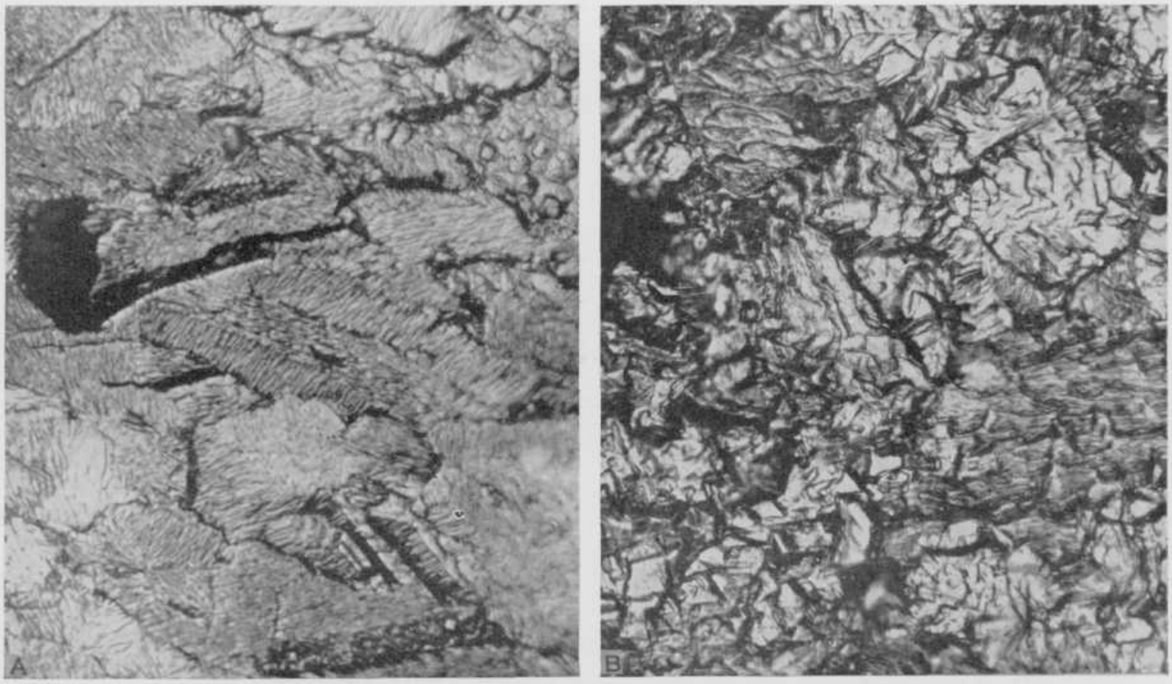

FIGURE 27. *Effect of testing conditions on structure of copper initially as annealed.*

Longitudinal sections etched in 3.5 parts glacial acetic acid and 4.5 parts nitric acid (conc.), and 2 parts absolute alcohol, ×750.

	Test		Remarks
	Tempera-ture	Strain rate	
	° F	*%/1,000 hr*	
A	300	36.4	Structure near axis of specimen 0.10 in. from position of complete fracture.
B	300	8.3	Do.

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

By Anton J. Streiff,[2 3] Laurel F. Soule,[2] Charlotte M. Kennedy,[2] M. Elizabeth Janes,[2 3] Vincent A. Sedlak,[2] Charles B. Willingham,[4] and Frederick D. Rossini[3]

This report describes the purification and determination of freezing points and purity of the following 29 hydrocarbons of the API-Standard and API-NBS series: 2,2,4,6,6-pentamethylheptane; 1,1,2-trimethylcyclopropane; *cis*-2-hexene; *cis*-3-hexene; 2-methyl-1-pentene; 4-methyl-1-pentene; 3-methyl-trans-2-pentene; 4-methyl-*cis*-2-pentene; 4-methyl *trans*-2-pentene; 4,4-dimethyl-1-pentene; 4,4-dimethyl-*trans*-2-pentene; 2,3,3-trimethyl-1-butene; *trans*-4-octene; 1-nonene; 1-decene; 1-undecene; 1,3-butadiene;1,2-pentadiene; 1,*cis*-3-pentadiene; 1,*trans*-3-pentadiene; 1,4-pentadiene; 2,3-pentadiene; 2-methyl-1,3-butadiene (isoprene); 1,5-hexadiene; 2,3-dimethyl-1,3-butadiene; 4-ethenyl-1-cyclohexene-(4-vinyl-1-cyclohexene); *cis*-decahydronaphthalene; *trans*-decahydronaphthalene; 2,3-dihydroindene (indan).

[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.
[3] Present address: Carnegie Institute of Technology, Pittsburgh, Pa.
[4] Present address: Mellon Institute of Industrial Research, Pittsburgh, Pa.

I. Introduction

Previous reports described the purification and determination of freezing points and purity of 114 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 [1,2,3,4,5].[5]

This report describes the purification and determination of freezing points and purity of an additional 29 hydrocarbons comprising 14 monoolefins, 9 diolefins, 2,2,4,6,6-pentamethylheptane, 1,1,2-trimethylcyclopropane, 4-ethenyl-1-cyclohexene (4-vinyl-1-cyclohexene), *cis*-decahydronaphthalene, *trans*-decahydronaphthalene, and 2,3-dihydroindene (indan).

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, on loan to qualified investigators for the measurement of properties.

II. Materials[6]

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: 1,1,2-trimethylcyclopropane; *cis*-2-hexene; *cis*-3-hexene; 4-methyl-1-pentene; 3-methyl-*trans*-2-pentene; 4,4-dimethyl-1-pentene; 4,4-dimethyl-*trans*-2-pentene; *trans*-4-octene; 1-nonene; 1-undecene; 1,4-pentadiene; 2,3-dihydroindene (indan).

By the Hydrocarbon Laboratory at the Pennsylvania State College, State College, Pa., under the supervision of F. C. Whitmore: 1,2-pentadiene; 2,3-pentadiene; 1,5-hexadiene; 2,3-dimethyl-1,3-butadiene.

By the Anglo-Iranian Oil Co., Ltd., Research Station, Sunbury-on-Thames, Eng., through S. F. Birch: 2,2,4,6,6-pentamethylheptane.

By the General Motors Corporation Research Laboratories, Detroit, Mich., through T. A. Boyd and W. G. Lovell: 2,3,3-trimethyl-1-butene.

By the Phillips Petroleum Co., Bartlesville, Okla., through F. E. Frey: 1,3-butadiene.

By the Office of Rubber Reserve, Washington, D. C.: 1,*cis*-3-pentadiene; 1,*trans*-3-pentadiene.

By purchase: 2-methyl-1-pentene; 4-methyl-*cis*-2-pentene; 4-methyl-*trans*-2-pentene; 1-decene; 2-methyl-1,3-butadiene (isoprene); 4-ethenyl-1-cyclohexene (4-vinyl-1-cyclohexene); *cis*-decahydronaphthalene; *trans*-decahydronaphthalene.

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

[5] Figures in brackets indicate the literature references at the end of this paper.
[6] The naming of the *cis* and *trans* isomers of the olefins listed below is described by Epstein and Rossini, Chem. Eng. News **26**, 2959 (1948).

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

Compound	Laboratory [a] providing starting material	Hydrocarbon charged for distillation		Distillation [b]									Volume of selected sample	
		Volume	Purity	Kind [c]	Azeotrope-forming substance [d]	Amount of hydrocarbon in the azeotropic distillate [e]	Distilling column number [b]	Number of theoretical plates [b] (approx.)	Reflux ratio [b] (approx.)	Rate of collection of distillate	Results plotted in figure	API Standard	API-NBS
		Liters	*Mole %*			*% by volume*				*ml/hour*		*ml*	*ml*
2,2,4,6,6-Pentamethylheptane	Anglo-Iranian	8.90	98.56 ±0.10	Reg			6	125	125/1	12.5	1		
		5.38	99.73 ±0.06	Reg			2A	200	160/1	4.5	2		
		3.75	99.76 ±0.06	Azeo	Me Carb.	76	12A	200	160/1	4.5	3	1,330	355
1,1,2-Trimethylcyclopropane	APIRP45	[f] 3.73	99.17 ±0.14	Reg			12A	200	160/1	4.5	4		
		0.63	99.62 ±0.12	Azeo	Acetone	67	13	130	145/1	8.5	5		300
cis-2-Hexene	APIRP45	3.71		Reg			4	200	160/1	4.5	6		
		1.85		Azeo	Ethanol	78	4	200	160/1	4.5	7	680	155
cis-3-Hexene	APIRP45	5.05	89.8 ±0.3	Reg			10A	200	200/1	6.0	8		
		2.48	98.70 ±0.16	Azeo	Methanol	73	15A	200	160/1	4.5	9		
		1.43	99.24 ±0.14	Reg			4	200	200/1	2.0	10	800	170
2-Methyl-1-pentene	APIRP6 [g]	[h] 6.20	79. ±5.	Reg			9	135	165/1	4.5	11		
		[i] 6.20	98.75 ±0.15	Reg			4	200	160/1	4.5	12	1,150	350
4-Methyl-1-pentene	APIRP45	[h] 4.50		Reg			13	130	145/1	8.5	13		
		[j] 3.98		Reg			3A	200	160/1	4.5	14	1,150	410
3-Methyl-*trans*-2-pentene	APIRP45	[h] 4.70		Reg			8	130	145/1	8.5	15		
		[j] 3.31		Reg			2A	200	160/1	4.5	16		
		1.76	99.45 ±0.12	Azeo	Ethanol	79	12A	200	160/1	4.5	17	750	175
4-Methyl-*cis*-2-pentene	APIR6 [g]	[j] 6.20		Reg			13	130	145/1	8.5	18		
		[k] 5.45		Reg			15A	200	160/1	4.5	19	1,350	250
4-Methyl-*trans*-2-pentene	APIR6 [g]	[j] 6.20		Reg			13	130	145/1	8.5	18		
		[k] 2.93		Reg			11A	200	160/1	4.5	20		
		1.66	99.55 ±0.09	Azeo	Ethanol	86	3A	200	160/1	4.5	21	770	200

See footnotes at end of table

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

Compound	Laboratory [a] providing starting material	Hydrocarbon charged for distillation		Distillation [b]								Volume of selected sample	
		Volume	Purity	Kind [c]	Azeotrope-forming substance [d]	Amount of hydrocarbon in the azeotropic distillate [e]	Distilling column number [b]	Number of theoretical plates [b] (approx.)	Reflux ratio [b] (approx.)	Rate of collection of distillate	Results plotted in figure	API Standard	API–NBS
		Liters	*Mole %*			*% by volume*				*ml/hour*		*ml*	*ml*
4,4-Dimethyl-1-pentene	APIRP45	6.90	99.34 ±0.12	Reg.			5	125	125/1	12.5	22		
		3.01	99.79 ±0.08	Reg.			11A	200	160/1	4.5	23	1,060	250
4,4-Dimethyl-*trans*-2-pentene	APIRP45	4.98	99.43 ±0.06	Reg.			12A	200	160/1	4.5	24	1,075	300
2,3,3-Trimethyl-1-butene	General Motors	15.00	98.64 ±0.12	Reg.			6	125	125/1	12.5	25		
		6.20	99.93 ±0.04	Reg.			11A	200	160/1	4.5	26	1,550	500
trans-4-Octene	APIRP45	4.66	93.8 ±0.2	Reg.			3A	200	160/1	4.5	27	1,115	350
1-Nonene	APIRP45	2.50	98.59 ±0.24	Reg.			4	200	160/1	4.5	28		
		2.06	99.70 ±0.18	Azeo.	Cell.	52	2A	200	160/1	4.5	29	1,150	355
1-Decene	APIRP6 [g]	12.00		Reg.			6	125	125/1	12.5	30		
		5.65	99.76 ±0.07	Reg.			12A	200	160/1	4.5	31	1,695	500
1-Undecene	APIRP45	4.10	99.71 ±0.08	Reg.			13	130	145/1	8.5	32	1,300	360
1,3-Butadiene	Phillips	2.90	99.83 ±0.06	Reg.			1A	150	120/1	2.25	33	1,860	440
1,2-Pentadiene	Penn State	1.66	95.7 ±0.3	Reg.			11A	200	180/1	4.0	34	470	150
1,*cis*-3-Pentadiene	Rubber Reserve	[l] 5.10		Reg.			9	135	185/1	4.0	35		
		[m] 4.57		Azeo.	Methanol	84	4	200	160/1	4.5	36	1,075	290
1,*trans*-3-Pentadiene	Rubber Reserve	[l] 5.10		Reg.			9	135	185/1	4.0	35		
		[m] 4.30		Azeo.	Methanol	85	4	200	160/1	4.5	37	1,050	220
1,4-Pentadiene	APIRP45	2.03	99.80 ±0.06	Reg.			1A	150	170/1	1.5	38	1,050	255
2,3-Pentadiene	Penn State	2.60	91.2 ±0.1	Reg.			9	135	185/1	4.0	39	950	285
2-Methyl-1,3-butadiene (Isoprene)	APIRP6 [n]	[h] 6.00	93.7 ±0.3	Reg.			13	130	145/1	8.5	40		
		[i] 4.16	99.87 ±0.06	Reg.			2A	200	160/1	4.5	41	1,900	400
1,5-Hexadiene	Penn State	1.94	99.84 ±0.08	Reg.			13	130	145/1	8.5	42	1,050	310
2,3-Dimethyl-1,3-butadiene	Penn State	2.30	98.17 ±0.10	Reg.			4	200	160/1	4.5	43	960	250
4-Ethenyl-1-cyclohexene (4-Vinyl-1-cyclohexene)	APIRP6 [o]	[p] 5.80		Reg.			14	125	125/1	12.5	44		
		[q] 5.80	99.84 ±0.07	Reg.			4	200	160/1	4.5	45	1,750	490
cis-Decahydronaphthalene	APIRP6 [r]	[s] 5.30		Reg.			7	130	145/1	8.5	46		
		[t] 5.30		Reg.			11A	200	160/1	4.5	47	1,100	300
trans-Decahydronaphthalene	APIRP6 [r]	[s] 5.30		Reg.			7	130	145/1	8.5	46		
		[t] 5.12		Reg.			11A	200	160/1	4.5	48	1,180	270
2,3-Dihydroindene (Indan)	APIRP45	2.67		Reg.			12	135	185/1	4.0	49		
		2.25	99.67 ±0.04	Reg.			15A	200	160/1	4.5	50	1,050	200

