The Viscosity of Acetone-water Solutions Up to Their Normal Boiling Points

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The viscosity of acetone-water liquid solutions has been measured over the entire concentration range at temperatures from 20°C. to generally within 1° to 10°C. of the normal boiling point. A capillary suspended-level viscometer was used, and the authors estimate that the results are accurate to ±0.2%. Both kinematic and absolute viscosities are given, and the results have been extrapolated to the boiling point of the solutions. The calibration of the Cannon-Ubbelohde viscometer is discussed in detail.

One of the aims of Research Project 1, Tray Efficiencies in Distillation Columns, sponsored by the American Institute of Chemical Engineers, was the determination of the effect of the physical properties of binary liquid systems on distillation efficiency. The viscosity of the system is required for the calculation of the liquid-phase diffusivity by the method of Wilke and Chang (*18*) and for the calculation of the liquid-phase Schmidt group. The physical properties are required at the boiling point, where the mass transfer is occurring, in order to relate the efficiency and the physical properties.

The viscosity of binary systems has been widely reported in the literature of the past sixty years, usually in connection with an attempt to devise equations for the prediction of the viscosity of mixtures, based on the viscosities of the pure components. Much of this work, however, is subject to errors resulting from the failure to apply the necessary corrections to the viscometer, for example, corrections for surface-tension effects or for kinetic-energy effects. In addition, not until recently (*15*) has there been available an accurate value for the viscosity of water for use as a primary standard for viscosity. With rare exseptions, viscosities of binary systems are not available at temperatures above 45°C.; for estimation of the boiling-point viscosity, an extrapolation of 15° to 60°C. would be required.

This investigation was designed to provide accurate liquid viscosity data for the acetone-water system over the entire concentration range. The investigation was carried out at temperatures from 20°C. to within 1° to 10° of the boiling point of the solution, and these data allowed estimation of the boiling-point viscosity with a high degree of accuracy.

EQUIPMENT AND PROCEDURE

Viscometer

General. After a survey of the various types of available viscometers and preliminary investigation of several, the Cannon-Ubbelohde viscometer was selected as best suited for this work, and two (size 25) were used. The instrument is of the "suspended-level" type, a modification the design of Ubbelohde (*17*). Its advantages include no significant variation of viscometer constant over the temperature range used, as well as no kinetic-energy correction for viscosities greater than 0.3 centistoke, and exact control of the filling volume is not required. It is capable of high precision (< ± 0.1% deviation) in routine use, with the precautions specified below.

The viscometer is shown in Figure 1. Sufficient sample (about 11 ml.) was introduced into tube *A* to fill bulb *D* to the lower filling line. This level was never higher than the upper filling line when the viscometer was mounted in the vertical position at the temperature of measurement. The viscometer, supported in the bath by rubber stopper cut to fit closely around tubes *A* and *B*, was aligned vertically and allowed to reach thermal equilibrium. In preliminary work the time required for the sample to flow between the two calibrated marks of bulb *F* was recorded at intervals to determine the minimum equilibration

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time. Entirely reproducible efflux times were obtained at all temperatures after 20 to 30 min., and 30 min. was adopted as the minimum equilibration time.

After the viscometer had been in the constant-temperature bath for the required time, tube *C* was closed by a small rubber tube attached to a 5-ml. glass syringe, and slight suction was applied to tube *B* by means of a rubber tube attached to a 50-ml. glass syringe. With some liquids it was necessary to draw the sample about halfway up tube *H* to prevent the entrance of air through the tube. When the liquid meniscus had traveled through bulb *F* and had about half-filled bulb *E*, suction was released, and tube *C* was opened, in that order, so that the sample dropped away from the lower end of the capillary and bulb *G* was emptied. The time required for the liquid meniscus to travel between the marks at the upper and lower ends of bulb *F* was recorded as the sample efflux time. Experiments which investigated the effect of filling bulb *E* to various levels failed to demonstrate any difference in efflux time, regardless of the initial level in *E*. Experiments in which the sample was forced up by slight pressure on tube *A*, designed to reveal any differences caused by application of pressure rather than suction to the sample, failed to show any difference in efflux time. Samples which were aerated before running had the same efflux time as nonaerated samples. The speed with which the sample was drawn up, as long as bubble formation in the liquid was avoided, had no detectable effect on efflux time. Allowing the sample to stand for 10 min. before running, after it had been drawn up into bulb *E* in an effort to dissipate any induced static charge, did not change the efflux time. Since the suction method was more convenient, it was used in all the following work.

