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Viscosity Measurements on Liquids to Pressures of 60 kbar

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A rudimentary capillary-type viscometer for measuring viscosities between 10^3 and 10^{10} poise at high pressures is described. Measurements are reported on petroleum ether to 57 kbar where the viscosity is approximately 10^{10} poise and on a 1:1 mixture of *n*-pentane–isopentane to 54 kbar where the viscosity is approximately 10^6 poise. Measurement precision of the technique is a few percent, but uncertainty in absolute determination at maximum pressure is only within a factor of two. Viscous stresses are shown to decay exponentially with time even at the highest pressures, and no effects of residual stress are observed.

I. INTRODUCTION

In a previous paper[1] the authors have discussed techniques for containing low-viscosity liquids within a capsule which in turn is placed within the solid-media pressure chamber of a hexahedral solid-media high-pressure system to obtain truly hydrostatic pressure to 60 kbar. Most ordinary liquids freeze to a crystalline form at pressures well below this limit, and those which do not crystallize become increasingly viscous and vitrify to a glass. A viscosity measurement thus becomes important in a practical sense to evaluate the suitability and limitations of various liquids for use as a hydrostatic pressure-transmitting medium at high pressures. Bridgman[2] measured the viscosities of a number of organic liquids to 30 kbar and found only six which are liquid at atmospheric pressure and room temperature and do not crystallize or vitrify at 30 kbar. Bridgman also indicated that these substances which did not crystallize were in a supercooled liquid state at the higher pressures, and that their viscosities increased exponentially at a rate of approximately one order-of-magnitude for each 5 kbar of pressure. The present work was initiated to select an appropriate liquid, find its limit of use, and remove uncertainties concerning the truly hydrostatic character of the liquids at these pressures. Since we were interested in achieving hydrostaticity at the highest possible pressure, the two liquids showing the greatest promise from Bridgman's work, petroleum ether and 1–1 *n*-pentane and isopentane mixture, were studied. Measurements were also made on isopropyl alcohol as a check on the technique and to make comparisons with the previous work of Bridgman. The uses of mixtures rather than pure substances is dictated by the knowledge that the pure liquids tend to crystallize below the pressures of interest.[3,4] We, of course, want to inhibit crystallization. We chose isopropyl alcohol for a comparison with Bridgman's work because it represented the highest viscosities measured by Bridgman.

Due to the inherent limitations imposed by the extreme pressures, and the relatively small-volume, present-day systems can maintain at these pressures, the viscometer must be of the most rudimentary type. As designed, the viscometer is usable only for rather high viscosities; in the work reported here measurements were above 10^4 poise. These measurements thus represent an extension both in pressure and in viscosity from Bridgman's work since his maximum viscosities were approximately 10^5 poise. No attempt has been made to measure lower viscosities using this technique although such measurements are feasible with minor changes. The present work is not intended as a highly precise study of pressure variation of viscosity, but rather as a practical investigation to determine appropriate relaxation times and limits of hydrostatic behavior when using particular liquids.

We will first describe the operation of the viscometer to make clear the approximations and the dependence on other measurements. The construction details will then be described before presenting the data on the liquids studied.

II. OPERATIONAL THEORY FOR THE VISCOMETER

A diagrammatic representation of the viscometer is shown in Fig. 1. Basically it consists of two cylindrical chambers C and C′ with enclosed volumes V and V′ connected by a capillary tube of prescribed length L and radius r. A time-dependent pressure difference can be established between the ends of the capillary, but no pressure difference can exist in the steady state. The geometrical location of the capillary is dictated by the limited space available in C′, which represents simply the liquid pressure capsule used in the hexahedral press.[1] The chamber C is a thick-walled metal container (brass in our case) which will hereafter be referred to as the viscometer. The viscometer is completely enclosed in C′. Both chambers C′ and C are filled with liquid at all times. We indicate the pressure in chamber C′ as P' and the pressure in C as P. At atmospheric pressure P_0 the dimensions of the viscometer are V_0, L_0, and r_0, where V_0 is the initial volume, L_0 is the initial length of the

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* Permanent address: Physics Department, Brigham Young University, Provo, Utah.

