial Research, Pittsburgh, Pennsylvania. 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); Isoprop., isopropanol.

[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 [4] for further details.

[g] Obtained by distillation from an Oklahoma petroleum.

[h] Obtained by distillation from commercial "pentenes" alkylates (pentenes + isobutane).

[i] This starting material consisted of a blend of 2,2,3,4-tetramethylpentane (approximately 25%), "S–4" reference fuel ("isooctane") (approximately 66%), and *n*-heptane (approximately 9%).

[j] Calculated from the measured freezing points of two separate lots which were blended together for this charge.

[k] The total volume of the API–Standard sample was 1,305 ml.

[l] The total volume of the API–NBS sample was 350 ml.

[m] This is a second lot of *n*-propylcyclopentane supplied by API Research Project 45.

[n] Fractions 272 to 292 from the distillation of isopropylcyclopentane in column 10 (see figure 31).

[o] This second lot of 1,1,3-trimethylcyclopentane was supplied by the API Research Project 45.

[p] Fractions 41 to 60 from the azeotropic distillation in column 12 (see fig. 36)

[q] This is a second lot of ethylcyclohexane supplied by the API Research Project 45.

[r] This is a third lot of ethylcyclohexane supplied by the API Research Project 45.

[s] Both *cis* and *trans* 1,2-dimethylcyclohexane were obtained from this material (see fig. 48).

[t] The distillation was begun with isopropanol as the azeotrope-forming substance, but because of the relatively small percentage of hydrocarbon in the azeotropic distillate, cellosolve was added to complete the distillation.

[u] The number of theoretical plates for this column was not determined.

III. Purification

The procedure followed in the process of purification and determination of purity was the same as that described in the previous report [1].

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 [4].

Figures 1 to 53, 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 ±0.01° C), the freezing point of selected fractions of hydrocarbon distillate (in air at 1 atm, usually with a precision near ±0.003° C), and the purity of the hydrocarbon distillate. The letters *W*, *X*, *Y*, and *Z* indicate the disposition of the material, as follows: *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.

As demonstrated in the previous report, 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. An example of a case where the purest material is at the very beginning of a distillation is shown in figure 27 on *trans*-1,3-dimethylcyclopentane, and an example of a case where the purest material is at the end of the distillation is shown in figure 17 for *sec*-butylbenzene.

IV. Freezing Points, Cryoscopic Constants, and Purity

Table 2 gives the following information for each of the 29 compounds, except as otherwise indicated: The kind of time-temperature curves, whether freezing or melting, used to determine the freezing point [7]; the freezing point of the actual sample, in air at 1 atm [7], for both the API-Standard and API-NBS lots; the calculated value of the freezing point for zero impurity [7]; 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 [7]; and the resulting calculated amount of impurity in the API-Standard and the API-NBS material.

TABLE 2.—*Freezing points and purity of 29 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: API-Standard	Freezing point of the actual selected sample, in air at 1 atm: API-NBS	Freezing point for zero impurity in air at 1 atm	Cryoscopic constant, A	Calculated amount of impurity in the actual selected sample [c]: API-Standard	Calculated amount of impurity in the actual selected sample [c]: API-NBS
PARAFFINS							
		° C	*° C*	*° C*	*deg⁻¹*	*Mole %*	*Mole %*
n-Nonone	F	−53.555	−53.550	−53.535±0.010	0.0388	0.08 ±0.04	0.06 ±0.04
2,2,5-Trimethylhexane	F and M	−105.856	−105.834	−105.780±0.015	[d].0265	.20 ±0.04	.14 ±0.04
2,4,4-Trimethylhexane	M	−113.434	−113.430	−113.380±0.020	.0535	.29 ±0.11	.27 ±0.11
2,2,3,3-Tetramethylpentane	F	−10.06	−10.05	−9.90 ±0.05	.0040	.064±0.020	.060±0.020
2,2,3,4-Tetramethylpentane	F	−121.221	−121.178	−121.09 ±0.05	.0027	.035±0.014	.024±0.014
2,2,4,4-Tetramethylpentane	F and M	−66.599	−66.580	−66.54 ±0.03	.0273	.16 ±0.08	.11 ±0.08
2,3,3,4-Tetramethylpentane	F and M	−102.137	−102.135	−102.123±0.010	.0369	.051±0.037	.044±0.037
3,3-Diethylpentane	M	−33.118	−33.116	−33.110±0.005	.0223	.018±0.011	.013±0.011
ALKYLBENZENES							
n-Butylbenzene (*B*)	M	−88.000	−87.993	−87.970±0.020	[e](0.0385)	0.12±0.08	0.09 ±0.08
Isobutylbenzene (*B*)	M	−51.523	−51.520	−51.48 ±0.03	.0306	.13±0.09	.112±0.09
sec-Butylbenzene (*B*)	M	−75.511	−75.493	−75.470±0.020	.0303	.12±0.06	.07 ±0.06
tert-Butylbenzene (*B*)	M	−57.876	−57.876	−57.850±0.015	.0218	.06±0.03	.06 ±0.03
ALKYLCYCLOPENTANES							
Ethylcyclopentane	M	−138.454	−138.452	−138.435±0.010	0.0303	0.06 ±0.03	0.05 ±0.03
1,1-Dimethylcyclopentane	F	−69.802	−69.802	−69.73 ±0.04	[d].004	.03 ±0.02	.03 ±0.02
cis-1,2-Dimethylcyclopentane	F	−53.927	−53.910	−53.85 ±0.04	.0042	.031±0.016	.025±0.016
trans-1,2-Dimethylcyclopentane	M	−117.63	−117.61	−117.57 ±0.03	[e](.0320)	.19 ±0.10	.13 ±0.10
trans-1,3-Dimethylcyclopentane	M	−133.767	−133.758	−133.680±0.020	.0455	.39 ±0.09	.35 ±0.09
n-Propylcyclopentane	M	−117.379	−117.378	−117.340±0.020	.0511	.20 ±0.10	.19 ±0.10
Isopropylcyclopentane	M	−111.433	−111.431	−111.375±0.020	.0346	.20 ±0.07	.19 ±0.07
1,1,2-Trimethylcyclopentane	F	−21.69	−21.68	−21.64 ±0.03	.0030	.015±0.009	.012±0.009
1,1,3-Trimethylcyclopentane	M	−142.56	−142.55	−142.44 ±0.08	.040	.48 ±0.32	.44±0.32
cis, cis, trans-1,2,4-Trimethylcyclopentane	M	−132.66	−132.64	−132.55 ±0.06	.0385	.42 ±0.23	.35±0.23
cis, trans, cis-1,2,4-Trimethylcyclopentane	M	−130.85	−130.84	−130.78 ±0.03	.0343	.24 ±0.10	.21±0.10
ALKYLCYCLOHEXANES							
Ethylcyclohexane	M	−111.335	----------	−111.300±0.020	0.0384	0.13±0.08	----------
1,1-Dimethylcyclohexane	F and M	−33.839	−33.654	−33.54 ±0.05	.0065	.19 ±0.03	0.07 ±0.03
cis-1,2-Dimethylcyclohexane	M	−50.047	−50.034	−50.00 ±0.03	.0051	.024±0.015	.017±0.015
trans-1,2-Dimethylcyclohexane	M	−88.203	−88.203	−88.180±0.020	.0365	.08 ±0.07	.08 ±0.07
cis,1,4-Dimethylcyclohexane	M	−87.445	−87.445	−87.425±0.015	.0283	.06 ±0.04	06 ±0.04
trans-1,4-Dimethylcyclohexane	F and M	−36.976	−36.964	−36.92 ±0.03	.0259	.14 ±0.08	.11 ±0.08

[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 [6].

[b] F indicates freezing and M indicates melting. See reference [7] for experimental details and the definition of the cryoscopic constant.

[c] The values in this column were calculated as described in reference [7], using the values of the cryoscopic constants and freezing points for zero impurity given in the preceding columns.

[d] This cryoscopic constant was determined by the procedure given on page 371 of reference [7].

[e] Not determined in this investigation. From the "*z*" tables of the American Petroleum Institute Research Project 44 [8].

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

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[8] American Petroleum Institute Research Project 44 at the National Bureau of Standards. Selected values of properties of hydrocarbons. Heat and entropy of fusion, freezing points, and cryoscopic constants. Tables 1z, 2z, 3z, 5z, and 6z.

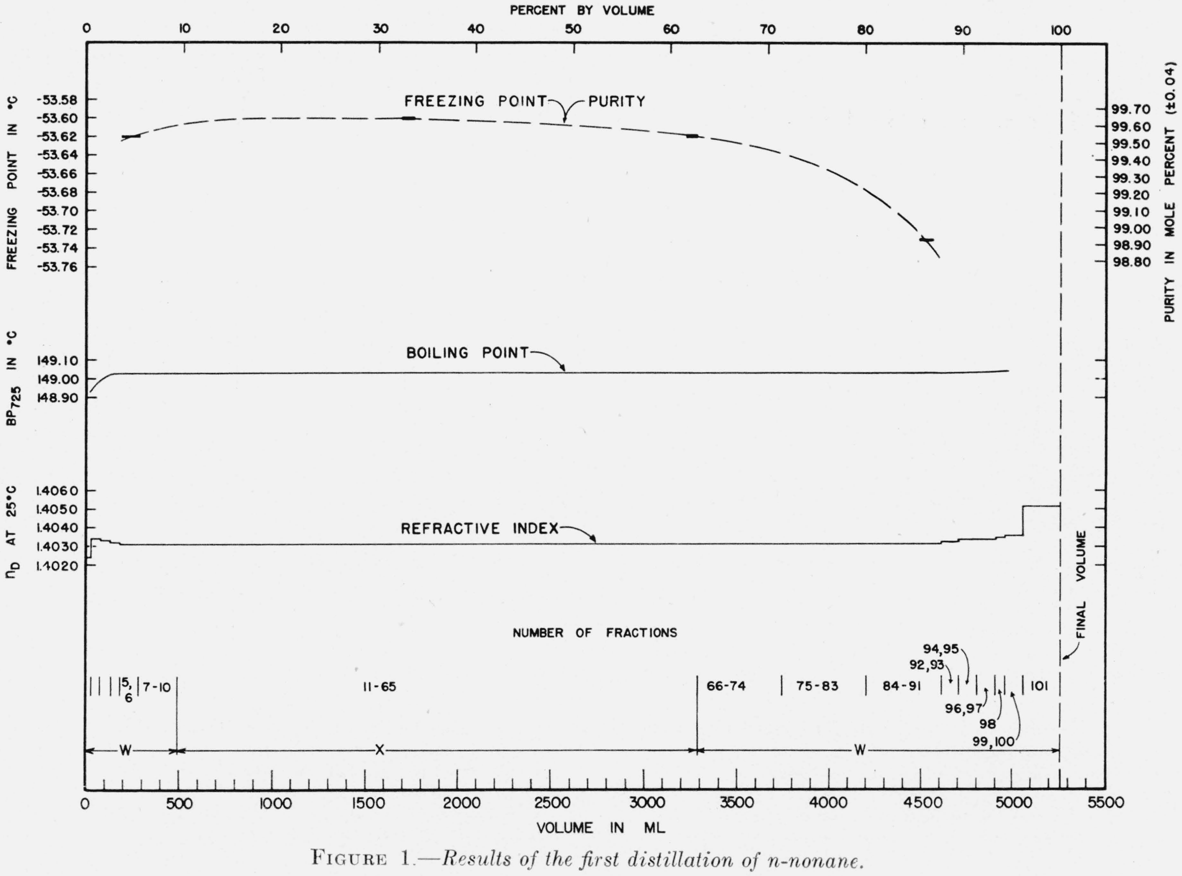

FIGURE 1.—*Results of the first distillation of n-nonane.*

Regular distillation at 725 mm Hg in still 14 (8/28/45 to 9/15/45).

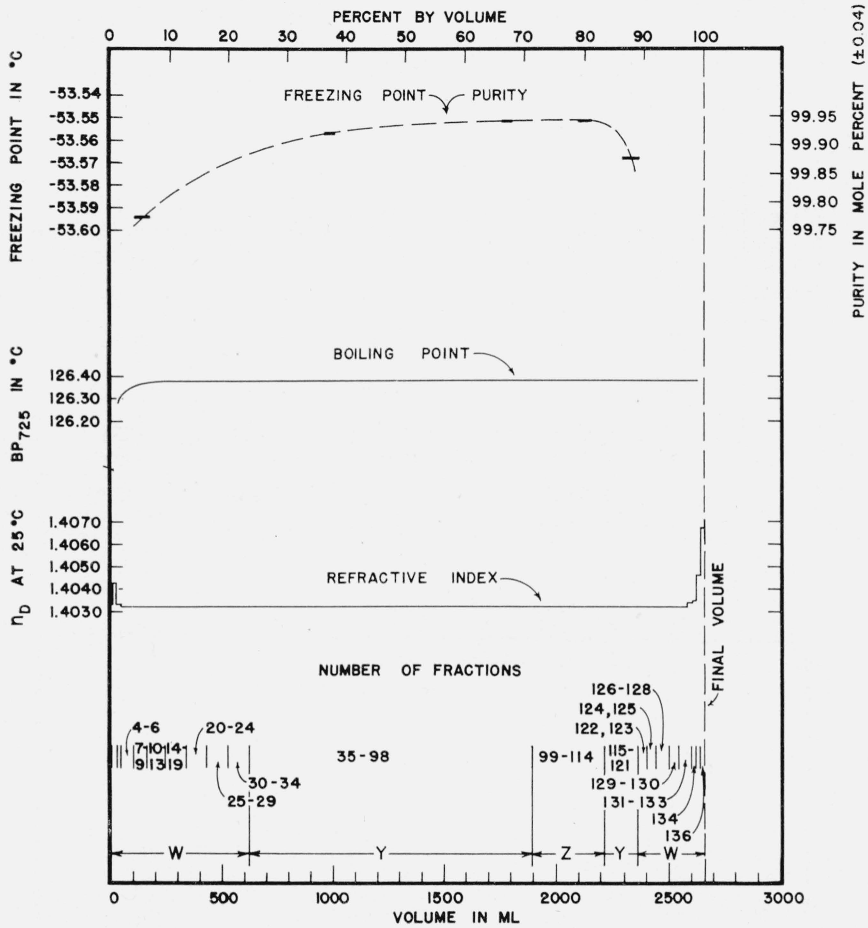

FIGURE 2.—*Results of the second and final distillation of n-nonane.*

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in still 13 (10/13/45 to 11/14/45).

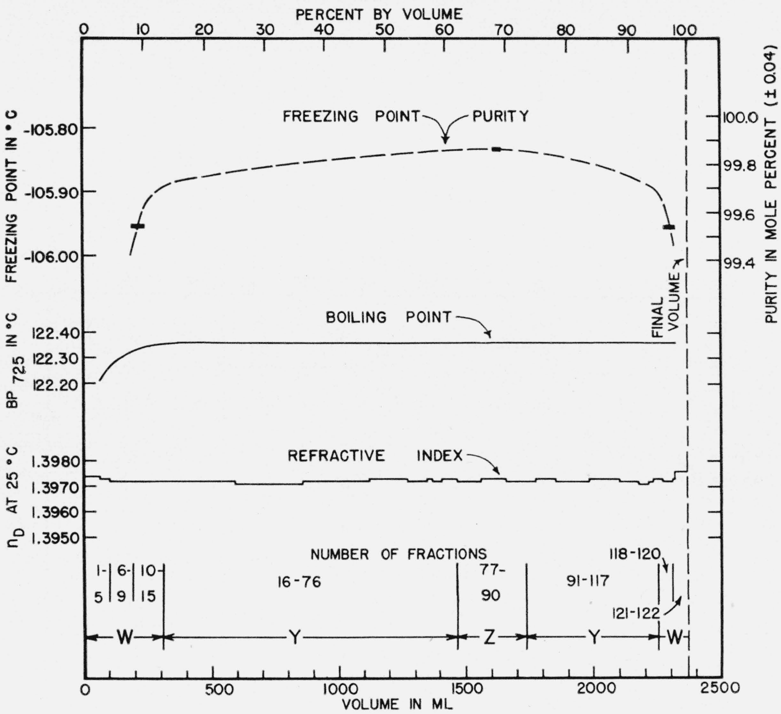

FIGURE 3.—*Results of the first and only distillation of 2,2,5-trimethylhexane.*

Regular distillation at 725 mm Hg in still 3 (6/26/44 to 8/6/44).

