side of the films were preserved in a similar way as a light pattern on a dark background which fixed-out only with difficulty.

Finally, four different samples of fluorine gave 25 photographs identical in appearance except for density, and showing altogether six clearly resolved maxima and minima (12 features) of a simple diatomic pattern extending to $s \approx 30$. The heavier pictures were taken with gas pressures ranging up to 1000 mm., nearly the most intense electron beam we could get, and only one or two tenth-second exposures. Because fluorine could not rapidly be removed from the apparatus by condensation in the liquid air traps, each exposure was followed by a high-pressure discharge of the electron tube. Neither this nor the rapid spread of the gas through the diffraction chamber seems to affect adversely the diffraction pattern obtained; excellent photographs of nitrogen and oxygen, which gave the correct interatomic distances (as known from band spectra) to within 0.01 Å. have been obtained[10] under similar circumstances. Our fluorine photographs are not as clean as those of, say, oxygen, but we feel that they are nevertheless entirely satisfactory.

Presentation of Measurements

Each of us has made two or more sets of measurements of the ring diameters on different groups of photographs made from the last four samples of fluorine. The corresponding values of $s_0 = (4\pi/\lambda)\sin(\varphi/2)$ were calculated and compared with the positions of the maxima and minima of $(\sin rs)/rs$, the simplified intensity function for a diatomic molecule. The resulting values are given in Table I where we have foregone a presentation of the observed s_0 values in order to give separately the results of the various sets of measurements. Each value of r given is obtained from a consecutive series of features, the measurements from the innermost and outermost rings only being rejected from the average. In the column headed "Average deviation" there is given for each set of measurements the average deviation from the value "Average r" of the values obtained from the individual features included in "Average r." The consistency of the values given in Table I is such that we believe our result to be as accurate as those obtained for other diatomic molecules by the electron-diffraction method.[10] We accordingly report for the normal state of the fluorine molecule $r_{\text{F-F}} = 1.435 \pm 0.010$ Å.

TABLE I

Observer	Features in av.	Readings on each	Features incl. in av.	Av. r, Å.	Av. dev., Å.
V.S.	7	6	2nd max.–5th max.	1.428	0.008
V.S.	7	5	2nd max.–5th max.	1.430	.004
M.T.R.	6	10	2nd max.–5th min.	1.437	.004
V.S.	8	5	2nd min.–5th max.	1.431	.004
V.S.	8	10	2nd min.–5th max.	1.436	.007
D.P.S.	7	10	2nd min.–5th max.	1.441	.008
D.P.S.	6	5	4th min.–6th max.	1.430	.004
M.T.R.	9	8	2nd max.–6th max.	1.435	.008
			Final average	1.434	
			Average deviation	0.004	

Summary

An electron diffraction reinvestigation of the internuclear distance in the fluorine molecule leads to the value 1.435 ± 0.010 Å., substantiating the provisional value found by Brockway.[5]

PASADENA, CALIFORNIA RECEIVED JULY 7, 1941

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[10] V. Schomaker and D. P. Stevenson, unpublished results.

[CONTRIBUTION FROM RESEARCH LABORATORIES, SHAWINIGAN CHEMICALS LTD.]

The Viscosity Function. III. Complete Viscosity Range*

BY ERNEST P. IRANY

It has been shown in the first paper of this series[1] that the viscosity functions of all *ideal mixtures*[2] follow one common law which, deduced by graphical means, is represented by the "ϕ scale" of viscosity. The observation that the temperature–viscosity functions of most pure liquids appear to obey the same or a closely related scale-law indicates its fundamental significance. The combined evidence very amply supports the conclusion that an *ideal* law governing the additivity of liquid viscosity is definable and that its expression by the ϕ scale is unique.

Thanks to abundant and very accurate viscosity data on pure liquids and their mixtures, the construction of the ϕ scale between about 0.25 and 50 centipoises encountered no great difficul-

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* Original manuscript received July 5, 1940.

[1] Irany, THIS JOURNAL, 60, 2106 (1938).

