A SYSTEM CORRELATING MOLECULAR STRUCTURE OF ORGANIC COMPOUNDS WITH THEIR BOILING POINTS. VII. NEW BOILING POINTS FOR CERTAIN PARAFFINS AND OLEFINS[1]

CORLISS R. KINNEY AND WILLIAM L. SPLIETHOFF

Received August 16, 1948

In 1938 a method was devised for calculating the boiling points of organic compounds from their structure (1). The method was based on the boiling points of hydrogen and the normal paraffin hydrocarbons, all other organic compounds being considered to be derivatives of these. Since then, several new methods have been published for obtaining the boiling points of hydrocarbons (2). In addition to these, which are limited to hydrocarbons, Klages has developed a general method very similar to the Kinney method (3).

In 1940, the boiling points of all of the paraffins whose atmospheric boiling points had been recorded were compared with the calculated values (4). Out of 108 paraffins, ten were found whose observed boiling points differed from the calculated by more than 10°. Since then, Butler (5) has resynthesized the third paraffin on this list, 2,4,6-trimethylheptane, and obtained the value 147.6° which is well within the 10° limit. Irving[2] has also prepared this hydrocarbon and obtained similar results. Consequently, this hydrocarbon should be removed from the list.

The boiling point of the fourth paraffin, 4,5-dimethyloctane, was incorrectly abstracted (6) and Francis[3] was the first to call attention to this error. The correct boiling point given by Festraete (7) was 160.5° which differs from the calculated by only 0.5°. Therefore, this hydrocarbon should be stricken from the list. The tenth paraffin, 4,8,12-trimethylhexadecane, should also be removed because it was subsequently shown that this hydrocarbon, crocetane, actually had 20 carbon atoms (8) instead of 19 for which the above structure was assigned. Using the new structure the calculated boiling point is 332.2° compared with the observed value of 334.0°.

Of the remaining seven paraffins the four appearing in Table I, numbers 5, 6, 7, and 9, have been resynthesized. The boiling points of all of these come well within the 10° limit and may, also, be removed from the original list.

The eighth paraffin on the list, 6-methyl-7-ethyldodecane, was prepared by Karrer (9) at the same time as the seventh and ninth hydrocarbons appearing in Table I. Since all three of Karrer's boiling points were uncorrected for barometric pressure and, apparently, for exposed thermometer thread as well, it seemed highly probable that the corrected boiling point of 6-methyl-7-ethyldodecane approximates the calculated value fairly closely as do 4-methyl-6-propyl-

---

[1] Presented at the New York meeting of the American Chemical Society, September 15, 1947.

[2] Lee Irving, University of Utah, 1943.

[3] Francis, private communication.

hendecane and 6-propyldodecane which were resynthesized. Therefore, it appears that 6-methyl-7-ethyldodecane should also be stricken from the 1940 list.

The two remaining paraffins, 3-methyl-3-ethylpentane and 2,3-dimethyl-3-ethylpentane,[4] constitute a separate case. 3-Methyl-3-ethylpentane was resynthesized by Barrowman,[5] but the same observed boiling point was obtained. Since this is 11.6° above the calculated value, the whole series of 3,3-disubstituted paraffins has been reexamined.

The calculated and observed boiling points of these hydrocarbons are given in Table II. It will be observed that the usual close agreement is obtained for the 3,3-dimethyl derivatives, but that markedly exalted boiling points are given by the 3-methyl-3-ethyl and the 3,3-diethyl derivatives. This exaltation of the boiling point is in sharp contrast with the depression in boiling point shown by 2,2-dimethyl derivatives for which a b.p.n. was assigned. In a similar way it is now proposed to assign a b.p.n. of 1.0 to account for the high boiling points of the 3-methyl-3-ethyl and the 3,3-diethyl structures. Thus, when calculating boiling points of compounds containing these groupings a b.p.n. of 1.0 should be added. In this way, the newly calculated boiling points appearing in Table II were obtained. It will be observed that the new values are in much better agreement with the observed boiling points. The first member of the 3,3-diethyl series shows an unusually high deviation (7.7°) but probably this is due to the discrepancy often shown by the first members of series.