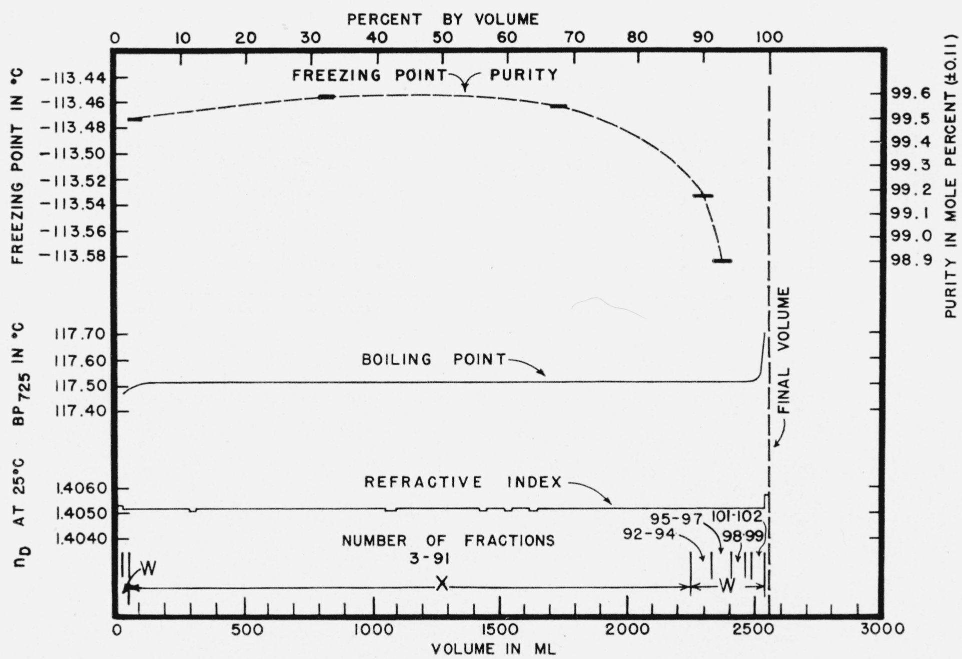

FIGURE 4.—*Results of the first distillation of 2,4,4-trimethylhexane.*

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in still 13 (11/16/44 to 12/6/44).

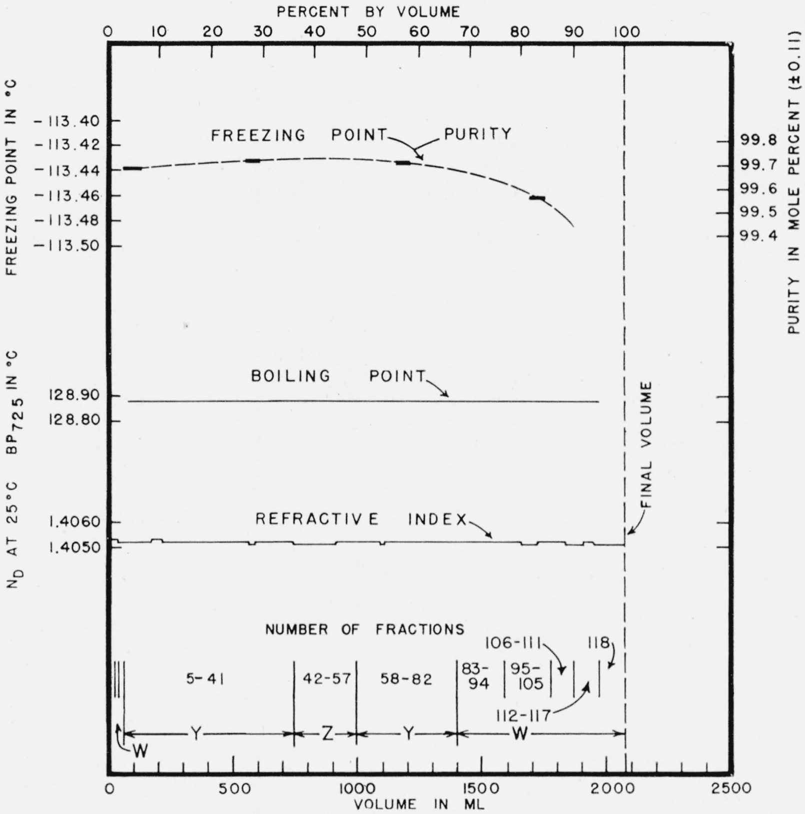

FIGURE 5.—*Results of the second and final distillation of 2,4,4-trimethylhexane.*

Regular distillation at 725 mm Hg in still 12 (6/21/45 to 7/13/45).

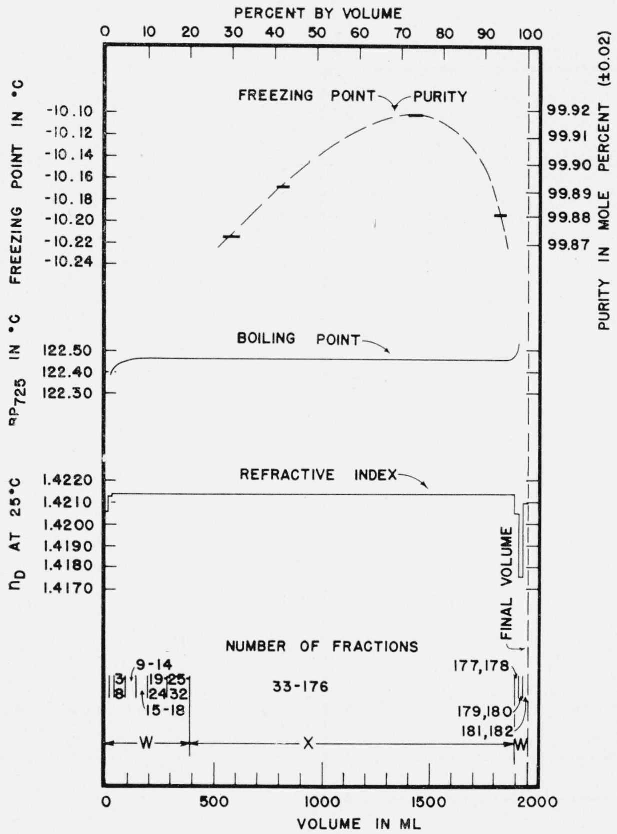

FIGURE 6.—*Results of the first distillation of 2,2,3,3-tetramethylpentane.*

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in still 10 (11/23/45 to 12/31/45).

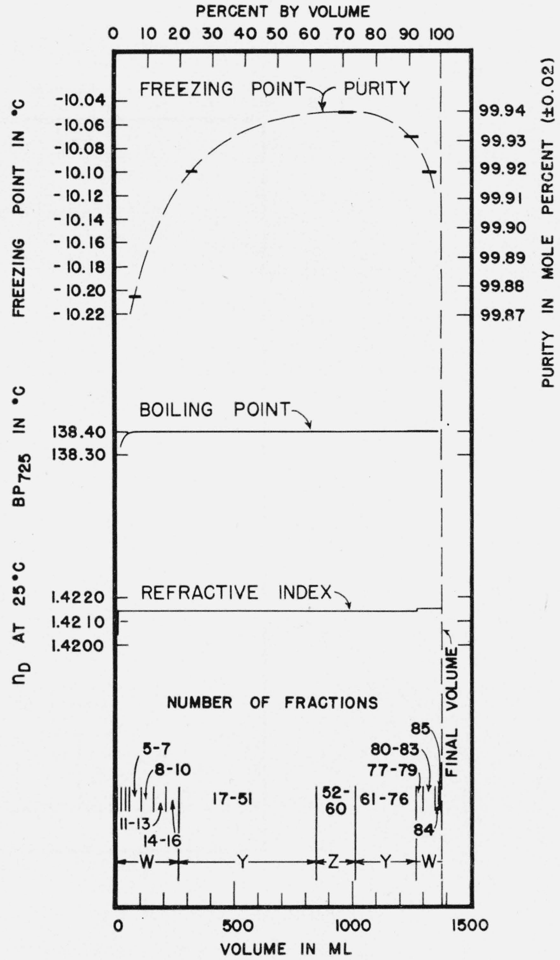

FIGURE 7.—*Results of the second and final distillation of 2,2,3,3-tetramethylpentane.*

Regular distillation at 725 mm Hg in still 11A (2/20/46 to 3/14/46)

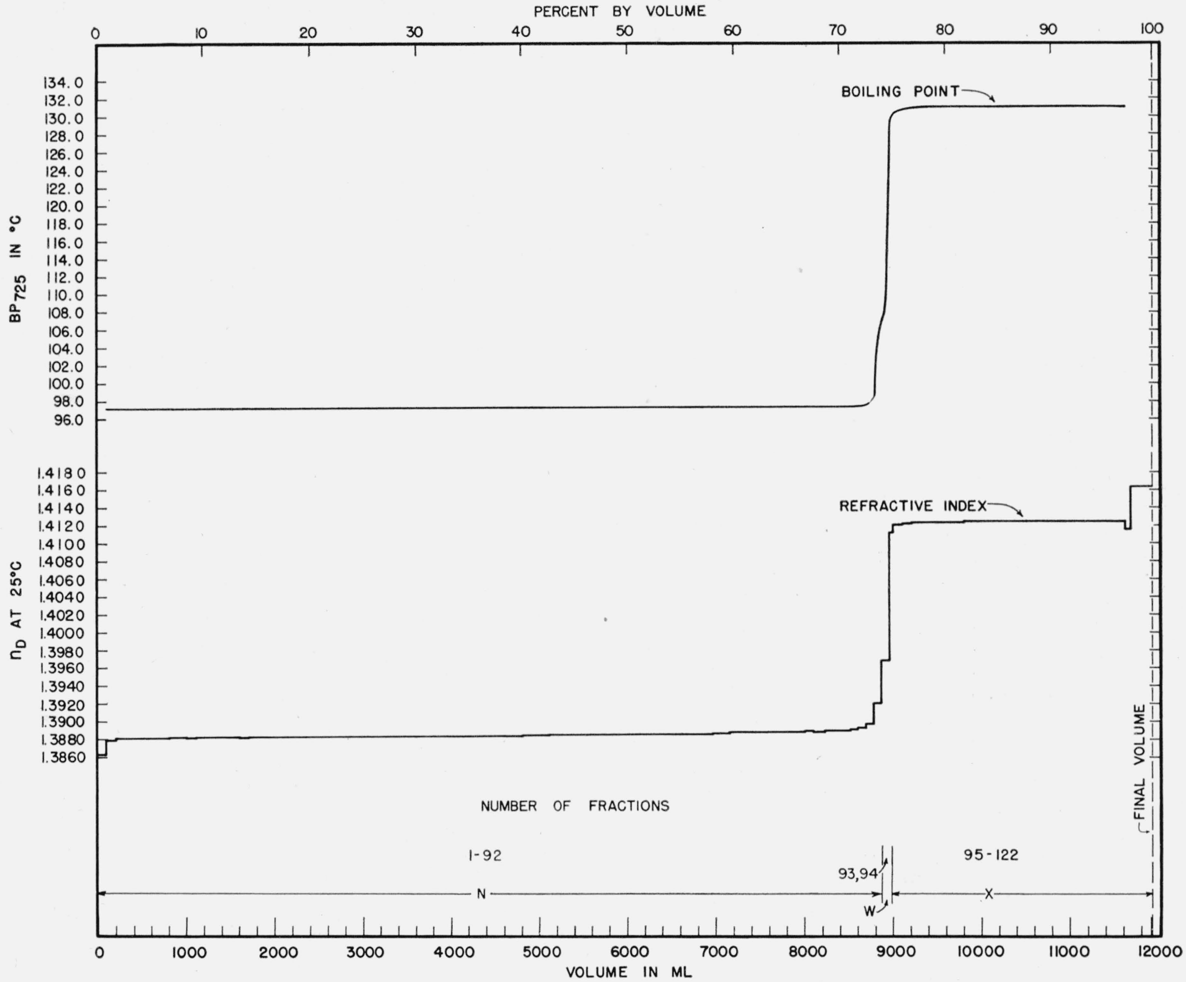

FIGURE 8.—*Results of the first distillation of 2,2,3,4-tetramethylpentane.*

Regular distillation at 725 mm Hg in still 6 (7/27/45 to 9/11/45).
The portion marked "N" was discarded. See footnote *i* of table 1.

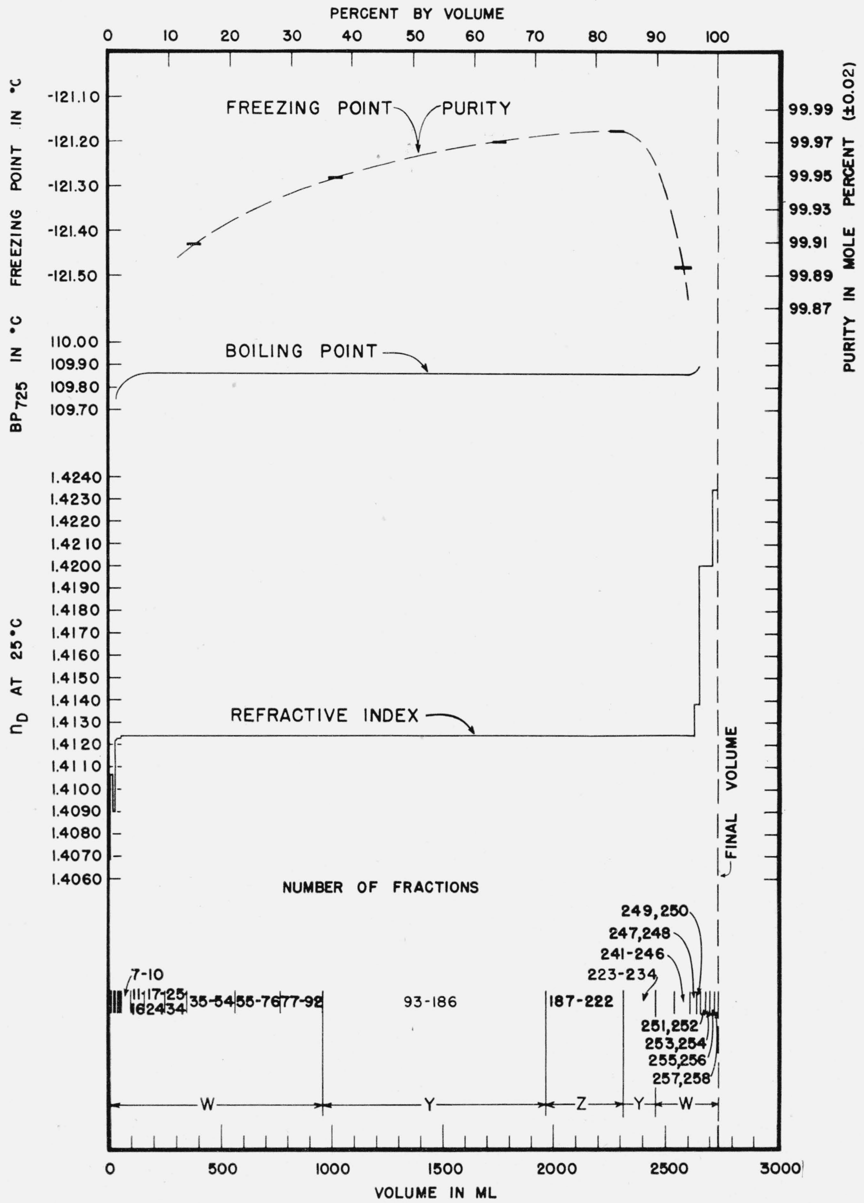

FIGURE 9.—*Results of the second and final distillation of 2,2,3,4-tetramethylpentane.*

Azeotropic distillation with ethylene glycol monomethyl ether at 725 mm Hg in still 10 (9/27/45 to 11/19/45).

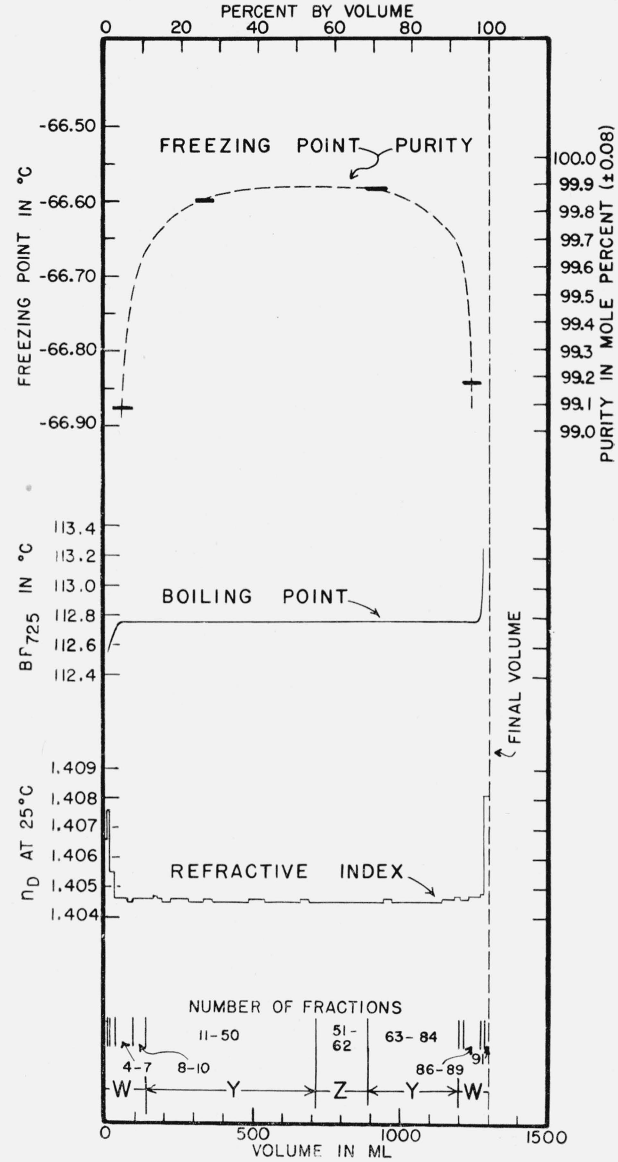

FIGURE 10.—*Results of the first and only distillation of 2,2,4,4-tetramethylpentane.*

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in still 4 (9/11/44 to 9/29/44).