[2] "A system is *ideal* if there are no changes of molecular state, *i. e.*, no association, dissociation or combination, on admixture of its components" (Findlay, "Osmotic Pressure," p. 30). These conditions may be considered fulfilled if admixture produces no calorimetric, gravimetric or cryoscopic effects. Many such systems are known and have been carefully studied; they provided the original data for the construction of the ϕ scale as well as proof of its validity. The terms *ideal* and *ideality* as used in the present work have no connection with the concept of an "ideal liquid" composed of spherical or perfectly symmetrical molecules.

ties. The task of extending the scale into the range of extremely high and extremely low viscosities does not merely call for a repetition of the same graphical technique but involves far more complex problems. Thus it becomes necessary again to prove that a unique law of ideal mixture and, independently, a unique viscosity-temperature relationship exist and that these laws can be expressed by continuous functional scales throughout the whole range of the liquid state. Given one such scale based exclusively on ideal mixture standards and another one based exclusively on ideal temperature functions, it may be possible to decide whether they are actually identical or merely similar. The importance of this distinction need not be emphasized.

The choice of standards of ideal behavior requires very careful consideration. Those criteria of ideality which are independent of viscosity measurements[2] steadily decrease in sensitivity as viscosity increases and become quite useless in the study of highly viscous systems. This restriction is the more serious as causes of non-ideality are certain to accumulate in substances of complex molecular structure. For these reasons it is necessary to base the standards of ideality entirely on the principles which the empirical but exact ϕ scale has revealed in its already known lower section. In the case of mixtures, systems of steep gradient, *i. e.*, composed of liquids of very dissimilar viscosities, may be used whose ideality can be ascertained within the known field but which also extend into the unknown range, providing new fixed points for the construction of the scale. Similarly, over-lapping viscosity-temperature functions can be used for the gradual determination of the pure temperature scale. The temperature functions of most pure liquids with but few exceptions, and those of all ideal mixtures conform with the fundamental scale function.

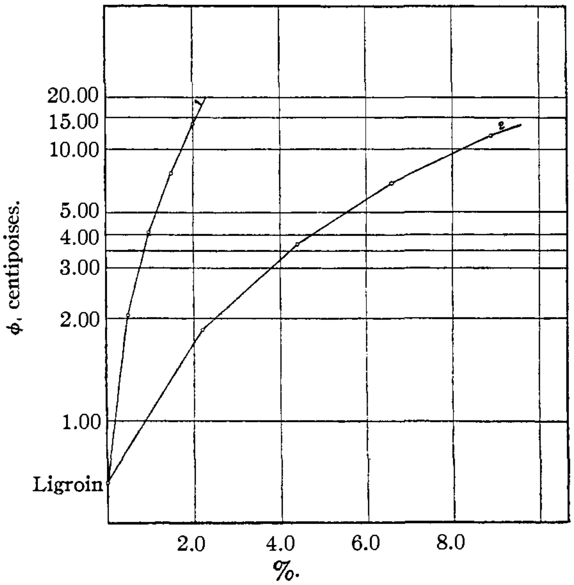

Fig. 1.—Polyisobutylene solutions in ligroin.

Mineral Oils.—One might expect to find suitable standards among the highly viscous mineral oils, in analogy with the lower aliphatic hydrocarbons which have been recognized as ideal.[1] However, many lubricating oils of various origins are non-ideal to a surprising extent, even in highly diluted solutions which can be examined within the existing range of the ϕ scale. These deviations may be ascribed to the accumulation, in large molecules, of structural groups ordinarily of negligible polarity, since groups recognized as strongly polar are known to be absent. This cumulative effect is most evident in certain macropolymers of seemingly well-known chemical constitution.[3] For example, poly-isobutylene, $(\text{—CH}_2\text{—CMe}_2\text{—})_x$, is a saturated paraffin of great chain length which should be ideal. The strong curvature in the ϕ function of its solutions in ligroin (Fig. 1) apparently is due to the combined effect of the numerous weakly electronegative methyl groups. A large number of viscous mineral oils show quite similar deviations in dilute solutions, but it is hardly possible to form a definite opinion concerning the structural groupings or constituents which are responsible for them.

American mineral oils can be divided into the mainly paraffinic mid-continent oils (Pennsylvania) and the naphthenic or asphaltic coastal oils (California, Gulf). The highly viscous fractions are probably modified to a large extent by condensation, polymerization, depolymerization and pyrolytic processes. The character and behavior of these oils is therefore determined by their source as well as by the method of refinement.