TABLE I

BOILING POINTS OF PARAFFINS 5, 6, 7, AND 9

NO.	STRUCTURE	BOILING POINT, °C.		
		Old Obsv'd	Calc'd	New Obsv'd
5	2,4,5,7-Tetramethyloctane	210.0	192.2	186.3
6	2,5,9-Trimethyldecane[a]	207.0	218.0	211.1
7	4-Methyl-6-propylhendecane	233.5[b]	249.6	242.6
9	6-Propyldodecane	242.0[b]	254.5	251.6

[a] 2,5,9-Trimethyldecane is better nomenclature than 2,6,9-trimethyldecane.

[b] 729 mm.

The high boiling points of 3-methyl-3-ethyl and 3,3-diethyl derivatives may be observed in other series of compounds, as for example, 4-methyl-4-ethyl-1-hexene (3-methyl-3-ethyl-5-hexene) among the olefins. Using the b.p.n. of 1.0 for the 3-methyl-3-ethyl grouping, the calculated boiling point comes within +1.7° of the observed instead of −7.2° without it.

Just what effect the 4-methyl-4-ethyl and 4,4-diethyl structures have on the boiling point is uncertain because no boiling points of these derivatives have been reported. The effect of the 4,4-dipropyl and 5,5-dibutyl structures is also unknown.

---

[4] This hydrocarbon appears on the 1940 list as 2,2-dimethyl-3-ethylpentane, but is an typographical error.

[5] S. R. Barrowman, University of Utah, 1944.

The normal boiling points of a number of new paraffins have been reported in the literature since 1940. Only four of these deviate from the calculated value by more than 10°. They are 2,4-dimethylnonane (10), 2,7-dimethyl-4,5-diethyloctane (11), 6,8-dimethyltridecane (12), and 2,2,3,3,4-pentamethylpentane (13). The deviations are respectively −10.3°, −11.3°, −18.1°, and +16.5°. Pressure and temperature corrections were not applied to the observed boiling points of the first two hydrocarbons; consequently, it seems quite likely that the corrected boiling points of these two hydrocarbons would fall well within the 10° limit. On the other hand, the boiling points of 6,8-dimethyltridecane and 2,2,3,3,4-pentamethylpentane vary considerably from the calculated and consequently the cause of this deviation should be investigated.

TABLE II

BOILING POINTS OF 3,3-DISTRIBUTED PARAFFINS

HYDROCARBONS	BOILING POINTS, °C.		
	Old Calc'd	Obsv'd	New Calc'd
3,3-Dimethylpentane	82.8	86.1[a]	
3,3-Dimethylhexane	110.6	112.0[a]	
3,3-Dimethylheptane	136.2	137.3[a]	
3,3-Dimethyloctane	160.0	161.2	
3-Methyl-3-ethylpentane	107.2	118.3[a]	116.7
2,3-Dimethyl-3-ethylpentane	130.8	142.0[a]	139.7
3-Methyl-3-ethylhexane	133.1	143.0[a]	141.9
3-Methyl-3-ethylheptane	157.1	163.9[b]	165.3
3-Methyl-3-ethyloctane	179.6	185.7[c]	187.3
3,3-Diethylpentane	129.9	146.5[a]	138.8
3,3-Diethylhexane	154.1	162.7[c]	162.4
3,3-Diethylheptane	176.9	186.7[c]	184.6
3,3-Diethyloctane	198.1	205.1[c]	205.5
3,3-Diethylnonane	218.3	222.1[c]	225.3

[a] A.P.I. Research Project 44, "Selected Values of the Properties of Hydrocarbons", Tables 2a, 3a, and 4a (1945), values to the nearest tenth of a degree.