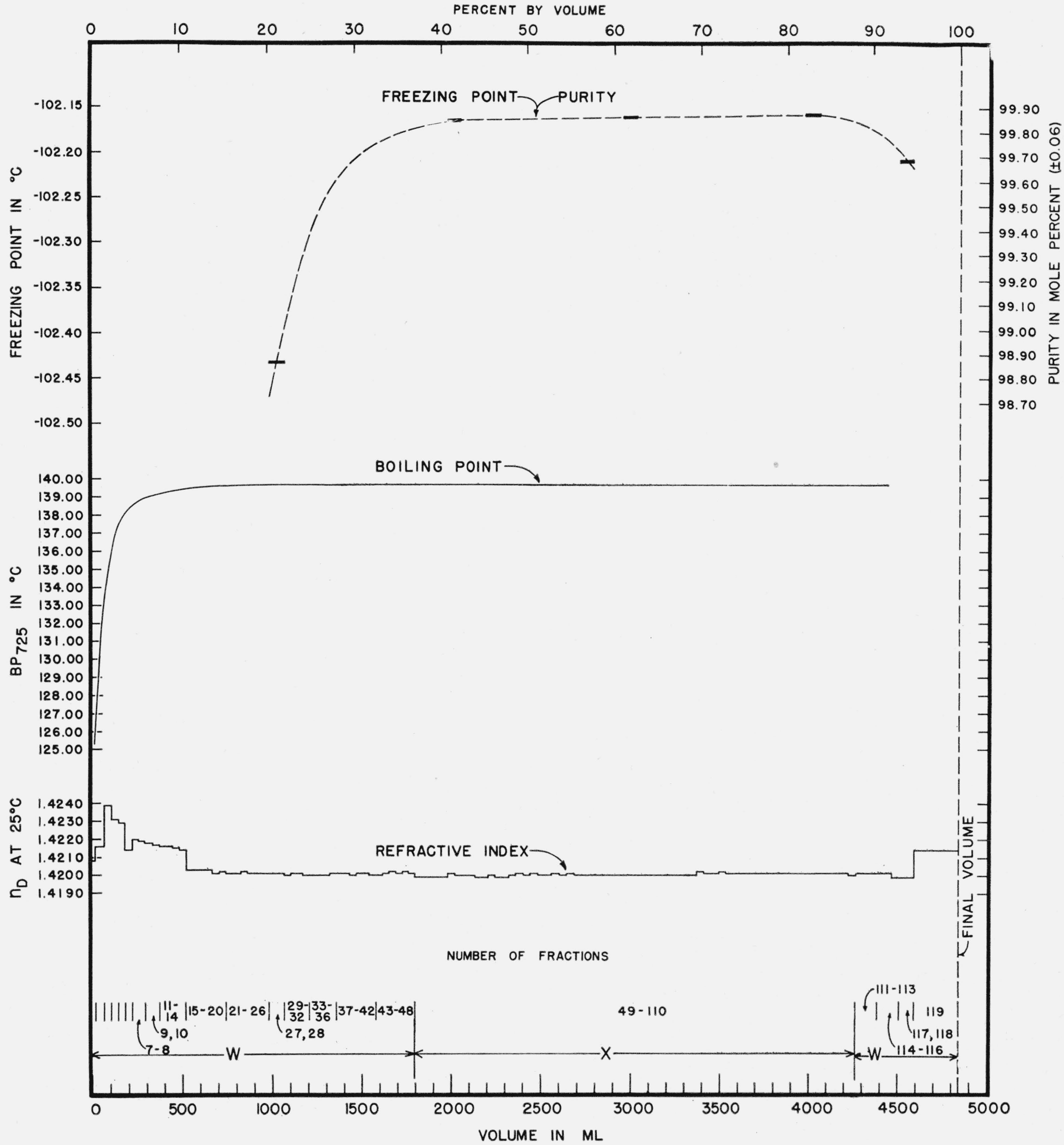

FIGURE 11.—*Results of the first distillation of 2,3,3,4-tetramethylpentane.*

Regular distillation at 725 mm Hg in still 8 (7/4/45 to 7/31/45).

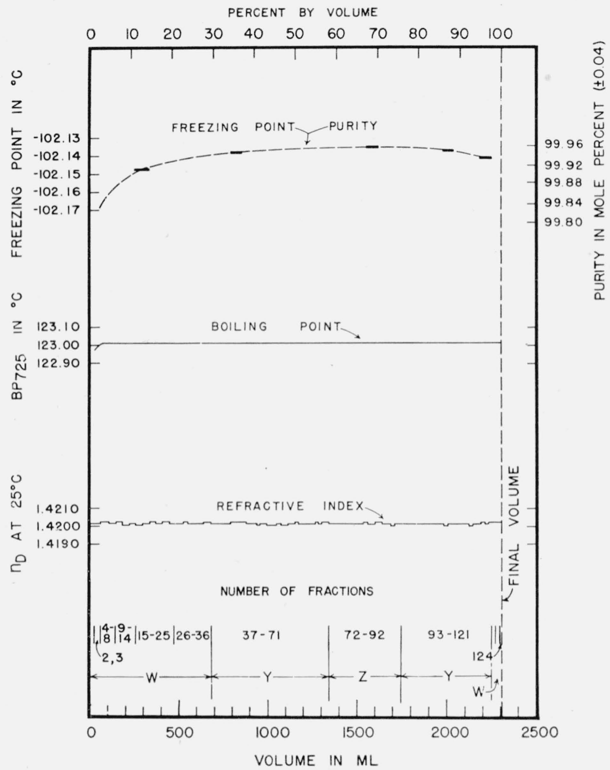

FIGURE 12.—*Results of the second and final distillation of 2,3,3,4-tetramethylpentane.*

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in still 8 (9/18/45 to 10/16/45).

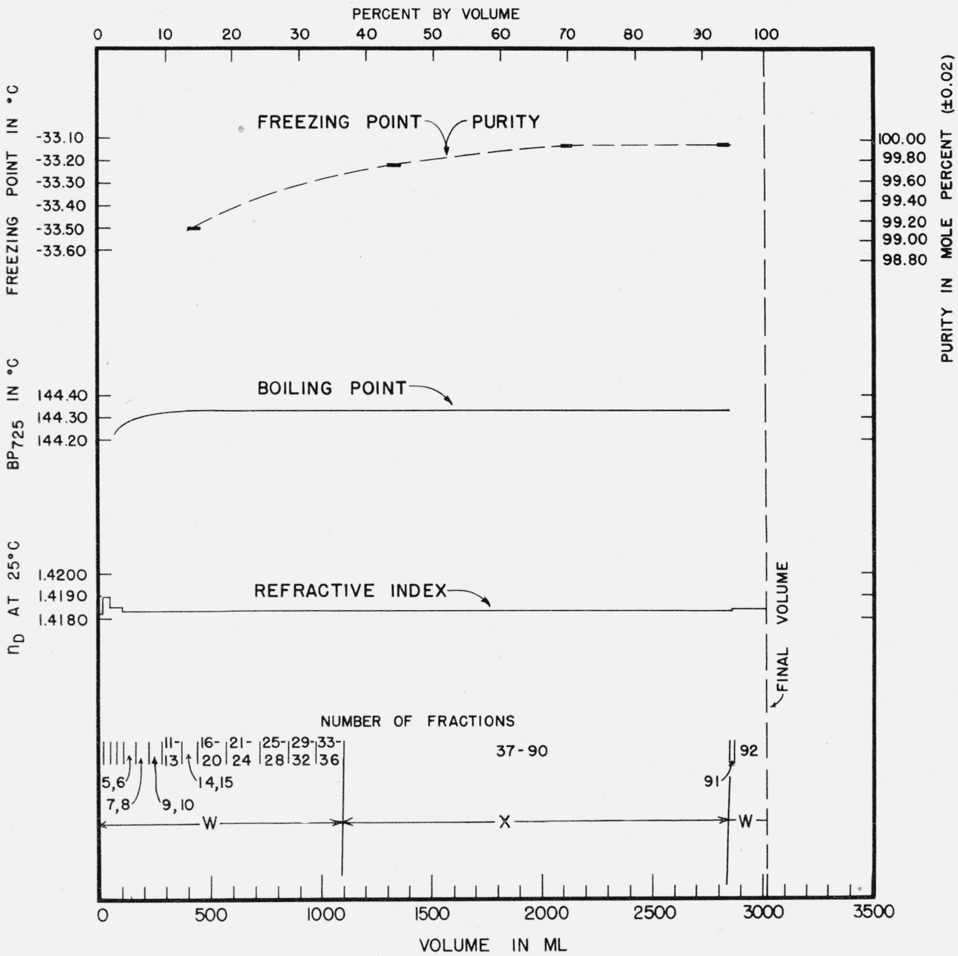

FIGURE 13.—*Results of the first distillation of 3,3-diethylpentane.*

Regular distillation at 725 mm Hg in still 13 (8/13/45 to 9/4/45).

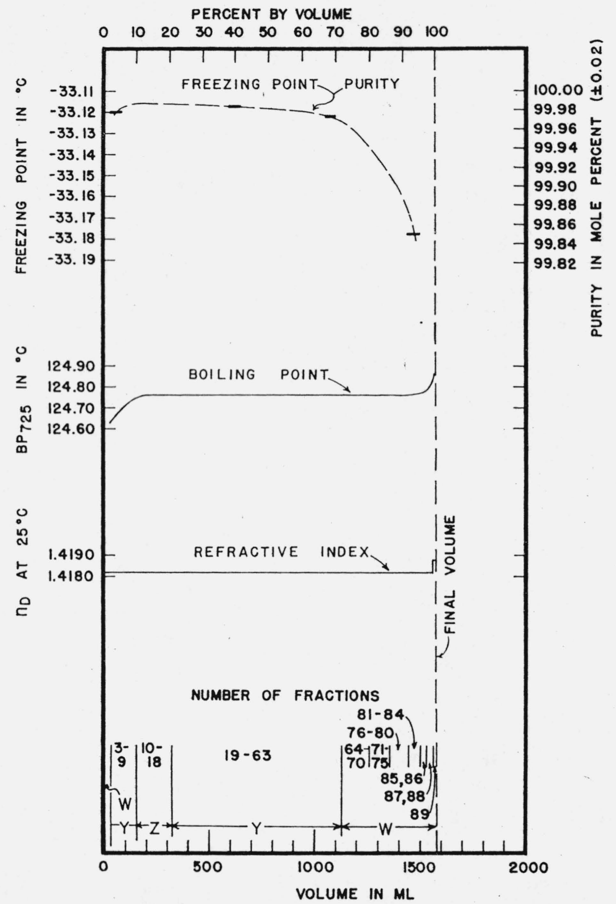

FIGURE 14.—*Results of the second and final distillation of 3,3-diethylpentane.*

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in still 13 (11/17/45 to 12/4/45).

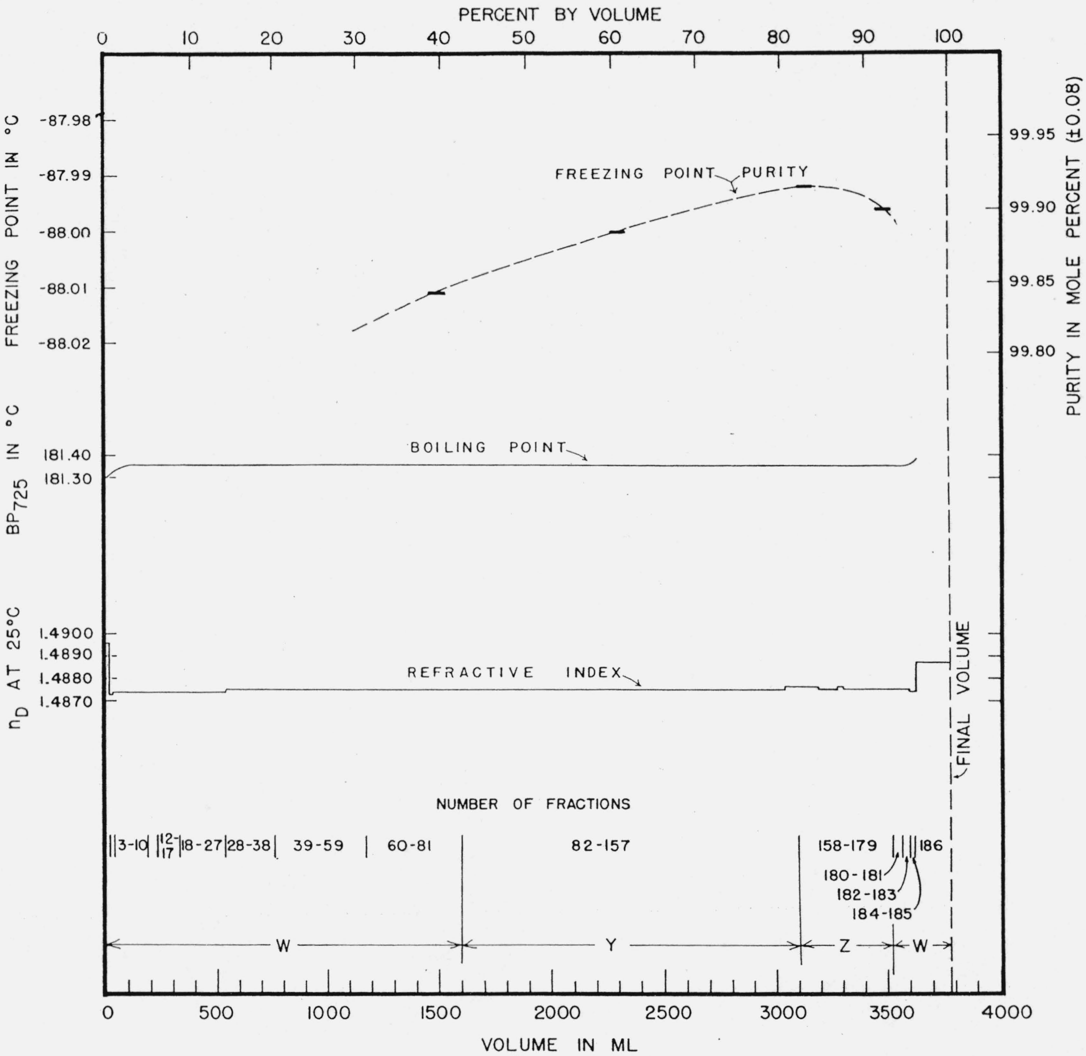

FIGURE 15.—*Results of the first and only distillation of n-butylbenzene.*

Regular distillation at 725 mm Hg in still 9 (7/17/45 to 8/18/45).

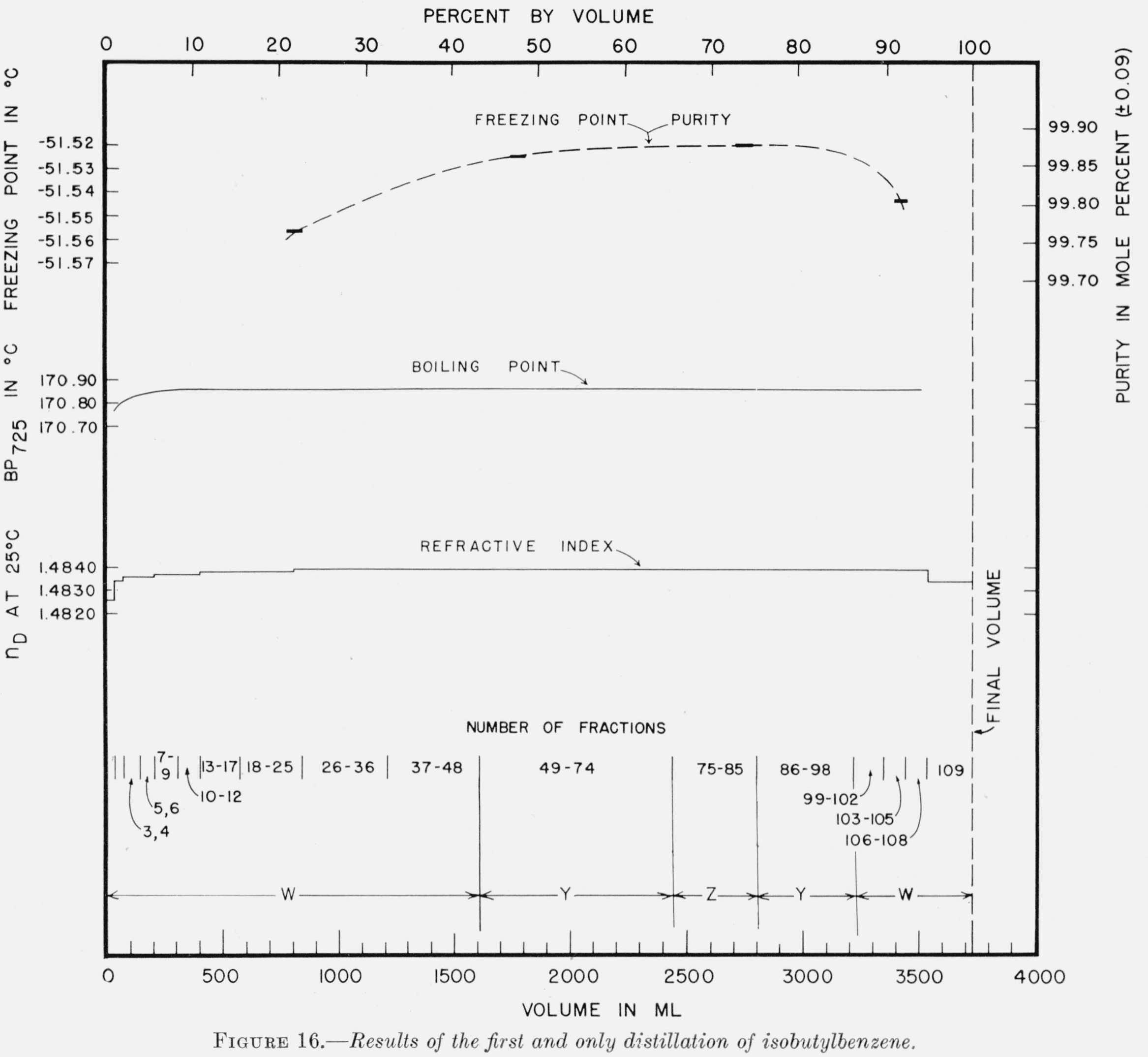

FIGURE 16.—*Results of the first and only distillation of isobutylbenzene.*

Regular distillation at 725 mm Hg in still 8 (8/27/45 to 9/16/45).