[a] The abbreviations represent the following laboratories APIRP45; American Petroleum Institute Research Project 45 at the Ohio State University, Columbus, Ohio. Penn State; Hydrocarbon Laboratory, Pennsylvania State College, State College, Pa. Anglo-Iranian; Anglo-Iranian Oil Co., Research Laboratories, Sunbury-on-Thames, England. General Motors; General Motors Corporation, Detroit, Mich. Phillips; Phillips Petroleum Co., Bartlesville, Okla. Rubber Reserve; Office of Rubber Reserve, Washington, D. C. APIRP6; American Petroleum Institute Research Project 6 at the National Bureau of Standards, Washington, D. C.

[b] See Reference [6] for further details.

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

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

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

[f] After 28 days of distillation of this material, the remaining hydrocarbon was converted to higher boiling isomers, and approximately 20% of the charge was not recoverable as 1,1,2-trimethylcyclopropane

[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] One of two charges of similar material, one of which was 6.2 liters, and the other of which was 15.0 liters. Both 4-methyl-*cis*-2-pentene and 4-methyl-*trans*-2-pentene were obtained from this material (see fig. 18).

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

[l] One of two similar charges. Both 1,*cis*-3-pentadiene and 1,*trans*-3-pentadiene were obtained from this material (see fig. 35).

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

[n] Obtained by purchase of commercially available material from Newport Industries, Inc., Pensacola, Fla.

[o] Obtained by purchase of commercially available material from Koppers Co., Inc., Pittsburgh, Pa.

[p] One of three similar charges.

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

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

[s] One of three similar charges. Both *cis*- and *trans*-decahydronaphthalene were obtained from this material (see fig. 46).

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

III. Purification

The procedures followed in the process of purification and determination of purity were the same as those described in the previous reports [2, 3, 4, 5].

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 [6].

Figures 1 to 50 inclusive show graphically the results of the distillation operations listed in table 1. These figures give each of the following properties 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 725 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; and Z, used for the API–NBS material.

As demonstrated in the previous reports [2, 3, 4, 5], 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.

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 a suitable impurity [7]; and the resulting calculated amount of impurity in the API–Standard and API–NBS material.

TABLE 2. *Freezing points and purity of 29 API-Standard and API-NBS hydrocarbons*

Compound	Kind of time-temperature observations used to determine the freezing point [a]	Freezing point of the actual selected sample in air at 1 atm		Freezing point for zero impurity in air at 1 atm	Cryoscopic constant [a] A	Calculated amount of impurity in the actual selected sample [b]	
		API-Standard	API-NBS			API-Standard	API-NBS
		° C	*° C*	*° C*	*Mole fraction/deg*	*Mole %*	*Mole %*
2,2,4,6,6-Pentamethylheptane	M	−66.967	−66.967	−66.92 ±0.03	0.0129	0.06 ±0.04	0.06 ±0.04
1,1,2-Trimethylcyclopropane	M	....	−138.209	−138.180 ±0.020	.0418	....	.12 ±0.08
cis-2-Hexene	M	−141.170	−141.146	−141.135 ±0.020	.0585	0.20 ±0.12	.06 ±0.05
cis-3-Hexene	F	−137.845	−137.839	−137.820 ±0.020	.0522	.13 ±0.08	.10 ±0.08
2-Methyl-1-pentene	M	−135.803	−135.791	−135.760 ±0.020	.0449	.19 ±0.09	.14 ±0.09
4-Methyl-1-pentene [c]	F	−153.69(I)	−153.68(I)	−153.63 ±0.04(I) −154.90 ±0.04(II)(u)	.03	.18 ±0.12	.15 ±0.12
3-Methyl-*trans*-2-pentene	M	−134.872	−134.866	−134.840 ±0.020	.0425	.14 ±0.09	.11 ±0.09
4-Methyl-*cis*-2-pentene	M	−134.447	−134.446	−134.430 ±0.015	.0461	.08 ±0.07	.07 ±0.07
4-Methyl-*trans*-2-pentene	M	−140.860	−140.826	−140.810 ±0.015	.0494	.25 ±0.07	.08 ±0.07
4,4-Dimethyl-1-pentene	F and M	−136.635	−136.635	−136.600 ±0.020	.0417	.15 ±0.08	.15 ±0.08
4,4-Dimethyl-*trans*-2-pentene	M	−115.263	−115.262	−115.235 ±0.010	.0327	.09 ±0.03	.09 ±0.03
2,3,3-Trimethyl-1-butene	F	−110.010	−109.994	−109.85 ±0.10	.00367	.06 ±0.04	.05 ±0.04
trans-4-Octene	M	−93.838	−93.838	−93.810 ±0.020	.0571	.16 ±0.11	.16 ±0.11
1-Nonene	M	−81.41	−81.41	−81.37 ±0.03	.0591	.24 ±0.18	.24 ±0.18
1-Decene	F	−66.329	−66.326	−66.310 ±0.020	.0594	.11 ±0.07	.09 ±0.07
1-Undecene	F	−49.206	−49.206	−49.185 ±0.020	.0421	.09 ±0.08	.09 ±0.08
1,3-Butadiene	F	−108.939	−108.937	−108.915 ±0.010	[d] .03560	.08 ±0.04	.08 ±0.04
1,2-Pentadiene	M	−137.326	−137.301	−137.26 ±0.03	.0612	.34 ±0.15	.21 ±0.15
1,*cis*-3-Pentadiene	F	−140.840	−140.836	−140.820 ±0.010	.0414	.08 ±0.04	.07 ±0.04
1,*trans*-3-Pentadiene	F	−87.501	−87.483	−87.470 ±0.010	.0264	.08 ±0.03	.03 ±0.03
1,4-Pentadiene	M	−148.288	−148.287	−148.275 ±0.010	.0521	.07 ±0.05	.06 ±0.05
2,3-Pentadiene	M	−125.690	−125.680	−125.650 ±0.020	.0384	.15 ±0.07	.11 ±0.07
2-Methyl-1,3-butadiene (isoprene)	M	−145.962	−145.962	−145.950 ±0.020	[d] .0330	.04 ±0.03	.04 ±0.03
1,5-Hexadiene	M	−140.702	−140.698	−140.680 ±0.015	.0505	.11 ±0.08	.09 ±0.08
2,3-Dimethyl-1,3-butadiene	M	−76.026	−76.020	−76.005 ±0.010	.0274	.06 ±0.03	.04 ±0.03
4-Ethenyl-1-cyclohexene (4-Vinyl-1-cyclohexene)	M	−108.748	−108.745	−108.720 ±0.020	.0367	.10 ±0.07	.09 ±0.07
cis-Decahydronaphthalene	M	−43.070	−43.048	−43.01 ±0.03	.0180	.11 ±0.05	.07 ±0.05
trans-Decahydronaphthalene	M	−30.415	−30.410	−30.400 ±0.015	.0291	.04 ±0.03	.03 ±0.03
2,3-Dihydroindene (indan)	F	−51.430	−51.410	−51.400 ±0.010	.0204	.06 ±0.02	.02 ±0.02

[a] F indicates freezing, and M indicates melting. See reference [7] for experimental details and the definition of the cryoscopic constant.
[b] 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 preceeding columns.
[c] This hydrocarbon has more than one crystalline form. The two forms indicated are labeled I and II in order of decreasing temperature of fusion (or freezing point). Form II will be, at its freezing point, in metastable equilibrium with the undercooled liquid, but will be unstable with respect to transition to the other solid form at the same temperature and pressure (1 atm). This is indicated by "u" in parentheses following the roman numeral.
[d] Not determined in this investigation. See reference [8].

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

V. References

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[7] A. R. Glasgow, Jr., A. J. Streiff, and F. D. Rossini, J. Research NBS **35**, 355 (1945) RP1676.
[8] A. R. Glasgow, Jr., N. C. Krouskop, J. Beadle, G. D. Axilrod, and F. D. Rossini, Anal. Chem. **20**, 410 (1948).

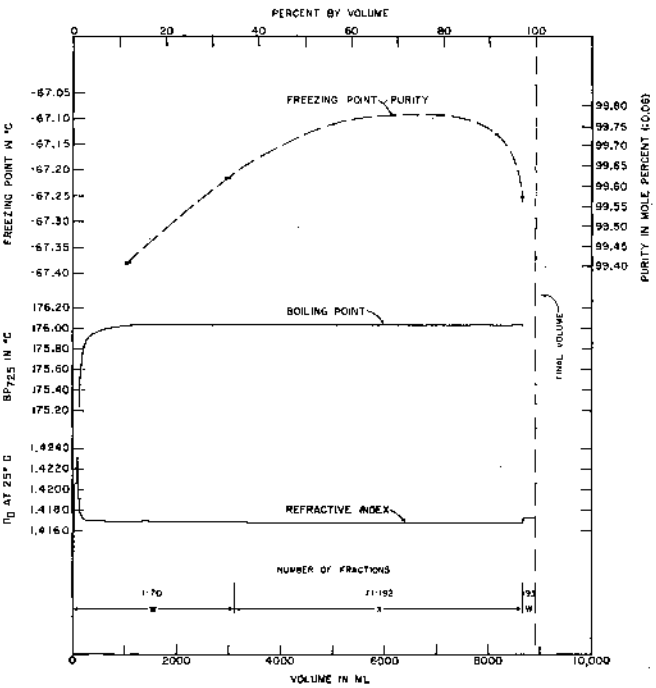

FIGURE 1. *First distillation of 2,2,4,6,6-pentamethylheptane.*

Regular distillation at 725 mm Hg in still 6(10/21/47 to 11/24/47).

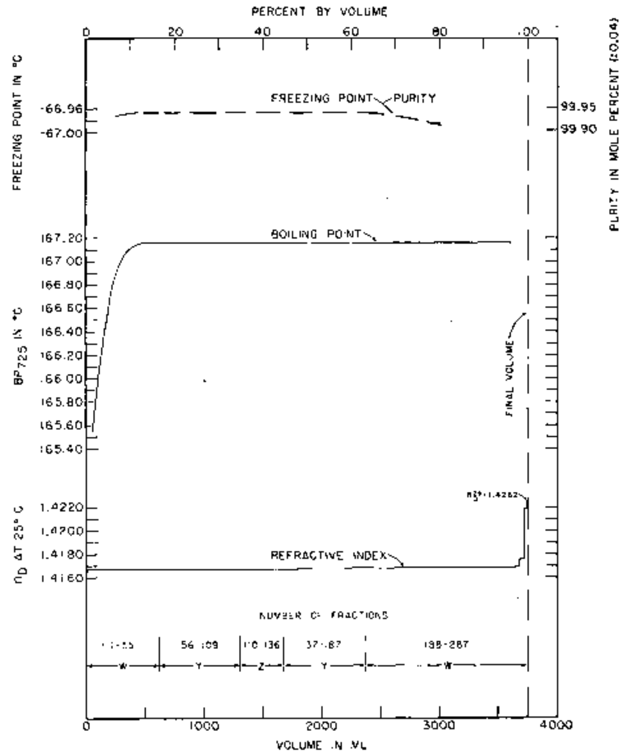

FIGURE 3. *Third and final distillation of 2,2,4,6,6-pentamethylheptane.*

Azeotropic distillation with diethylene glycol monomethyl ether at 725 mm Hg in still 12A (3/25/48 to 5/19/48).