[1] J. D. Barnett and C. D. Bosco, Rev. Sci. Instr. 38, 957 (1967).

[2] P. W. Bridgman, *Collected Experimental Papers* (Harvard University Press, Cambridge, Mass., 1964), Vol. VI, Paper 166, pp. 3903–3914.

[3] L. E. Reeves, G. J. Scott, and S. E. Babb, Jr., J. Chem. Phys. 40, 3662 (1964).

[4] S. H. Gelles, J. Chem. Phys. 48, 526 (1968).

capillary, and r_0 is the initial radius of the capillary hole. V, L, and r are functions of pressure. We assume for simplicity that $V' \gg V$ and that the pressure P' can be changed at will by manipulating the applied load of the hexahedral press. We further assume that the compressibility K_l of the liquid and the compressibility K_m of the viscometer metal are known at the ambient pressure P. The pressures P and P' are assumed continuously measurable.

Consider the viscometer with volume V at an ambient pressure P filled with liquid also at pressure P. As pressure is applied to the liquid in the outer capsule C′, the viscometer elastically deforms. If the compressibilities of the liquid and metal of which the viscometer is constructed were equal, the change in the applied pressure P' would be immediately transferred to the liquid inside the viscometer, and no pressure differential between ends of the capillaries would appear. If, however, the liquid is more compressible than the metal, which is the typical situation, the viscometer will elastically deform, and the pressure P inside will instantaneously increase to a value determined by the elastic properties of both the liquid and the metal. Furthermore, the pressure P will be less than P', the difference being supported by the metal viscometer. A pressure difference thus exists across the capillary, and viscous flow results. The pressure difference equalizes in time and allows the viscometer to relax to a state of hydrostatic compression at the higher pressure.

A complete solution of the elastic deformation of the viscometer would determine the instantaneous volume V of C in terms of the pressure, the dimensions of the viscometer, and the elastic coefficients of the metal. Such a solution for a thick-walled, hollow, closed-end cylinder is not simple. We assume a solution of the form

$$V/V_0 = 1 - A(P'-P) - K_m P', \qquad (1)$$

where the value of A is to be evaluated experimentally. This type of expression is suggested by the analysis[5] of the deformation of two different physical approximations to the thick-walled, finite-length cylinder: (a) A hollow sphere with inside and outside radii a and b, respectively, yields

$$\frac{V}{V_0} = 1 - \left[\frac{3}{4\mu[1-(a^3/b^3)]} + \frac{K_m}{[(b^3/a^3)-1]}\right] \times (P'-P) - K_m P' \qquad (2)$$

and (b) a thick-walled, finite-length cylinder in which the longitudinal stress is assumed constant and equal to the outside pressure P' and the radial stress is a function of r only yields

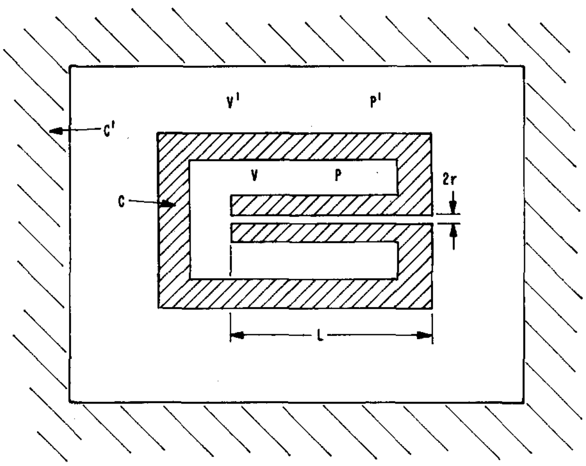

FIG. 1. Diagrammatic cross section of cylindrical viscometer giving nomenclature for mathematical derivations.

$$\frac{V}{V_0} = 1 - \left[\frac{1}{\mu[1-(a^2/b^2)]} + \frac{2K_m}{3[(b^2/a^2)-1]}\right] \times (P'-P) - K_m P', \qquad (3)$$

where μ and K_m are the rigidity modulus and compressibility of the metal and a and b are the inside and outside radii, respectively. We expect the measured value of A [Eq. (1)] obtained experimentally to be of the order given by the bracketed expressions of Eqs. (2) and (3). The values of A and K_m in Eq. (1) are in reality functions of pressure rather than constants as the linear theory suggests. This pressure variation can most conveniently be accounted for by introducing "instantaneous" values A', K_m', and K_l' rather than A, K_m, and K_l where the primed quantities relate to changes in volume relative to the ambient volume rather than to V_0, as will be seen below.