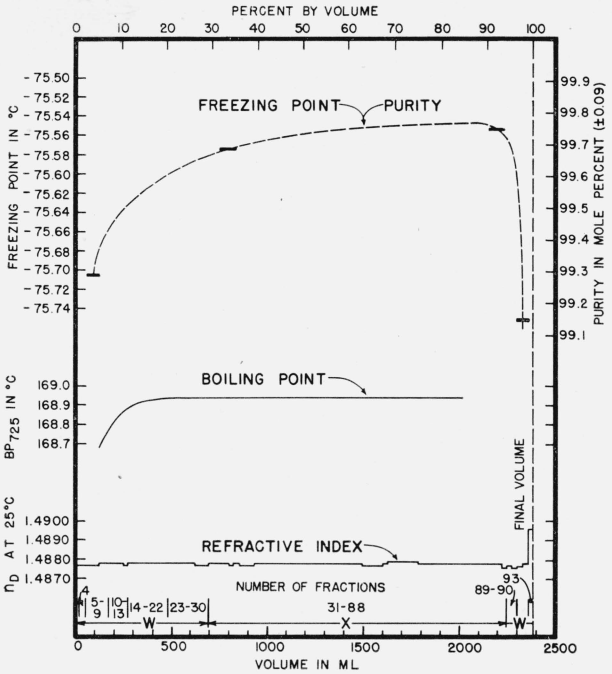

FIGURE 17.—*Results of the first distillation of sec-butylbenzene.*

Azeotropic distillation with diethylene glycol monomethyl ether at 725 mm Hg in still 8 (10/2/44 to 10/19/44).

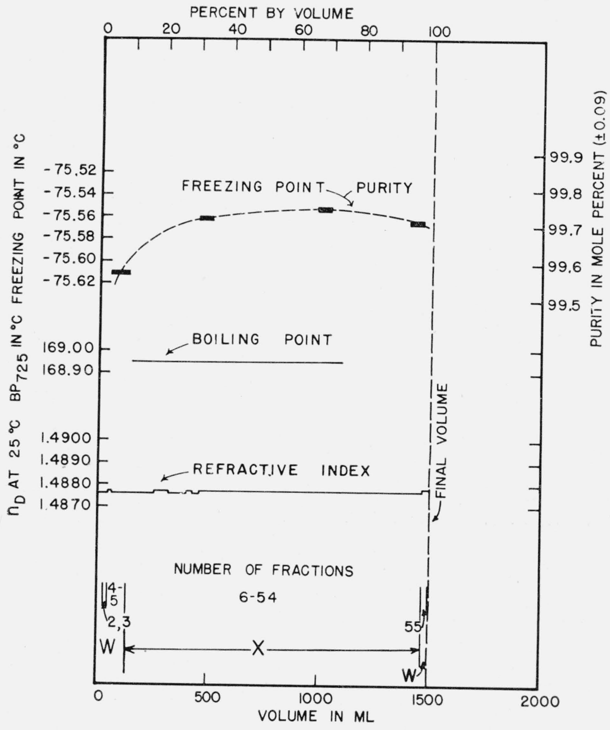

FIGURE 18.—*Results of the second distillation of sec-butylbenzene.*

Azeotropic distillation with diethylene glycol monomethyl ether at 725 mm Hg in still 13 (6/29/45 to 7/12/45).

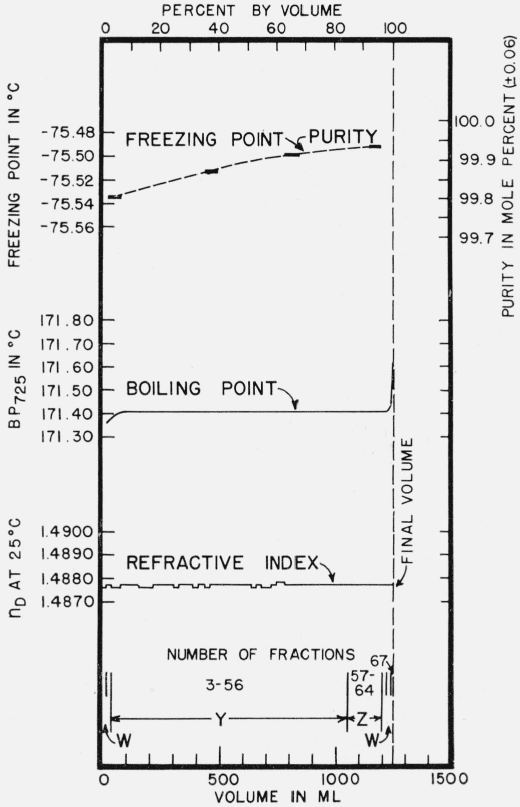

FIGURE 19.—*Results of the third and final distillation of sec-butylbenzene.*

Regular distillation at 725 mm Hg in still 12 (8/17/45 to 9/4/45).

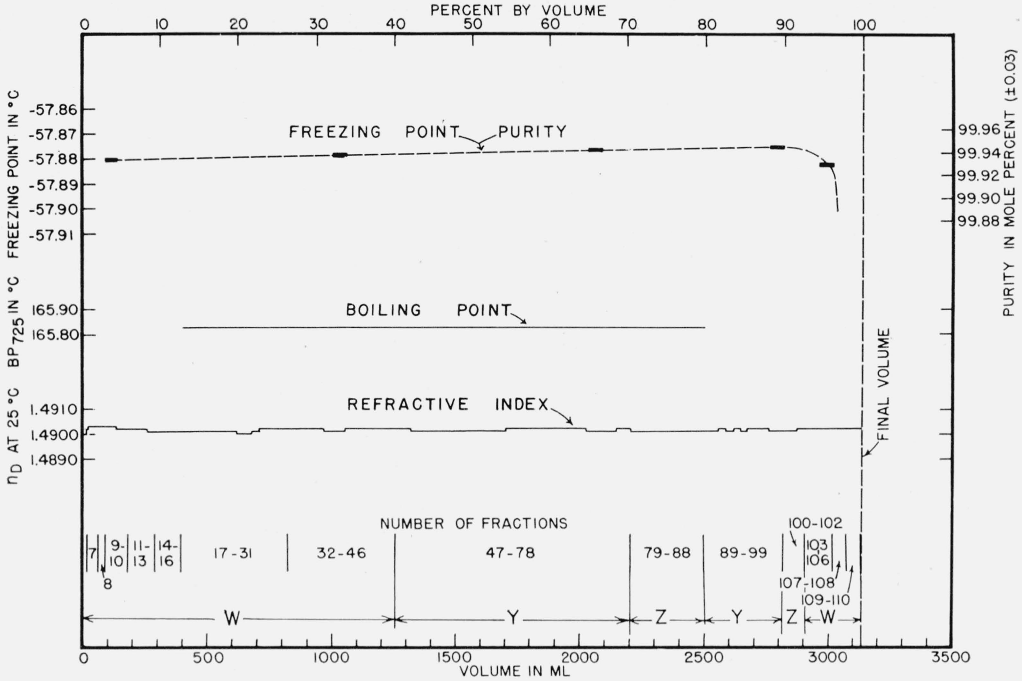

FIGURE 20.—*Results of the first and only distillation of tert-butylbenzene.*

Azeotropic distillation with diethylene glycol monomethyl ether at 725 mm Hg in still 8 (10/19/44 to 11/9/44).

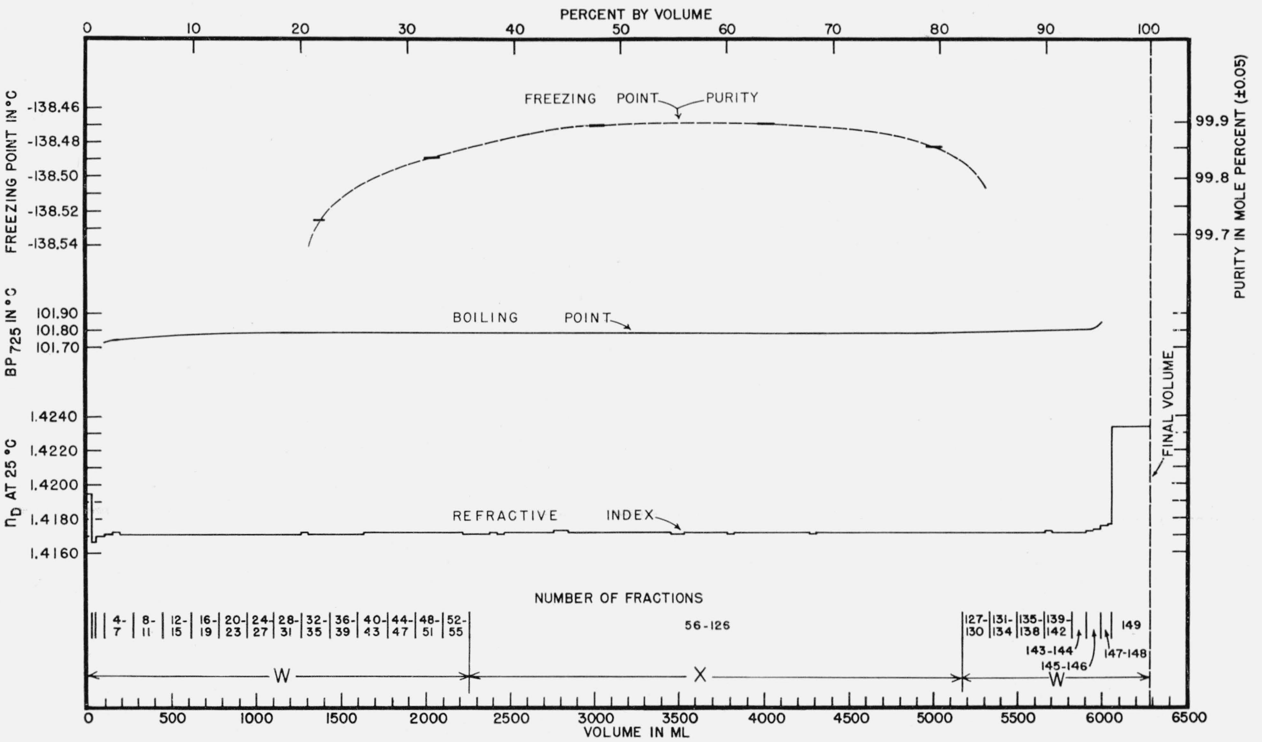

FIGURE 21.—*Results of the first distillation of ethylcyclopentane.*

Regular distillation at 725 mm Hg in still 15 (9/20/44 to 10/10/44).

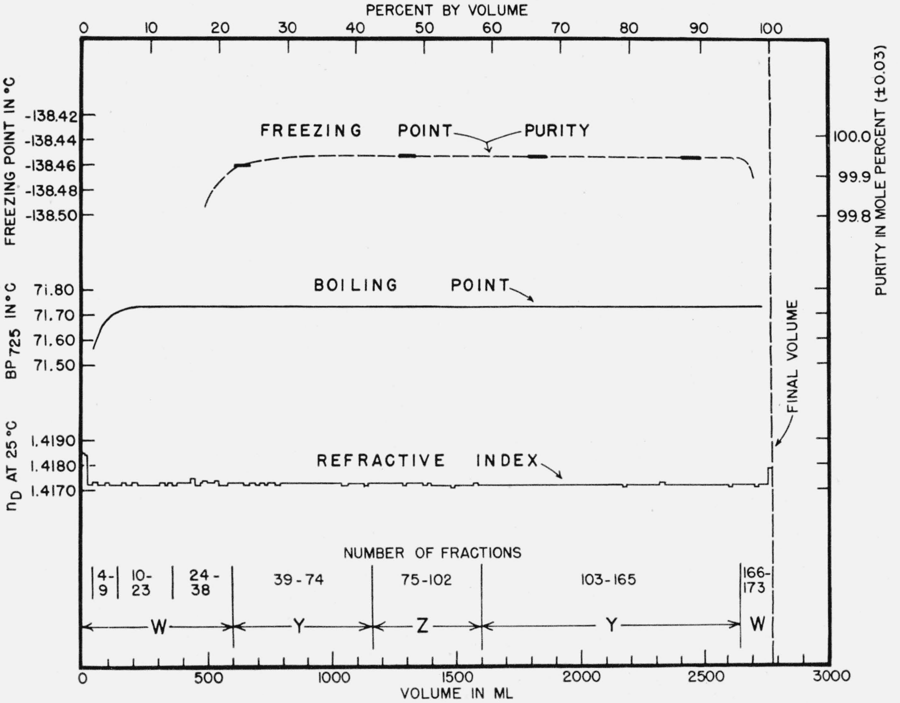

FIGURE 22.—*Results of the second and final distillation of ethylcyclopentane.*

Azeotropic distillation with ethanol at 725 mm Hg in still 13 (3/7/45 to 4/5/45).

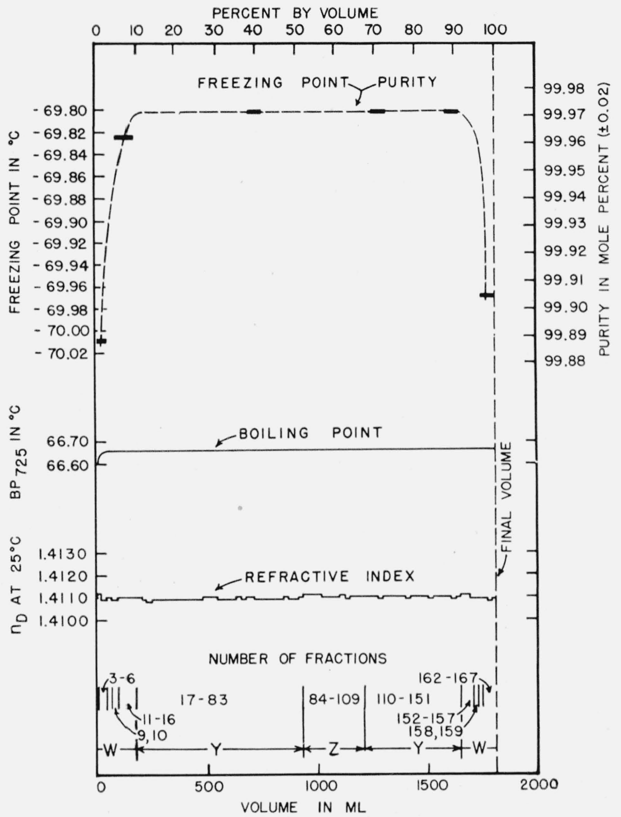

FIGURE 23.—*Results of the first and only distillation of 1,1-dimethylcyclopentane.*

Azeotropic distillation with ethanol at 725 mm Hg in still 9 (8/27/45 to 9/28/45).