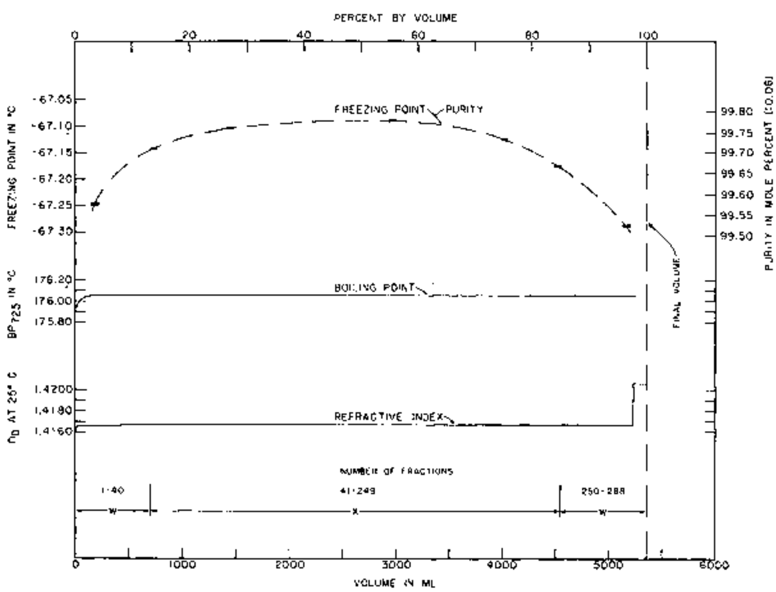

FIGURE 2. *Second distillation of 2,2,4,6,6-pentamethylheptane.*

Regular distillation at 725 mm Hg in still 2A(1/8/48 to 3/1/48).

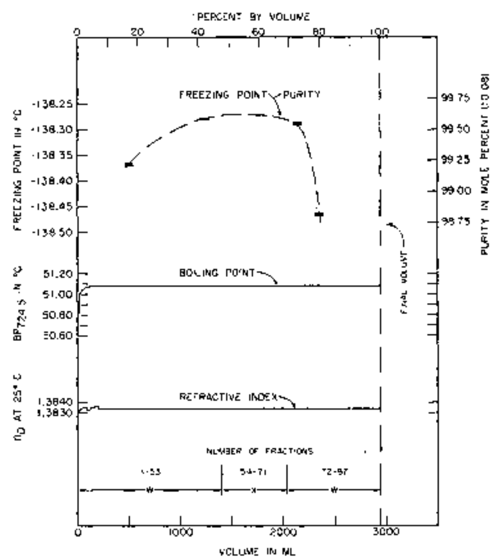

FIGURE 4. *First distillation of 1,1,2-trimethlcyclopropane.*

Regular distillation at 725 mm Hg in still 12A(7/18/47 to 8/16/47).

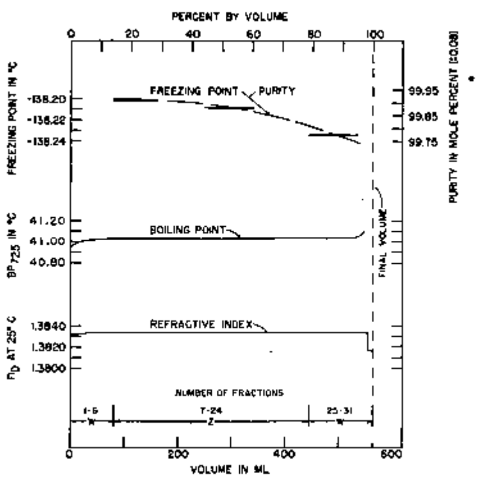

FIGURE 5. *Second and final distillation of 1,1,2-trimethylcyclopropane.*

Azeotropic distillation with acetone at 725 mm Hg in still 13(9/3/47 to 9/10/47).

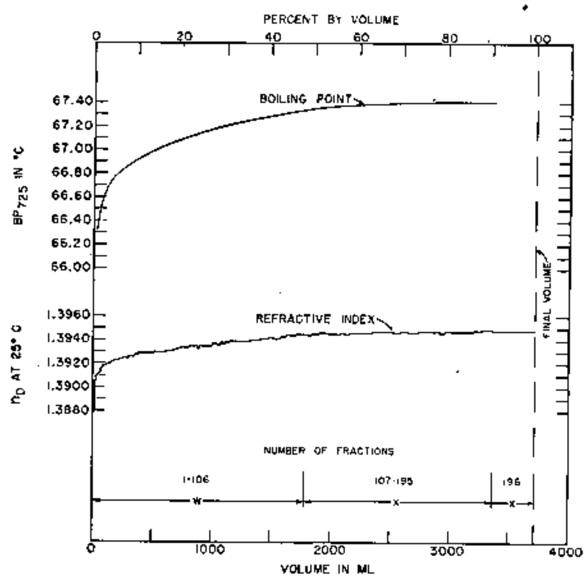

FIGURE 6. *First distillation of cis-2-hexene.*

Regular distillation at 725 mm Hg in still 4(12/24/48 to 1/27/49). The residue from the distillation (marked "x''") was azeotropically distilled with ethanol and the hydrocarbon portion added to fractions 107 to 195 for redistillation.

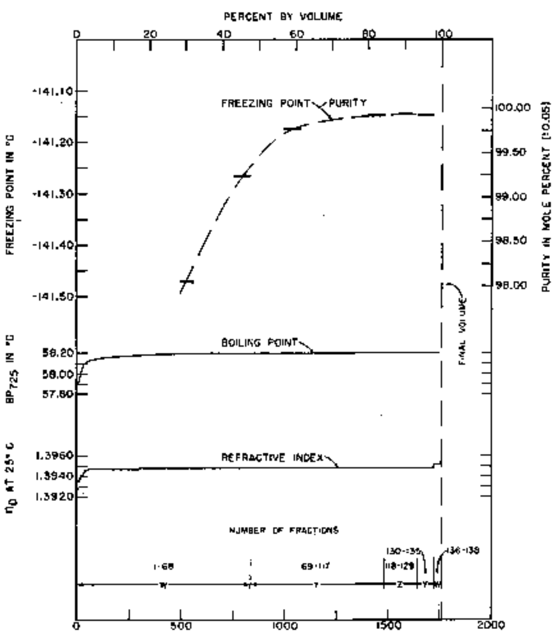

FIGURE 7. *Second and final distillation of cis-2-hexene.*

Azeotropic distillation with ethanol at 725 mm Hg in still 4(3/7/49 to 4/1/49).

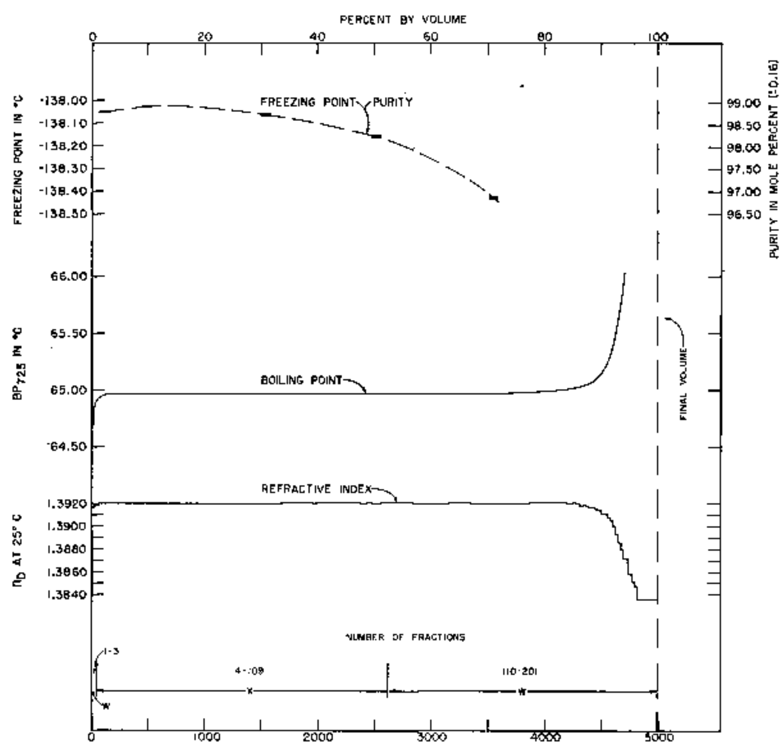

FIGURE 8. *First distillation of cis-3-hexene.*

Regular distillation at 725 mm Hg in still 10A(9/30/48 to 11/15/48).

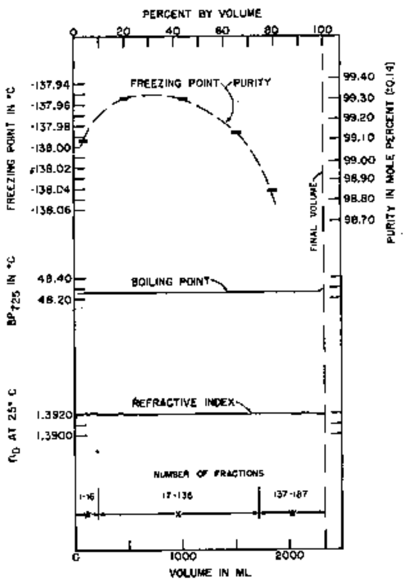

FIGURE 9. *Second distillation of cis-3-hexene.*

Azeotropic distillation with methanol at 725 mm Hg in still 15A (12/3/48 to 1/5/49).

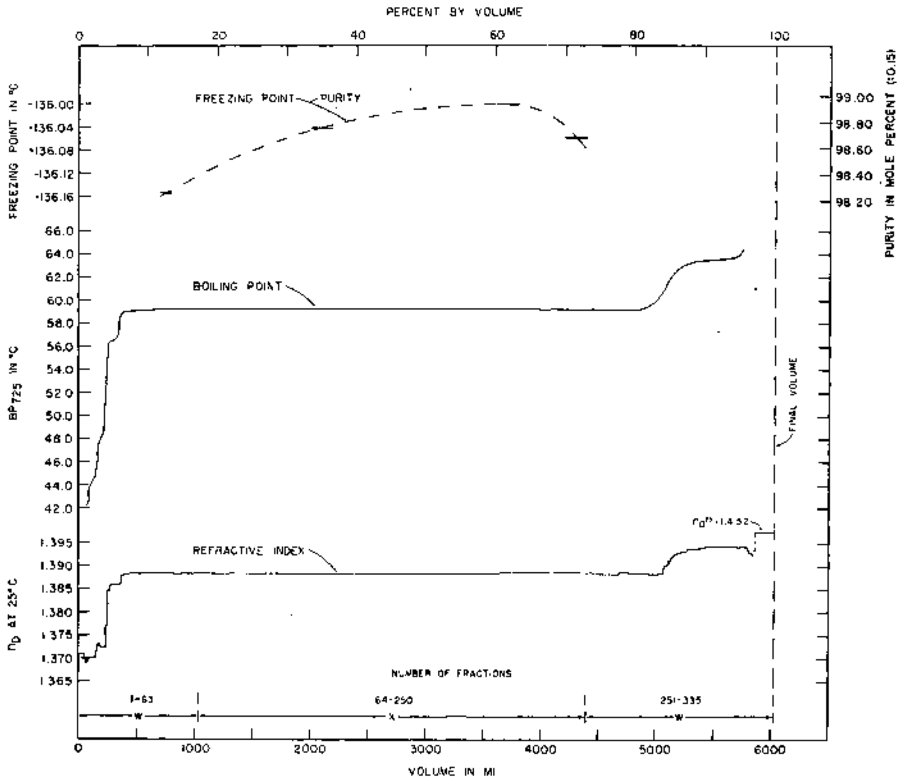

FIGURE 11. *First distillation of 2-methyl-1-pentene.*

Regular distillation at 725 mm Hg in still 9 (8/1/47 to 10/7/47). This is one of two similar distillations.

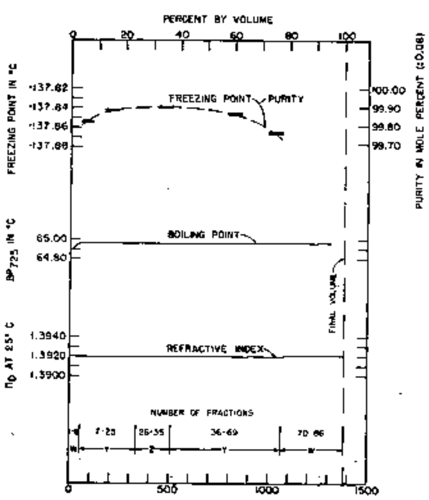

FIGURE 10. *Third and final distillation of cis-3-hexene.*

Regular distillation at 725 mm Hg in still 4 (2/4/49 to 3/4/49).