In order to consider the liquid exchange through the capillary, we must obtain an expression for the amount of liquid contained within the viscometer C occupying a volume V at a given pressure P. If this amount of liquid were at atmospheric pressure, it would occupy a volume V_{0l} which is different than the volume V_0 of C at atmospheric pressure. In terms of V_{0l} we may write

$$V_l = V_{0l}\left[1 - \int_0^P K_l dP\right] \qquad (4)$$

in which we have allowed the liquid compressibility K_l to vary with pressure.

Having expressions for the volume of the capsule [Eq. (1)] and for the volume of the enclosed liquid [Eq. (4)] we are prepared to write the differential equation which governs the time variations of P

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[5] See, for example, L. D. Landau and E. M. Lifshitz, *Theory of Elasticity* (Addison-Wesley Publ. Co., Reading, Mass., 1959), pp. 17–21.

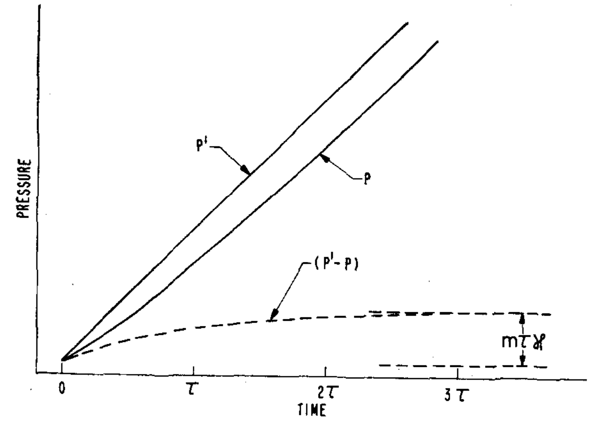


FIG. 2. Measurable pressure variations associated with an idealized viscosity determination using a linear increase of pressure P' with time.

associated with variations of P'. If $(P'-P)$ is nonzero, a differential volume dV_f of liquid will flow through the capillary in a differential time dt. This flow results in a change in pressure inside the viscometer within the same time interval, thus causing both a differential change in viscometer volume dV_v associated with deformation–relaxation and a differential change in the volume dV_l of the enclosed liquid due to compressibility. In equation form

$$dV_f = dV_v - dV_l.$$

The sign of dV_l is such as to make the magnitude of dV_v and dV_l additive. Taking differentials of the well-known expression for viscous flow through a capillary along with differentials from Eqs. (1) and (4) above yields

$$\begin{aligned}\pi r^4(P'-P)dt/8L\eta &= -V_0[A\,d(P'-P)+K_m dP'] + V_{0l}K_l dP \\ &= -V[A'd(P'-P)+K_m' dP' - K_l' dP], \quad (5)\end{aligned}$$

where η is the viscosity of the liquid, L is the length, and r is the radius of the capillary. Straightforward manipulations yield the desired differential equation

$$\frac{d(P'-P)}{dt} + \frac{P'-P}{S\eta} = \kappa\frac{dP'}{dt}, \quad (6)$$

where

$$S = 8LV(A'+K_l')/\pi r^4 \quad (6a)$$

and

$$= (K_l'-K_m')/(A'+K_l'). \quad (6b)$$

Since P' can be changed at will, we analyze two particularly useful special cases, each of which lends itself to a particular method of taking data. From an initial equilibrium condition of $P'=P=P_1$, P' is caused to increase or decrease linearly in time according to the equations $P'=P_1+mt$. The solution to (6) under these conditions yields

$$P'-P = m\tau\kappa[1-e^{-t/\tau}], \quad \text{where} \quad \tau = S\eta. \quad (7)$$