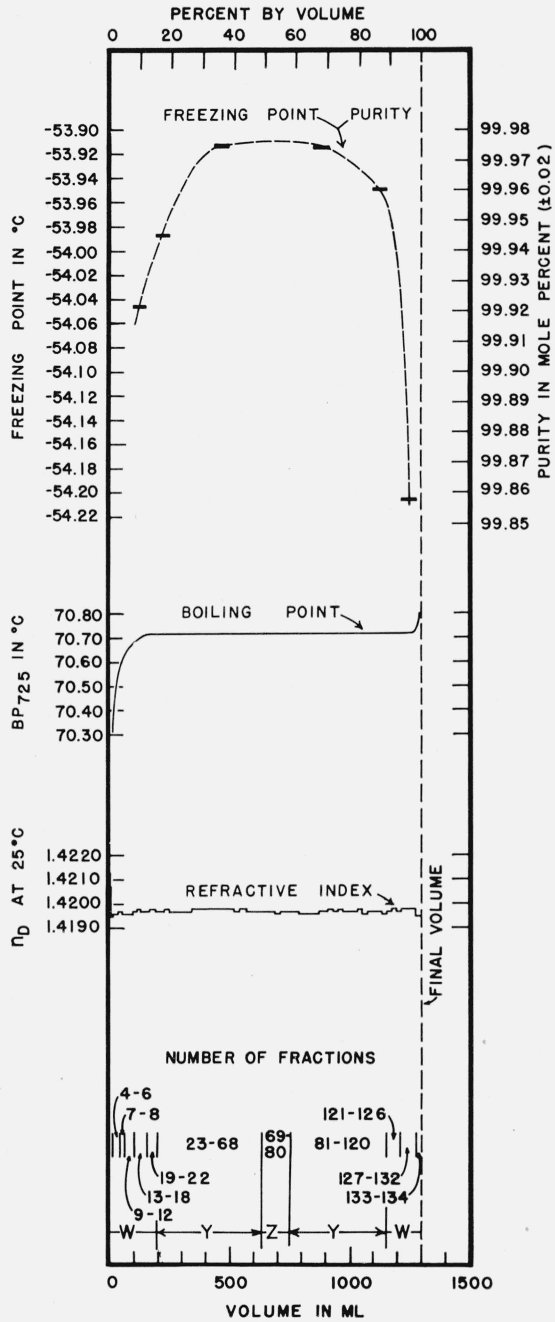

FIGURE 24.—*Results of the first and only distillation of cis-1,2-dimethylcyclopentane.*

Azeotropic distillation with ethanol at 725 mm Hg in still 9 (9/29/45 to 10/24/45).

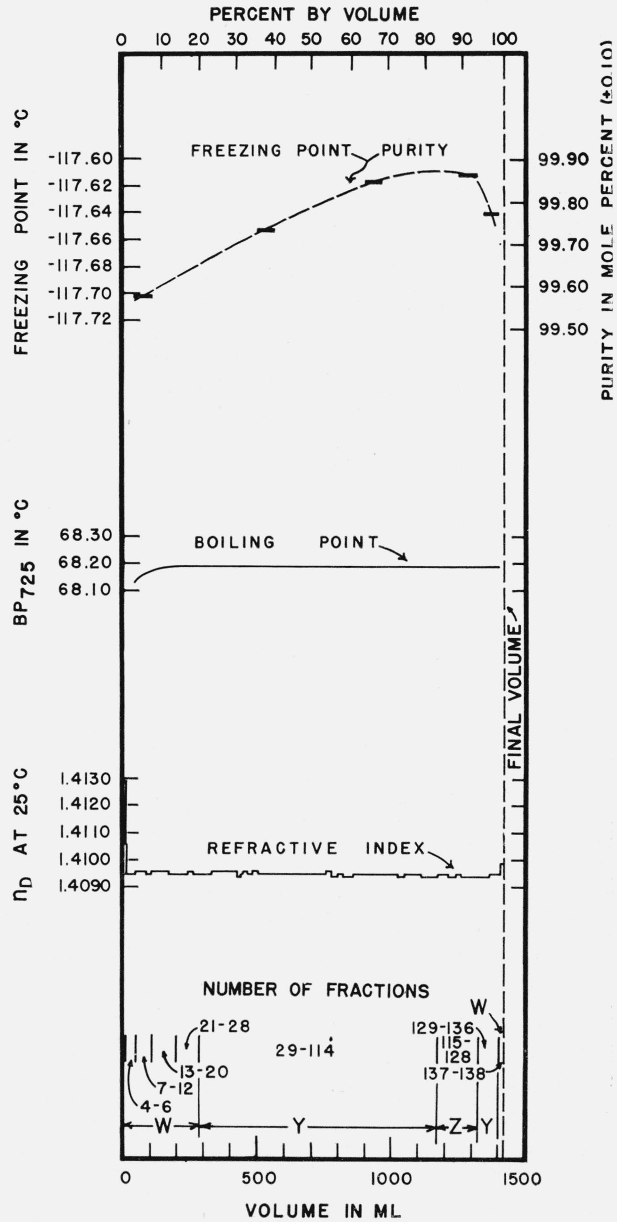

FIGURE 25.—*Results of the first and only distillation of trans-1,2-dimethylcyclopentane.*

Azeotropic distillation with ethanol at 725 mm Hg in still 4 (10/2/45 to 10/28/45).

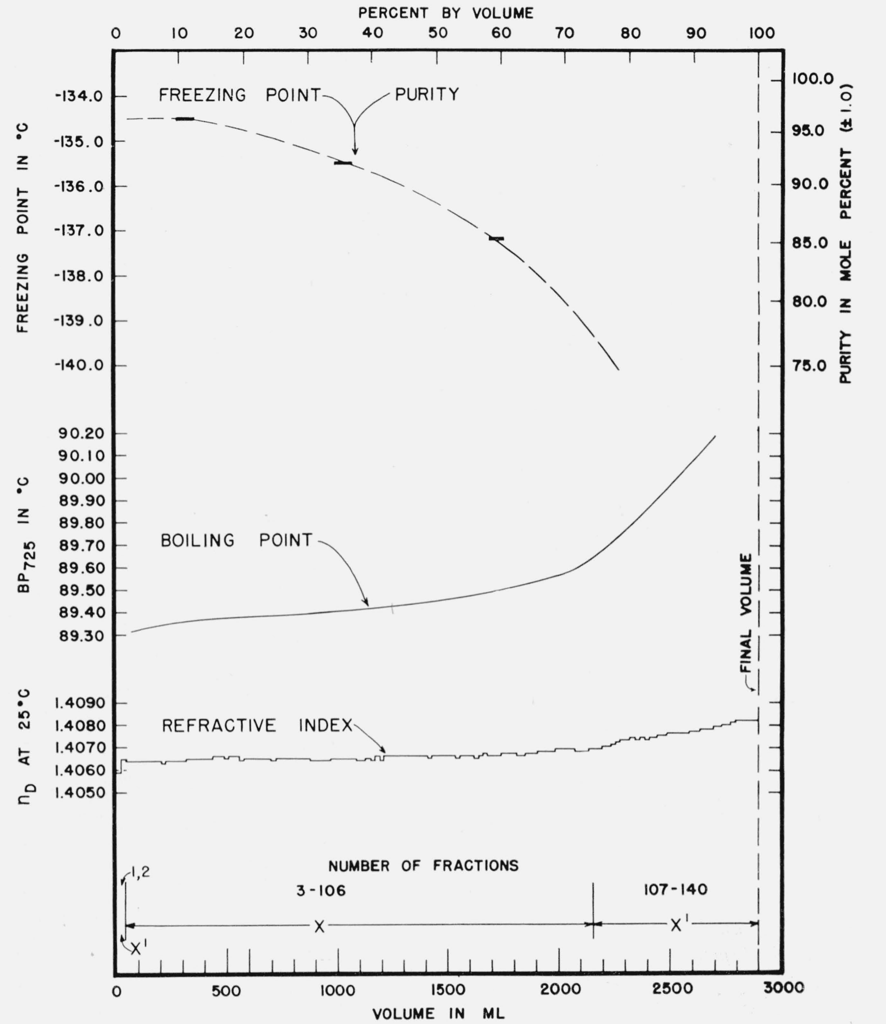

FIGURE 26.—*Results of the first distillation of trans-1,3-dimethylcyclopentane.*

Regular distillation at 725 mm Hg in still 4 (11/6/44 to 12/1/44).
Fractions 1, 2, and 107 to 140 (marked "X′") were retained for further processing by the API Research Project 6.

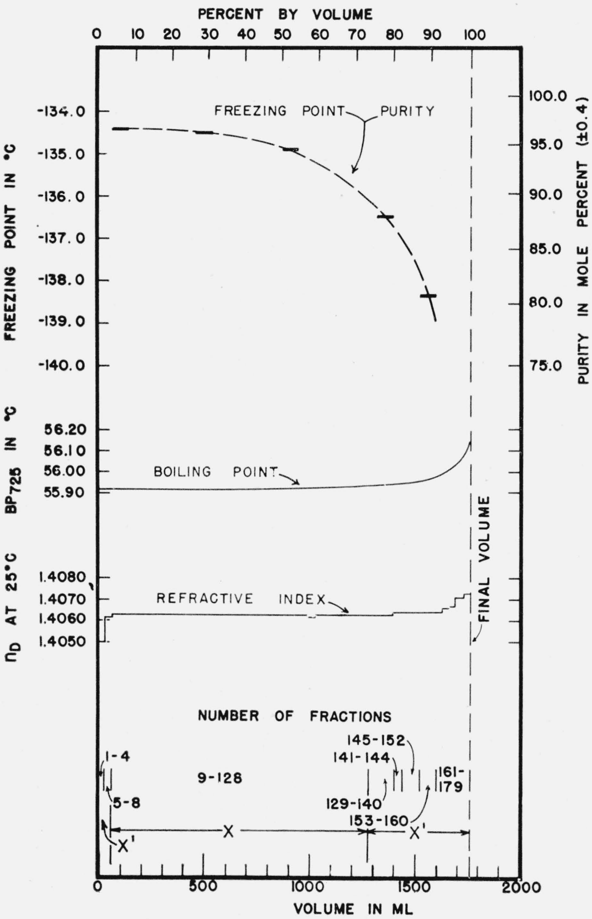

FIGURE 27.—*Results of the second distillation of trans-1,3-dimethylcyclopentane.*

Azeotropic distillation with methanol at 725 mm Hg in still 10 (5/26/45 to 7/4/45).
Fractions 1 to 8 and 153 to 179 (marked "X'") were retained for further processing by the API Research Project 6.

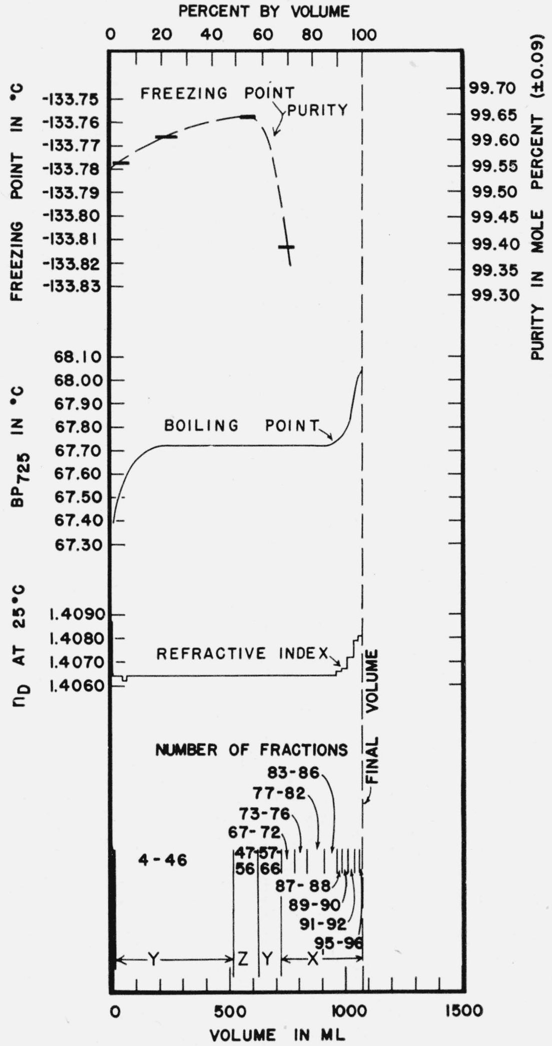

FIGURE 28.—*Results of the third and final distillation of trans-1,3-dimethylcyclopentane.*

Azeotropic distillation with ethanol at 725 mm Hg in still 4 (10/29/45 to 11/20/45).
Fractions 67 to 96 (marked "X'") were retained for further processing by the API Research Project 6.

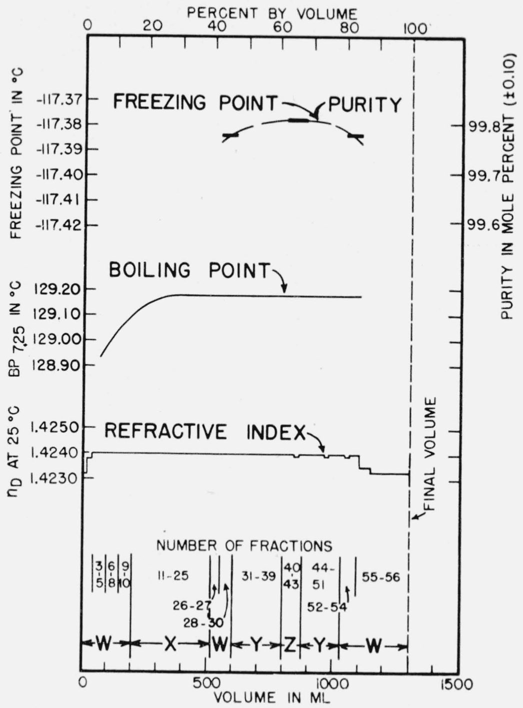

FIGURE 29.—*Results of the first distillation of n-propylcyclopentane.*

Regular distillation at 725 mm Hg in still 4 (6/12/44 to 6/24/44).

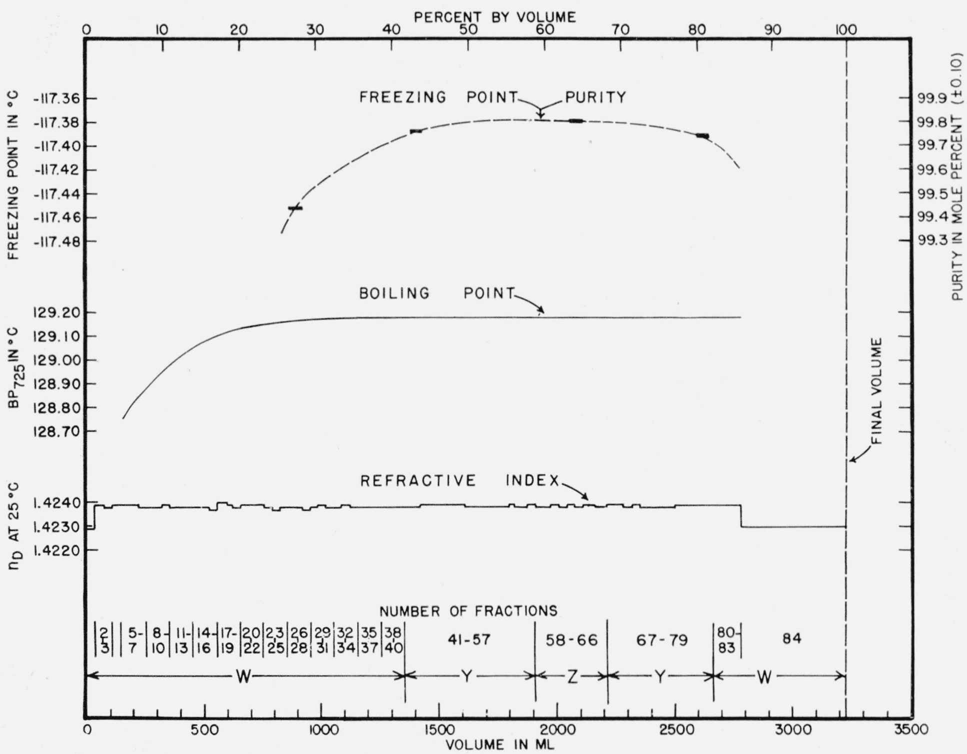

FIGURE 30.—*Results of the second and final distillation of n-propylcyclopentane.*

Regular distillation at 725 mm Hg in still 13 (12/7/44 to 12/22/44).
See table 1 for the composition of the charge for this distillation.