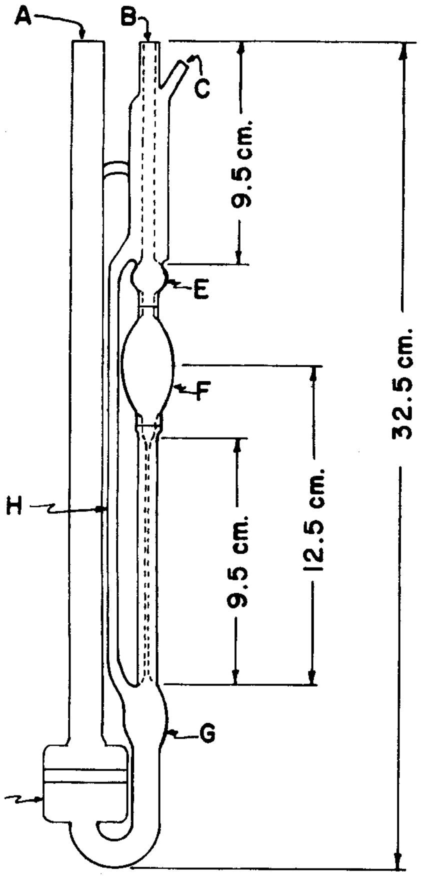

Fig. 1. Cannon-Ubbelohde viscometer.

At least three, and usually four, separate observations of efflux time were made with each sample. For the calibration runs, at least six observations were made. These multiple runs always agreed within ±0.1% and usually showed even less variation. More reproducible results were obtained when the viscometer walls were wet by the sample prior to the recorded runs.

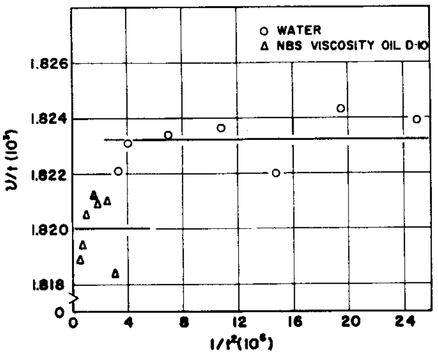

Fig. 2. Calibration curve, viscometer A19.

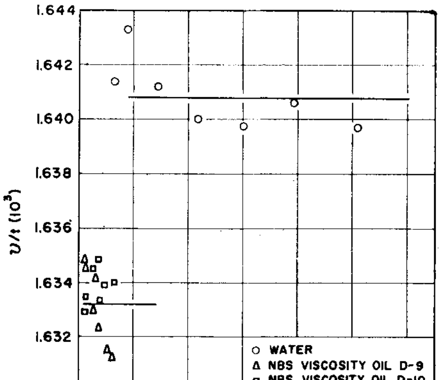

Fig. 3. Calibration curve, viscometer A43.

Samples for analysis were obtained by inserting the tip of a 6-in. 20-gauge stainless-steel hypodermic syringe needle into the center of bulb *F*, Figure 1, and withdrawing about 1 ml. with a 5-ml. glass syringe. A sample was withdrawn and analyzed prior to the first run, once during the series of runs, and following the final run in order to detect any change in sample concentration during the run. No appreciable differences in concentration were found, even at the highest temperatures used, and the average of these analyses was reported.

Temperature Control. Very careful temperature control is required for accurate and reproducible results. For temperatures between 20° and 40°C., a Model A Fisher Isotemp Bath provided control within ±0.005°C. Cooling was provided by a four-coil copper tube attached to a refrigerated water-methanol line. For temperatures between 50° and 90°C., an 18-liter water bath equipped with a circulating pump and heating coils and a tungsten-mercury thermoregulator attached to a relay provided control within ±0.02°C. The latter bath was illuminated by a circular fluorescent lamp.

Temperatures were measured during each run by a mercury-in-glass thermometer graduated in 0.1°C. intervals; the temperature was estimated to 0.01°C. by the use of a cathetometer. The true bath temperature and the thermometer in use were checked daily by reference to a thermometer which had been calibrated by the National Bureau of Standards and compared with at least one other N.B.S.-calibrated thermometer. The ice-point readings of these thermometers were checked at intervals.