The results of an idealized experiment of the measurable pressures for this case are shown in Fig. 2. When $t \ll \tau$, we have $P'-P=m\kappa t$, and during this period

$$(P'-P)/(P'-P_1) = \kappa.$$

This is experimentally realizable when the viscosity is sufficiently high that pressure changes in C' can be effected in times much less than τ. The ratio κ is thus measurable, and the quantity A' can be determined to within the accuracy to which $(K_l'-K_m')$ is known. The uncertainty in $(K_l'-K_m')$ is propagated through A' and S to the viscosity determination and represents the dominant uncertainty in the measurement at high pressures.

The asymptotic value of $m\tau\kappa$ obtained for $(P'-P)$ from Eq. (7) when $t \gg \tau$ is experimentally measurable when the viscosity is very low, and if a value of κ is known, the measurement yields good viscosity data. In practice, these asymptotic measurements are made at the lower pressures where τ is much less than the pumping time. Later measurements at higher pressures using $t \ll \tau$ yield κ. For these low-viscosity measurements, the assumption is made that κ is a slowly varying function with pressure, and within the accuracy of the experiments the variation can be neglected.

At the higher pressures a second method of taking data is used based upon a second special time variation of P'. In this case, P' is rapidly changed from an equilibrium condition where $P'=P=P_1$ to a higher or lower value P_2 and then held constant in time at this value. The rapid change is made in a linear manner, and thus, during the change the analysis given above is applicable and yields κ. Since P' is constant after this time, the solution of Eq. (6) becomes

$$P'-P = (P_2-P_3)e^{-t/\tau} = \kappa(P_2-P_1)e^{-t/\tau},$$

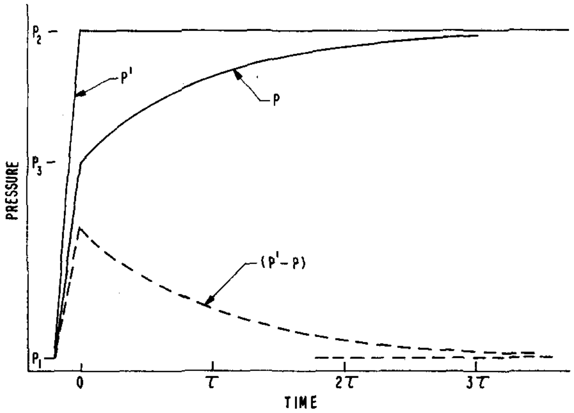


FIG. 3. Measurable pressure variations associated with an idealized viscosity determination using a constant pressure P' following a rapid linear increase.

where time is measured from the moment the pressure change is concluded, the P_3 is the inside pressure resulting from elastic deformation of the viscometer. Pressures existing in an idealized measurement are indicated in Fig. 3 for a κ value of 0.4. A graphical plot on semilog paper of $(P'-P)$ vs t yields τ from which the viscosity is calculated.

One should note that the proportionality constant S relating the time constant τ to the viscosity varies with pressure only through the quantity $(A'+K')$ since the ratio LV/r^4 is independent of a uniform compression of the viscometer.

III. VISCOMETER CONSTRUCTION AND USE

Three separate viscometers of differing dimensions have been used in this work above 50 kbar. All have essentially the geometry indicated in Fig. 1 with varying dimensions to change the proportionality constant S between τ and η. The outside diameter of all three viscometers was approximately 0.8 cm and each had a wall thickness of approximately 1.5 mm. The overall length varies between 1 and 2 cm. Coils made with cotton-wrapped or silk-wrapped 0.125-mm-diam manganin wire were used as the pressure sensors, referred to hereafter as manganin gauges. The viscometers were constructed of brass in two sections as indicated in Fig. 4. The electrical feedthrough (4) was provided by simply using a commercial two-component Epoxy cement surrounding a copper wire. The viscometer body itself was used as the other contact for the inner manganin gauge. After the inside manganin gauge was wound and electrically connected as indicated, the two brass pieces of the viscometer were brazed together using a soldering iron and soft solder. Care was necessary to prevent overheating of the Epoxy during this brazing. A loose-fitting Teflon cover was placed over the outside rear end of the viscometer, and the outside manganin gauge was wrapped on this Teflon cover. As discussed below, it is advantageous to make the two manganin gauges of approximately equal resistance. The completed viscometer was seasoned at 140°C. for several hours to anneal the manganin coils. No effort was made to calibrate the manganin gauges of each viscometer on an absolute scale, but this is an obvious refinement.