Ideal Mixtures and Temperature Functions of Mineral Oils.—Various oils, including paraffinic and naphthenic types, were first examined as to ideality in mixture with low-viscosity diluents. The solvents used were technical heptane (Eastman), ligroin, kerosene, etc., which are composed of normal hydrocarbons and known to be ideal.[1]

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(3) Irany, This Journal, **61** 1734 (1939).

Most paraffinic oils have non-ideal functions. Their mixtures with aliphatic solvents rise above, and those with aromatics fall below, linearity in the known part of the ϕ scale. Typical data are given in Table I and in the ϕ scale diagram, Fig. 2.

TABLE I

PARAFFINIC OILS. MIXTURES WITH LOW-VISCOSITY DILUENTS

Vol.% oil	Sp. gr.	Viscosity	Vol.% oil	Sp. gr.	Viscosity
Oil no. 1 + Heptane, 20°			Oil no. 1 + Benzene, 20°		
0	0.715	0.47	0	0.878	0.65
25.3	.757	1.15	25.2	.875	1.26
49.8	.756	3.41	50.4	.875	3.26
75.9	.840	19.9	75.7	.877	14.5
100	.880	244	100	.880	244
Oil no. 2 + Kerosene, 20°			Oil no. 3 + Ligroin, 20°		
0	0.814	1.92	0	0.768	0.66
25.6	.842	6.15	30.7	.807	2.69
38.7	.855	12.47	49.6	.833	8.35
40.2	.856	14.01	69.8	.857	39.9
65.3	.883	85.8	100	.888	1516
80.6	.899	388			
100	.920	5110			

TABLE II

PARAFFINIC OILS, TEMPERATURE FUNCTIONS

°C.	Sp. gr.	Visc.	Sp. gr.	Visc.	Sp. gr.	Visc.
	Oil no. 1		Oil no. 2		Oil no. 4	
0	0.891	3140	...	..	0.899	1215
10	.886	524	...	..	...	....
20	.880	244	0.920	5110	.883	197
30	...	....	.913	1860	...	....
37.7	.868	87.1	...	..	...	....
40	...	....	.907	797	.874	57.3
50	.860	47.8	...	..	...	....
60	.855	30.3	...	..	...	....
75	.845	17.4	...	..	...	....

TABLE III

NAPHTHENIC OILS, MIXTURES WITH LOW-VISCOSITY DILUENTS

Vol.% oil	Sp. gr.	Viscosity	Vol.% oil	Sp. gr.	Viscosity
Oil no. 5 + Heptane, 0°			Oil no. 5 + Heptane, 20°		
0	0.732	0.57	0	0.715	0.47
25.0	.782	1.39	24.8	.768	1.01
50.0	.833	4.96	49.8	.820	2.98
74.9	.883	42.2	74.8	.871	15.50
100	.932	3080	100	.919	390
Oil no. 6 + Heptane, 0°			Oil no. 6 + Heptane, 20°		
0	0.732	0.57	0	0.715	0.47
25.1	.785	1.54	24.9	.768	1.09
50.1	.834	6.35	49.8	.818	3.66
75.0	.885	77.3	74.8	.868	25.3
90.1	.911	888	89.9	.898	162
100	...	....	100	.917	988
Oil no. 7 + Heptane, 20°			Oil no. 7 + Kerosene, 20°		
0	0.715	0.47	0	0.814	1.93
24.9	.755	1.00	26.3	.828	3.90
49.9	.794	2.72	51.0	.842	9.33
74.9	.831	11.26	75.2	.854	27.9
100	.868	127.3	90.1	.862	64.8
			100	.868	127.3

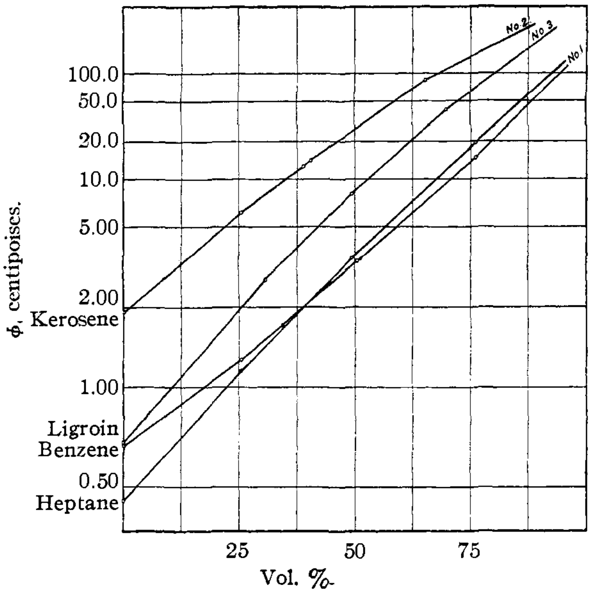

Fig. 2.—Paraffinic oils, mixtures, data Table I.