Timing. Some of the efflux times were measured with stopwatches graduated in 0.2-sec. intervals; later, stopwatches graduated in 0.1-sec. intervals became available, and they were used for all subsequent work, including the calibrations runs. All stopwatches were calibrated against the National Bureau of Standards time signal, and no correction larger than 0.05% was required. While the efflux time was noted, the meniscus was observed through a cathetometer telescope in order to provide magnification and to avoid parallax.

Cleaning. Maintaining a scrupulously clean surface within the viscometer was essential. During every run the drainage characteristics of bulbs *E*, *F*, and *G* were carefully observed, and if any irregularities were noted in drainage or in reproducibility of results, the sample was discarded, and the viscometer was removed for cleaning. If both the sample and the viscometer were free from grease and dirt, the liquid drained from bulbs *E*, *F*, and *G* in a completely smooth and uniform film. Any rivulets or drops that formed during or after drainage resulted in erratic efflux times and required a thorough cleaning of the viscometer and a fresh, clean sample.

The cleaning procedure which proved most satisfactory consisted in soaking the viscometer for 24 hr. in concentrated cleaning solution (concentrated sulfuric acid, saturated with potassium dichromate), followed by rinsing eight times with conductivity water and soaking for 24 hr. in conductivity water to remove adsorbed chromic acid. Drying overnight in an air oven at 105°C. completed the process. This cleaning procedure was routinely carried out at least once a week. The viscometer was cleaned daily by multiple rinses with conductivity water and dried at 105°C.

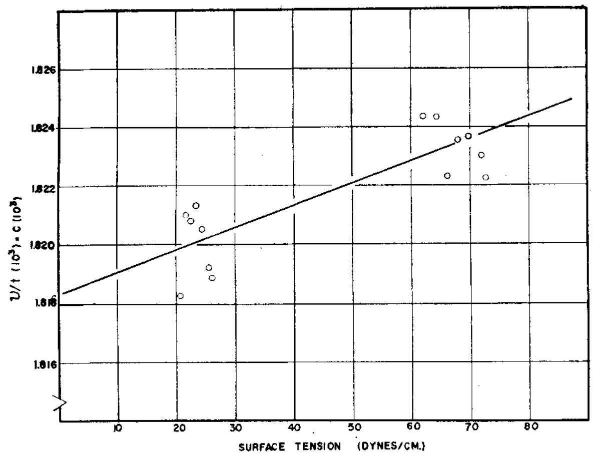

Fig. 4. Variation of viscometer constant with surface tension, viscometer A19.

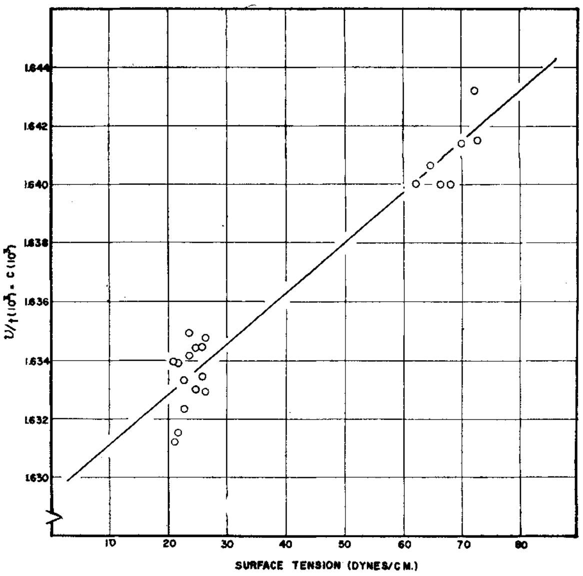

Fig. 5. Variation of viscometer constant with surface tension, viscometer A43.

Solvents

The water used as a calibration standard and for preparation of aqueous samples was daily prepared from once-distilled water (Barnstead still) by redistillation in an all-quartz still equipped with an air-cooled condenser, the middle cut (one-third the starting volume) being retained. Such water had a specific conductance of 1 × 10^{-6} reciprocal ohms at most and is referred to as *conductivity water*.

The acetone was analyzed by refractive index, and its density was determined. From these data the actual composition of the starting material was found (*16*) to be 99.54 mole % acetone, 0.45 mole % water. Distillation of the material in a fractional-distillation column packed with ⅛-in. Pyrex helices (packing 1 × 90 cm.) showed a boiling range of 56.1° to 56.2°C. (747 to 750 mm.).

