THE AMERICAN MINERALOGIST

JOURNAL OF THE MINERALOGICAL SOCIETY OF AMERICA

VOL. 16 FEBRUARY, 1931 No. 2

IMMERSION LIQUIDS OF LOW REFRACTION

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Larsen[1] lists twenty-three minerals having indices lower than 1.45. Winchell[2] in his recent volume lists twenty-five in this range. Among these are such common minerals as opal, fluorite, and chrysocolla, as well as the familiar natron and cryolite. Forty-two optically known artificial inorganic substances[3] also have indices below 1.45. All these substance have hitherto been excluded, for practical purposes, from those which can be easily identified by the immersion method, chiefly because all liquids of corresponding indices have certain properties which render their use difficult, if not impossible. Most of those listed[4] have a solvent action on the crystals in question. This is particularly true of water, the alcohols, and their mixtures. Other liquids are known but these have such high vapor pressures that it is impossible to obtain reliably constant mixtures of any two to give intermediate values.

The writers have endeavored to fill the low index gap by fractionating and determining the optical properties of the lower petroleum distillates. Except for the lowest individual fractions, these liquids are ideal immersion media. They dissolve no known inorganic compounds; they may be obtained with almost any index without mixing; the index is constant on evaporation, and they are stable. The lowest members suffer the disadvantage of being very

[1] Esper S. Larsen, The Microscopic Determination of the Nonopaque Minerals, *U. S. G. S. Bulletin* **679**, p. 163, 1921.

[2] N. H. Winchell and A. N. Winchell, Elements of Optical Mineralogy, Part III, **1929**, p. 118 and p. 124.

[3] Alexander N. Winchell, The Optic and Microscopic Characters of Artificial Minerals, *University of Wisconsin Studies in Science*, No. **4**, p. 162, and pp. 168–9, 1927.

[4] Albert Johannsen, Manual of Petrographic Methods, **1918**, pp. 260–1. Esper S. Larsen, *op. cit.*, p. 15. Emile M. Chamot and Clyde W. Mason, Handbook of Chemical Microscopy, Vol. **I**, p. 386, 1930.

volatile, but even they have the very real advantage of not changing index appreciably on evaporation. They are no less accurate media than the upper members, but they allow the microscopist very little time to make observations before they have evaporated from the slide; there is the further attendant difficulty that their rapid evaporation lowers the temperature.

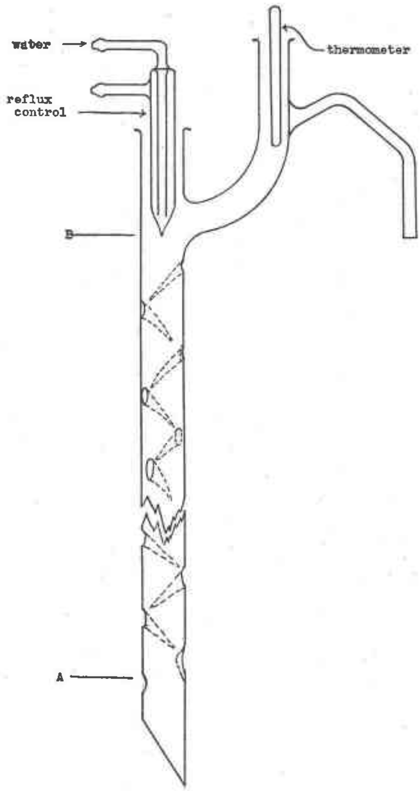


FIG. 1.

Several of the lower paraffins may be had on the market, but the number obtainable is insufficient to cover the desired range, and they can not be mixed to obtain intermediate liquids of lasting indices. For the purpose of immersion media, better liquids, although chemically less pure,[5] are obtained by re-fractionating com-

[5] This, while a fraction may have the boiling point of, say, nonane, it will not be composed of any single species of nonane nor indeed a mixture of nonane species,

mercial petroleum fractions, such as petroleum ether, the ligroins, gasolene, and kerosene. Fractions may be obtained between any desired boiling point limits, and if these be sufficiently narrow, the indices will change only very slightly on evaporation. The extent of these changes is indicated in Table IV; the first column giving the indices of representative liquids before evaporation, and the second, the indices after evaporation to one half the original volume in case this was possible, and if not, after air had been bubbled through them for eight hours.