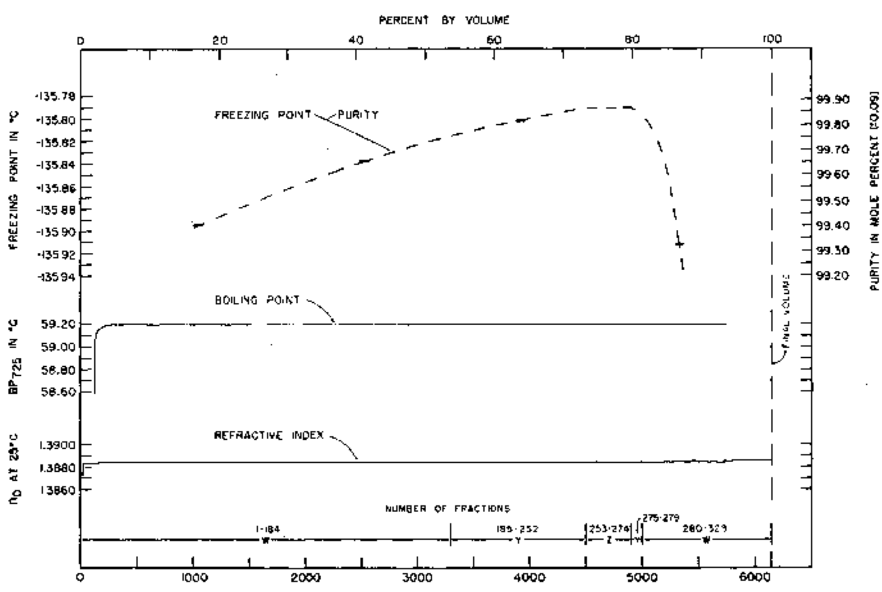

FIGURE 12. *Second and final distillation of 2-methyl-1-pentene.*

Regular distillation at 725 mm Hg in still 4 (1/2/48 to 3/1/48). See footnote i of table 1.

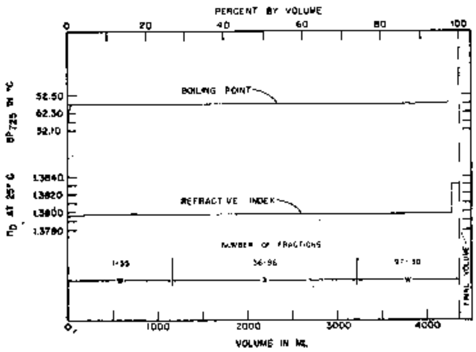

FIGURE 13. *First distillation of 4-methyl-1-pentene.*

Regular distillation at 725 mm Hg in still 13(4/4/47 to 4/28/47). One of two similar distillations.

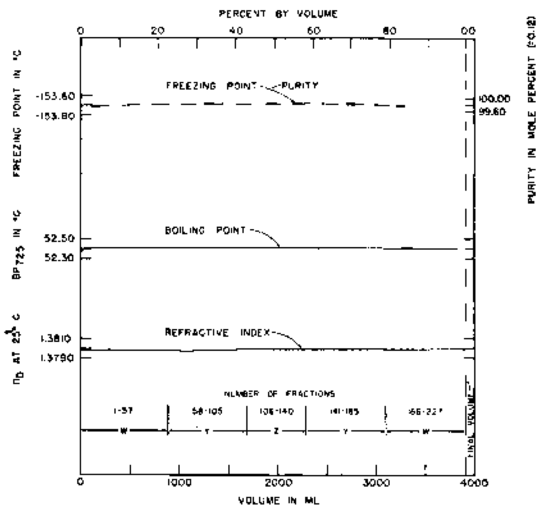

FIGURE 14. *Second and final distillation of 4-methyl-1-pentene.*

Regular distillation at 725 mm Hg in still 3A (5/27/47 to 7/7/47). See footnote i of table 1.

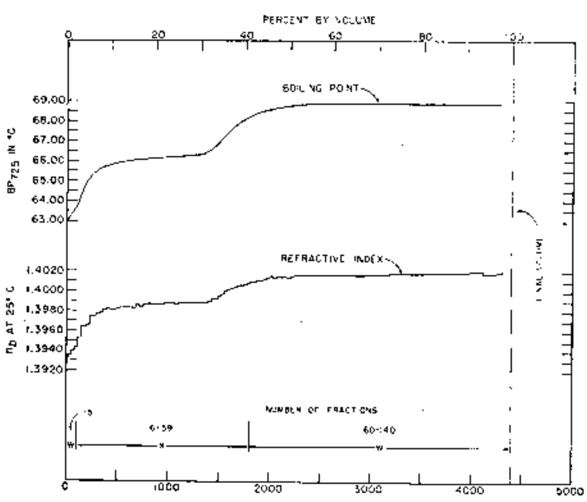

FIGURE 15. *First distillation of 3-methyl-trans-2-pentene.*

Regular distillation at 725 mm Hg in still 8(3/2/48 to 3/29/48). One of two similar distillations.

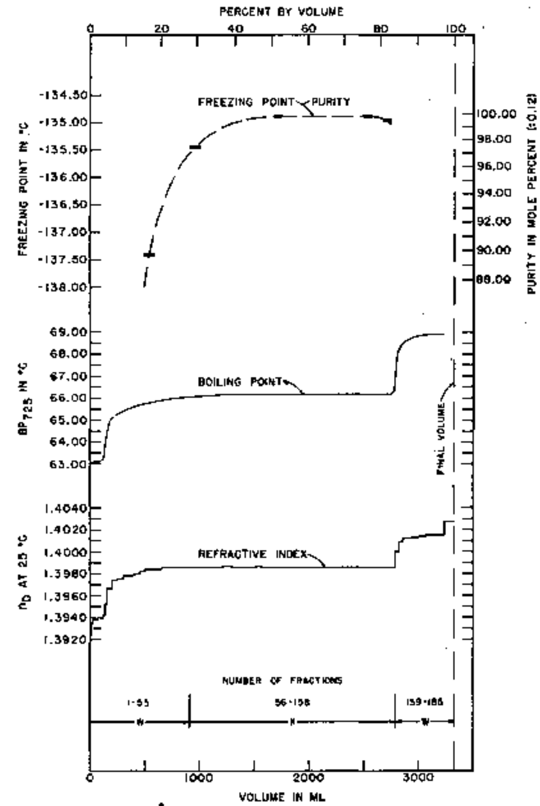

FIGURE 16. *Second distillation of 3-methyl-trans-2-pentene.*

Regular distillation at 725 mm Hg in still 2A (4/19/48 to 5/24/48). See footnote i of table 1.

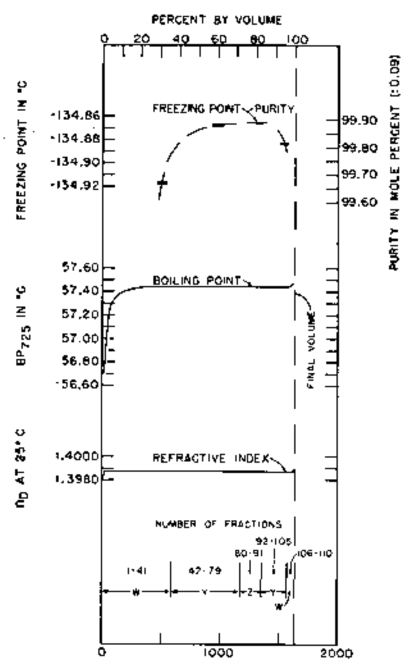

FIGURE 17. *Third and final distillation of 3-methyl-trans-2-pentene.*

Azeotropic distillation with ethanal at 725 mm Hg in still 12A (7/21/48 to 8/10/48).

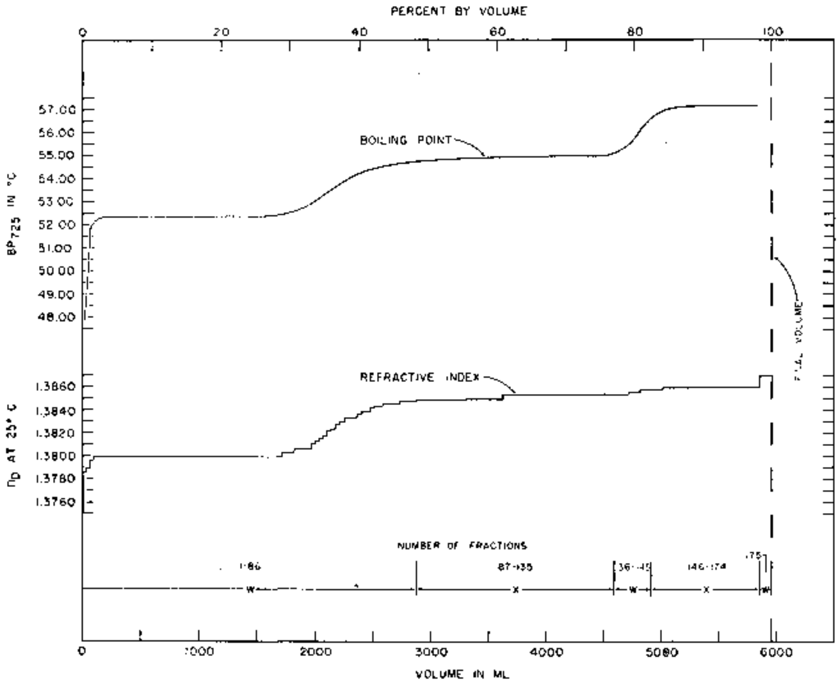

Figure 18. *First distillation of 4-methyl-2-pentene (cis+trans).*

Regular distillation at 725 mm Hg in still 13 (6/26/47 to 7/28/47). One of two distillations of similar material. See footnote j of table 1. Fractions 87 to 135 were redistilled to obtain 4-methyl-*cis*-2-pentene (see fig. 19), and fractions 146–174 were redistilled to obtain 4-methyl-*trans*-2-pentene (see fig. 20).

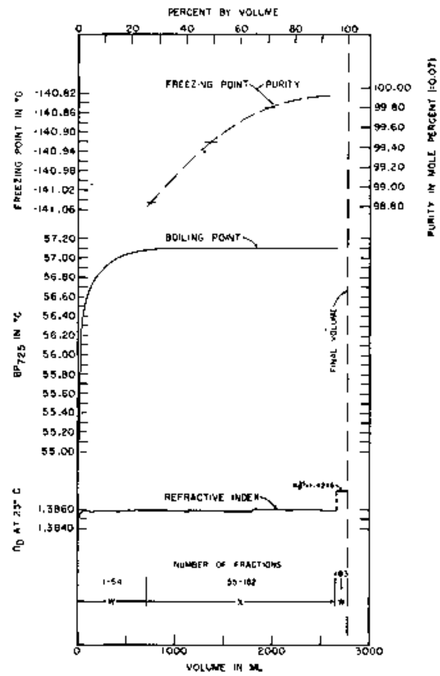

Figure 20. *Second distillation of 4-methyl-trans-2-pentene.*

Regular distillation at 725 mm Hg in still 11A (2/5/48 to 3/15/48). See footnote k of table 1.

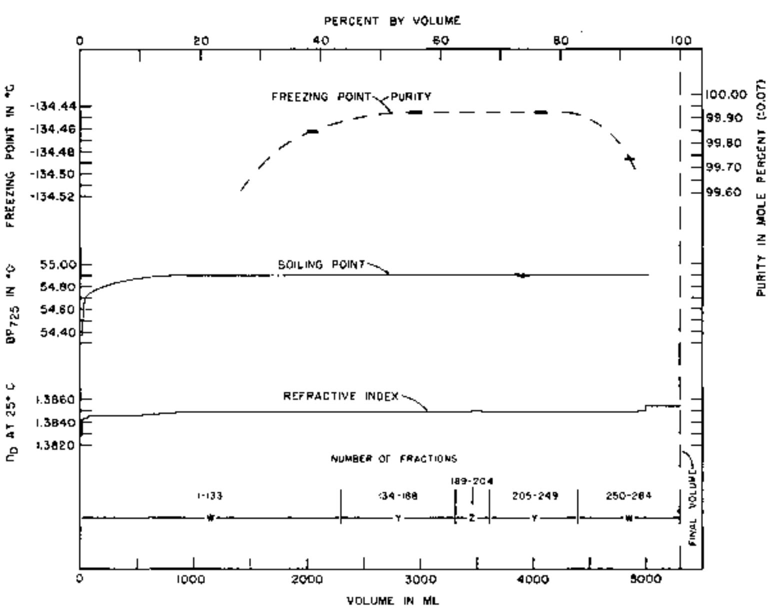

Figure 19. *Second and final distillation of 4-methyl-cis-2-pentene.*

Regular distillation at 725 mm Hg in still 15A (1/23/48 to 3/16/48). See footnote k of table 1.