[b] Previously reported as 156.3° by Campbell and Eby, *J. Am. Chem. Soc.*, **62**, 1800 (1940).

[c] Boiling points of new paraffins; see the Experimental Part.

A reexamination of the olefins on the 1940 list (4) gave results similar to those obtained for the paraffins. 3-Decene,[6] 5-decene (14), and 4,8-dimethyl-4-nonene have been resynthesized. The new observed boiling points are given in Table III.

2,3-Dimethyl-2-butene (15)[6] and 2,3,5-trimethyl-2-hexene (16) have also been resynthesized, with little change of the boiling point in the first case, but with an increase of 12.3° in the latter. These hydrocarbons are members of the 2,3-dimethyl-2-olefin series and in addition to these members, the whole series has been resynthesized up to 2,3-dimethyl-2-octene (16). On reexamining the data available for all the $R_2C = CR_2$ olefins, it was found that raising the b.p.n.

---

[6] F. K. Balli, University of Utah, 1943.

for this type of olefin linkage from 2.8 to 3.2 brought the boiling points of all of the known members of the series well within the 10° limit. In view of these new data, it is recommended that the b.p.n. of the tetrasubstituted double bond be raised to 3.2.

2,2-Dimethyl-4-ethyl-3-hexene was found to be another incorrectly abstracted compound on consulting the original paper (17). The correct structure was 4,4-dimethyl-3-ethyl-2-pentene and the calculated boiling point for this structure agrees very well with the observed boiling point, although the observed range of 8° makes the true boiling point rather uncertain.

The first three olefins of the 1940 list, 3-methylene-2,2,4-trimethyl-4-ethylhexane, 3-methylene-2,2,4,4-tetramethylhexane, and 3-methylene-2,2,4,4,5-pentamethylhexane[7] may be considered to be examples of hydrocarbons in which four or more alkyl groups are attached to adjacent carbon atoms of the main chain of six carbon atoms (4). These structures were assigned the b.p.n. of 1.0. If this b.p.n. is used in calculating the boiling points of the methylene derivatives, the deviations of the observed boiling points from the calculated become −1.0°, +3.2° and −1.0° respectively. If the same b.p.n. is also applied to the fourth olefin on the list, 4-methylene-3,3,5,5-tetramethylheptane, for which there are no paraffinic analogs, its calculated boiling point comes within 5.4° of the observed value. Consequently, it seems very likely that this b.p.n. should be used in calculating the boiling points of olefins with bunched side chains such as these, even though the side chains may be partly alkenyl radicals. If this is done, one of the known olefins, 3-methylene-2,2,4-trimethylhexane, has a calculated boiling point of 158.8° as compared with an observed boiling range of 146–150°. Although the mid-point of this range is more than 10° from the calculated, the 10° limit comes within the observed boiling range and it is not considered likely that the true boiling point of this compound is too far from the calculated. It should also be pointed out that the first compound on the 1940 list, 3-methylene-2,2,4-trimethyl-4-ethylhexane, is an example of a 3-methyl-3-ethyl hydrocarbon for which an additional b.p.n. of 1.0 should be added. The use of this additional b.p.n. brings the calculated boiling point to within 1.0° of the observed value.

TABLE III

NEW BOILING POINTS FOR RESYNTHESIZED OLEFINS

OLEFIN	CALC'D B.P.	NEW OBSV'D B.P.	DEVIATION
3-Decene	171.3	173.3	+2.0
5-Decene	171.3	169.6	−1.7
4,8-Dimethyl-4-nonene	184.6	182.3	−2.3

The two remaining olefins on the list, 2,4,6-trimethyl-3-heptene and 2,4,7-trimethyl-4-octene, were prepared by Tuot (18). The boiling point of the second

---

[7] This nomenclature is employed because "the longest chain in the molecule must be used in calculating the boiling point," ref. 4, p. 559.

of these, 2,4,7-trimethyl-4-octene, comes within the 10° limit if the reported boiling point is corrected for the low observed pressure. The first hydrocarbon, 2,4,6-trimethyl-3-heptene, was resynthesized[8] and the new observed boiling point also fell within the prescribed limit, 148.2° as compared with 156.2°.