In the writers' distillations two columns were used, both of the Vigreux type. A diagram of one is shown in Fig. 1. The larger was thirty inches from *A* to *B* and twenty millimeters inside diameter; the smaller, twenty inches from *A* to *B* and twenty-four millimeters inside diameter. The projections are not 90° apart but about 100°. This prevents the gases going directly up the perpendicular channels which would otherwise exist. It is necessary to exercise great care that each projection is so placed that the liquid dropping from its point will fall at the base of the second projection below; thus the fractionation is much better and the rate of distillation may be much more rapid without "choking." The reflux control must be supplied from a constant pressure device and the rate of flow of the water must be controlled by a needle valve. The water must, of course, be very nearly constant in temperature.

Five commercial distillates were used: petroleum ether, two ligroin's (Eastman's No. 513 and No. 1628), gasolene, and kerosene. All fractions obtained between 150° and 200° were redistilled once and all those below 150° twice. The limits are shown in Table II. The tables and graphs indicate concisely the results obtained.[6]

Table I indicates the relation between the boiling point and the volume of each fraction obtained on the first distillation, starting with a liter in each case. Under kerosene the second distillation is also listed. It is regretted that this information was not obtained for the whole series; however, that for kerosene will serve to indicate

but will be a complex mixture of hydrocarbons concerning the exact nature of which little is known. It is impossible to separate such a mixture into its pure components because of the great similarity of properties. For this reason it is an ideal immersion medium.

[6] Due to the differences in commercial distillates produced at different times and by various manufacturers, the values given may be expected to differ somewhat in attempts at duplication. The values given will, however, serve as welcome guides when the preparation of a set of liquids of any desired range is required

TABLE I

Volume of Fraction on First Distillation 1000 cc. Used in Each Case					Volume of Fraction on Second Distillation of Kerosene	
Boiling point	Petroleum ether	Ligroin No. 513	Ligroin No. 1628	Gasolene		
20– 25	60					
25– 30	150					
30– 35	160					
35– 40	190			40		
40– 45	50			40		
45– 50	40			30		
50– 55	20			30		
55– 60	20			20		
60– 65	30			30		
65– 70	60			60		
70– 75	25	85		120		
75– 80	20	90		60		
80– 85	30	180		60		
85– 90		230	55	70		
90– 95	855	170	200	60		
95–100		85	270	60		
100–105		30	75	50		
105–110		10	50	40		
110–115			60	40		
115–120		880	50	30		
120–125			50	20		
125–130			45			
			855	860		
135–140					10	30
140–145					5	10
145–150					10	15
150–155					25	10
155–160					5	20
160–165					15	20
165–170					50	30

TABLE I (*continued*)

Volume of Fraction on First Distillation 1000 cc. Used in Each Case					Volume of Fraction on Second Distillation of Kerosene	
Boiling point	Petroleum ether	Ligroin No. 513	Ligroin No. 1628	Gasolene		
170–175					40	30
175–180					30	50
180–185					20	30
185–190					70	45
190–195					40	50
195–200					30	45
200–205					40	30
205–210					20	10
210–215					40	45
215–220					40	40
220–225					85	75
225–230					30	30
230–235					100	90
235–240					50	45
240–250					40	30
250–260					50	45
					845	825

what might be expected. Fig. 2 shows the values for kerosene graphically. Table II and Fig. 3 show the index at different temperatures for the final fractions. In Fig. 4 the index at 22°C is plotted against the upper boiling point limit. Whether the changes in slope are due to changes in equilibrium of the distilling column or to the constitution of the liquid we are not prepared to say, nor is it a matter of any importance to the work. Table III lists the dispersion, $n_F - n_C$, at 22°C for various indices.