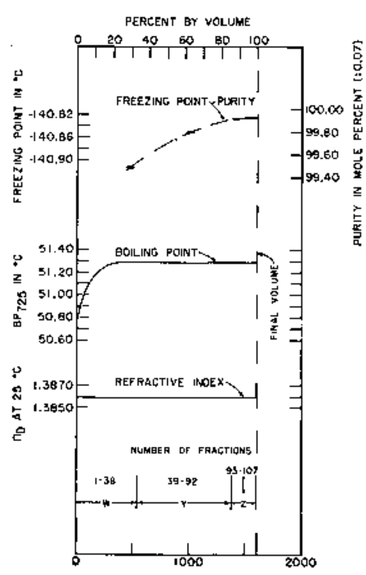

Figure 21. *Third and final distillation of 4-methyl-trans-2-pentene.*

Azeotropic distillation with ethanol at 725 mm Hg in still 3A (7/14/48 to 8/1/48).

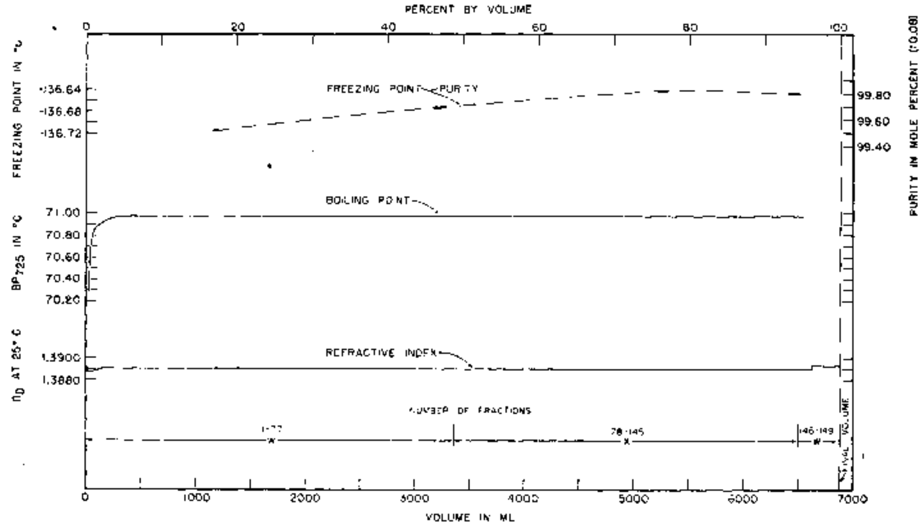

Figure 22. ***First distillation of 4,4-dimethyl-1-pentene.***

Regular distillation at 725 mm Hg in still 5 (9/19/47 to 10/20/47).

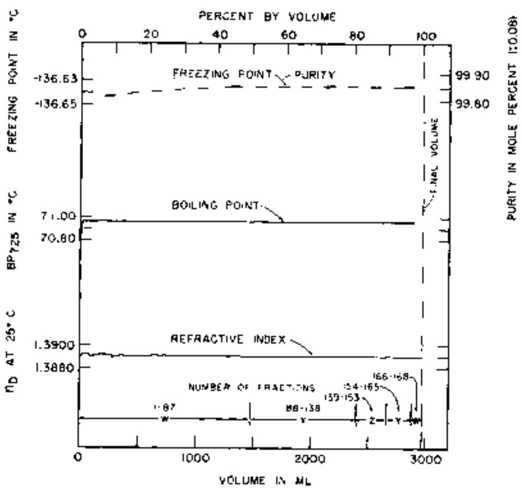

Figure 23. ***Second and final distillation of 4,4-dimethyl-1-pentene.***

Regular distillation at 725 mm Hg in still 11A (10/30/47 to 12/1/47).

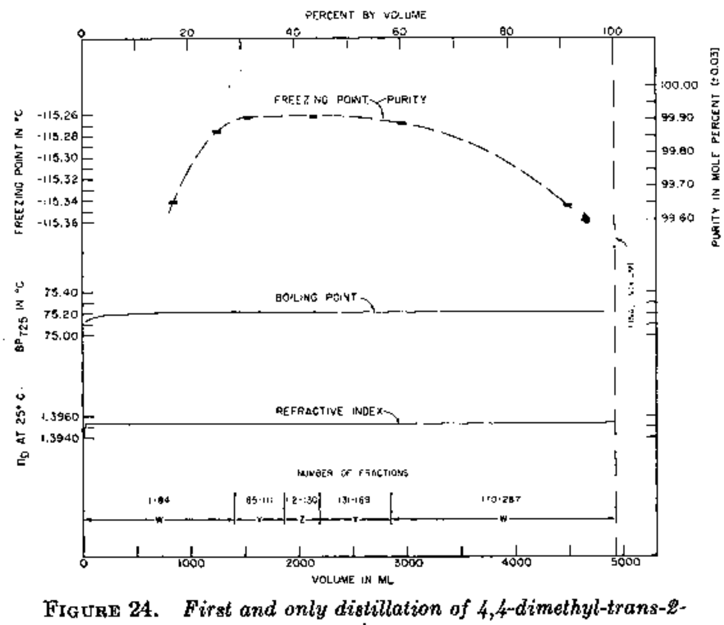

Figure 24. ***First and only distillation of 4,4-dimethyl-trans-2-pentene.***

Regular distillation at 725 mm Hg in still 12A (1/19/49 to 3/9/49).

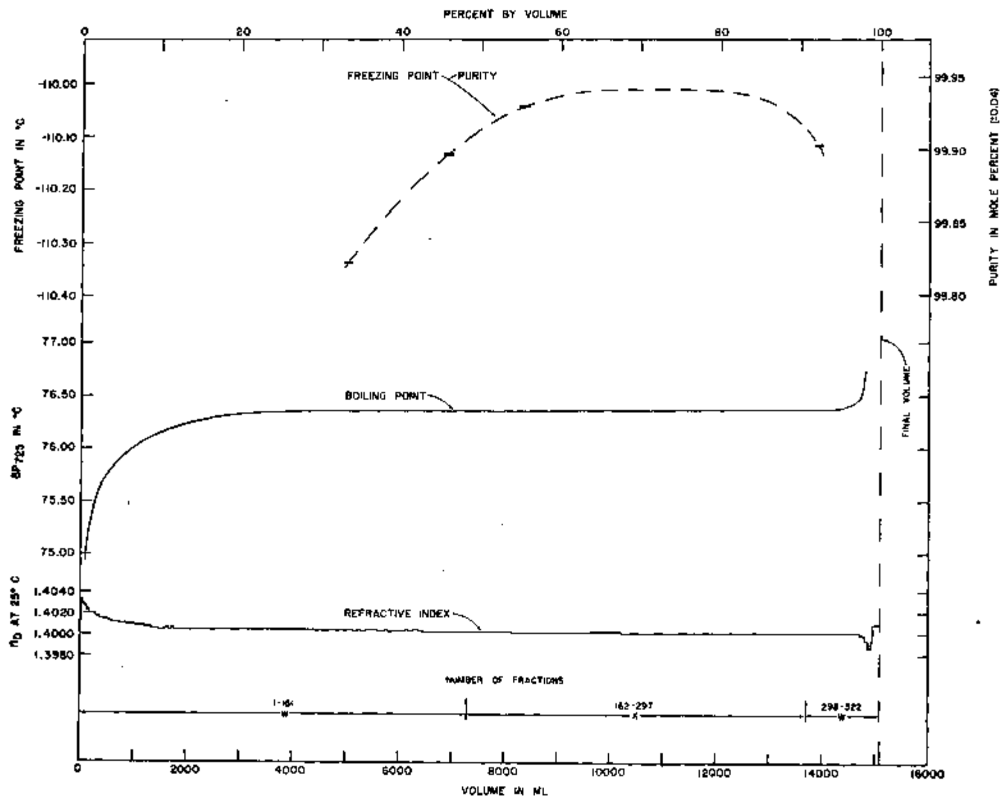

FIGURE 25. ***First distillation of 2,3,3-trimethyl-1-butene.***

Regular distillation at 725 mm Hg in still 6 (8/22/47 to 10/20/47).

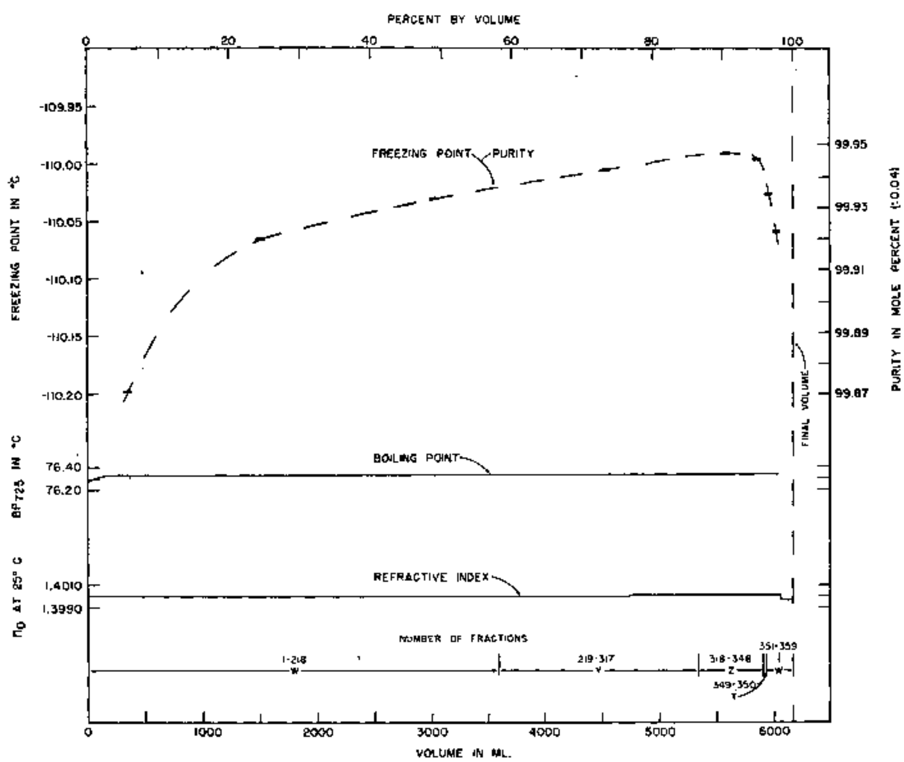

FIGURE 26. ***Second and final distillation of 2,3,3-trimethyl-1-butene.***

Regular distillation at 725 mm Hg in still 11A (12/3/47 to 2/4/48).

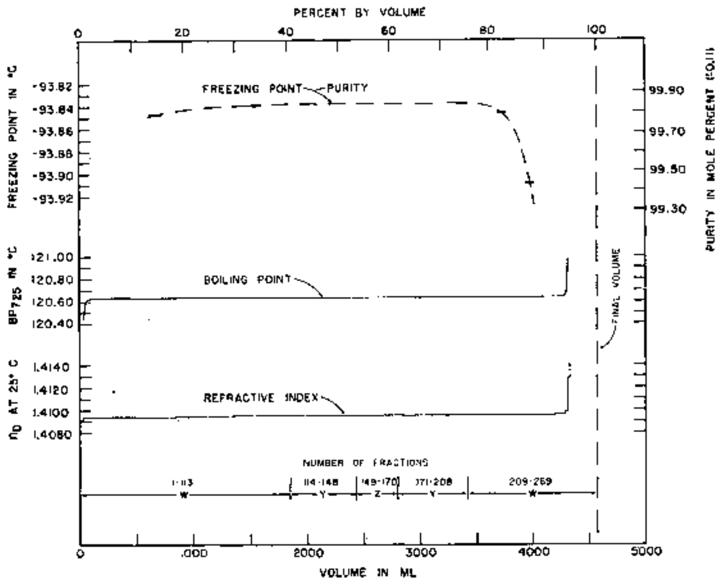

Figure 27. *First and only distillation of trans-4-octene.*
Regular distillation at 725 mm Hg in still 3A (10/6/47 to 11/21/47).

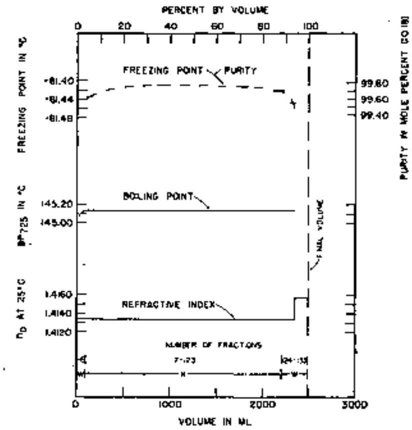

Figure 28. *First distillation of 1-nonene.*
Regular distillation at 725 mm Hg in still 4 (10/7/47 to 11/4/47).