Since 1940, boiling points for a number of new olefins have appeared. Five of these have boiling points deviating by more than 10° from the calculated. For two of them, 5,5-dimethyl-3-heptene (19) and 2,2,3,4,5,5-hexamethyl-3-hexene (20), boiling ranges of 8° and 13° respectively were reported, and in view of the uncertainty regarding their true normal boiling points the reported ranges are of little value. Also, 2,5-dimethyl-4-hendecene (21) was reported as being impure; consequently, the true boiling point of this hydrocarbon is uncertain. The fourth olefin, 2,2,5-trimethyl-3-hexene (22), and the fifth, 2,2,5,5-tetramethyl-3-hexene (13), have observed boiling points of 11.1° and 14.7° lower respectively than the calculated values, taking into consideration the 2,2-dimethyl structures occurring in both of them. Since these hydrocarbons have related structures it may be possible that their structure requires a b.p.n. to account for the abnormally low boiling points which have been reported. Further verification of these boiling points should be made, however.

TABLE IV

NEW 3,3-DISUBSTITUTED PARAFFINS

	ANALYSIS				B.P. AT 760 MM., °C.	n_D^{20}	d_4^{20}
	Calc'd		Found				
	C	H	C	H			
3-Methyl-3-ethylheptane	84.5	15.5	84.4	15.4	163.9	1.4205	0.7478
3-Methyl-3-ethyloctane	84.6	15.4	84.1	15.5	185.7	1.4250	.7528
3,3-Diethylhexane[a]	84.5	15.5	84.4	15.1	162.7	1.4261	.7624
3,3-Diethylheptane	84.6	15.4	84.2	15.4	186.7	1.4299	.7703
3,3-Diethyloctane	84.7	15.3	84.4	15.5	205.1	1.4321	.7713
3,3-Diethylnonane	84.8	15.2	84.5	15.4	222.1	1.4365	.7758

[a] Valentine and Spliethoff, The Pennsylvania State College, 1948.

In view of the several methods that have been developed for the prediction of the boiling points and other physical properties of compounds, particularly hydrocarbons, values reported on the literature should be very carefully checked before publication. Relatively simple calculations may save other investigators a great deal of time.

EXPERIMENTAL

The methods of making the hydrocarbons described below are only briefly outlined since no unusual procedures or modifications were used. Some of the data were abstracted from theses completed at the University of Utah and in these cases the boiling points were corrected for exposed thermometer stem. Pressures were held at 760 mm. ± 1 mm. by added

---

[8] Lento and Spliethoff, The Pennsylvania State College, 1948.

air pressure from a ballast tank, excepting for the boiling points of 3-methyl-3-ethylpentane and 3-decene which were corrected for low barometric pressure by the method of Hass and Newton (23).

The remaining boiling points were obtained on one degree (or less) fractions using a 35-plate column, 12 mm. by 40 cm. and packed with $\frac{1}{16}$-inch stainless steel helices. The boiling points were determined in a modified semimicro Cottrell boiling point apparatus (24). Pressures were maintained at 760 mm. ± 0.05 mm. and temperatures were read with an accuracy of ±0.2° using a calibrated copper-constantan thermocouple.

The refractive indices were measured with a Bausch and Lomb Abbé-type refractometer. The compensating prisms of this instrument were calibrated by the manufacturer to give values at the wave length of the sodium D-line. The sample prisms were maintained at a temperature of 20.0° ± 0.1° by circulating water from a constant-temperature bath. The scale readings were calibrated against a standard test plate having a refractive index of 1.5137 supplied with the instrument.

