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THE RING METHOD OF MEASURING SURFACE TENSION FOR LIQUIDS OF HIGH DENSITY AND LOW SURFACE TENSION

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ABSTRACT

The existing correction tables applicable to the measurement of surface tension by the ring method are not extensive enough to be used with liquids of high density coupled with low surface tension, as for example, the fluorocarbons. The convenience and rapidity of the ring method make it worthwhile to extend the tables to the region in question. The tables therefore, have been extended by fitting a combined hyperbolic and linear equation to the curves derived from existing tables. This equation produces values in agreement with existing tables to better than 0.05 percent. The extrapolated tables were verified experimentally over their whole range by comparing measurements of the surface tensions of five fluorocarbon liquids made by the differential capillary-rise method (which is independent of corrections) with measurements on the same liquids made by the ring method. The agreement was as good as that associated with the original tables.

PROBLEM STATUS

This is a final report on one phase of the problem; work on other phases is continuing.

AUTHORIZATION

NRL Problem C09-01R
NR 409-010

THE RING METHOD OF MEASURING SURFACE TENSION FOR LIQUIDS OF HIGH DENSITY AND LOW SURFACE TENSION

INTRODUCTION

The method of determining surface tension by measuring the force necessary to detach a ring from a liquid surface has come into widespread use because of its rapidity and convenience. Although this method does not produce results of highest accuracy except with the most tedious efforts, with reasonable care, it is possible to produce results with an error of less than 0.5 percent.

The ring method was placed on a firm basis in the classical work of Harkins and co-workers (1,2). These authors measured the surface tensions of some liquids by both the capillary-rise and ring methods and compared the two values. They prepared a table of correction factors which reconciled the values obtained by the two methods. Freud and Freud (3) analyzed the ring method from the theoretical standpoint and found excellent agreement between the empirically derived tables and the theory of the ring method. The tables are therefore being widely used and indeed are nearly indispensable if reasonable accuracy is required in using the ring method.

Harkins et al. (1,2) found that the correction factor, F , for the ring method depended on R^3/V and R/r where R is the radius of the ring, r the radius of the wire of the ring, and V the maximum volume of liquid lifted above the plane of the liquid surface. Their tables cover the range of R^3/V from 0.30 to 3.50 and of R/r from 30 to 80. For most purposes this range is adequate and permits the choice of a ring whose dimensions are such as to introduce the fewest experimental difficulties.

The liquid fluorocarbons and many derivatives which have only recently come into prominence, have comparatively low surface tensions coupled with high densities. Therefore, when a ring of convenient size (4 to 6 cm in circumference) is used, one finds that R^3/V is larger than 3.50 and that the proper correction factor lies outside the scope of the tables. Figure 1 shows the limits of the region where rings 6 and 12 cm in circumference may be used without exceeding the limit of the tables at $R^3/V \leq 3.50$. For $R/r = 30$, combinations of surface tension and density yielding values of R^3/V less than 3.50 lie above the limiting line in each of the two cases represented. For R/r greater than 30 the limiting lines are displaced upward, further restricting the useful areas. It is obvious from Figure 1 that for successful use of the existing tables for the determination of the surface tension of the fluorocarbons, one must resort to rings at least 12 cm in circumference. The use of rings of this size introduces serious problems in making and maintaining the rings flat and level. It is therefore of general interest to extend the Harkins and Jordan tables to regions of $R^3/V > 3.50$ so that the ring method might be used for the fluorocarbons and similar liquids with rings of moderate size.

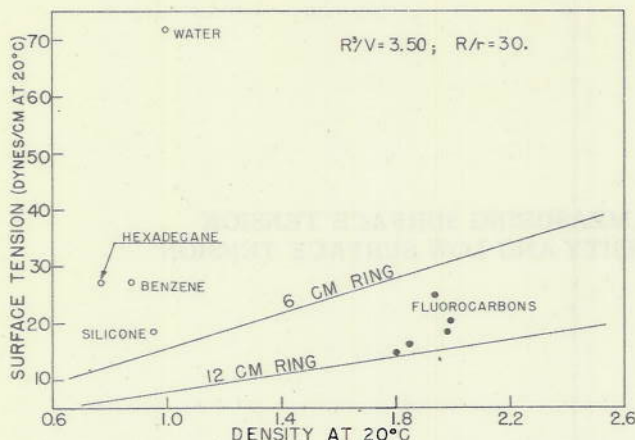


Figure 1 - Limiting values of surface tension and density for application of Harkins and Jordan correction tables