The naphthenic oils, on the other hand, have ideal functions. Their mixtures with low-boiling hydrocarbons give perfectly straight lines within the known extent of the ϕ scale, as shown in Fig. 3 (Table III). Hydrogenation does not affect the linear course, as exemplified by sample no. 7. Based on these observations in the low-viscosity range, the naphthenic oils may be accepted as

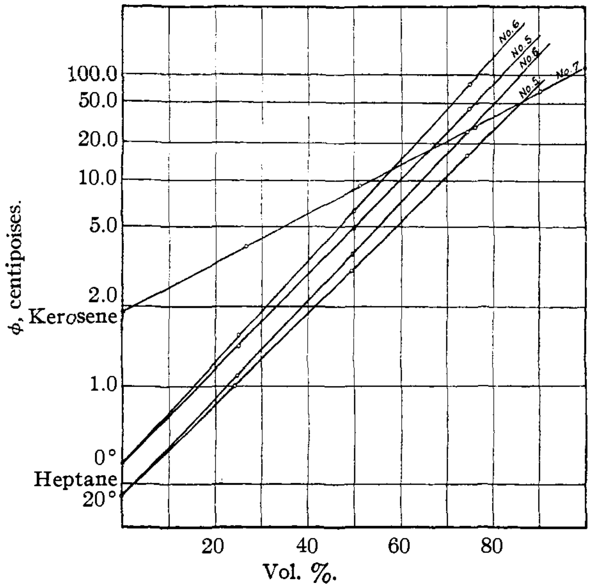

Fig. 3.—Naphthenic oils, mixtures, data Table III.

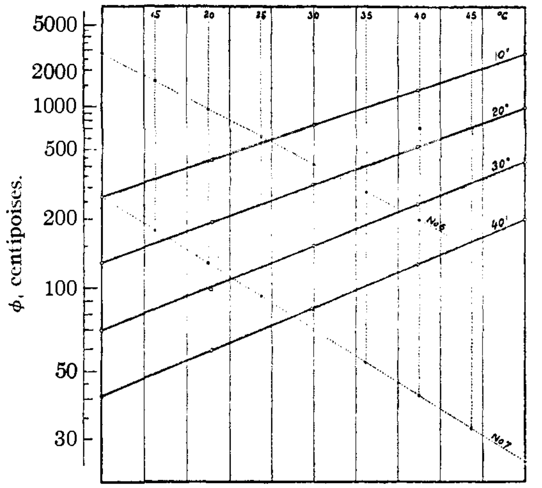

Fig. 4.—Mixture of viscous naphthenic oils, data Table V.

standards for the extension of the ideal mixture scale into the field of high viscosities.

The viscosities of various naphthenic oils were measured over a wide temperature range, see Table IV. Two of these oils (no. 6 and no. 7), of high but very different viscosities, were mixed in varying proportions and examined at 10, 20, 30 and 40° (Table V). In Fig. 4 the temperature functions of the single components and the isotherms of their mixtures are plotted into a common viscosity scale. The accurate linearity achieved in the temperature as well as mixture functions confirms that, over a wide range of high viscosities, the two functions are indistinguishable.

The temperature functions of all oils surveyed, including naphthenic as well as paraffinic types, appear linear in the ϕ scale of Fig. 4, as shown in Fig. 5. The same applies to six machine oils, not further specified, whose viscosities between 10 and 48° are recorded.[4]

TABLE IV

NAPHTHENIC OILS, TEMPERATURE FUNCTIONS

°C.	Oil no. 5 Sp. gr.	Oil no. 5 Visc.	Oil no. 6 Sp. gr.	Oil no. 6 Visc.	Oil no. 7 Sp. gr.	Oil no. 7 Visc.
−5	0.935	5990	...	..	0.884	989
−3	.933	4780	...	..	...	...
0	.932	3080	...	..	.881	561
5	...	....	0.926	4970	.877	382
10	.925	1012	.923	2760	.874	263
15	...	....	.920	1610	.871	177
20	.919	390	.917	988	.868	127.3
25	...	....	.914	626	.865	...
30	...	....	.911	414	.862	69.8
35	...	....	.908	286	.859	53.5
37.7	.909	105.8	...	..	...	...
40	...	....	.905	200	.856	40.5
45	...	....	...	..	.853	32.5
50	.900	50.7	...	..	.850	25.5
55	...	....	...	..	.847	21.2
60	.895	30.9	...	..	.844	17.0
65	...	....	...	..	.841	14.6
75	.886	17.2	...	..	.835	10.4