Densities were measured in a Nicol type pycnometer that had been calibrated to four decimal places with an uncertainty of ±0.0001 using distilled water as the calibrating medium. The pycnometer was placed in a constant-temperature bath maintained at 20.0° ± .05°. After twenty minutes, the volume of liquid was adjusted to the calibration mark and allowed to come to room temperature. All weighings were made on a magnetically-damped analytical balance which had been calibrated and adjusted against a set of weights certified by the National Bureau of Standards. The weights used were also calibrated against the same standard set using the same balance.

2,4,6-Trimethylheptane.[2] Diisobutyl ketone was treated with methylmagnesium iodide, giving methyl diisobutyl carbinol. On distillation at normal pressure, the carbinol dehydrated to the corresponding olefin, which was reduced with sodium and alcohol until a negative test for unsaturation was obtained with alkaline permanganate. The product, 2,4,6-trimethylheptane, boiled at 145.6–146.1° (corr.) at 760 mm. ± 1 mm. The index of refraction was n_D^{20} 1.4209.

2,4,5,7-Tetramethyloctane.[2] 2-Chloro-4-methylpentane was treated with sodium in the Wurtz reaction. The product, 2,4,5,7-tetramethyloctane, boiled at 186.2–186.3° (corr.) at 760 mm. ± 1 mm. The index of refraction was n_D^{20} 1.4229.

2,5,9-Trimethyldecane.[9] Chloroacetone was treated with two equivalents of isoamyl magnesium bromide, giving 2,5,9-trimethyl-5-decanol, which was dehydrated. The olefin was hydrogenated in a Parr machine with Adams catalyst to yield 2,5,9-trimethyldecane which boiled at 211.1° at 760 mm. The index of refraction was n_D^{20} 1.4244 and the density was d_4^{20} 0.7546.

Anal. Calc'd for $C_{13}H_{28}$: C, 84.8; H, 15.2.
Found: C, 84.6; H, 15.2.

4-Methyl-6-propylhendecane. 4-Methyl-6-propyl-6-hendecanol was prepared following Karrer's method (9). After dehydrating the alcohol, the olefin was hydrogenated in a Parr machine with Adams catalyst. 4-Methyl-6-propylhendecane had the boiling point 242.6° at 760 mm. The index of refraction was n_D^{20} 1.4389 and the density d_4^{20} 0.7798.

Anal. Calc'd for $C_{15}H_{32}$: C, 84.9; H, 15.1.
Found: C, 84.7; H, 14.6.

6-Propyldodecane.[10] 6-Propyl-6-dodecanol was prepared using Karrer's directions (9). This was dehydrated to the corresponding olefin which distilled at 244.6–245.6° (corr.) at 643.8 mm. The index of refraction was n_D^{20} 1.4405 and the density was d_4^{23} 0.7791. The olefin was reduced with sodium and alcohol until a negative test for unsaturation was obtained with alkaline permanganate. A fraction boiling at 240–242° at 649.7 mm. was redistilled at 760 mm. ± 1 mm. and boiled at 250.6–252.6° (corr.). The index of refraction was n_D^{18} 1.4332 and the density was d_4^{19} 0.7721.

---

[9] M. L. Briggs, Nancy Hoeflich, and W. L. Spliethoff, The Pennsylvania State College, 1944.

[10] J. R. Morandi, University of Utah, 1943.

3-Methyl-3-ethylpentane.[5] 3-Methyl-3-ethylpentane was synthesized from 3-chloro-3-methylpentane and diethylzinc (25). Its boiling point was 118.8° (corr.), the index of refraction was n_D^{20} 1.4089, and the density d_4^{20} 0.7230.

3,3-Disubstituted paraffins. The following new 3,3-disubstituted paraffins given in Table II were made by reacting a tertiary halide with a primary alkyl Grignard reagent (26). The boiling points were determined in the modified semimicro Cottrell apparatus (24) as described above. Their physical properties are given in Table IV.