pull on the ring ($Mg/4\pi R$) and D and d are the densities of the lower and upper phases, respectively (liquid-air, water-oil, etc.). By means of their equation they were able successfully to extrapolate the Harkins and Jordan tables to regions of R^3/V smaller than 0.30; this region is useful in applying the ring method to measurements of interfacial tension where V is likely to be large because of the small differences in density between two liquid phases. When the Zuidema and Waters equation was applied to the raw data for the fluorocarbons however, discrepancies in F were found at $R/r = 54$ which ranged from 4 percent at $R^3/V = 4.6$ to 7 percent at $R^3/V = 7.0$. In this region, $P/(D-d)$ ranges from 15 to 10 and the Zuidema and Waters function moves more slowly than required by the experimental data. In fact, the function has a limit of $F \rightarrow 0.84$ at $P/(D-d) \rightarrow 0$ whereas experiment shows the need of $F < 0.83$ at $P/(D-d) < 15$.

EXTRAPOLATION OF THE HARKINS AND JORDAN TABLES

The Harkins and Jordan tables were extrapolated by finding equations describing the curves plotted from the tables and extending the curves by means of these equations for $R/r = 40, 50$, and 60 from $R^3/V = 3.5$ to 7.5 . Lagrangian interpolation was employed to obtain the curves for $R/r = 52, 54, 56$, and 58 . The extrapolated tables were tested experimentally at $R/r = 54.0$ where the interpolation is most distant from the extrapolated curves and the results are described below. Table 1 shows the extrapolated tables for $R^3/V = 3.5$ to 7.50 . The intervals were chosen as 0.25 since the function changes fairly slowly in this region and interpolation is easy. Following Harkins and Jordan, the deciles of R/r are given to four decimal places and the interpolated values to three. A detailed description of the method used in this extrapolation is given in the appendix. Figure 2 shows the curves for $R/r = 40, 50$, and 60 in the extrapolated region; the dotted lines are in the adjacent region of the Harkins and Jordan tables.

Extrapolation of the tables to $R^3/V = 7.50$ for $R/r = 40, 50, 52, 54, 56, 58$, and 60 has been accomplished by the present work. These values of R/r were considered especially useful since rings 6 cm in diameter with values of R/r between 52 and 56 are commercially available. The procedures outlined below may be used to extend the extrapolations beyond $R^3/V = 7.50$ or to other values of R/r .

Zuidema and Waters (4) analyzed the tabulated data of Harkins and Jordan and presented an equation which described the data closely. They showed that the correction factor, F , is a parabolic function of $P/(D-d)$ where P is the maximum

TABLE 1
Correction Factors, F, for the Ring Method

R^3/V	R/r						
	40	50	52	54	56	58	60
3.50	0.8063	0.8407	0.847	0.852	0.858	0.863	0.8672
3.75	0.8002	0.8357	0.842	0.848	0.853	0.858	0.8629
4.00	0.7945	0.8311	0.837	0.843	0.849	0.854	0.8590
4.25	0.7890	0.8267	0.833	0.839	0.845	0.850	0.8553
4.50	0.7838	0.8225	0.829	0.835	0.841	0.847	0.8518
4.75	0.7787	0.8185	0.825	0.832	0.838	0.843	0.8483
5.00	0.7738	0.8147	0.822	0.828	0.834	0.840	0.8451
5.25	0.7691	0.8109	0.818	0.825	0.831	0.837	0.8420
5.50	0.7645	0.8073	0.815	0.821	0.828	0.834	0.8389
5.75	0.7599	0.8038	0.811	0.818	0.825	0.830	0.8359
6.00	0.7555	0.8003	0.808	0.815	0.821	0.827	0.8330
6.25	0.7511	0.7969	0.805	0.812	0.818	0.825	0.8302
6.50	0.7468	0.7936	0.801	0.808	0.815	0.822	0.8274
6.75	0.7426	0.7903	0.798	0.806	0.813	0.819	0.8246
7.00	0.7384	0.7871	0.795	0.803	0.810	0.816	0.8220
7.25	0.7343	0.7839	0.792	0.800	0.807	0.813	0.8194
7.50	0.7302	0.7807	0.789	0.797	0.804	0.811	0.8168