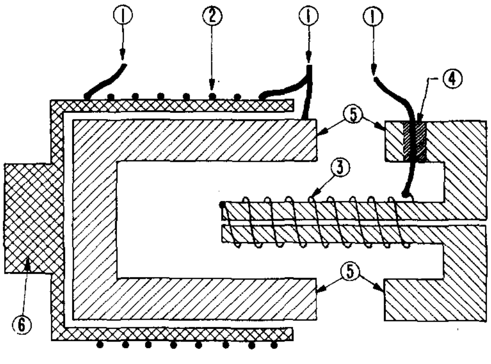


FIG. 4. Cross section of cylindrical viscometer showing details of construction. (a) Copper lead wires to extend out of capsule; (2) outside manganin gauge, M_0; (3) inside manganin gauge, M_i; (4) epoxy electrical feedthrough; (5) surfaces to be brazed to seal viscometer; (6) Teflon insulating sheath.

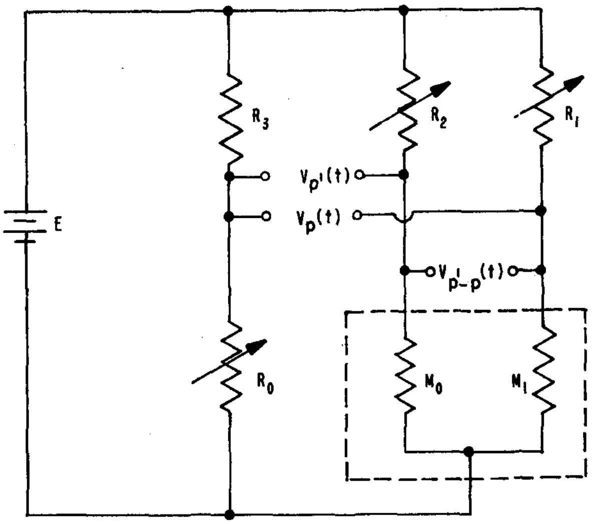


FIG. 5. Parallel Wheatstone-bridge circuit used for recording pressure changes detected by manganin gauges.

The time variations of the manganin resistance were measured by connecting them into a parallel Wheatstone bridge circuit as shown in Fig. 5 from which voltages proportional to $(P'-P)$, P', or P could be detected as desired. Voltages were measured using a Keithly 150A microvolt meter, and the output was recorded as a function of time. The resistances R_1, R_2, and R_3 as generally used were approximately 100 times M_0, M_i, and R_0 in order to make the voltage output of the Wheatsone bridge effectively linear with change of resistance M_0 and M_i.

A typical recording of the output of the microvoltmeter for a viscosity determination is shown in Fig. 6 along with the semilog plot showing the exponential decay with time. Note the zero shift after the pumping period. This shift can be reduced to a negligible amount by making the outside and inside manganin resistances of equal value and both as large as is feasible. This zero shift, we believe, is attributed to lead resistance effects and could be completely eliminated by using a four-lead resistance measuring technique.

IV. RESULTS AND DISCUSSION OF ACCURACY

A total of five independent viscosity experiments were performed involving three different liquids. Each experiment constituted a series of individual viscosity measurements over a convenient pressure interval. The determinations within a given experiment all had systematic errors in common, but were independent determinations at each pressure. Viscosity measure-