TABLE II

INDEX OF REFRACTION AT DIFFERENT TEMPERATURES

Boiling point	22°C	30°C	40°C	Boiling point	22°C	30°C	40°C
30– 32	1.3548			110–112	1.4074	1.4034	1.3983
32– 34	1.3562			112–114	1.4077	1.4038	1.3990
34– 35	1.3572	1.3521		114–116	1.4076	1.4037	1.3990
35– 37	1.3582	1.3533		116–118	1.4072	1.4032	1.3984
37– 40	1.3611	1.3562		118–120	1.4074	1.4038	1.3983
40– 42	1.3643	1.3593	1.3547	120–122	1.4080	1.4040	1.3993
54– 56	1.3739	1.3692	1.3642	122–124	1.4093	1.4052	1.4007
60– 62	1.3760	1.3716	1.3655	124–126	1.4108	1.4063	1.4018
62– 64	1.3776	1.3731	1.3672	126–128	1.4122	1.4080	1.4037
65– 67	1.3793	1.3750	1.3692	128–130	1.4146	1.4103	1.4059
67– 68	1.3838	1.3790	1.3737	130–135	1.4187	1.4145	1.4098
67– 70	1.3838	1.3790	1.3738	135–140	1.4212	1.4172	1.4128
70– 71	1.3889	1.3841	1.3788	140–145	1.4226	1.4188	1.4140
71– 72	1.3903	1.3857	1.3801	145–150	1.4241	1.4203	1.4157
74– 76	1.3967	1.3923	1.3868	150–155	1.4280	1.4241	1.4198
76– 78	1.3989	1.3945	1.3890	155–160	1.4308	1.4273	1.4224
78– 80	1.4013	1.3970	1.3915	160–165	1.4330	1.4293	1.4248
80– 82	1.4037	1.3991	1.3840	165–170	1.4349	1.4311	1.4268
86– 88	1.3993	1.3950	1.3899	170–175	1.4360	1.4323	1.4280
88– 90	1.3994	1.3953	1.3900	175–180	1.4381	1.4343	1.4300
90– 92	1.3991	1.3948	1.3897	180–185	1.4398	1.4360	1.4318
92– 94	1.3986	1.3946	1.3893	185–190	1.4411	1.4376	1.4330
94– 95	1.3992	1.3950	1.3900	190–195	1.4428	1.4393	1.4348
95– 96	1.4000	1.3957	1.3906	195–200	1.4444	1.4409	1.4368
96– 97	1.4008	1.3967	1.3917	200–210	1.4468	1.4433	1.4393
97– 98	1.4011	1.3870	1.3923	210–215	1.4479	1.4443	1.4400
98–100	1.4027	1.3983	1.3936	215–220	1.4491	1.4459	1.4417
100–102	1.4039	1.3998	1.3950	220–225	1.4500	1.4469	1.4424
102–104	1.4052	1.4011	1.3963	225–230	1.4520	1.4487	1.4443
104–106	1.4059	1.4021	1.3970	230–235	1.4530	1.4497	1.4453
106–108	1.4068	1.4030	1.3978	235–240	1.4550	1.4518	1.4473
108–110	1.4073	1.4034	1.3982	240–250	1.4569	1.4537	1.4495
				250–260	1.4593	1.4563	1.4518

TABLE III

DISPERSION AT 22°C

Index	Dispersion
1.3500	.0058 ± .0001
1.3600	.0062 ± .0001
1.3700	.0064 ± .0001
1.3800	.0068 ± .0001
1.3900	.0070 ± .0001
1.4000	.0073 ± .0001
1.4100	.0076 ± .0001
1.4200	.0080 ± .0001
1.4300	.0083 ± .0001
1.4400	.0086 ± .0001
1.4500	.0090 ± .0001
1.4600	.0092 ± .0001

TABLE IV

CHANGE OF INDEX ON EVAPORATION

Before	After	Change	
1.3562	1.3561	−.0001	
1.3776	1.3783	+.0007	
1.3838	1.3843	+.0005	Evaporated to ½ volume.
1.3991	1.3992	+.0001	
1.4059	1.4057	−.0002	
1.4093	1.4091	−.0002	
1.4212	1.4210	−.0002	
1.4381	1.4381	.0000	Air bubbled through for 8 hours.
1.4479	1.4479	.0000	
1.4530	1.4530	.0000	