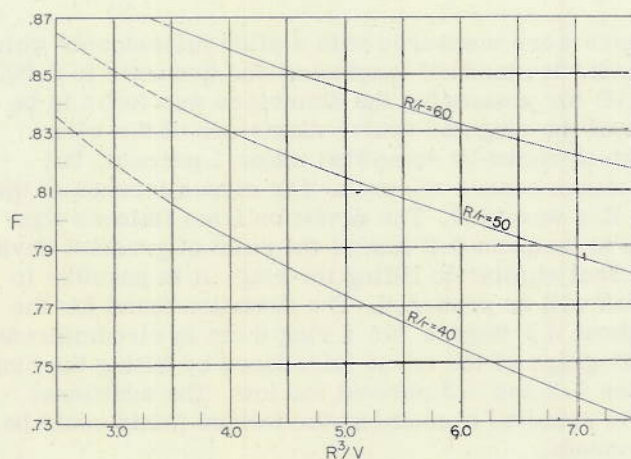


Figure 2 - Correction factor, F, as a function of R^3/V . Dotted lines are from Harkins and Jordan tables

carefully tested to make certain that the contact angle with glass was zero since the method depends on this being the case.

The capillary-rise cell used in these measurements had large and small capillaries whose diameters were 0.1 and 0.05 cm, respectively. These dimensions insured that the rise would be at least 0.3 cm even for liquids of surface tensions as low as 15 dynes/cm. The rise was measured with a slide micrometer accurate to 0.0001 cm so that this

EXPERIMENTAL

The surface tensions of a number of liquids were measured at $20.00 \pm 0.03^\circ\text{C}$ by the differential capillary-rise method of Sugden (5) and by the ring method at $20.0 \pm 0.1^\circ\text{C}$ and the results were compared. The liquids were chosen to give a wide range of values of R^3/V .

Results obtained for three pure compounds using the differential capillary-rise method were in excellent agreement with the most reliable values given in the literature. The surface tension of water was measured as 72.75 dynes/cm (6); benzene, 28.86 dynes/cm (6); n-hexadecane, 27.60 dynes/cm (7).

The other liquids of this study were

measurement was good to 0.03 percent for the smallest rise and proportionately better for liquids of higher surface tension.

The same capillary-rise cell was used for all the liquids reported here. Difficulties were encountered in cleaning the cell after measurements on the fluorocarbons. These liquids are insoluble or difficultly soluble in conventional solvents, and are not destroyed by oxidizing acids. It was necessary to rinse out the cell with $\text{CF}_3\text{-CCl}_3$ (Freon-113) which is a good low-boiling solvent for fluorocarbon liquids. This liquid not only effectively dissolved and removed all the fluorocarbon oil left on the walls of the cell but was itself readily removed by blowing clean dry air through the cell. The cell was subsequently boiled in nitric-sulfuric acid (1:2) for 10-15 minutes until it was hydrophilic. The acid was removed by ten rinses with steaming, freshly distilled water, and the cell was dried at 110°C . With this method of cleaning, no difficulty was encountered in obtaining reproducibility between separate measurements to within 0.02-0.03 percent. Every surface tension value given here is the average of at least three independent measurements.

To "calibrate" the ring, four liquids were chosen whose F values fell within the scope of the Harkins and Jordan tables. Of five 6-cm rings obtained from the manufacturer, all showed deviations from perfect planarity. Moreover, where the rings were welded to the supporting stirrups more metal had been removed than was actually necessary to make the rings smooth. This had the effect of reducing the diameter of the wire at two points in every ring. Both these effects tend to make the ring break away too early so that the values of the surface tension are invariably low by the same amount percentagewise. For the work reported here, a ring was chosen which had a value of $R/r = 54.0$ so that the extrapolated tables could be tested at a point where the interpolations were most distant from the deciles.