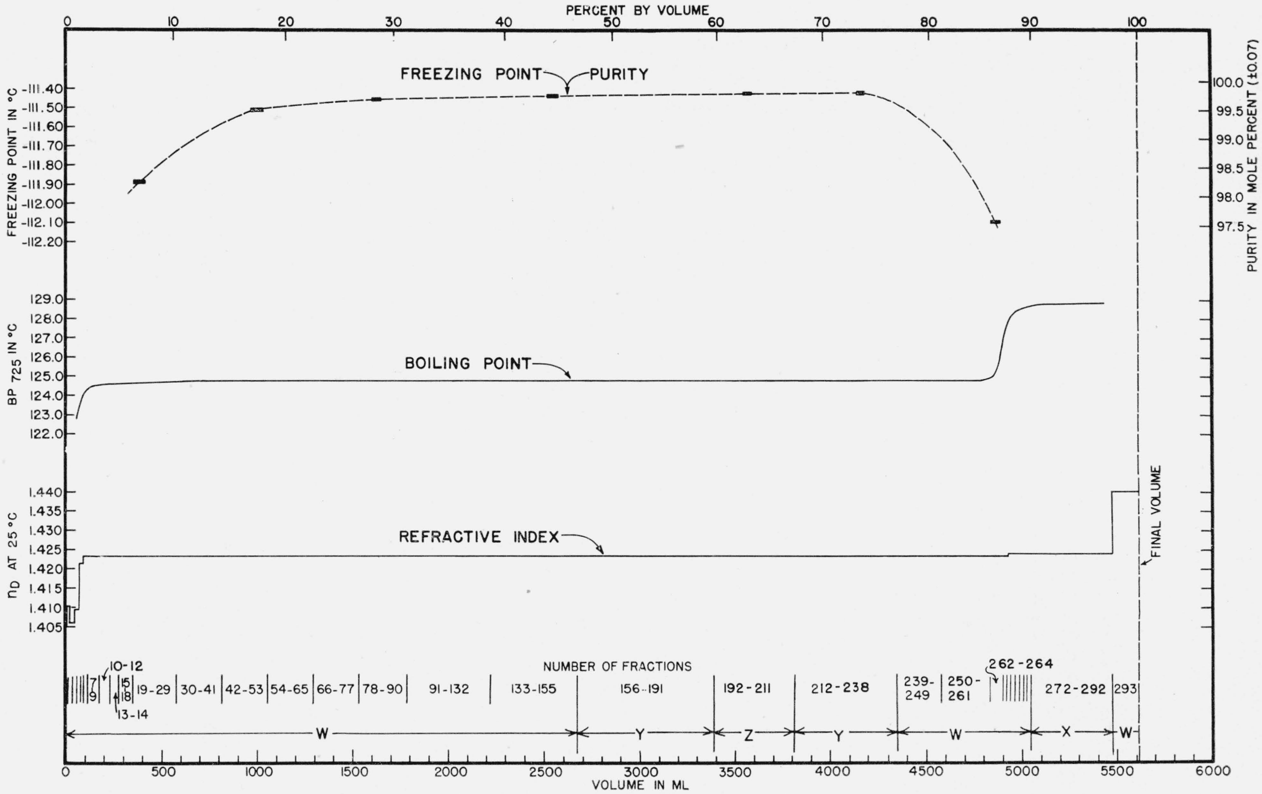

FIGURE 31.—*Results of the first and only distillation of isopropylcyclopentane.*

Regular distillation at 725 mm Hg in still 10 (7/3/44 to 8/20/44.)
The portion marked "X" was used as part of a charge of *n*-propylcyclopentane (see fig. 30 and footnote *n* of table 1).

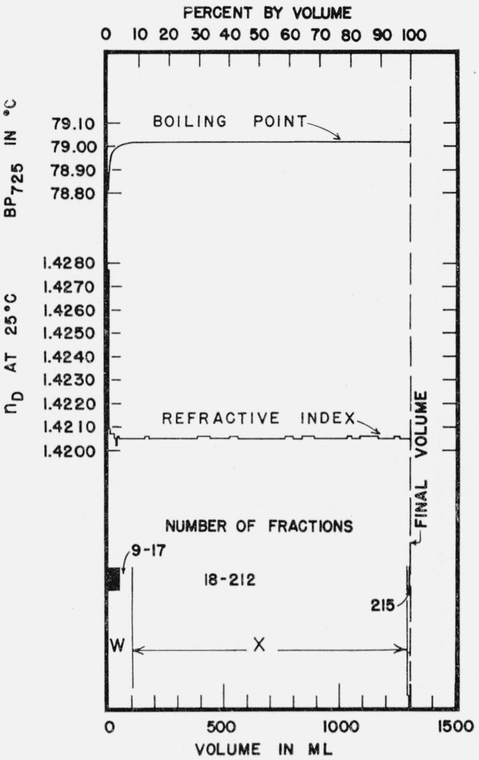

Figure 32.—*Results of the first distillation of 1,1,2-trimethylcyclopentane.*

Azeotropic distillation with isopropanol at 725 mm Hg in still 9 (11/23/45 to 1/7/46).

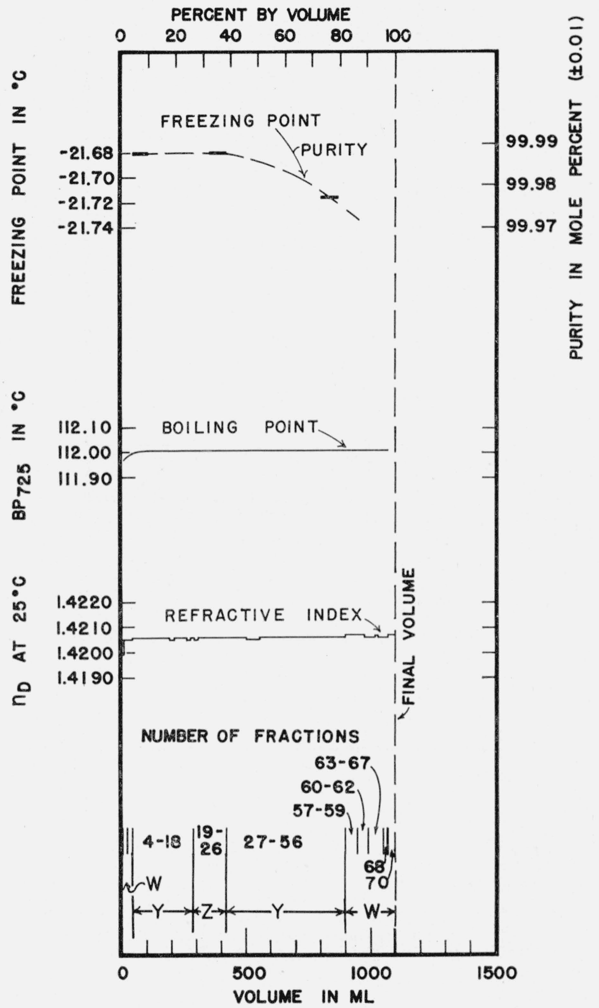

Figure 33.—*Results of the second and final distillation of 1,1,2-trimethylcyclopentane.*

Regular distillation at 725 mm Hg in still 3 (1/22/46 to 2/16/46).

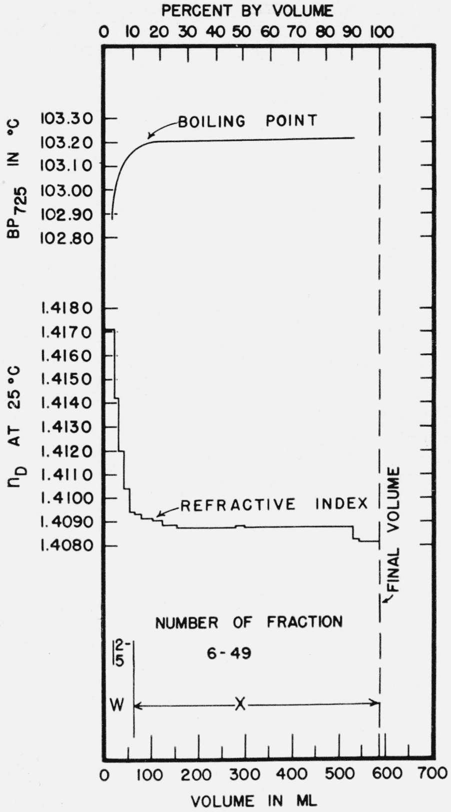

FIGURE 34.—*Results of the first distillation of 1,1,3-trimethylcyclopentane.*

Regular distillation at 725 mm Hg in still 3 (10/6/44 to 10/16/44).

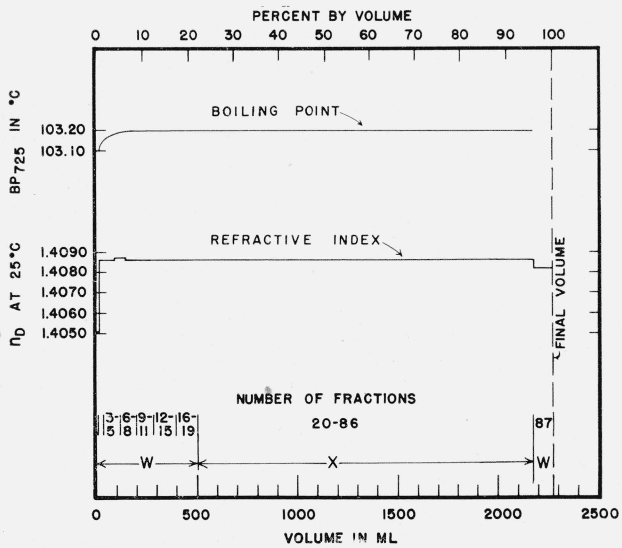

FIGURE 35.—*Results of the second distillation of 1,1,3-trimethylcyclopentane.*

Regular distillation at 725 mm Hg in still 12 (3/13/45 to 4/5/45).
See table 1 for the composition of the charge for this distillation.

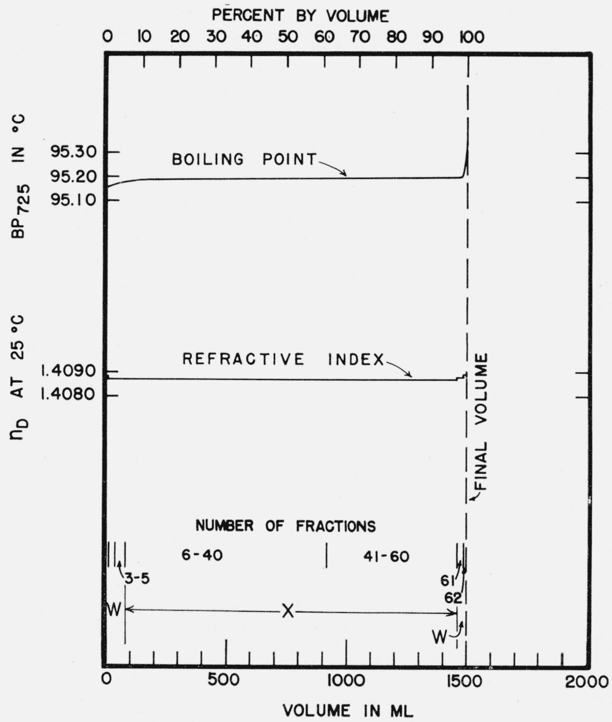

FIGURE 36.—*Results of the third distillation of 1,1,3-trimethylcyclopentane.*

Azeotropic distillation with ethylene glycol monomethyl ether at 725 mm Hg in still 12 (10/1/45 to 10/25/45).

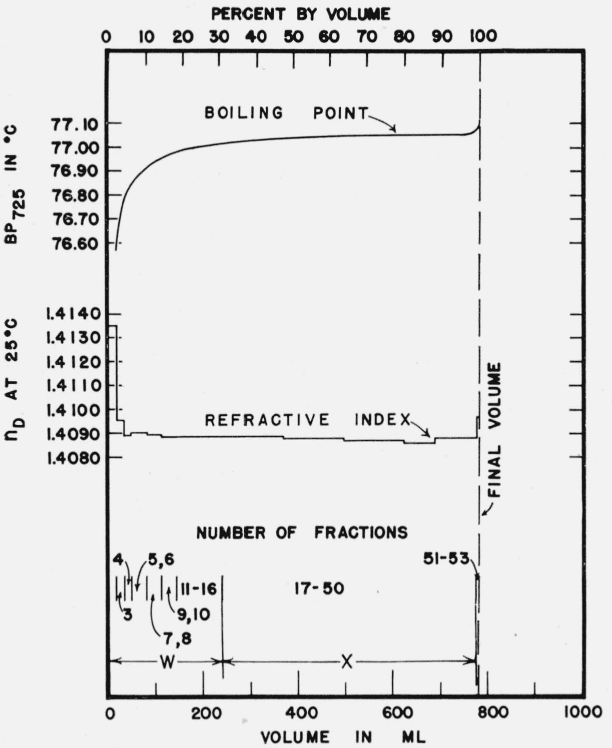

FIGURE 37.—*Results of the fourth distillation of 1,1,3-trimethylcyclopentane.*

Azeotropic distillation with isopropanol at 725 mm Hg in still 7 (11/17/45 to 11/27/45).

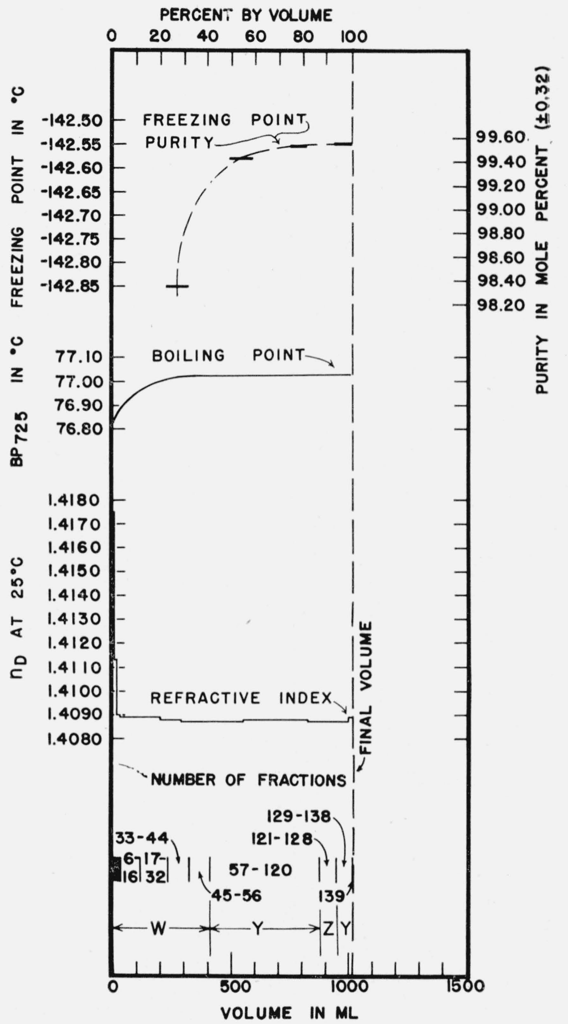

FIGURE 38.—*Results of the fifth and final distillation of 1,1,3-trimethylcyclopentane.*

Azeotropic distillation with isopropanol at 725 mm Hg in still 4 (1/3/46 to 1/29/46).

See table 1 for composition of the charge for this distillation.

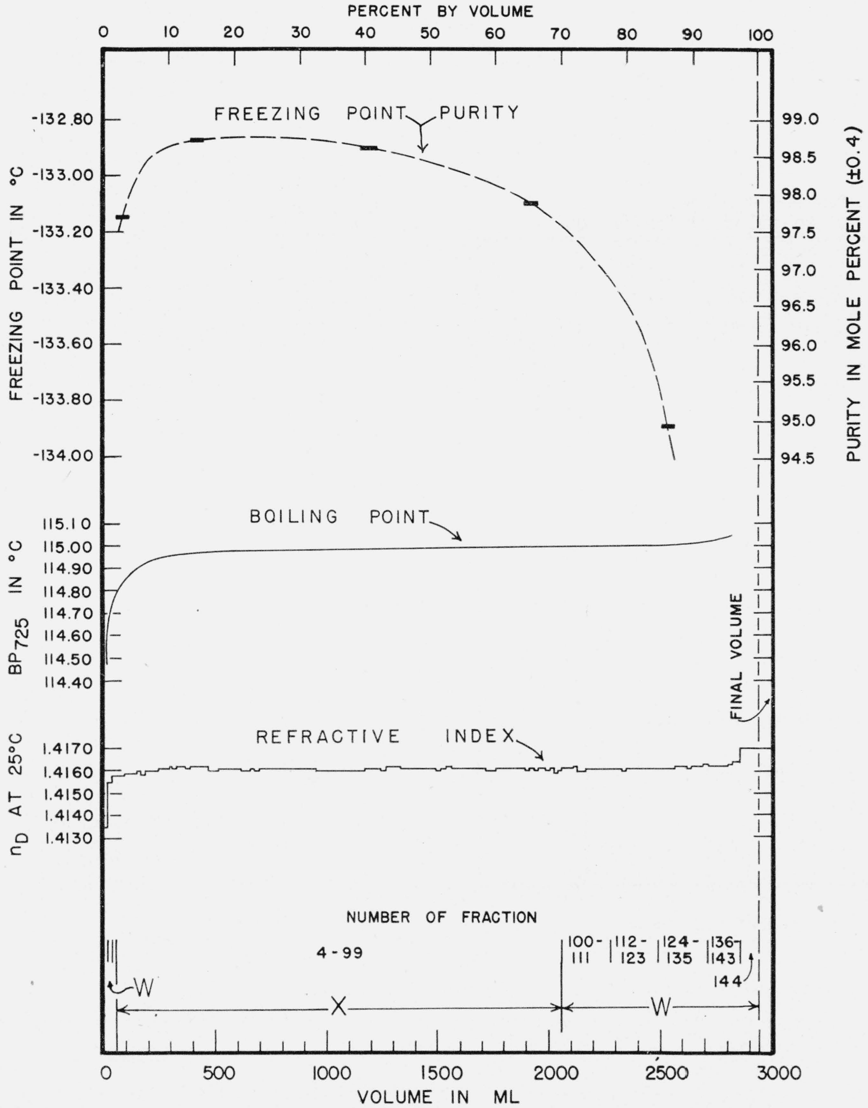

FIGURE 39.—*Results of the first distillation of cis, cis, trans-1,2,4-trimethylcyclopentane.*

Regular distillation at 725 mm Hg in still 4 (12/5/44 to 1/5/45).