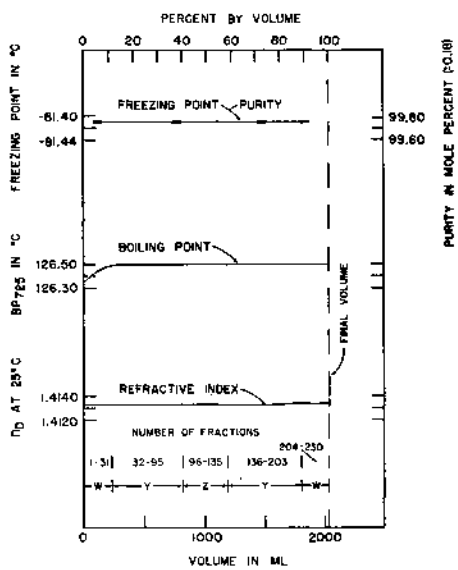

Figure 29. *Second and final distillation of 1-nonene.*
Azeotropic distillation with ethylene glycol monoethyl ether at 725 mm Hg in still 2A (11/26/47 to 1/7/48).

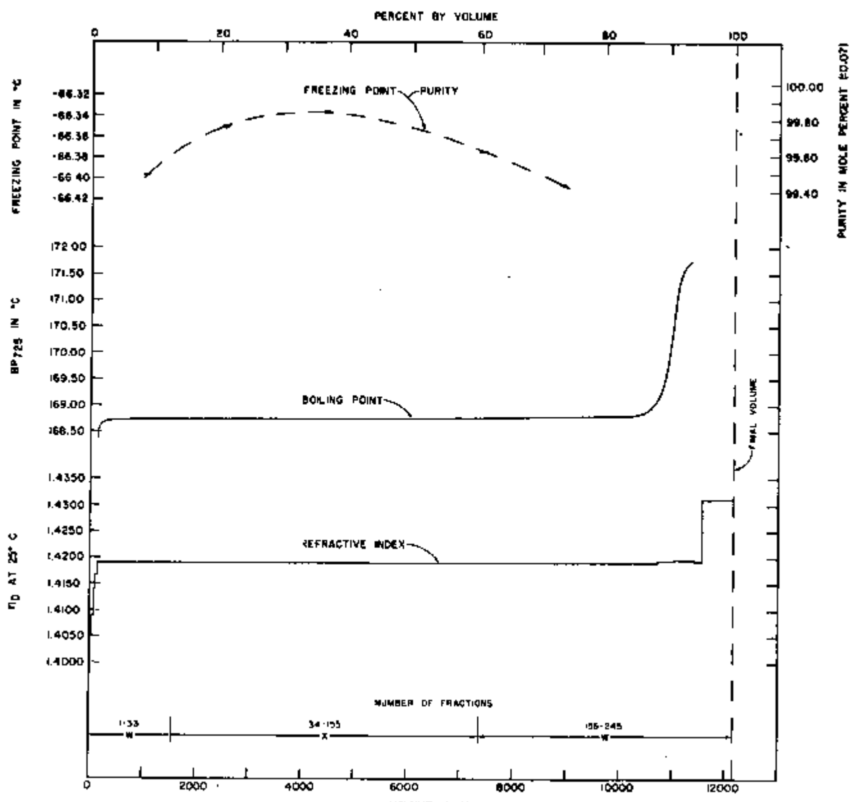

Figure 30. *First distillation of 1-decene.*
Regular distillation at 725 mm Hg in still 6 (5/22/47 to 7/7/47).

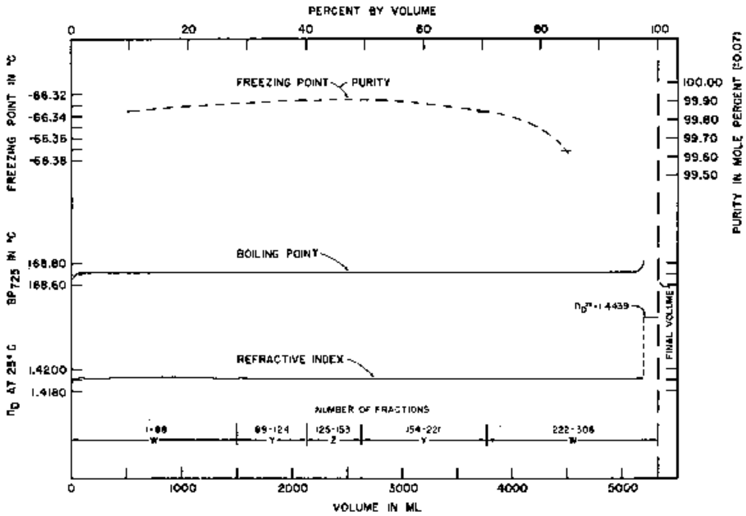

Figure 31. *Second and final distillation of 1-decene.*
Regular distillation at 725 mm Hg in still 12A (10/27/47 to 1/12/48).

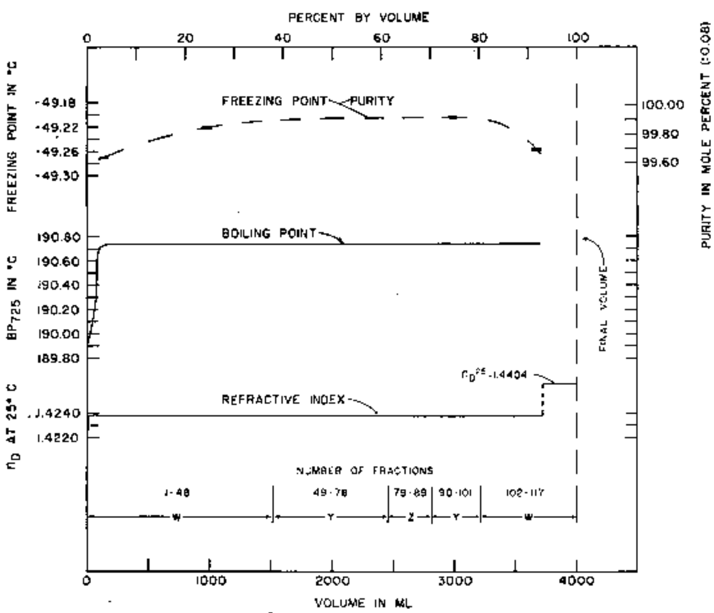

Figure 32. *First and only distillation of 1-undecene.*
Regular distillation at 725 mm Hg in still 13 (4/20/48 to 5/12/48).

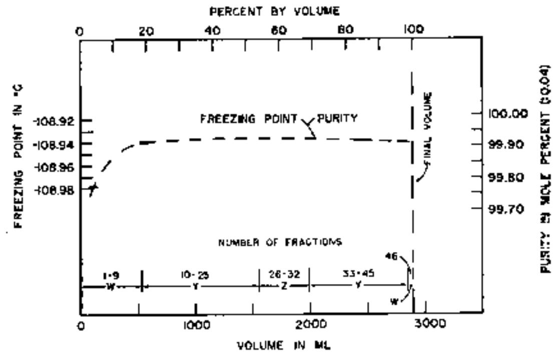

Figure 33. *First and only distillation of 1,3-butadiene.*
Regular distillation at 1 atm in still 1A (10/8/47 to 11/28/47).

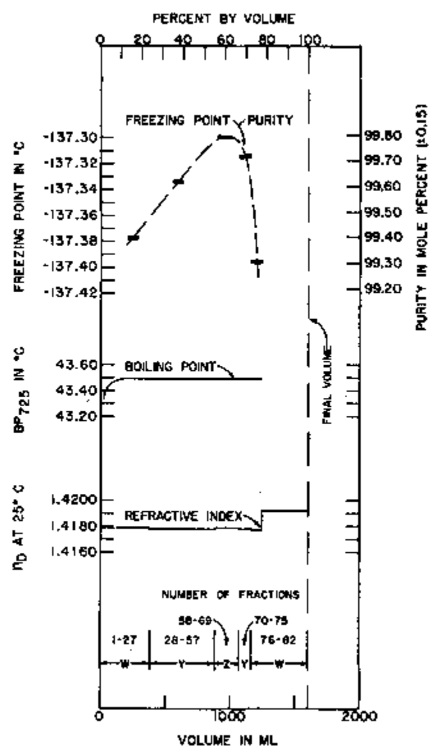

Figure 34. *First and only distillation of 1,2-pentadiene.*
Regular distillation at 725 mm Hg in still 11A (7/14/48 to 8/3/48).

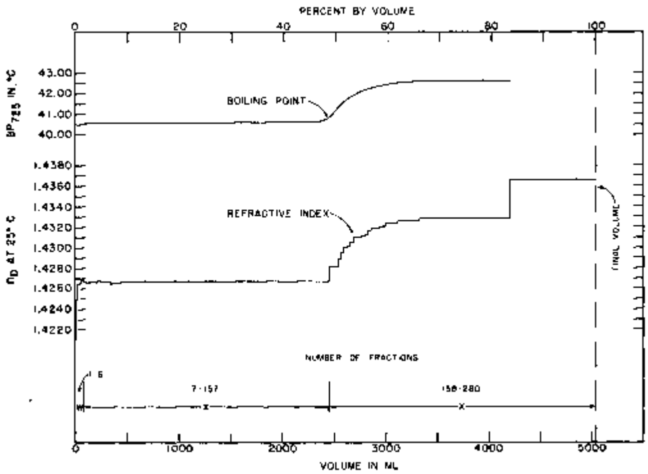

Figure 35. *First distillation of 1,3-pentadiene (cis+trans).*

Regular distillation at 725 mm Hg in still 9 (5/14/48 to 7/6/48). One of two similar distillations. See footnote 1 of table 1. Fractions 7 to 157 were redistilled to obtain 1,*trans*-3-pentadiene (see fig. 37), and fractions 158 to 280 were redistilled to obtain 1,*cis*-3-pentadiene (see fig. 36).

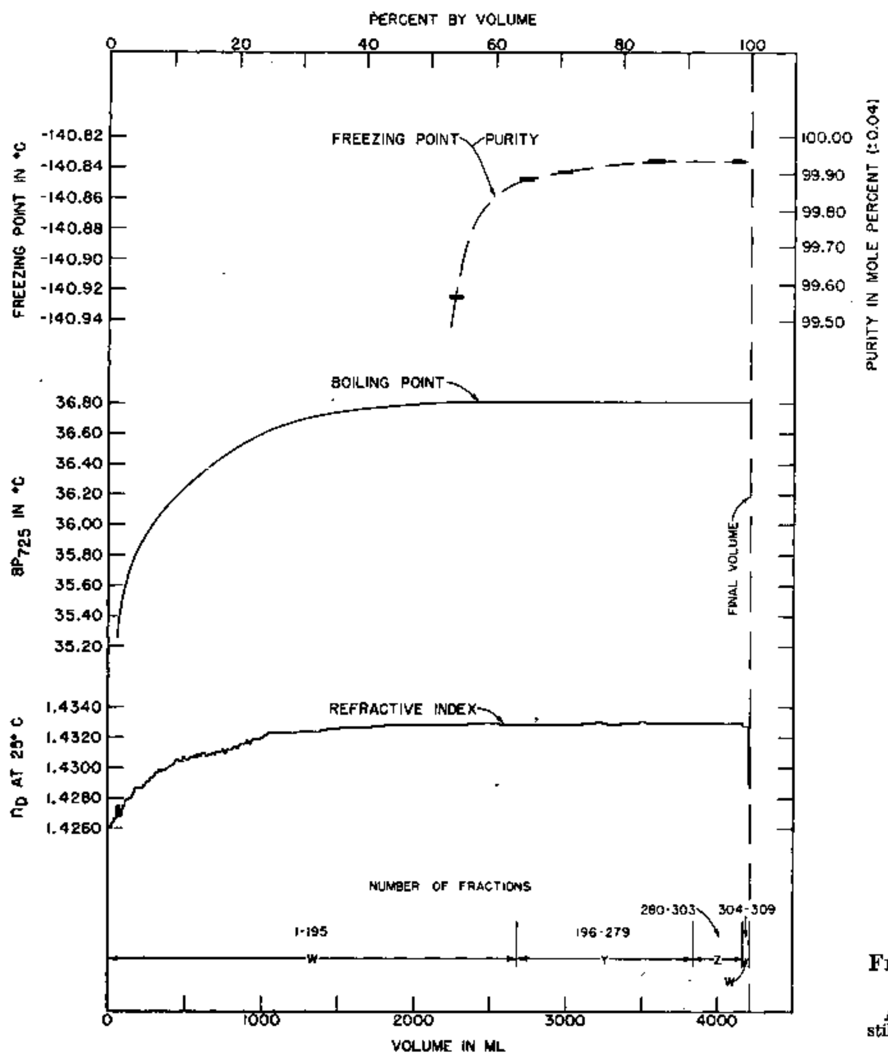

Figure 36. *Second and final distillation of 1,cis-3-pentadiene.*

Azeotropic distillation with methanol at 725 mm Hg in still 4 (9/30/48 to 11/23/48). See footnote m of table 1.