The diameters of the ring and the wire were measured with a slide micrometer which had been compared with a Bureau of Standards standard length and was accurate to 0.0001 cm. The method of Harkins and Jordan (2) for measuring the diameters was found to be satisfactory. Averages of six diameters of the ring and twelve diameters of the wire were taken. The ring was found to be out-of-round by somewhat under 1 percent, but Harkins and Jordan claim that this is a minor source of error. The circumference of the ring used was found to be 5.990 cm and R/r was 54.0. The deviation from flatness was measured with a cathetometer and found to be about 0.2 mm at the point of greatest deviation. If deviation from flatness has an effect similar to tilting the ring, it is possible to estimate the magnitude of the error which will be produced. The deviation found for the ring is equivalent to an angle of tilt of about 0.5 degree for a ring 6 cm in circumference. Referring to the Harkins' and Jordan's (2) graph of the error introduced by tilting the ring, it is clear that the results will be between 0.2 and 0.3 percent too low. The additional contribution to the error by the excessive removal of metal at the welded joints could be estimated from the calibrating measurements.

Table 2 shows the discrepancies between the values obtained by the capillary-rise method and by the ring method for the four liquids which could be measured with the help of the existing tables. The ring method gave values about 0.4 percent low with no apparent systematic differences in the error over the whole range of R^3/V . Since no other errors known to the authors were involved in these measurements, it is reasonable to assume that the 0.4 percent error was due to deviation from flatness of the ring (contributing about half the error) and a too small radius of the wire at the welded joints.

A chainomatic balance especially designed for surface-tension measurements was used in this study. Incorporated in it was a platform which could be lowered smoothly and slowly while weight was added by means of a rider and chain. The balance was

TABLE 2
Determination of Ring Correction

Compound	R^3/V	Surface Tension (Dynes/cm, 20°C)				Difference (%)
		Diff. Cap. Rise	Ring	Difference * (%)	Ring (Corr.)	
Water	0.92	72.75	72.57	-0.25	72.85	+0.14
Benzene	1.93	28.86	28.76	-0.34	28.87	+0.04
Hexadecane	1.80	27.60	27.44	-0.57	27.55	-0.18
Hexadecamethyl-heptasiloxane	3.14	18.61	18.53	-0.43	18.60	-0.05

*Average correction 0.40%

sensitive to 0.1 mg and the rider and chain were calibrated with certified weights before use. To obtain good results it was found necessary to insulate the balance from vibration and to fit it with a magnetic damper. The ring was held in a pin chuck fitted with a circular eye which hung from a free swinging hook attached to an agate knife edge. The ring was leveled by bringing a dish of mercury close to it and sighting between the ring and the mercury surface. Very small deviations from the level could be determined in this fashion. This method was found to be easier and more convenient than the method of Harkins and Jordan (2) which comprised using a metal mirror leveled with a spirit level.

The liquid to be measured was held in a thick walled Petrie dish 10 cm in diameter. The edge of the dish was finely ground to a good fit with a cover of plate glass. The cover was slotted to permit the wires supporting the ring to pass through. After the ring and cover were in position, the slot was covered except where the wires passed through. This arrangement worked satisfactorily in minimizing evaporation of even the most volatile liquids studied.