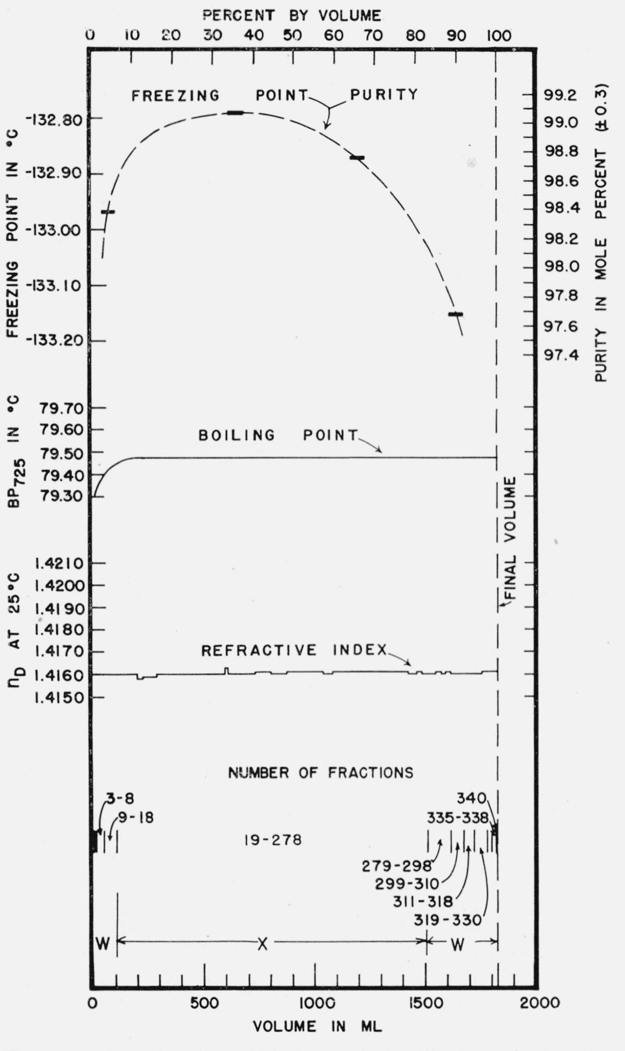

FIGURE 40.—*Results of the second distillation of cis, cis, trans-1,2,4-trimethylcyclopentane.*

Azeotropic distillation with isopropanol at 725 mm Hg in still 11 (6/21/45 to 8/21/45).

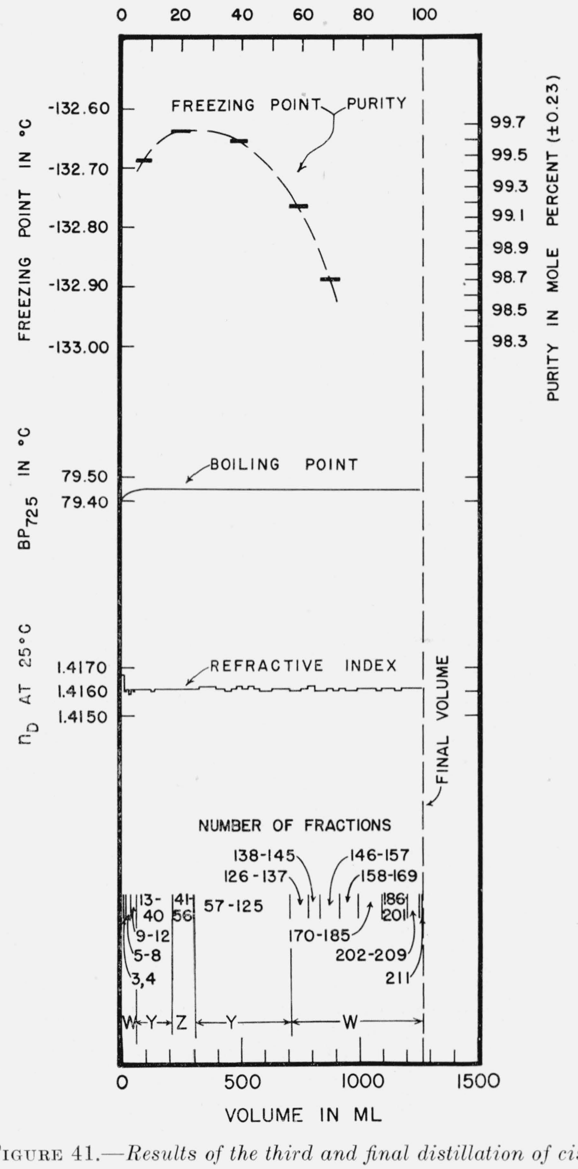

FIGURE 41.—*Results of the third and final distillation of cis, cis, trans-1,2,4-trimethylcyclopentane.*

Azeotropic distillation with isopropanol at 725 mm Hg in still 4 (11/21/45 to 1/3/46).

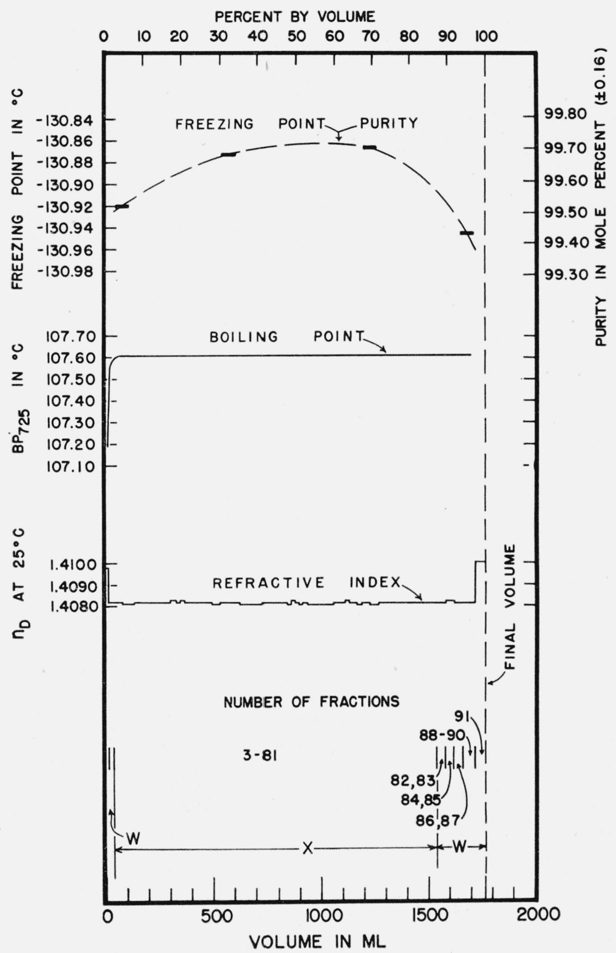

FIGURE 42.—*Results of the first distillation of cis, trans, cis-1,2,4-trimethylcyclopentane.*

Regular distillation at 725 mm Hg in still 2 (7/19/45 to 9/2/45).

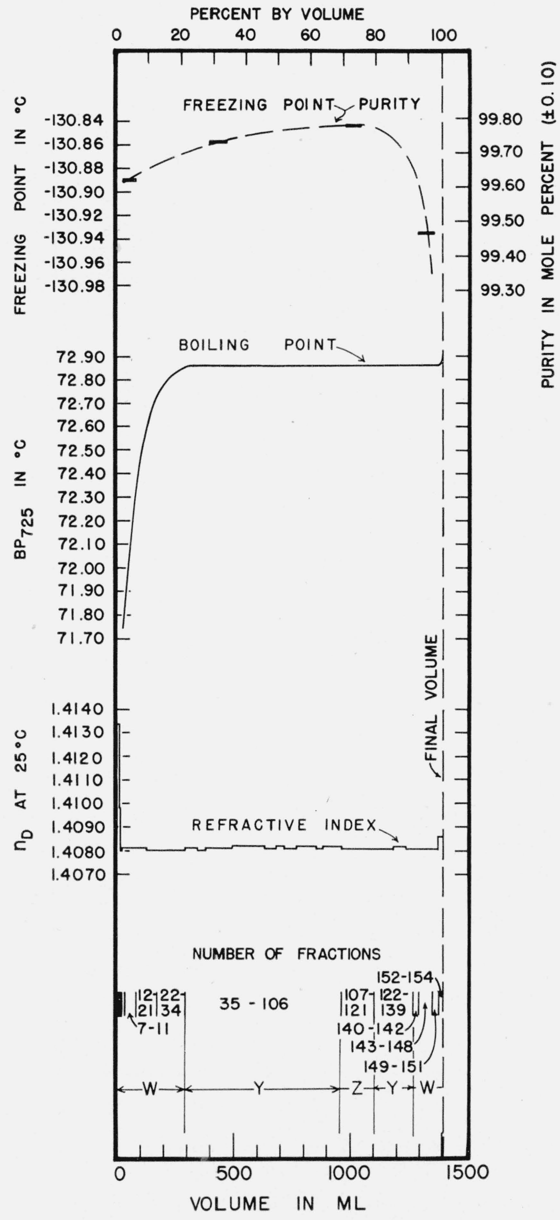

FIGURE 43.—*Results of the second and final distillation of cis, trans, cis-1,2,4-trimethylcyclopentane.*

Azeotropic distillation with ethanol at 725 mm Hg in still 9 (10/26/45 to 11/23/45).

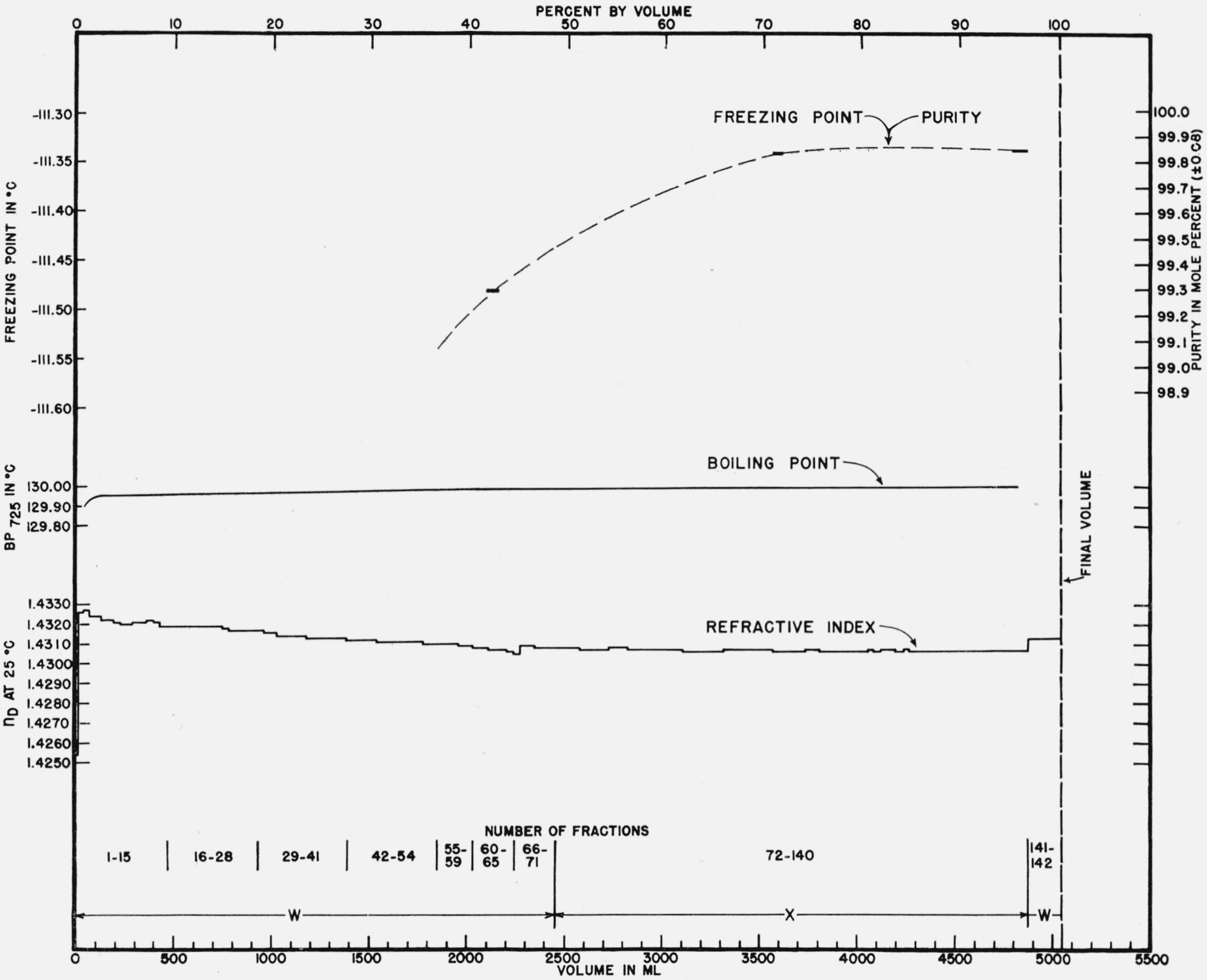

FIGURE 44.—*Results of the first distillation of ethylcyclohexane.*

Regular distillation at 725 mm Hg in still 8 (7/20/44 to 8/15/44).

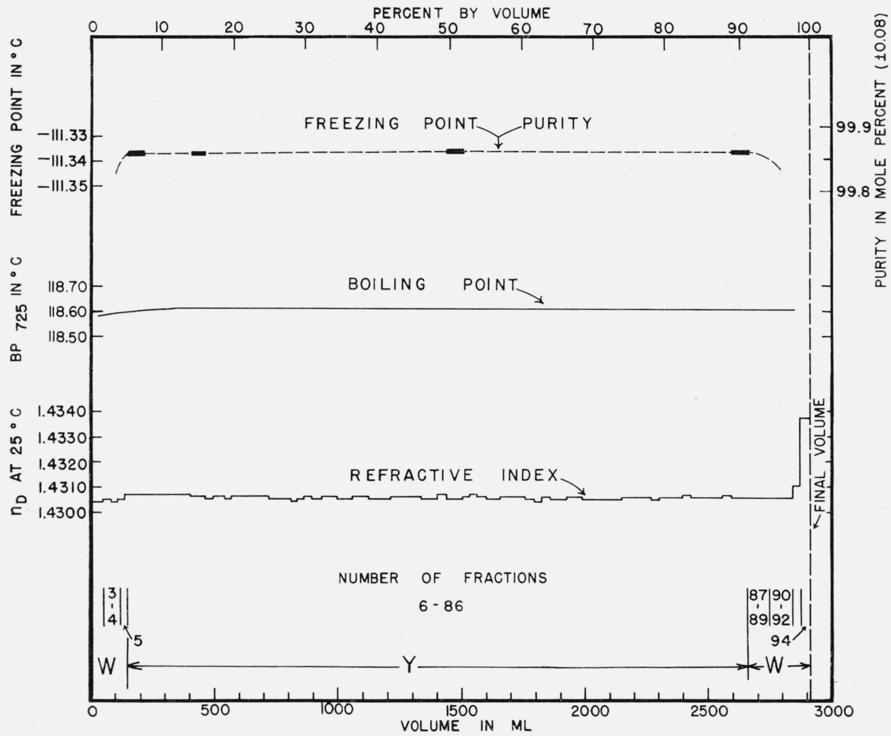

FIGURE 45.—*Results of the second and final distillation of ethylcyclohexane.*

Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in still 15 (12/16/44 to 1/3/45). See table 1 for composition of the charge for this distillation.

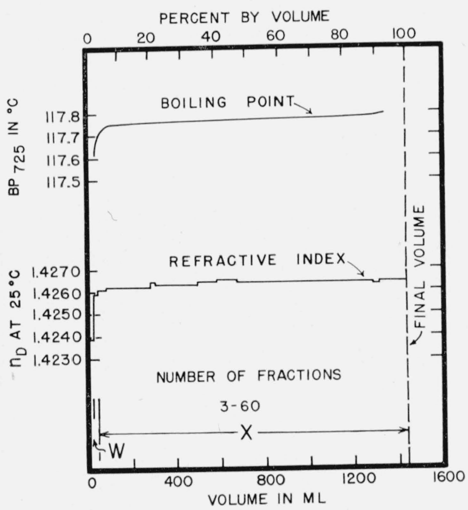

FIGURE 46.—*Results of the first distillation of 1,1-dimethylcyclohexane.*

Regular distillation at 725 mm Hg in still 12 (2/22/45 to 3/11/45).

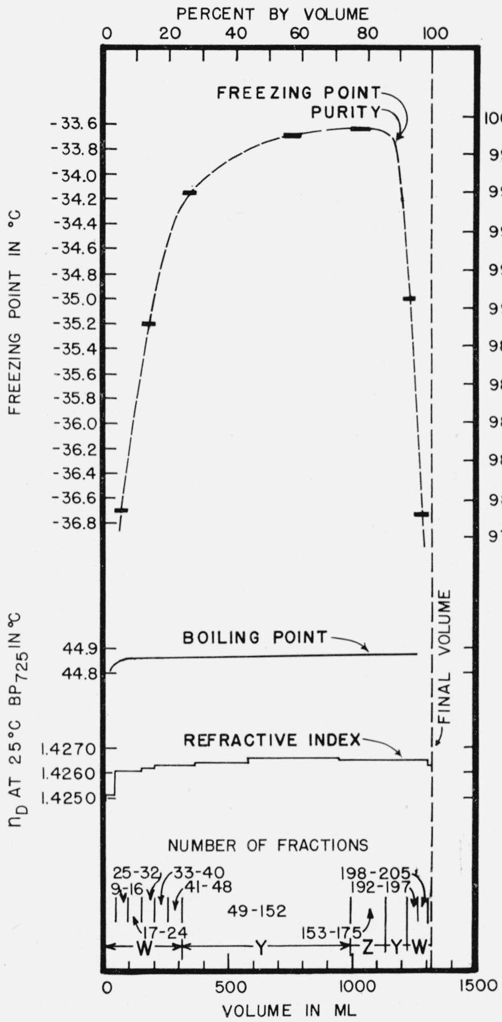

FIGURE 47.—*Results of the second and final distillation of 1,1-dimethylcyclohexane.*

Azeotropic distillation with ethanol at 725 mm Hg in still 9 (3/30/45 to 5/7/45).