For calibration in the high-viscosity range (0.9 to 2.5 centistokes), samples of standard viscosity oil *D* (calibrated at six to eight temperatures over the range 20° to 100°C.) were obtained from the National Bureau of Standards. Benzene, acetic acid, and toluene were purified for use in comparing results obtained with the two viscometers.

Calibration

With routine capillary viscometers, viscosity is usually calculated from the modified Poiseuille equation

$$\nu = Ct - \frac{B}{t} \qquad (1)$$

where

ν = kinematic viscosity, centistokes
t = efflux time, sec.
C, B = viscometer constants, dependent on the Reynolds number

Discussion of the derivation of this simplified equation may be found in Cannon and Fenske (*5*) and Swindells, Hardy, and Cottington (*15*). Calibration of the Cannon-Ubbelohde viscometer consists in the evaluation of C and B. Although C and B vary with Reynolds number (*4*), the design of most commercial viscometers

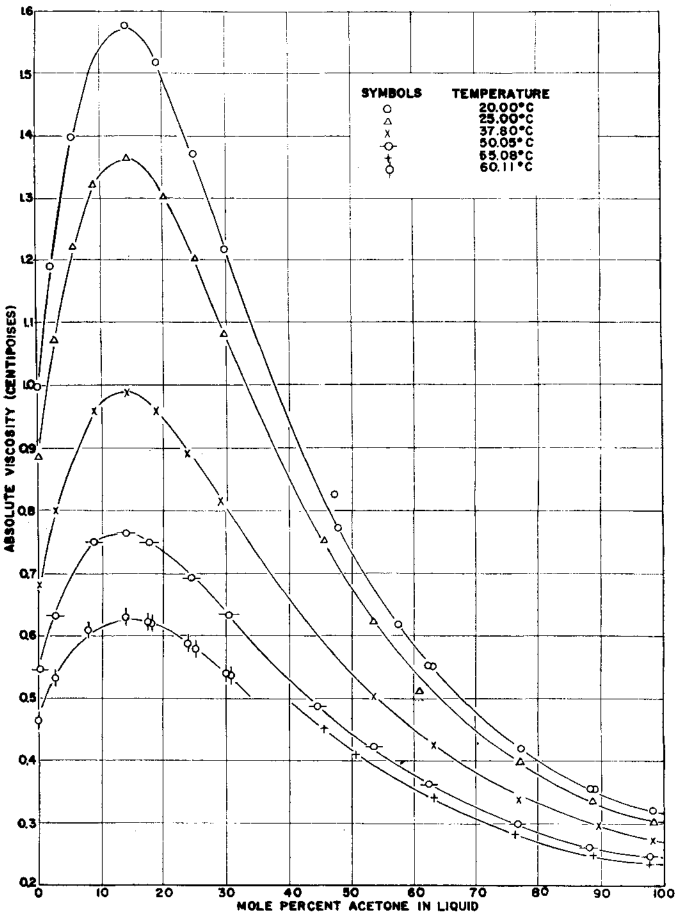

Fig. 6. Viscosity of acetone-water solutions.

is such that for most of their useful range (efflux time greater than 200 sec.) C is constant, and B = 0. Thus Equation (1) reduces to

$$\nu = Ct \qquad (2)$$

To define this range and to evaluate the quantities B and C, one can rewrite Equation (1):

$$\frac{\nu}{t} = C - \frac{B}{t^2} \qquad (3)$$

The efflux times of liquids of different known viscosities are measured (in this case water at several temperatures and N.B.S. calibrated oil at several temperatures). The quantity ν/t is then plotted vs. $1/t^2$; B is the slope of the tangent to the curve at a given value of $1/t^2$, and C is the intercept of the tangent at a value of $1/t^2 = 0$. Where $B = 0$, a horizontal line results, and C is constant.