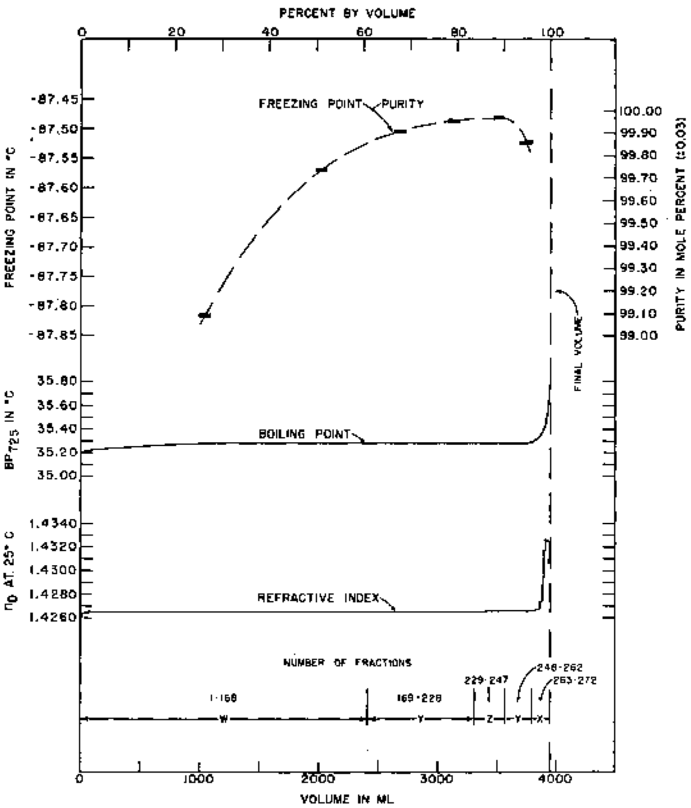

FIGURE 37. *Second and final distillation of 1,trans-3-pentadiene.*

Azeotropic distillation with methanol at 725 mm Hg in still 4 (8/11/48 to 9/28/48). See footnote m of table 1.

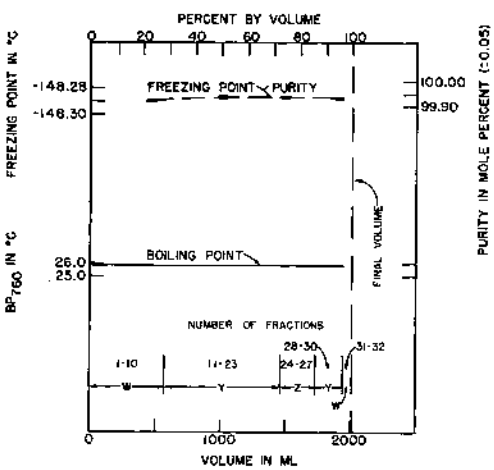

FIGURE 38. *First and only distillation of 1,4-pentadiene.*

Regular distillation at 725 mm Hg in still 1A (8/25/48 to 10/4/48).

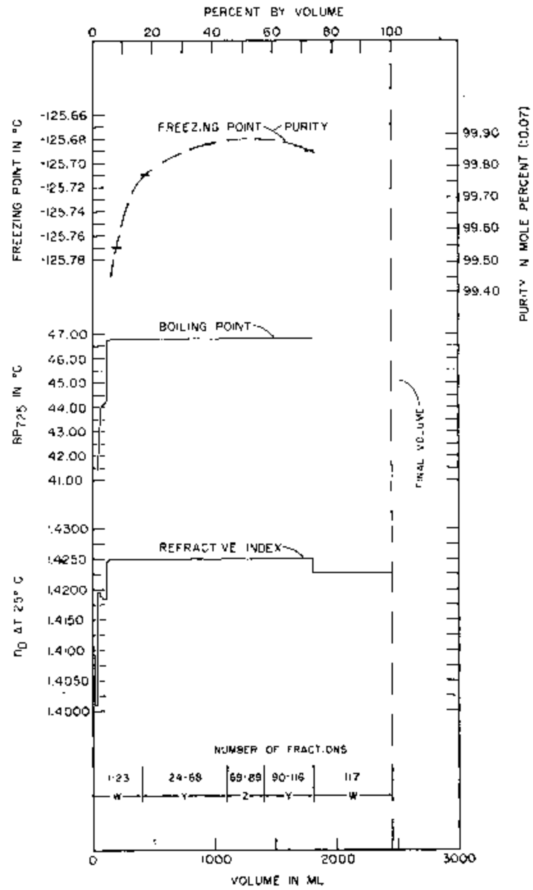

FIGURE 39. *First and only distillation of 2,3-pentadiene.*

Regular distillation at 725 mm Hg in still 9 (7/8/48 to 8/3/48).

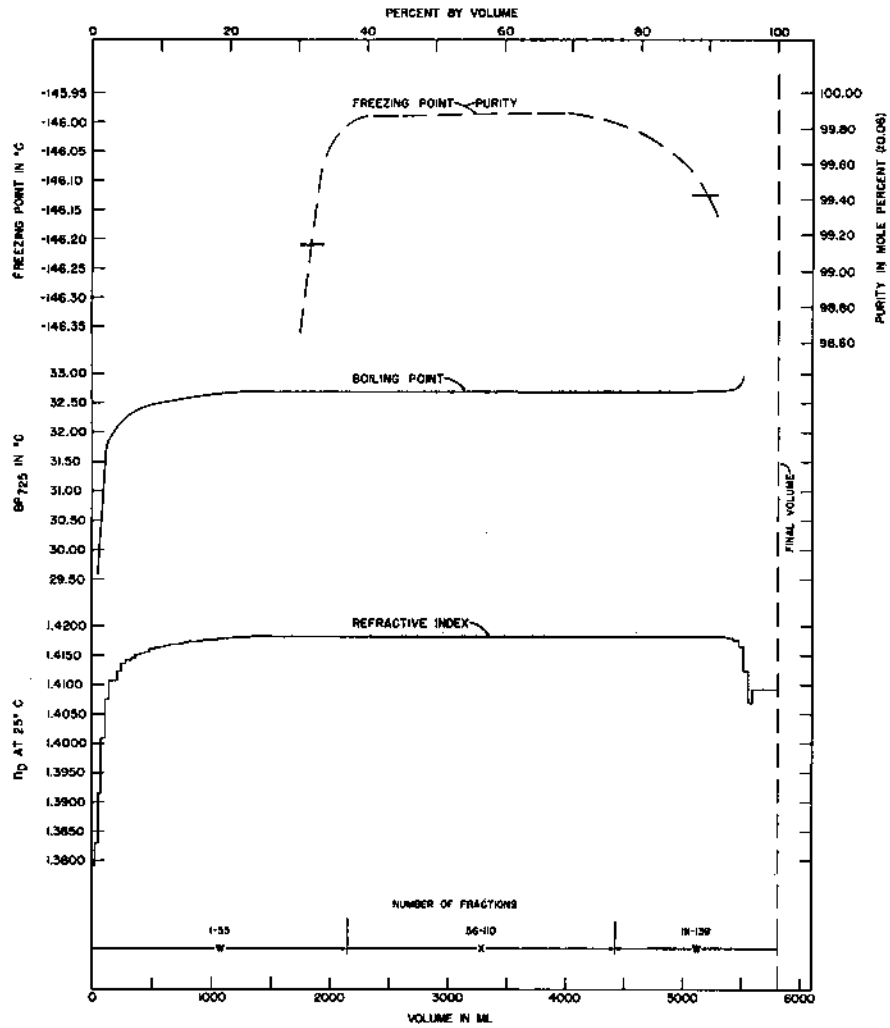

FIGURE 40. *First distillation of 2-methyl-1,3-butadiene (isoprene).*

Regular distillation at 725 mm Hg in still 13(1/20/47 to 2/20/47). One of two similar distillations.

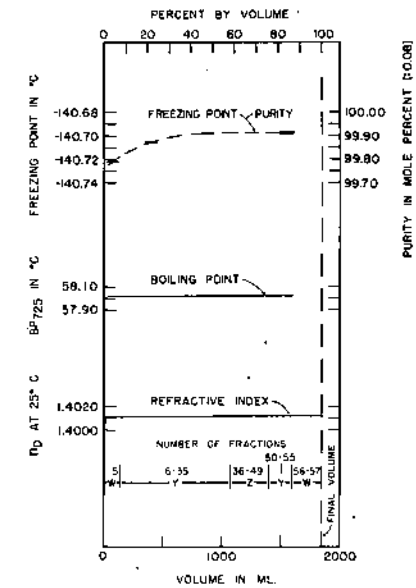

FIGURE 42. *First and only distillation of 1,5-hexadiene.*

Regular distillation at 725 mm Hg in still 13(11/7/47 to 11/25/47).

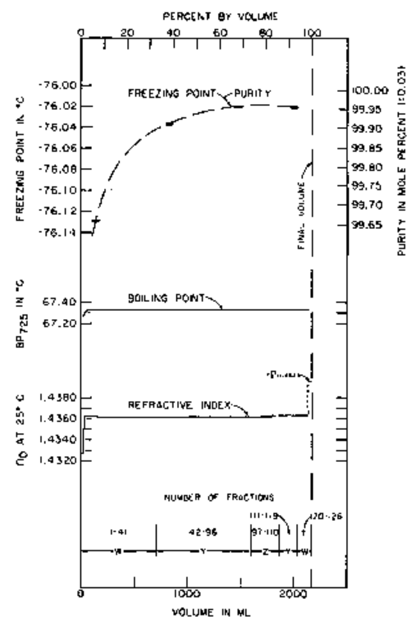

FIGURE 43. *First and only distillation of 2,3-dimethyl-1,3-butadiene.*

Regular distillation at 725 mm Hg in still 4(11/26/48 to 12/20/48).

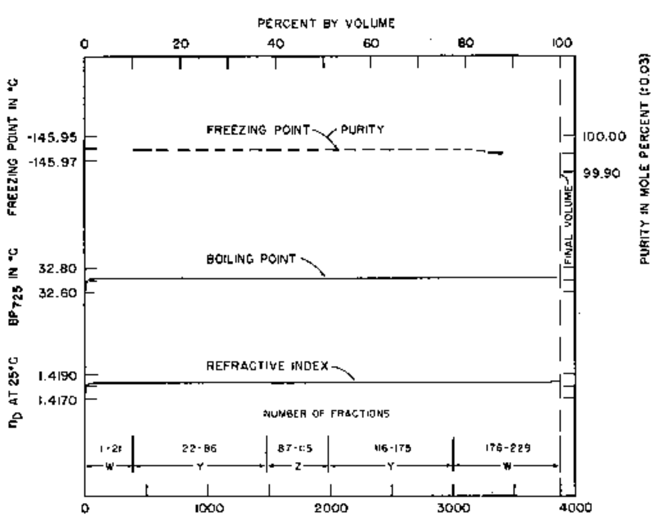

FIGURE 41. *Second and final distillation of 2-methyl-1,3-butadiene (isoprene).*

Regular distillation at 725 mm Hg in still 2A(10/7/47 to 11/17/47). See footnote i of table 1.

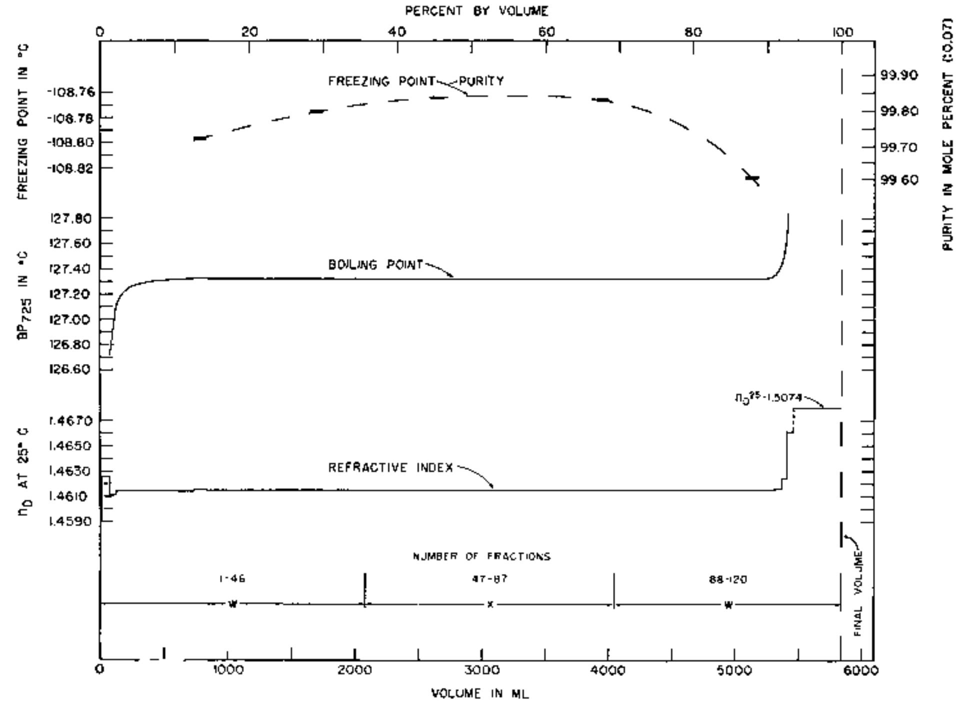

FIGURE 44. *First distillation of 4-ethenyl-1-cyclohexene (4-vinyl-1-cyclohexene).*

Regular distillation at 725 mm Hg in still 14(12/2/47 to 12/23/47). One of three similar distillations.