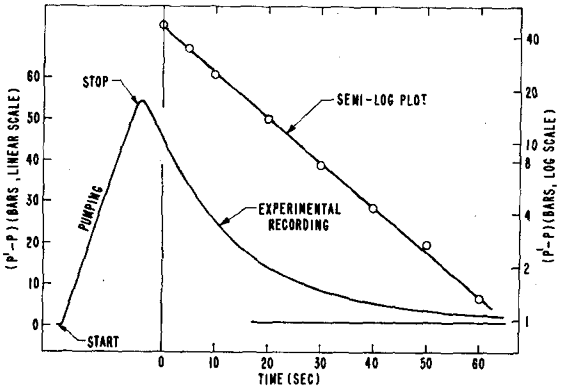
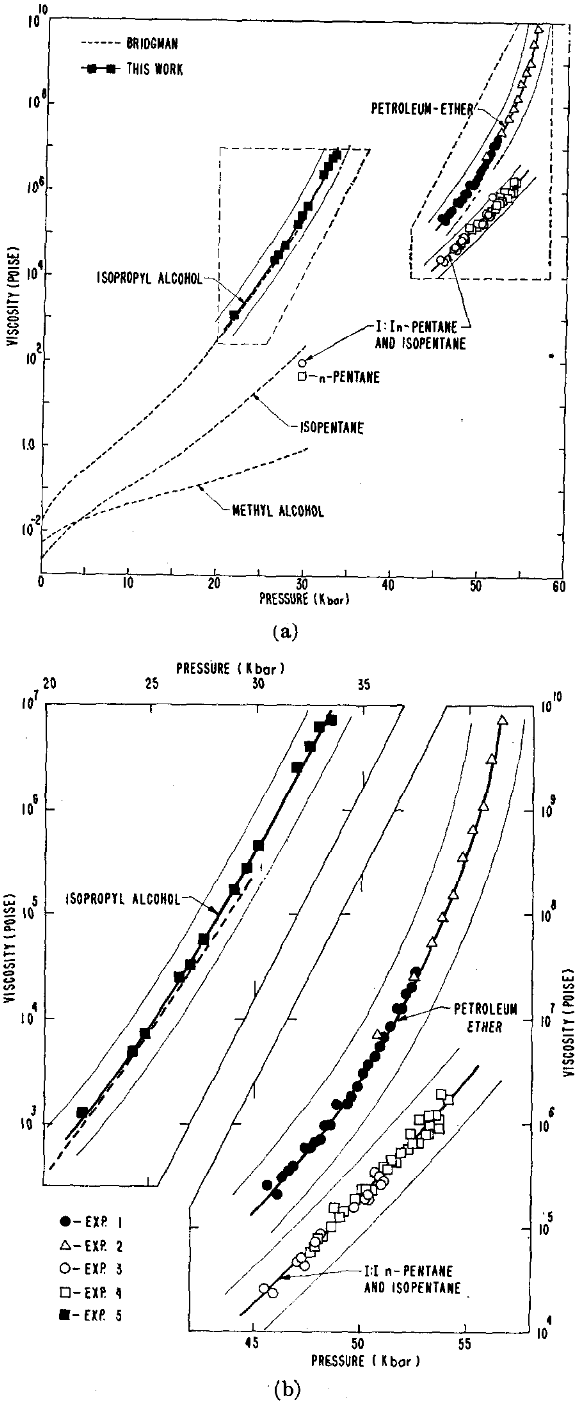


FIG. 6. Typical recorder trace of $(P'-P)$ pressure differences es a function of time and corresponding semilog plot to illustrate axponential variation. Points were simply taken from recording.

ments obtained from these five experiments are shown in Fig. 7 along with data on these and related substances reported by Bridgman.[2] Bridgman's data on methyl alcohol are given because it appears to be a very promising liquid at higher pressures. Bridgman reports only one viscosity determination above 12 kbar for 1:1 *n*-pentane isopentane mixture and one determination for pure *n*-pentane. These points are also shown in Fig. 7(a) for reference. Scientific data associated with each experiment and critical viscometer dimensions are given in Table I for each set of data in Fig. 7. The experiments are numbered in the chronological order in which they were performed, and due to improved understanding, experimental technique, and instrumentation, the accuracy and reliability also improved with each experiment. Light lines in Fig. 7 indicate estimated limits of maximum uncertainty in reported viscosity values on an absolute scale.