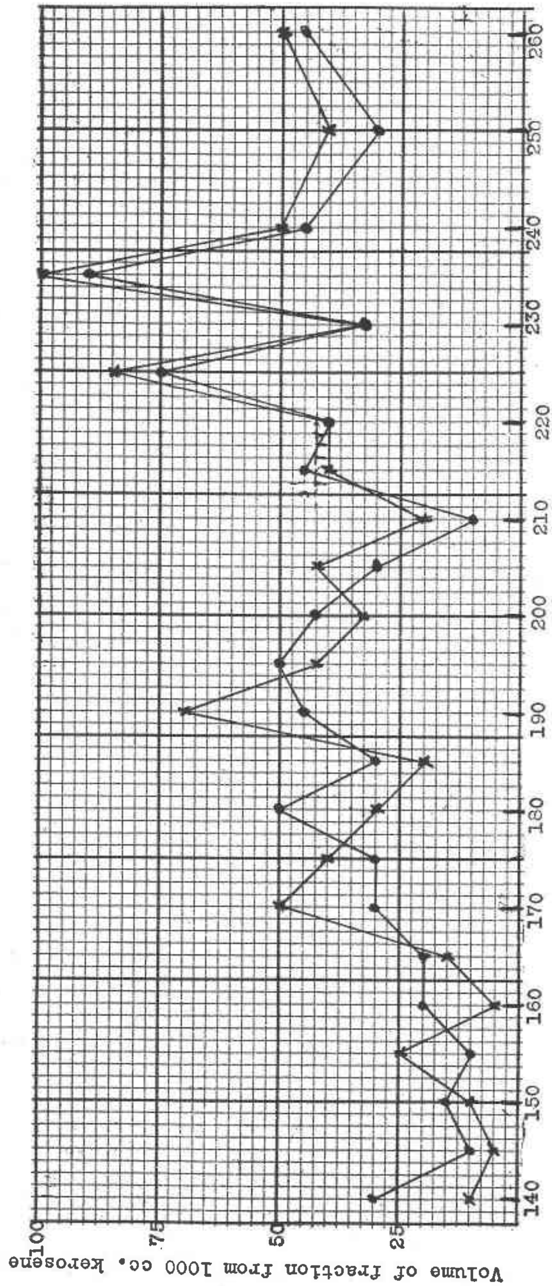
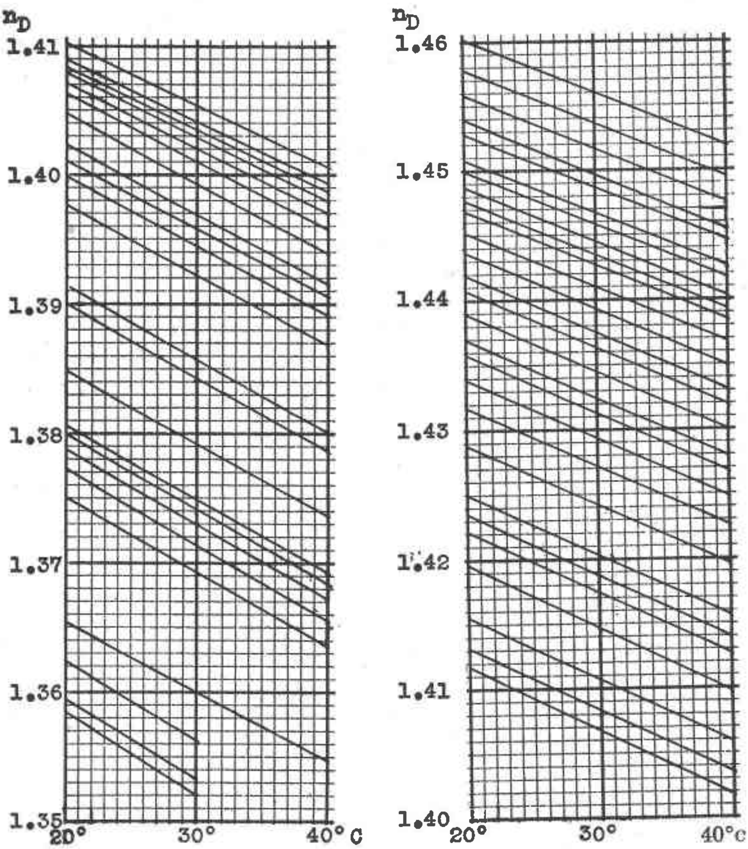
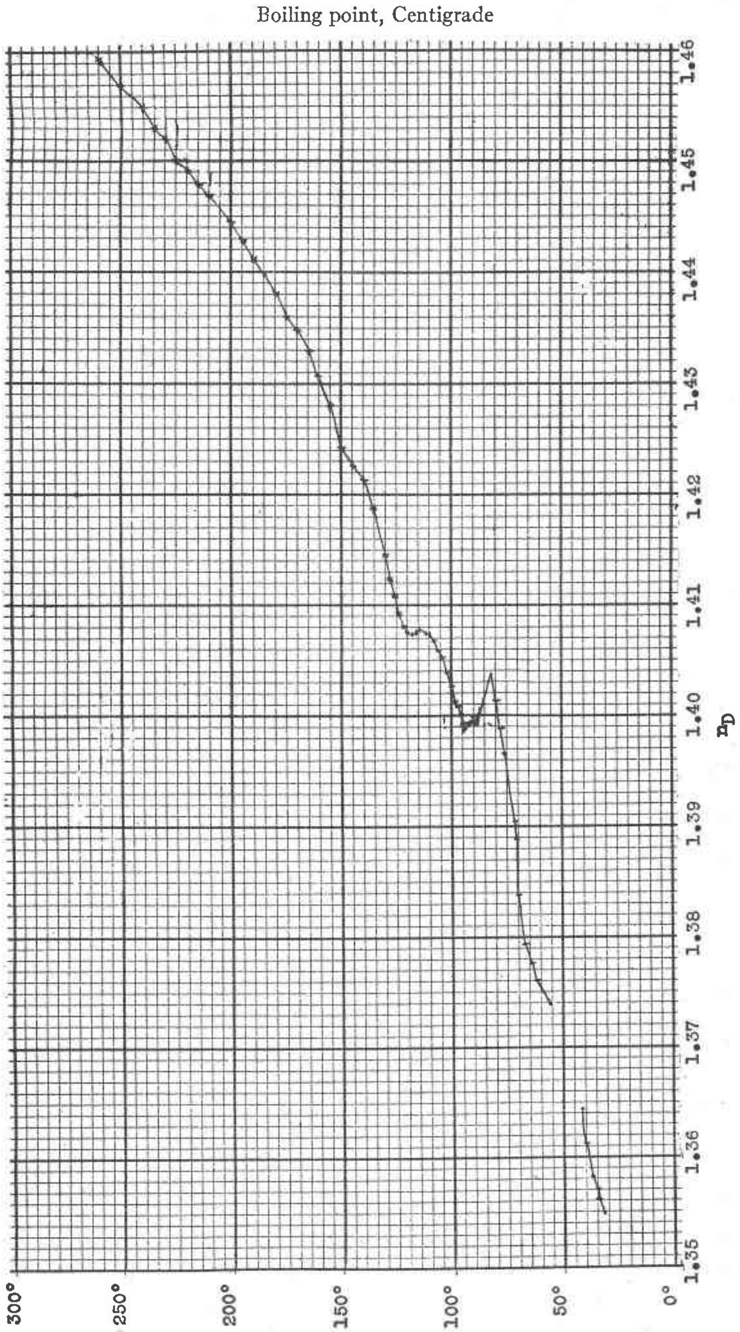


FIG. 2. Volume of fractions derived from one liter of kerosene at different temperatures.
× first distillation
• second distillation



Indices of fractions at different temperatures

FIG. 3. Variation of refractive index with temperature.



Index of Final Fractions at 22°C.

FIG. 4. The refractive index of fractions as a function of the boiling point.