The ring was flamed, leveled as described above, and the zero of the balance determined. A clean Petrie dish containing the liquid to be measured was placed on the platform and the liquid surface raised to within 2-3 mm of the ring. The cover was put in place and the slot covered. The balance case was closed and the whole system allowed to come to thermal equilibrium for one hour to insure that the space above the liquid would become saturated with vapor. The liquid was then raised till it touched the ring. Weight was added and the platform simultaneously lowered to keep the balance pointer on zero. The beam arrest was lowered very little so that the pointer could swing only about half a division on either side of zero. This was done to prevent the ring from becoming completely detached at the end point of the measurement. In this way successive measurements could be made quickly since the position of the platform need only be changed a small amount between measurements. Weight was added and the platform lowered until the pointer could no longer be brought back to zero by lowering the platform. The chain was then backed off 2-3 mg and the platform raised a little. Then weight was added 0.1 mg at a time with at least 5 seconds between additions for the nonviscous liquids and 10 seconds for the viscous liquids. The platform was lowered very slowly at the same time always keeping the pointer at zero. When addition of 0.1 mg made the pointer swing irreversibly to the left, the weight was recorded as the maximum pull on the ring. Successive measurements made in this fashion were invariably within 0.1 mg of each other. At least four measurements were made at 15-minute intervals. The whole procedure was repeated for each fluid at least twice on different days to insure that no topical variables were affecting the measurements. The separate measurements on different days always checked to within 0.3 mg. The average of the three separate measurements is the value reported in Tables 2 and 3.

TABLE 3
Comparison of Surface Tension Measurements by the Differential Capillary Rise and Ring Methods for Liquids of High Density and Low Surface Tension

Compound	R^3/V	Surface Tension (Dynes/cm, 20°C)			Difference (%)
		Diff. Cap. Rise	Ring	Ring (Corr.)	
Fluorolube (Hooker)*	4.61	25.03	24.85	24.95	-0.32
Fluoro Lubricant (DuPont FCD 330)	5.75	20.15	20.06	20.14	-0.05
Fluoro Kerosene (DuPont FCD 329)	6.16	18.31	18.17	18.24	-0.38
Perfluorodimethylcyclohexane	6.65	16.00	15.95	16.01	+0.06
Perfluoromethylcyclohexane	6.97	14.70	14.69	14.75	+0.34

*Polymonochlorotrifluoroethane

RESULTS

Table 2 shows the differences in the values obtained by the differential capillary-rise and ring methods for four liquids when R^3/V falls in the range of the Harkins and Jordan correction tables. There is a systematic error (due to imperfection of the ring) averaging 0.40 percent. When this correction is applied to the values obtained by the ring method, the percentage difference between the two methods is reduced to less than 0.2 percent. Harkins and Jordan consider that for values of R/r other than deciles the tables are accurate to 0.4 percent with a probable error of less than 0.3 percent.

Table 3 gives the differences in the values obtained by the two methods for five liquids when R^3/V is larger than 3.50. In this case the raw data for the ring method were corrected with the extrapolated tables shown above. It is evident that when the 0.4 percent correction determined earlier is applied to the fluorocarbon data, the residual differences are all less than 0.4 percent over the range of R^3/V from 4.61 to 6.97. It is obvious that as the surface tension decreases, the sensitivity of the balance (0.1 mg) assumes greater importance percentagewise so that differences of 0.01 dynes/cm (0.1 mg \approx ca 0.01 dynes/cm) are equivalent to 0.05 percent at surface tensions of 20 dynes/cm and proportionately more at lower surface tensions.

CONCLUSIONS

The tables of correction for the ring method of determining surface tension of Harkins and Jordan (2) have been extrapolated to $R^3/V = 7.50$ for values of R/r of 40, 50, 52, 54, 56, 58, and 60. The extrapolations were checked experimentally and proved to have the same order of accuracy as the Harkins and Jordan tables for $R/r = 54.0$. For the other values of R/r tabulated here, the agreement should be at least as good.

These tables may therefore be used with commercially available rings 6 cm in circumference to measure the surface tensions of liquids of high density and low surface tension by the ring method. For highest accuracy, it is necessary to determine the errors inherent in the ring by calibration with liquids of known surface tensions.

The ring method is therefore now available for measuring surface and interfacial tensions from regions of high interfacial tension and low density, by the extrapolation of Zuidema and Waters (4), to regions of low surface tension and high density, by the present present extrapolation.