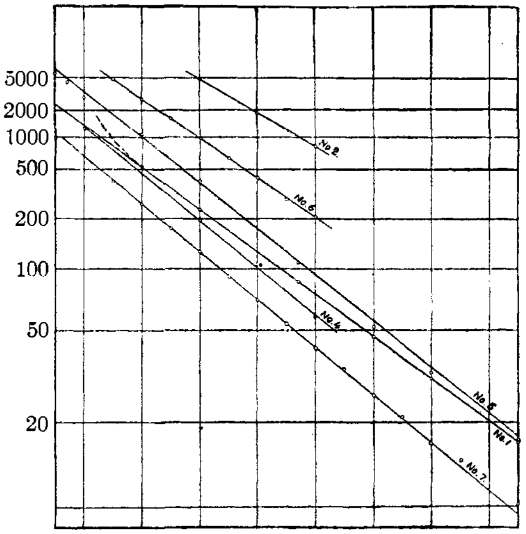

Fig. 5.—Temperature functions, data Tables II and IV.

TABLE V

NAPHTHENIC OILS, MIXTURES OIL NO. 6 + OIL NO. 7

Vol.% no. 6	Sp. gr.	Visc.	Vol.% no. 6	Sp. gr.	Visc.
	10°			20°	
0	0.874	263	0	0.868	127.3
25.8	.887	443	25.8	.880	193
50.0	.899	742	49.9	.893	306
74.9	.911	1395	74.9	.904	525
100	.923	2760	100	.917	988
	30°			40°	
0	0.862	69.8	0	0.856	40.5
25.8	.876	99.0	25.8	.869	58.9
49.8	.887	150	49.9	.881	83.8
74.8	.899	243	74.9	.893	125
100	.911	414	100	.905	200

The Low Viscosity Range.—Viscosity measurements on pure hydrocarbons, *e. g.*, butane between −23.6 and 34.5°,[5] methane, ethane, ethylene, propane, propylene and nitrogen between about −200 and −100°,[6] have supplied standards for the accurate extension of the pure temperature scale down to 0.1 centipoise.

Data on ideal mixtures are not available in sufficient number for the construction of the scale to

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(4) Landolt, Börnstein and Roth, "Physikalisch-chemische Tabellen," Berlin, 1912, p. 81.

(5) Kuenen and Wisser, *cf.* Evans, *J. Inst. Petroleum Tech.*, **24**, 50 (1938).

(6) Gerf and Galkov, *J. Tech. Phys.* (U. S. S. R.), **10**, 725 (1940).

a comparable level of low viscosity. However, as far as reliable mixture standards can be applied, a gradual but definite divergence from the pure temperature scale has become evident. The lower the viscosity, the more undeniable appears the fact that the mixture function and the temperature function of viscosity are not identical.

Discussion.—It has been possible, by the use of a large number of exact measurements, to construct the two different scales into which, for the above reason, the ϕ scale must be divided. Retaining the symbol ϕ for the ideal mixture (volume) function of viscosity, the ideal temperature function or scale may be designated by Θ. Within their respective definitions these scales seem to be absolutely precise at all viscosity levels; there can be no doubt about the uniqueness and the basic significance of the laws they express, even though the postulate of the identity of these laws must be abandoned.

As it would be inconvenient to present the whole length of the two scales in an illustration without sacrifice in resolvability, factors are given in Table VI which allow the construction of any scale section with the aid of an interpolation curve. These factors represent the length of the scale from the ordinate of 1.00 centipoise to various other viscosity levels, taking the distance between 1 and 100 cp. as an arbitrary unit of reference.