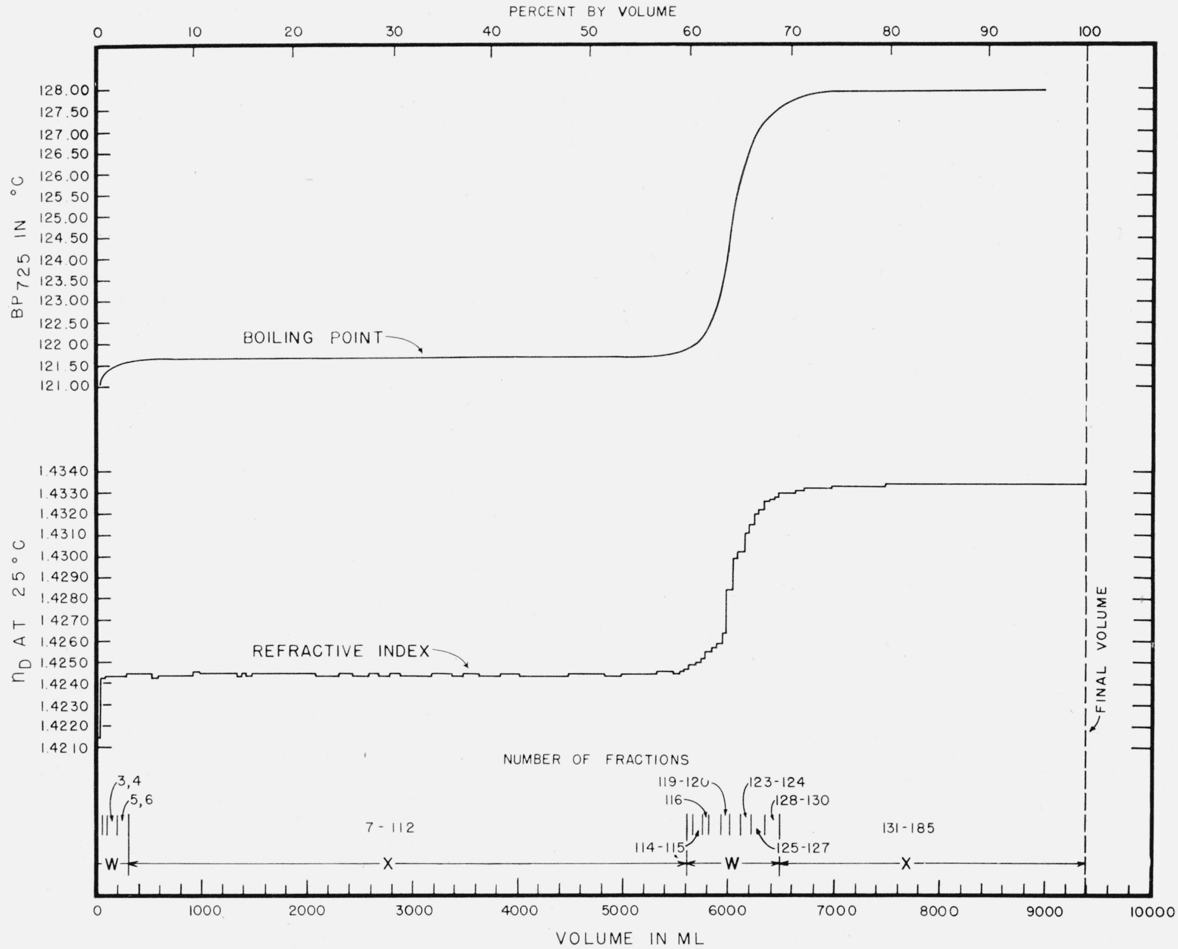

FIGURE 48.—*Results of the first distillation of cis- and trans-1,2-dimethylcyclohexane.*

Regular distillation at 725 mm Hg in still 5 (1/15/45 to 2/16/45).

Fractions 7 to 112 were redistilled to obtain *trans*-1, 2-dimethylcyclohexane (see fig. 50). Fractions 131 to 185 were redistilled to obtain *cis*-1,2-dimethylcyclohexane (see fig. 49).

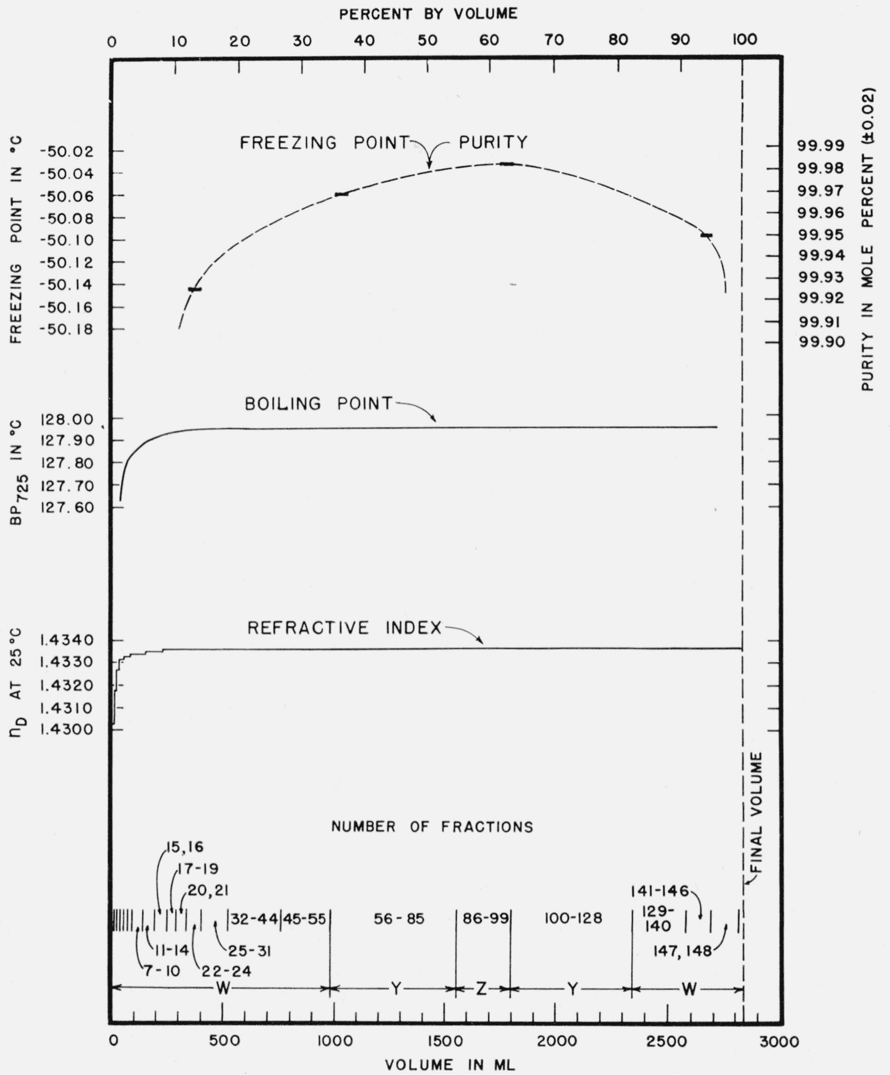

FIGURE 49.—*Results of the second and final distillation of cis-1,2-dimethylcyclohexane.*

Regular distillation at 725 mm Hg in still 9 (5/7/45 to 6/4/45).

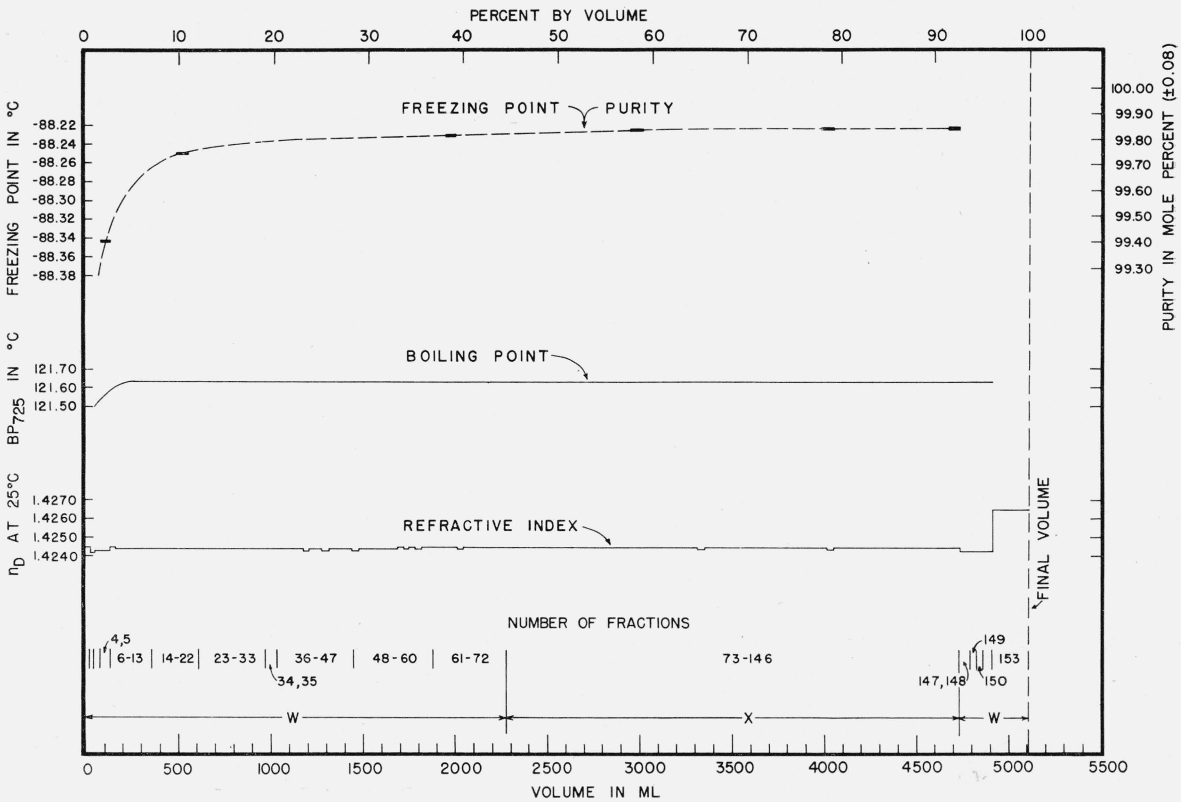

FIGURE 50.—*Results of the second distillation of trans-1,2-dimethylcyclohexane.*

Regular distillation at 725 mm Hg in still 7 (3/21/45 to 4/23/45).

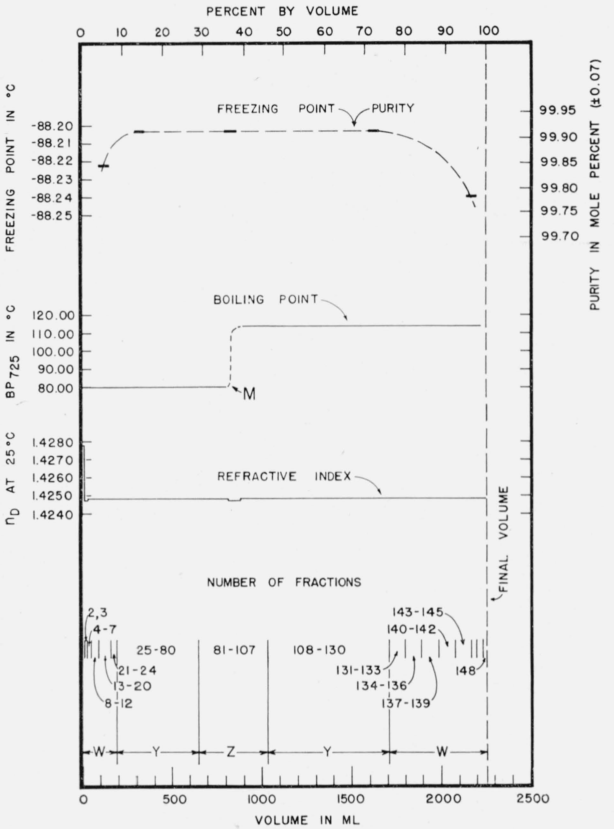

FIGURE 51.—*Results of the third and final distillation of trans-1,2-dimethylcyclohexane.*

Azeotropic distillation with isopropanol and ethylene glycol monoethyl ether at 725 mm Hg in still 13 (9/5/45 to 10/11/45).
The distillate preceding the point marked "M" was distilled with isopropanol as the azeotrope-forming substance, and the remainder of the hydrocarbon was distilled with ethylene glycol monoethyl ether (see footnote *t* of table 1).

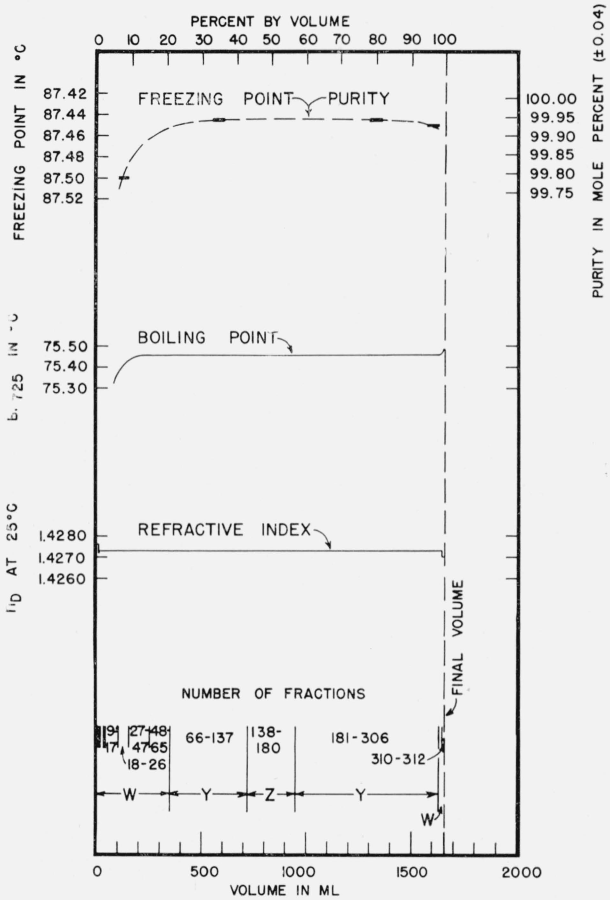

FIGURE 52.—*Results of the first and only distillation of cis-1,4-dimethylcyclohexane.*

Azeotropic distillation with ethanol at 725 mm Hg in still 9 (1/8/46 to 3/7/46).

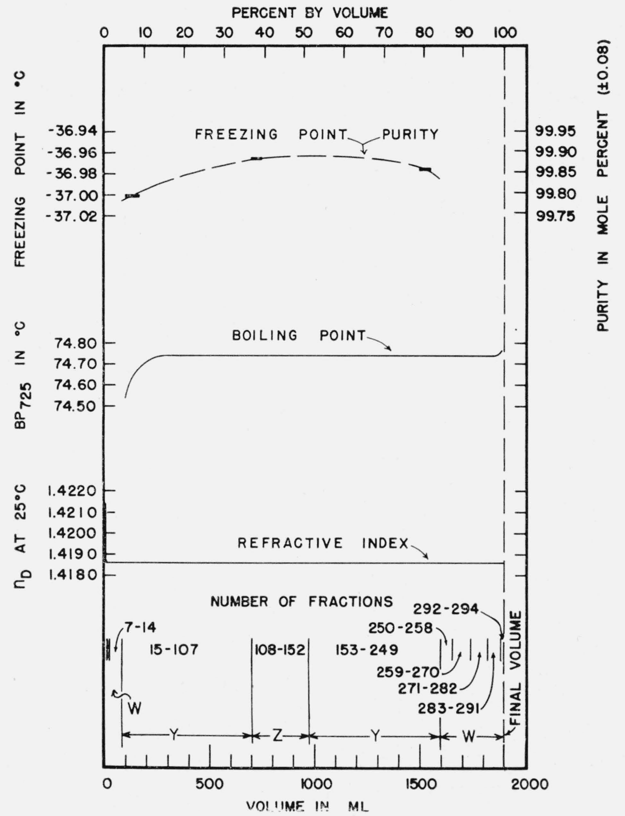

FIGURE 53.—*Results of the first and only distillation of trans-1,4-dimethylcyclohexane.*

Azeotropic distillation with ethanol at 725 mm Hg in still 10 (1/5/46 to 2/28/46).

WASHINGTON, June 15, 1946.