The initial intent of this work was not in the spirit of a quantitative, absolute viscosity determination for specified fluids but rather to determine limits of hydrostaticity at these pressures. The reproducibility and apparent sensitivity actually observed, however, suggest that this investigation might well be used for such a fundamental viscosity study. For this reason we shall make a rather crude analysis of the errors involved and mention some specifics about each experiment to enable the reader to better evaluate the technique for quantitative measurement.

Experiments 1 and 2 were exploratory in nature to see if the general concept of the measurement was valid. They were performed before the complete analysis given above, including the deformation, was formulated. Pressures were determined simply from a load vs pressure calibration of the hexahedral press. The reported viscosity values on petroleum ether are thus rather inaccurate, the dominant uncertainty being associated with a pressure uncertainty of approximately 2 kbar. A relative pressure scale shift of 0.6 kbar between experiments one and two was necessary to make the petroleum ether curve of Fig. 7 continuous. Experiments 3 and 4 were performed not only to determine hydrostatic limits of 1:1 *n*-pentane isopentane mixture but to check our understanding of the



(a)

(b)

FIG. 7. (a) Viscosity measurements on three liquids studied as compared with previous data by Bridgman. Estimated limits of accuracy on an absolute basis is indicated. (b) Details of (a) showing in detail measured points to indicate precision and reproducibility in each experiment.

TABLE I. Viscometer dimensions and other significant parameters associated with each experiment.

Experiment	Liquid	r_0 (mm)	L_0 (mm)	V_0 (mm^3)		S (poise/sec)	Shortest τ measured	Longest τ measured
1	Petroleum ether	0.17	8.7	62	0.17	8.5×10^4	2.8	320
2	Petroleum ether	0.38	8.7	62	0.17	2.1×10^6	3.3	3510
3	1:1 *n*-pentane and isopentane	0.17	15.2	137	0.10	1.7×10^4	1.6	42
4	1:1 *n*-pentane and isopentane	0.17	15.2	142	0.10	1.6×10^4	3.7	76
5	Isopropyl alcohol	0.17	15.2	144	0.25	8.5×10^3	0.17	543

operation of the viscometer, determine reproducibility in individual viscosity measurements, and to check reproducibility between completely independent experiments. Pressure was determined using the outside manganin coils as an absolute gauge with pressures based on a low-pressure calibration of a gauge made from the same spool of manganin wire. As seen in Fig. 7, consistency between experiments three and four was good without a relative pressure scale shift. The extrapolation of a low-pressure manganin calibration curve to 50 kbar is probably uncertain to 1.0 kbar. If the same manganin gauge were used in different experiments, pressure reproducibility could be of the order of 10 bar even though a calibration at high pressure on an absolute scale is not known better than 0.5 kbar at these pressures.

Experiment 5 was performed to obtain data for comparison with Bridgman's independent work, and thus evaluate the reliability of the technique. Using the uncalibrated manganin gauge as mentioned above, pressure uncertainty is approximately 0.5 kbar.

Systematic uncertainties in the viscosity measurement can be attributed to three areas: (1) compressibility of the liquid and deformation of the viscometer, (2) dimensions of the viscometer, and (3) determination of the time constant. Our estimates of the magnitudes of these errors for 1:1 *n*-pentane isopentane and for isopropyl alcohol are given along with suggested improvement.

The only available liquid compressibility data at these pressures is due to Bridgman,[6] and he measured only pure substances. We used a value of $K_l'=2.0\times10^{-6}$/bar for both petroleum ether and the 1:1 *n*-pentane isopentane mixture based on Bridgman's data of similar liquids, but we feel that this value could be in error by as much as 30%. Bridgman's data on pure substances is probably no better than 10%. Uncertainty of approximately 40% and 20% in the determined viscosities of the 1:1 *n*-pentane isopentane and isopropyl alcohol, respectively, was attributed to uncertainty in K_l' and the associated distortion problem. This uncertainty represents the most serious limitation of the technique at the higher pressures. This error is more significant at the higher pressures where K_l' is small since the analysis given in Sec. I breaks down as the compressibility of the liquid approaches the compressibility of the metal of the viscometer. The evaluation of the constants in Eq. (6) associated with the elastic deformation of the viscometer also become less certain. In the case of isopropyl alcohol, however, when K_l' was large, the experimentally determined value of A' agreed within approximately 40% of values calculated from Eqs. (2) and (3). This is not unreasonable considering the crude models which Eqs. (2) and (3) represent, and implies consistency in our analysis.