* * *

APPENDIX

Method of Extrapolation

The equations for R/r equal to 40, 50, and 60 were obtained by fitting to the Harkins and Jordan tables (2) equations made up of a hyperbolic component and a straight-line component. An exponential component was used tentatively on the table at $R/r = 50$ but was discarded in the final computations since it had no effect on the region of interest. In the example described ($R/r = 50$), R^3/V will be designated as x , and the correction factor, F , will be designated as y . The data for the curve in question were plotted at intervals of 0.1x and a hyperbolic component was recognized. The data were divided into two groups of 16 points each and the averages of the x and y values were taken. These averages were substituted in the equation

$$a + bx = x/y, \quad (1)$$

to give

$$a + 1.15b = 1.15/0.92774,$$

and

$$a + 2.75b = 2.75/0.85904,$$

which, when solved, gave $a = -0.17040$ and $b = 1.2261$. Substituting these values in Equation (1) and equating for y gave

$$y_I = \frac{x}{1.2261x - 0.17040}. \quad (2)$$

First approximations for y were computed from Equation (2) and residuals formed from the equation

$$y_I - y = \text{Residue}. \quad (3)$$

The residues plotted at intervals of 0.1x showed the presence of an exponential component and a straight-line component. Since in the region of interest, the straight-line component appeared more prominent, a straight line was written to describe this component. In the portion of the curve which approaches a straight line from $x = 2.4$ to 3.5 the residues at 0.1x intervals were averaged in two groups and substituted in the equation

$$y = cx + d, \quad (4)$$

giving

$$0.00016667 = 2.65c + d,$$

and

$$0.0060833 = 3.25c + d.$$

Solving these equations and substituting for c and d gave the equation

$$y = -0.010417x + 0.027771. \quad (5)$$

By adding Equations (2) and (5) the following equation for the second approximation of y is obtained:

$$y_{II} = \frac{x}{1.2261x - 0.17040} - 0.010417x + 0.027771. \quad (6)$$

Residues were taken by subtracting the original values of y from values as computed by Equation (6). In the region of interest the agreement was within the accuracy requirements, but the exponential component was still evident in the region of lower x values ($R^3/V < 2.4$). Therefore, an equation of the form $y = fe^{gx}$ was written. In writing this equation values at $x = 0.6$ and 2.2 were substituted in the equation in the form

$$\ln y = \ln f + gx,$$

to give

$$-2.1804 = \ln f + 0.6g,$$

and

$$-6.3771 = \ln f + 2.2g,$$

which, when solved, gave $f = 0.54521$ and $g = -2.6230$.

The equation,

$$y = 0.54521e^{-2.6230x}, \quad (7)$$

was added to Equation (6) to give the third approximation as follows:

$$y_{III} = \frac{x}{1.2261x - 0.17040} - 0.010417x + 0.027771 - 0.54521e^{-2.6230x}. \quad (8)$$

Values of F computed from (8) with the exception of $R^3/V = 0.4$ and 0.5 were in error by 0.5 percent or less throughout, with the error decreasing as R^3/V increases. The error reduces to 0.1 percent at $R^3/V = 1.5$ and to about 0.05 percent above $R^3/V = 2.0$. Furthermore, the second approximation, Equation (6), agrees with the Harkins and Jordan tables to better than 0.05 percent for values of $R^3/V > 2.4$. Since the region of interest is above $R^3/V = 2.4$, the exponential component was neglected in all of the computations involved in making Table 1. The data for $R/r = 40, 50$, and 60 were computed from equations of the same form as Equation (6) and were tabulated to four decimal places. From these data three-point Lagrangian interpolation was used to compute data to three decimal places for $R/r = 52, 54, 56$, and 58 .

Table 4 shows the agreement of Equation (6) with the Harkins and Jordan tables for $R/r = 50$ and $R^3/V > 2.4$.

TABLE 4
Correction Factors, F, for the Ring Method at $R/r = 50$

R^3/V	H and J*	Eq. (6)
2.4	0.8680	0.8685
2.5	0.8651	0.8654
2.6	0.8624	0.8625
2.7	0.8598	0.8596
2.8	0.8570	0.8569
2.9	0.8545	0.8543
3.0	0.8521	0.8518
3.1	0.8494	0.8495
3.2	0.8472	0.8471
3.3	0.8449	0.8450
3.4	0.8424	0.8428
3.5	0.8404	0.8407

*Harkins and Jordan

* * *

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