TABLE VI

Abs. viscosity centipoises	ϕ Scale (Mixtures)	Θ Scale (Temp.)	Abs. viscosity centipoises	ϕ Scale (Mixtures)	Θ Scale (Temp.)
0.100	...	−2.7	40.0	0.866	0.905
.200	−0.705	−1.41	50.0	.915	.930
.300	− .530	−0.940	60.0	.938	.949
.400	− .392	− .655	80.0	.975	.980
.500	− .289	− .462	100	1.000	1.000
.600	− .213	− .322			
.800	− .093	− .127	150	1.044	1.035
			200	1.074	1.056
1.00	0.000	0.000	300	1.109	1.084
1.50	.148	.193	500	1.150	1.115
2.00	.246	.304	600	1.165	1.124
3.00	.373	.436	800	1.184	1.140
4.00	.455	.520	1000	1.201	1.156
5.00	.510	.575	1500	1.225	1.169
6.00	.550	.615	2000	1.241	1.181
8.00	.612	.672	3000	1.271	1.199
10.0	.661	.714	5000	1.293	1.219
15.0	.738	.783			
20.0	.784	.822	Infinite		1.69
30.0	.846	.875			

Both scales differ very distinctly from logarithmic functions in that they possess finite length. This is quite obvious in the case of the Θ scale because of its linear relation to temperature which, as far as the liquid state is concerned, cannot extend beyond absolute zero and the critical temperature. However, numerous theoretical and empirical formulations of the viscosity–temperature function propose exponential terms of the general type

$$\eta = Ae^{B/T} \qquad (1)$$

in which η is absolute viscosity and T, absolute temperature; A and B are variously defined individual constants. Substituting the viscosity function which, by definition, is the law of the Θ scale

$$\Theta - \Theta_{cr} = \frac{T}{T_{cr}} (\Sigma - \Theta_{cr}) \qquad (2)$$

where Σ is the total length of the scale and T_{cr}, Θ_{cr} are the respective critical quantities, the constants can be determined so as to conform with the factors of Table VI. The resulting formula

$$\Theta - \Theta_1 = \frac{1.69}{1 + 1.38/\log \eta} \qquad (3)$$

represents the Θ scale very accurately between about 0.5 cp. and infinite viscosity. The asymptotic limit of the latter can be very accurately set at a level 1.69 units above that of 1 centipoise. The failure of equation (3) to represent the Θ scale at low viscosity levels indicates that the functions of the type (1) become invalid upon approach to the critical region; consequently, their various theoretical premises[7] may be regarded as of limited validity.

The following oils were used in the present survey. The numbers refer to tables and figures.

No. 1. Paraffinic oil (Pennsylvania). Sample by courtesy of Dr. R. B. Dow and Dr. H. A. Everett, Pennsylvania State College. This oil has been studied in earlier work, particularly on viscosity under high pressure, by Dow, *J. Applied Phys.*, **8**, 367 (1937).

No. 2. Standard Viscosity Oil No. 7, U. S. Bureau of Standards.

No. 3. Commercial "Bright Stock" (Pennsylvania). Sample by courtesy of Dr. R. B. Dow, Pennsylvania State College. Used in high pressure work by Dibert, Dow and Fink, *J. Applied Phys.*, **10**, 113 (1939).

No. 4. Liquid Petrolatum (Heavy). Commercial grade.

No. 5. Naphthenic Oil (California). See No. 1.

No. 6. Naphthenic Oil (California). Sample by courtesy of Imperial Oil Limited.

No. 7. Naphthenic Oil (Colombia), Hydrogenated. Sample by courtesy of Imperial Oil Limited and Standard Oil Company of New Jersey.

Viscosity measurements were carried out in a series of four capillary type viscometers[8] which were calibrated against absolute standards and against each other. All viscosities are given in centipoises.

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(7) Second Report on Viscosity and Plasticity, Academy of Sciences, Amsterdam, 1938; Kincaid, Eyring and Stearn, *Chem. Rev.*, **28**, 301 (1941).

(8) British Standards Institution, Viscometers No. 1–No. 4.

Pressure Functions of Mineral Oils.—The existence of a fundamental or *ideal* viscosity–pressure function has been made apparent in the Π scale of viscosity.[1] The general validity of this scale up to about 30 cp. is well supported by Bridgman's viscosity measurements on simple organic liquids under extremely high pressures. As in the case of the ϕ scale, the extension of the Π scale into the range of high viscosities depends primarily on correct criteria of ideal behavior, and the only source of such criteria are the regularities found in the lower section of the scale. Thus it may be postulated that the viscosity–pressure functions of a liquid are acceptable as standards for the construction of the Π scale if all isotherms appear linear in the same diagram and if they converge toward a common point of intersection whose abscissa represents the internal pressure of the liquid.[9]

This is very well realized in Fig. 6 which shows the measurements by Dibert, Dow and Fink[10] on a series of paraffinic oils at various temperatures.