The uncertainties in the dimensions of the viscometer are minor except for the capillary radius which has a r^4 dependence in Eq. (6). Estimated uncertainty is approximately 10% for our viscometers, but with proper care this could easily be reduced to 1% or 2%.

Any systematic error in the measurement of the time constant should be indicated in Fig. 7 for the 1:1 *n*-pentane isopentane mixture since measurements were made on both increasing and decreasing pressure cycles. The deviations appear to be random with a standard deviation of approximately ten per cent. In a well-controlled hydrostatic system at lower pressure this would be of the order of 1%.

Adding these uncertainties, we expect the viscosity of 1:1 *n*-pentane isopentane mixture to be uncertain to less than 60% on an absolute scale and the isopropyl alcohol to be uncertain less than 40%. Error associated with pressure uncertainty of course adds to this. It appears to the authors that the technique described here could have applications where K_l' was well known, and in this case refinements would permit measurements to approximately 5% or less.

V. CONCLUSIONS

Perhaps the most significant result of this work for the practical high-pressure researcher is the demonstration that there exists a truly hydrostatic media usable as a high-pressure environment to approximately 60 kbar

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[6] P. W. Bridgman, *Collected Experimental Papers* (Harvard University Press, Cambridge, Mass., 1964), Vol. VI, Paper 138, pp. 3463–3488.

providing appropriate relaxation times are taken into account. In this connection, it is important to realize that the time constants measured in this work are related to a very restricted flow geometry and that relaxation times in a bulk material would be two or three orders-of-magnitude shorter than the time constants measured here. It should also be realized that if a high viscosity liquid is used as an environment, shear stress is produced in the liquid and in the sample during changes in pressure, but these shear stresses relieve in a characteristic time, and only after sufficient time are hydrostatic conditions established. The data reported here indicate what time scales are required when using a particular liquid, to keep shear levels below specified limit.

The analysis of the viscometer assumed a stress-independent coefficient of viscosity, and the fact that the semilog exponential decay curves were effectively straight lines implies that this is true even to the maximum pressures and viscosities studies. There was no indication of residual stress within the pressure sensitivity of approximately 1 bar, but no special effort was taken to establish the total absence of residual stress.

In this experiment, we report viscosity measurements from approximately 10^3 poise to 10^{10} poise covering an overall range of 10^7. It appears, however, that with some effort viscometers with S values differing by as much as 10^5 could be constructed, and since time constants from 0.1 sec to 10^4 sec can be measured, the over-all range could probably be extended to 10^{10} from 10 poise to 10^{11} poise. If pressure chambers were available with larger volumes, even lower viscosities could be measured. We have demonstrated the consistency of the technique with previous work at lower pressures and have effectively doubled the pressure range over which viscosity measurements have been made.

In light of the estimated uncertainties, the agreement of our measurements with Bridgman's isopropyl alcohol is better than could be expected but is satisfying and indicated the consistency of the two methods. We do not intend, however, to base the reliability of the technique on agreement with Bridgman's data since Bridgman stated that his data for isopropyl alcohol were taken in three sections using different viscometers and that an additive constant was applied to the higher viscosities to yield a smooth curve. Although the uncertainties indicated in Table I are not satisfying when compared to atmospheric measurements of viscosity, it should be noted that more precise viscosity measurements on an absolute basis are not justified practically until improved pressure calibration techniques and an improved experimental pressure scale are available. Furthermore, improved viscosity analysis would be meaningless until improved compressibility measurements are available on liquids in question. The experimental dependence of a viscosity measurement on compressibility of the liquid using this technique is somewhat inconsequential in a theoretical sense since any theoretical analysis of viscosity changes will be associated with volume changes rather than pressure, and thus reliable compressibility data would be required for any meaningful fundamental study independent of how the viscosities were measured.

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