3-Decene.[6] 3-Decene was prepared by the method of Schmidt and Boord (27) for making hexenes. α,β-Dibromobutyl ethyl ether was made from butyraldehyde using Swallen and Boord's procedure (28) for α,β-dibromoethyl ethyl ether. After washing the crude product with water and drying with calcium chloride, it was treated with *n*-hexylmagnesium bromide. The crude product of this reaction, α-hexyl-β-bromobutyl ethyl ether, was refluxed with *n*-propyl alcohol and zinc dust for 14.5 hours. The filtrate from this reaction was poured into cold water. The olefin was dried and distilled from sodium. On fractionation, a cut boiling at 173.0–173.5° (corr. 760 mm.) was collected as 3-decene. The index of refraction was n_D^{20} 1.4221 and the density was d_4^{20} 0.7447.

4,8-Dimethyl-4-nonene. 2-Methyl-1-pentanol was oxidized to 2-methylpentanal which was treated with isoamylmagnesium bromide. The resulting carbinol was dehydrated with fused potassium acid sulfate and the olefin distilled from sodium. Fractionation in the 35-plate column yielded a cut which boiled at 182.3° at 760 mm. The index of refraction was n_D^{20} 1.4329 and the density d_4^{20} 0.7567.

Anal. Calc'd for $C_{11}H_{22}$: C, 85.7; H, 14.3.
Found: C, 85.4; H, 14.5.

2,3-Dimethyl-2-butene.[6] This olefin was prepared by dehydrating dimethylisopropylcarbinol with anhydrous oxalic acid. The carbinol was made from acetone and isopropylmagnesium bromide. After distilling from sodium the olefin was fractionated through a ten plate column and the fraction boiling at 68.0–68.7° at 645.8 mm. was taken as the heart cut. The boiling point at 760 mm. was calculated by the method of Hass and Newton (23) to be 72.75–74.45° or 74.1° for the midpoint.

2,4,6-Trimethyl-3-heptene.[8] This olefin was prepared by the dehydration of 2,4,6-trimethylheptanol-4 with potassium acid sulfate. The tertiary alcohol was prepared from isobutylmagnesium bromide and methyl isobutyl ketone. After refluxing the olefin with sodium, it was fractionated in a semimicro column packed with $\frac{1}{16}$-inch stainless steel helices. Two fractions, boiling from 145–148°, were selected as the heart cut. The boiling point at 760 mm. was found to be 148.2°, the index of refraction was n_D^{20} 1.4201 and the density was d_4^{20} 0.7366.

Anal. Calc'd for $C_{10}H_{20}$: C, 85.7; H, 14.3.
Found: C, 85.3; H, 14.5.

Acknowledgment. The authors wish to thank Mr. C. D. Nuebling, Sr., for the analyses reported in this paper and Dr. R. D. Hinkel for the use of the semimicro Cottrell boiling point apparatus. We are also indebted to the Council on Research of The Pennsylvania State College for a part of the funds used in this work.

SUMMARY

The boiling points of ten paraffins not agreeing with the calculated boiling point have been reexamined. All of these have been corrected excepting two which are 3-methyl-3-ethyl and 3,3-diethyl paraffins. Additional new members of these series have been synthesized, and it has been established that these paraffins have abnormally high boiling points. Therefore, a boiling point number has been assigned these structures.

Also, the boiling points of 12 olefins not in agreement with the calculated

values have been reexamined. New boiling points published for olefins containing the tetrasubstituted double bond require that the boiling point number of this linkage be raised from 2.8 to 3.2. All of the remaining olefins have been accounted for by literature correction, new boiling points, or by the application of paraffin boiling point numbers. Two new olefins, 2,2,5-trimethyl-3-hexene and 2,2,5,5-tetramethyl-3-hexene, appear to have abnormally low boiling points.