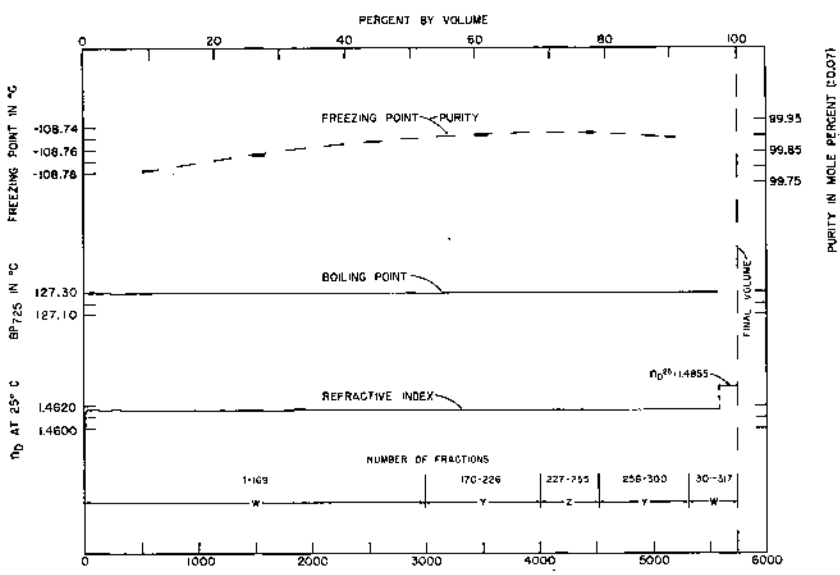

FIGURE 45. *Second and final distillation of 4-ethenyl-1-cyclohexene (4-vinyl-1-cyclohexene).*

Regular distillation at 725 mm Hg in still 4(5/3/48 to 6/28/48). See footnote q of table 1.

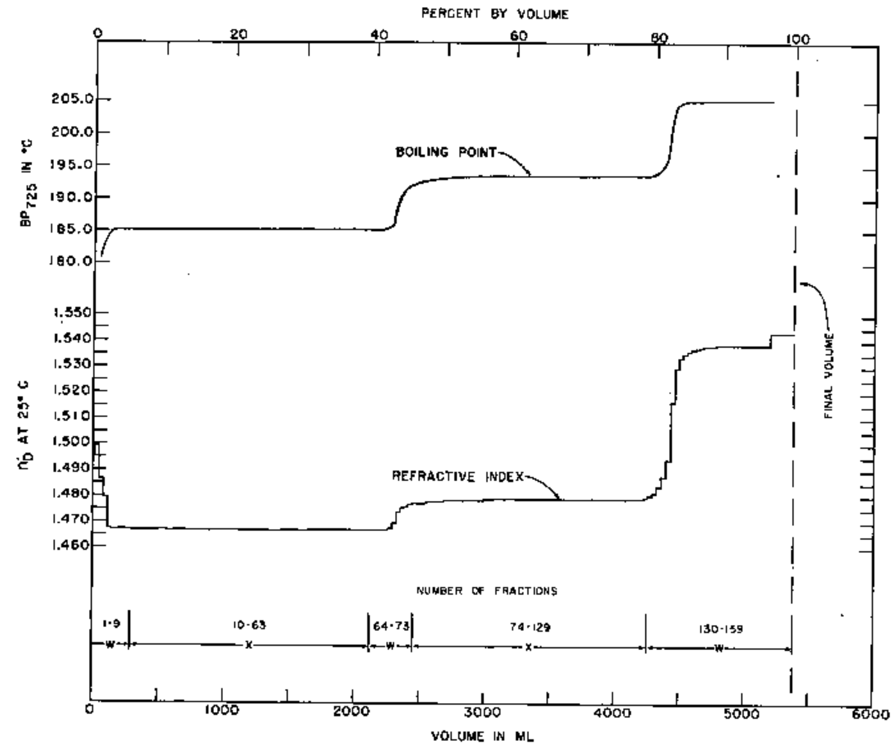

FIGURE 46. *First distillation of decahydronaphthalene (cis+trans).*

Regular distillation at 725 mm Hg in still 7(12/22/47 to 1/21/48). One of three similar distillations. See footnote s of table 1. Fractions 10 to 63 were redistilled to obtain *trans*-decahydronaphthalene (see fig. 48), and fractions 74 to 129 were redistilled to obtain *cis*-decahydronaphthalene (see fig. 47).

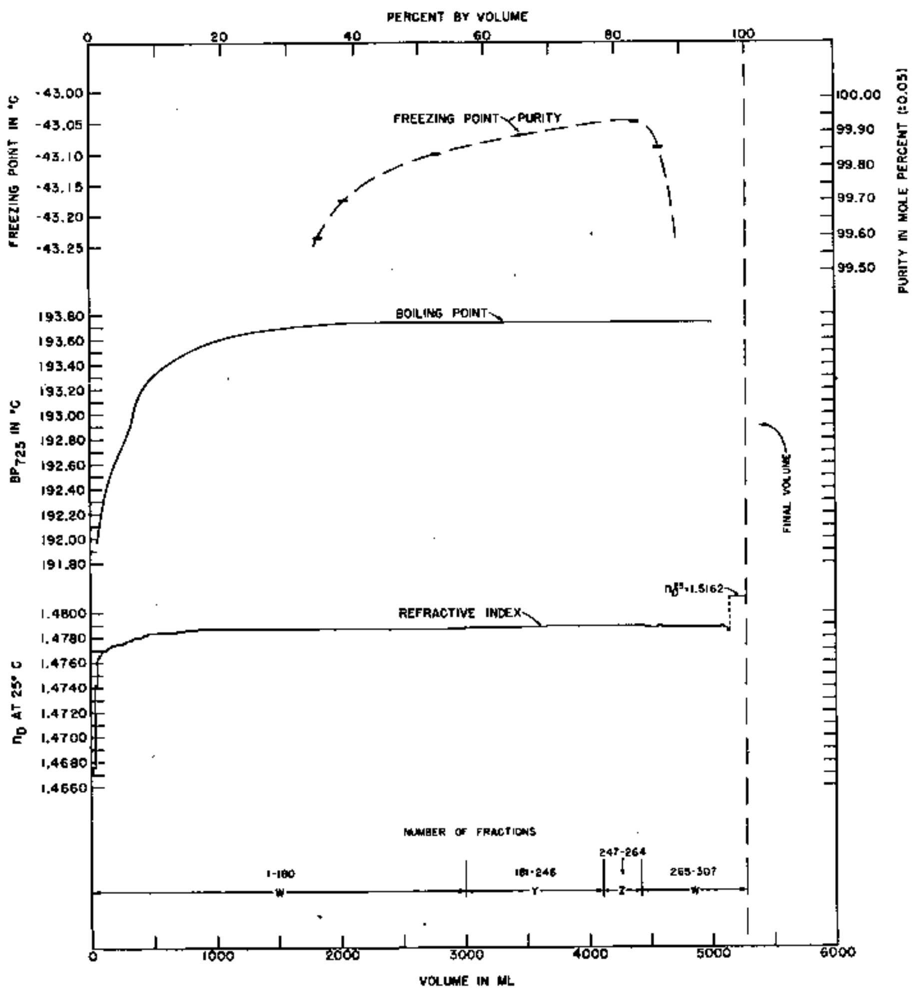

FIGURE 47. *Second and final distillation of cis-decahydronaphthalene.*

Regular distillation at 725 mm Hg in still 11A (5/10/48 to 7/12/48). See footnote t of table 1.

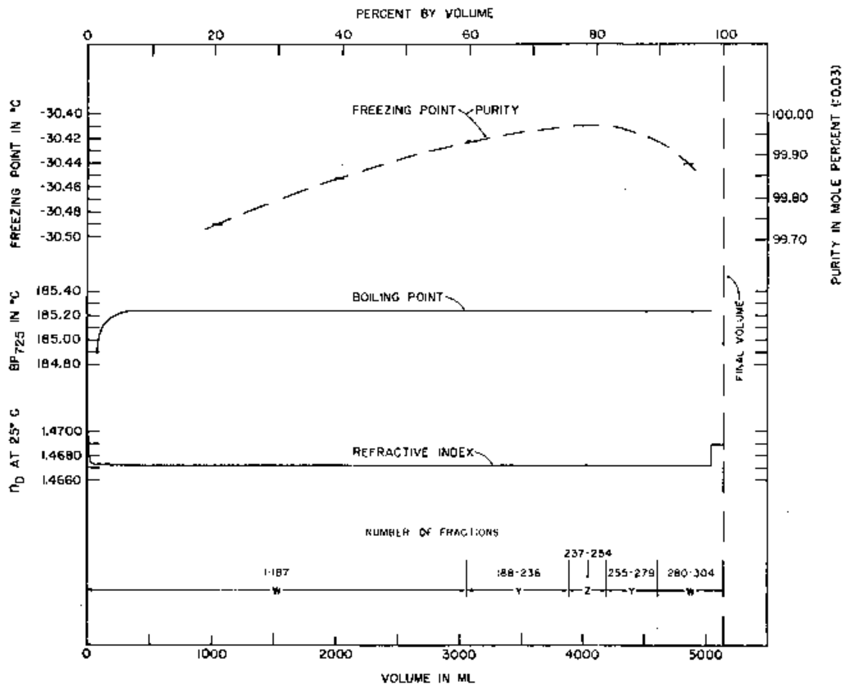

FIGURE 48. *Second and final distillation of trans-decahydronaphthalene.*

Regular distillation at 725 mm Hg in still 11A (3/18/48 to 5/10/48). See footnote t of table 1.

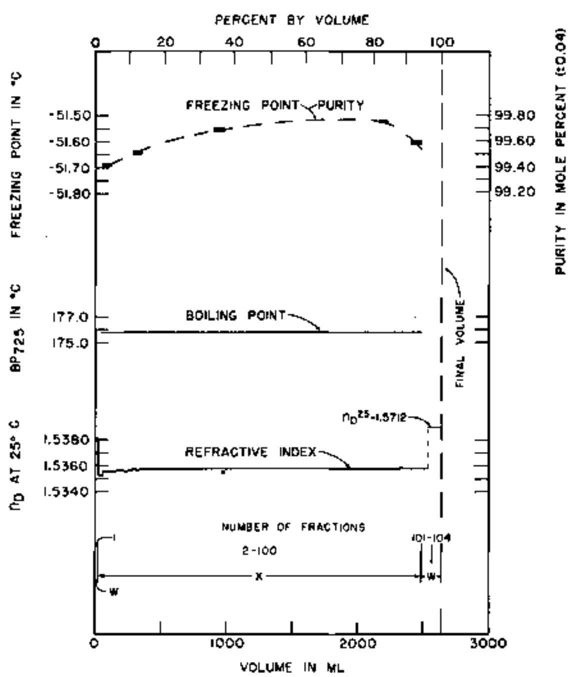

FIGURE 49. *First distillation of 2,3-dihydroindene (indan).*

Regular distillation at 725 mm Hg in still 12(5/15/45 to 6/13/45).

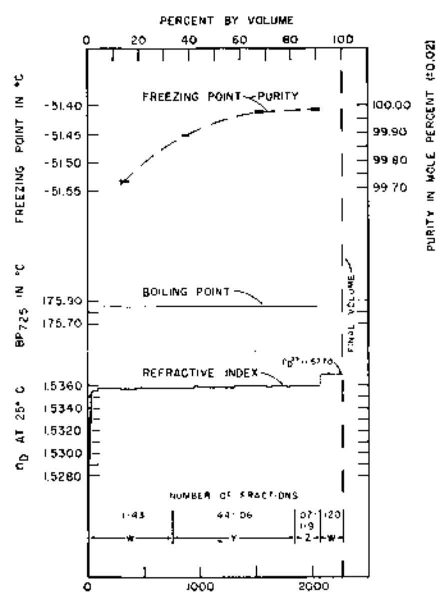

FIGURE 50. *Second and final distillation of 2,3-dihydroindene (indan).*

Regular distillation at 725 mm Hg in still 15A. (3/18/48 to 4/9/48).

WASHINGTON, May 8, 1950.