Conductivity water and N.B.S. viscosity oil D were the calibration standards used here. At every temperature six efflux-time observations were made on each of at least two samples of each standard. Deviation in efflux time between repeated runs seldom amounted to more than 0.3 sec. in up to 1,500 sec., and was never greater than 0.1%. The viscosities and average efflux times for each standard in the two viscometers, $A19$ and $A43$, are available from the American Documentation Institute (*1*); Figures 2 and 3 show the resulting curves. The deviation of the experimental points from the horizontal lines drawn through their averages was 0.04 to 0.06%. It is evident that B may be neglected over the range of t indicated. An efflux time of 200 sec. is the arbitrary minimum recommended by the A.S.T.M. (*2*). No efflux time less than 199.3 sec. was observed in the calibration.

Since the oil and water calibration points (Figures 2 and 3) did not fall on the same straight line, it was assumed that a surface-tension correction must be applied. The surface tension of oil D was measured by the capillary-height method (*8*) at the temperatures at which the viscometers were calibrated; the surface tension of water at these temperatures is well known (*8*). Figures 4 and 5 show ν/t [from Equation (2) it is seen that $\nu/t = C$ over the range of t used here] plotted as a function of surface tension. The cluster of points at the lower end of each line represents the oil runs, and the points at the higher end of the line represent the water calibration runs. Figures 4 and 5 were considered to be the calibration curves in this work. Figures 2 and 3 were useful only to demonstrate that $B = 0$. As long as the surface tension is known to 2 or 3 dynes/cm., any possible error in C due to the surface-tension correction will be within the precision of the C constant determination itself. The surface tension of all the acetone-water solutions the viscosities of which are reported here has been determined (*8*). From the surface tension, a C constant was selected from Figures 4 and 5, and the kinematic viscosity was calculated by means of Formula (2).

Comparison of Viscosities. When a series of purified solvents (benzene, toluene, acetic acid, and acetone) was run at several temperatures for each solvent in both viscometers, comparative results, calculated from the constants of Figures 4 and 5, did not differ from one another by more than ±0.15%. The average difference in calculated viscosity when the two viscometers were used was ±0.06%. The viscometers were used interchangeably throughout the investigation.

Discussion of the Calibration. The results of the calibrations reported here demonstrate the inadequacy of Equation (1) to describe the conditions of flow in the Cannon-Ubbelohde viscometers used. Only when the surface-tension correction was made in the manner described here could the calculated viscosities measured with different Cannon-Ubbelohde viscometers be made to agree to better than ±0.5 to 1.0%, although the results with either viscometer could be reproduced to better than ±0.05%. Although the calibration method presented here may be lacking in theoretical rigor, it was the only method found that would give the same viscosity for a particular sample with two different viscometers.

In later runs a third Cannon-Ubbelohde viscometer having approximately twice the capillary diameter of A19 and A43 was calibrated in the same manner as described here. The results of all three viscometers could be brought into agreement only by using a surface-tension correction of the type presented here.