STATE COLLEGE, PENNSYLVANIA

REFERENCES

(1) KINNEY, *J. Am. Chem. Soc.*, **60**, 3932 (1938).
(2) (a) EGLOFF, SHERMAN, AND DULL, *J. Phys. Chem.*, **44**, 730 (1940); (b) FRANCIS, *Ind. Eng. Chem.*, **33**, 554 (1941); **35**, 442 (1943); (c) TAYLOR, PIGNOCCO, AND ROSSINI, *J. Research Natl. Bur. Standards*, **34**, 413 (1945); (d) CORBIN, ALEXANDER, AND EGLOFF, *Ind. Eng. Chem.*, **38**, 156 (1946); (e) WIENER, *J. Am. Chem. Soc.*, **69**, 17 (1947); (f) PLATT, *J. Chem. Phys.*, **15**, 419 (1947).
(3) KLAGES, *Ber.*, **76**, 788 (1943).
(4) KINNEY, *Ind. Eng. Chem.*, **32**, 562 (1940), Table IV.
(5) (a) BUTLER, Diss. Ohio State Univ. (1940); (b) DOSS, "Physical Constants of the Principal Hydrocarbons," The Texas Co., N. Y., 4th Ed., 1943.
(6) *Chem. Zentr.*, **104**, I, 45 (1933).
(7) FESTRAETE, *Bull. soc. chim. Belg.*, **41**, 328 (1932).
(8) KUHN AND L'ORSA, *Ber.*, **64**, 1732 (1931).
(9) KARRER, *Helv. Chim. Acta*, **20**, 1180 (1930).
(10) JACQUEMAIN, *Ann. chim.*, **19**, 522 (1944).
(11) PETROV AND KAPLAN, *J. Gen. Chem. (U.S.S.R.)*, **12**, 99 (1942).
(12) SHIRM AND BESENDORF, *Arch. Pharm.*, **280**, 64 (1942).
(13) HOWARD, MEARS, FOOKSON, POMERANTZ, AND BROOKS, *J. Research Natl. Bur. Standards*, **38**, 365 (1947).
(14) CAMPBELL AND O'CONNOR, *J. Am. Chem. Soc.*, **61**, 2897 (1939).
(15) BROOKS, *J. Research Natl. Bur. Standards*, **24**, 44 (1940).
(16) CLINE, Diss. Ohio State Univ. (1940); ref. (5) (b).
(17) NAZAROV, *Ber.*, **69**, 21 (1936).
(18) TUOT, *Compt. rend.*, **197**, 1434 (1933); *Bull. soc. chim.*, **9**, 889 (1942).
(19) PETROV, *J. Gen. Chem. (U.S.S.R.)*, **9**, 2129 (1939).
(20) NAZAROV, *Ann. Leningrad State Boulnoff Univ. Chem. Ser.*, **1**, 123 (1935).
(21) PETROV, PAVLOV, AND MAKAROV, *J. Gen. Chem. (U.S.S.R.)*, **11**, 1104 (1941); *Chem. Abstr.*, **37**, 3731 (1943).
(22) KUHN AND HOFFER, *Ber.*, **67**, 357 (1934); HENNE, CHANAN, AND TURK, *J. Am. Chem. Soc.*, **63**, 3474 (1941).
(23) HASS AND NEWTON, p. 1757, "Handbook of Chemistry and Physics," 29th Edition, 1945, Chemical Rubber Company, Cleveland, Ohio.
(24) HINKEL, Ph.D. Thesis, The Pennsylvania State College, 1946.
(25) WIBAUT, *Rec. trav. chim.*, **58**, 329 (1939); **59**, 1220 (1940).
(26) CAMPBELL AND EBY, *J. Am. Chem. Soc.*, **62**, 1800 (1940).
(27) SCHMIDT AND BOORD, *J. Am. Chem. Soc.*, **54**, 759 (1932).
(28) SWALLEN AND BOORD, *J. Am. Chem. Soc.*, **52**, 654 (1930).