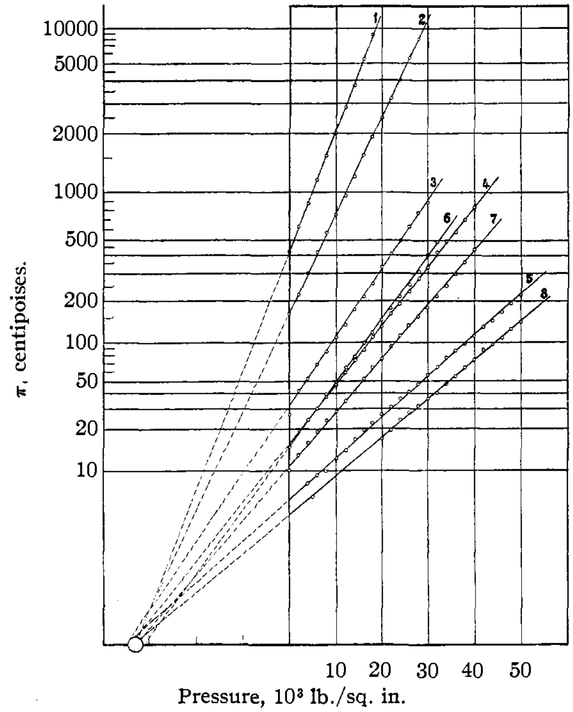

Fig. 6.—Paraffinic mineral oils, pressure function: 1, 2, "Commercial Bright Stock" at 100 and 130°F.; 3, 4, 5, high b. p. fraction of "Solvent Refined Neutral"; 6, 7, 8, same, low b. p. fraction (Dibert, Dow and Fink[10]).

The rather sharply defined region where all lines intersect indicates an internal pressure of about 32,000 lb./sq. in. (2300 kg./sq. cm.) as common to oils of the paraffinic type. Other data[11] confirm this estimate very closely.

The available measurements on naphthenic oils,[11] shown in Fig. 7, are not entirely conclusive as to strict linearity. The appreciable concavity of the lines may be connected with the more aromatic character of these oils, since the highly viscous aromatic materials extracted from paraffinic oils[11] show the same trend. The internal pressure of the naphthenic oils seems to be much lower than that of the paraffinic group, about 20,000 lb./sq. in. (1450 kg./sq. cm.).

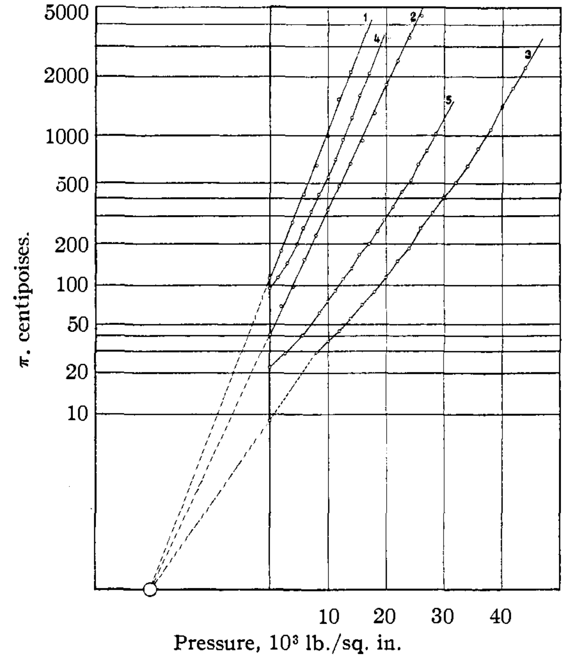

Fig. 7.—Naphthenic mineral oils, pressure function: 1, 2, 3, California oil at 100°F., 130°F., 210°F. (Dow[4]); 4, 5, acetone extract from paraffinic oil at 100°F., 155°F. (Thomas, Ham and Dow[11]).

The scale of Fig. 6, which, tentatively, may be accepted as the correct Π scale, is interesting because it approaches a purely logarithmic function in the higher viscosity levels.