TABLE 1. VISCOSITY OF LIQUID ACETONE-WATER SOLUTIONS

Mole % acetone	ρ* g./cc.	ν, cs.	η, cp.	Mole % acetone	ρ*, g./cc.	ν, cs.	η, cp.
20.00°C.				25.00°C.			
0	0.9982053	1.0038	1.0020	0	0.9970470	0.8931	0.8905‡
2.51	0.9886	1.2125	1.1987	2.92	0.9852	1.0954	1.0792
5.87	0.9766	1.4415	1.4077	5.83	0.9743	1.2596	1.2272
14.63	0.9477	1.6702	1.5828	9.13	0.9625	1.3809	1.3291
18.07	0.9367	1.6531	1.5485	14.57	0.9444	1.4541	1.3733
25.25	0.9159	1.5044	1.3779	20.14	0.9262	1.4133	1.3090
30.55	0.9022	1.3577	1.2249	25.08	0.9140	1.3259	1.2119
47.7	0.8621	0.9593	0.8270	30.1	0.8980	1.2123	1.0886
48.4	0.8607	0.9018	0.7762	46.2	0.8603	0.8749	0.7527
58.2	0.8431	0.7393	0.6233	53.8	0.8455	0.7389	0.6247
63.0	0.8352	0.6619	0.5528	61.6	0.8323	0.6133	0.5104
63.6	0.8343	0.6588	0.5496	77.5	0.8097	0.4896	0.3964
77.5	0.8150	0.5169	0.4213	89.4	0.7953	0.4219	0.3355
89.2	0.8010	0.4412	0.3534	99.2	0.7851	0.3860	0.3030
90.1	0.8000	0.4410	0.3528	(100)	0.7844	(0.3840)†	(0.3012)†
99.2	0.7970	0.4024	0.3207				
(100)	0.7899	(0.3985)†	(0.3148)†				
37.80°C.				50.05°C.			
0	0.99304	0.6857	0.6809‡	0	0.98781	0.5530	0.5464‡
2.97	0.9797	0.8221	0.8054	2.87	0.9739	0.6521	0.6351
9.12	0.9546	1.0101	0.9643	9.10	0.9464	0.7860	0.7439
14.47	0.9349	1.0635	0.9943	14.49	0.9249	0.8259	0.7639
19.05	0.9190	1.0496	0.9646	18.20	0.9111	0.8228	0.7497
24.20	0.9025	0.9944	0.8974	24.90	0.8889	0.7841	0.6970
29.9	0.8860	0.9247	0.8193	30.7	0.8715	0.7320	0.6379
45.6	0.8484	0.7029	0.5963	45.6	0.8347	0.5842	0.4876
53.8	0.8326	0.6070	0.5054	54.2	0.8182	0.5138	0.4204
63.9	0.8153	0.5202	0.4241	63.5	0.8025	0.4497	0.3609
77.6	0.7949	0.4286	0.3407	77.5	0.7807	0.3818	0.2981
90.5	0.7790	0.3766	0.2934	89.3	0.7655	0.3422	0.2620
99.2	0.7705	0.3494	0.2692	99.0	0.7528	0.3241	0.2440
(100)	0.7698	(0.3465)†	(0.2690)†	(100)	0.7550	(0.3235)†	(0.2442)†
55.08°C.				60.11°C.			
46.3	0.8273**	0.5467	0.4523	0	0.98243**	0.4738	0.4658‡
50.9	0.8185**	0.5012	0.4102	2.92	0.9729**	0.5506	0.5357
63.9	0.7961**	0.4302	0.3425	8.03	0.9434**	0.6441	0.6083
77.0	0.7753**	0.3669	0.2845	14.38	0.9172**	0.6891	0.6320
89.4	0.7593**	0.3298	0.2504	17.74	0.9044**	0.6896	0.6237
98.9	0.7500**	0.3105	0.2329	18.11	0.9031**	0.6894	0.6226
(100)	0.7488**	(0.3095)†	(0.2318)†	24.20	0.8811**	0.6650	0.5859
				25.32	0.8774**	0.6587	0.5779
				30.3	0.8627**	0.6264	0.5404
				31.4	0.8598**	0.6257	0.5380

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*Interpolated from the results of Thomas and McAllister (*16*) measured on the same acetone lot. The water densities were calculated from those reported by J. H. Perry, "Chemical Engineers Handbook," McGraw-Hill Book Company, Inc., New York (1950), p. 175.

†Extrapolated by extension of the experimental results.

‡The viscosities of water were calculated by the modified Cragoe equation (J. F. Swindells, personal communication):

$$\log_{10}\frac{\eta_t}{\eta_{20^\circ}} = \frac{1.3272(20 - t) - 0.001053(20 - t)^2}{t + 105}$$

where t = temperature, °C., and with the accepted [Swindells, J. F., J. R. Coe, Jr., and T. B. Godfrey, *J. Research, Natl. Bur. Standards*, **48**, 1 (1952)] value for $\eta_{20^\circ}{}^{H_2O} = 1.0020$ cp. Absolute viscosity was then converted to kinematic viscosity by the relationship

$$\nu = \frac{\eta}{\rho}$$

**Extrapolated from the results of Thomas and McAllister (*16*).

TABLE 2. VISCOSITY OF LIQUID ACETONE-WATER SOLUTIONS AT THEIR NORMAL BOILING POINTS

Mole % acetone	B.P.*, °C.	ρ†, g./cc.	η, cp.
0	100.0	0.95835	0.282
1.0	93.0	0.9567	0.329
2.0	86.5	0.9551	0.370
3.0	81.3	0.9533	0.407
4.0	77.6	0.9500	0.437
5.0	75.0	0.9472	0.462
6.0	72.8	0.9435	0.486
7.0	71.0	0.9400	0.510
8.0	69.5	0.9364	0.532
9.0	68.1	0.9331	0.543
10.0	66.7	0.9298	0.560
12.5	65.7	0.9202	0.576
15.0	63.4	0.9119	0.597
17.5	62.7	0.9030	0.595
20.0	62.2	0.8949	0.591
30.0	61.0	0.8623	0.539
40.0	60.4	0.8368	0.473
50.0	59.8	0.8155	0.403
60.0	59.3	0.7983	0.340
70.0	58.8	0.7823	0.304
80.0	58.2	0.7678	0.268
90.0	57.4	0.7560	0.246
100.0	56.2	0.7476	0.231