Summary

The task of extending the functional viscosity scales beyond the limited range determined in the first paper of the series, demands rigorous criteria of ideality. Calorimetric, gravimetric or cryoscopic evidence becomes too uncertain in the

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(9) Under conditions well below the critical region, the internal pressure may be considered very little dependent on temperature and, at least within a limited temperature interval, characteristic of the liquid.

(10) Dibert, Dow and Fink, *J. Applied Phys.*, **10**, 113 (1939).

(11) Dow, *ibid.*, **8**, 367 (1937); Thomas, Ham and Dow, *Ind. Eng. Chem.*, **31**, 1267 (1939).

complex substances which possess high viscosity. However, very sensitive criteria of ideality are available by postulating the continuity of those principles and regularities which have been revealed in the lower ranges of the scales.

It has been proved that an ideal mixture function and an ideal temperature function can be recognized as continuous and unique throughout the range of practical viscometry, from about 0.1 to about 5000 centipoises. These scale functions of viscosity are, however, different; the earlier postulate of their identity[1] has been abandoned and replaced by a quantitatively defined relationship. The pressure scale (Π) has been extended tentatively to nearly 10,000 centipoises.

The three viscosity scales (Φ, Θ and Π) are derived from experimental data on individual liquids each of which can cover only a limited range of liquid conditions. These sections of the basic viscosity functions have been joined into continuity. It is possible to define a fictitious "*ideal liquid*" capable of passing through all conceivable variations of the liquid state without limitation, in accord with the fundamental law embodied in the three scales. The latter provide the most extensive empirical evidence to test the validity of mathematical formulations of the viscosity functions.

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QUEBEC, CANADA

RECEIVED MAY 22, 1941

[CONTRIBUTION FROM THE DEPARTMENT OF PHYSICS, LEHIGH UNIVERSITY]

Studies of the Preparation and Allotropic Transformation of Lead Monoxide[*]

BY MAX PETERSEN

When measurements of the magnetic susceptibility of lead oxides were undertaken, it was found that the observed results depended on the manner of preparing the oxide. The chemical literature regarding these oxides is somewhat discordant. Lead monoxide may be prepared dry by the vacuum decomposition of hydrated lead oxide or of lead carbonate; but disagreement still persists as to which crystalline form, yellow or red, reresults from these decompositions.[1,2,3,4] Nor is it settled how the one crystal form is to be converted to the other. This paper reports some evidence on these points; the related magnetic observations will form another report.

The disagreement first mentioned has its origin in the manner of preparing the hydrate or in its consequent form; it has but one known crystal lattice. The author has used hydrates prepared as directed by (a) Pleissner[1] or Eberius,[5] (b) Hüttig,[3] (c) Kohlschütter and Roesti,[2] and (d) Müller.[6] One solution was always added to the other in a very fine vigorous jet driven by nitrogen under pressure, both having been cooled below 5°. Since the difference in the precipitates is involved in the results, it must be described.

(a) Following Eberius the barium hydroxide solution was added to the lead acetate. The former was meticulously drawn clear of suspensions from the flask in which it had been prepared and excess solids had been allowed to settle for a day or more. The hydrate so obtained is coarse, well-developed, cross-formed crystals sometimes attaining great ornateness. (b) Hüttig's preparation "$PbO/H_2O(1)$" was a fine powder which barely showed the cross-form of the crystals. (c) Kohlschütter's preparation appeared as small rudimentary crosses, coarser than (b) but not approaching (a). (d) The Müller hydrate was the finest precipitate obtained. By comparison of its Brownian movement with that of some zinc oxide of known size, the particle size of this hydrate was usually 0.2 to 0.4 μ.

Dehydration was always done in a good Hyvac vacuum, using an all-glass system save at the pump connection. The hydrate, usually about 10 g., was spread on the bottom of a 125-ml. Erlenmeyer flask. This flask was fused to a 12-mm. Pyrex tube about 50 cm. long leading to a 3.5 × 60 cm. phosphorus pentoxide drying tube, connected to the pump by a short rubber tube. An electric furnace lined with heavy copper plate maintained the desired temperature, usually be-

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(3) G. Hüttig and B. Steiner, *Z. anorg. allgem. Chem.*, **197**, 257 (1931).

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(5) E. Eberius, Thesis, Leipzig, 1931.

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