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*These temperatures were obtained from the data of A. S. Brunjes and M. J. P. Bogart [*Ind. Eng. Chem.*, **35**, 258 (1954)], D. F. Othmer and R. F. Benenati [*ibid.*, **37**, 299 (1945)], and J. C. Chu ["Distillation Equilibrium Data," Reinhold Publishing Corporation, New York (1950), p. 24]. More weight was given the data of Brunjes and Bogart, since they showed more internal consistency.

†Thomas and McAllister (*16*).

Analytical Procedure

All analyses were made by determining the refractive index (n_D^{25}) of the solution with a Bausch and Lomb Precision Refractometer and reading the acetone concentration from a standard curve of refractive index vs. mole percentage of acetone. The standard curve was prepared by determining the refractive indices of solutions which had been prepared by mixing weighed samples of acetone and water. All weights used in calculating the compositions of the solutions were the corrected vacuum weights. The chain, the rider, and the brass weights used in the analytical balance were checked against National Bureau of Standards calibrated weights, and corrections were noted.

The refractive index–composition curve has a maximum at 40 to 42 mole % acetone and, for this reason, refractive index alone is not a suitable analysis in the region of 35 to 45 mole % acetone. The accuracy of the analysis is within 0.02 to 0.05 mole % in the region of 0 to 26 and 98 to 100 mole % acetone; the accuracy is within 0.1 mole % for the remainder of the curve. The refractive index found for solutions between 0 and 100 mole % acetone may be obtained from the American Documentation Institute (*1*).

RESULTS

The viscosity of the acetone-water system at various temperatures is given in Table 1. The absolute viscosities were calculated from the measured kinematic viscosities and the liquid densities. The surface tensions of the solutions, used for the selection of the proper *C* constant, were those reported by Howard and McAllister (*8*). The absolute viscosity results are shown graphically in Figure 6.

From the data of Table 1, viscosity-vs.-temperature curves were prepared and extrapolated to the normal boiling points. The average extrapolation required was 9°C., but from 8 to 100 mole % acetone the extrapolation covered less than 9°C. The viscosities at the boiling points are given in Table 2.

DISCUSSION

Two factors impede comparison of these viscosities with those found in the literature. In 1953 a new value for the viscosity of water at 20°C. (1.002 centipoise) was adopted (*15*) as the primary standard for viscosity determinations, and all work carried out prior to that time had been based on higher values (1.005, 1.009 centipoise). This error cannot be corrected by multiplying observed viscosities by the ratio of the two 20°C. values; correction would require recalculation of the calibration constants of the viscometers used, and, in most cases, sufficient data for this correction have not been reported. An error of greater magnitude has been introduced by the failure of earlier workers to use anhydrous acetone. Only within the past decade (*16*) has the density of absolutely anhydrous acetone been available for comparison. A comparison of the reported viscosity and density of the "100%" acetone of various investigators (*3, 7, 9, 10, 11, 12, 14*) is given in reference *1*. The densities indicate that the starting material of these workers was not anhydrous, and on the assumption that the only contaminant was water, the compositions ranged from 99.3 to 96.7 mole % acetone. It is apparent from these results that the acetone concentrations which were reported would be subject to increasingly large errors as the acetone percentage increased. For these reasons a tabular comparison of the viscosities found by earlier workers (*3, 6, 10, 13, 14*) and those found here is not presented. In general, however, the values from 0 to 50 mole % acetone agree within about 1.0% with those reported here; previous work in the range of 50 to 100 mole % acetone may be from 2 to 5% high. An exception is the work reported by Sandonnini (*14*); these viscosities are from 40 to 80% higher than those reported here. The viscosities reported in Table 1 are thought to have an absolute accuracy of ±0.2%. The viscosities reported in Table 2 are given to three significant figures only, because the boiling temperature at the given composition is not known to greater accuracy than ±0.1°C., and hence the boiling-point viscosity is no more accurate than shown.

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