

Torsional Crystal Technique for the Measurement of Viscosities of Liquids at High Pressure

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The damping effect of a viscous medium on a torsionally vibrating quartz crystal has been used to measure the viscosities of liquids at high pressures. Results have been obtained for benzene, cyclohexane, carbon tetrachloride, isopentane and n-pentane at temperatures of 30 or 50°C and pressures up to 7000 kg cm⁻² in systems where the liquid did not freeze. Good agreement with existing high pressure data for these systems is obtained.

It is difficult to extend conventional techniques of high accuracy such as the capillary flow method to determine the viscosity of liquids at high pressures. Most of the effort in this field has therefore been confined to falling weight or rolling ball techniques. Even with these methods substantial problems remain in either rotating the high pressure system with the viscometer or uncoupling from it after each pressure change. It is therefore pertinent to explore alternative methods of determining the viscosity of liquids which do not present great difficulties at pressures up to about 10 000 bar and temperatures of the order of 100°C. In the present work, application of the torsional crystal technique to high pressure viscometry is examined.

THEORY OF METHOD

When a suspended quartz crystal of length l with axis parallel to its length has an alternating voltage applied to electrodes located longitudinally in the four quadrants between the Y and Z axes, mechanical torsion is produced which generates pure shear waves. In a vacuum, the crystal then resonates at a characteristic frequency f_0 , which for the fundamental mode is given by

$$f_0 = (2l(\rho_Q\sigma)^{\frac{1}{2}})^{-1}, \quad (1)$$

where $\sigma = 2.42 \times 10^{-12}$ cm² dyn⁻¹, is the elastic modulus for torsion and ρ_Q is the density of quartz. Both f_0 and R_0 , the resistance at resonance in a vacuum, vary slowly with temperature.

When the crystal is placed in a viscous medium, the propagated wave is damped according to the equation for plane waves,

$$p = p_0 \exp \left[-\left(\frac{\pi f \rho}{\eta} \right)^{\frac{1}{2}} z \right], \quad (2)$$

where z is the distance from the surface of the crystal, and η and ρ are the viscosity and density of the medium respectively. The attenuation of a wave, $(\pi f \rho / \eta)^{\frac{1}{2}}$,

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in benzene at 25°C for a crystal 5 cm long and 0.5 cm diam. having a resonant frequency of 39 kHz is approximately 416 nepers cm^{-1} . The amplitude of the wave is therefore reduced to e^{-1} of its initial value at a distance of 2.4×10^{-4} cm from the crystal surface. This distance is small compared to the crystal radius so that plane wave theory is applicable.

The degree of damping in a viscous fluid can be readily measured in terms of an impedance loading on the crystal which produces a change in the crystal resistance at resonance, $\Delta R = R - R_0$ and in the resonant frequency, $\Delta f = f_0 - f$. For Newtonian fluids, these changes are related to the viscosity and density of the fluid by the equations:

$$R - R_0 = \Delta R = K_1(\pi f \eta \rho)^{\frac{1}{2}}, \quad (3)$$

$$f_0 - f = \Delta f = K_2(\pi f \eta \rho)^{\frac{1}{2}}. \quad (4)$$

K_1 and K_2 are electromechanical constants given by

$$K_1 = \frac{2R(r^{-1} + l^{-1})}{\pi \rho_Q(f_2 - f_1)} \left(1 - \frac{2\Delta f}{f_0}\right) \quad (5)$$

and

$$K_2 = (r^{-1} + l^{-1})/\pi \rho_Q, \quad (6)$$

where r and l are the crystal radius and length respectively, and f_2 and f_1 are the frequencies above and below the resonant frequency where the crystal resistance is $2R$. The half-conductance points, (f_1 and f_2), are clearly indicated in fig. 5. The factor $(1 - 2\Delta f/f_0)$ in eqn (5) is a correction for the increase in the effective moment of inertia of the crystal due to the reaction of the fluid, which is of the order of 0.1 % in the liquids investigated.

EXPERIMENTAL

FACTORS AFFECTING MEASUREMENTS

CRYSTALS AND LEADS

Extensive investigations of the torsional crystal technique for measuring viscosity have been made by Mason¹ who devised the method, by Rouse *et al.*² and Lamb and coworkers.³ In the present work the crystals used were supplied by Brush Clevis Co. and had a length of 5 cm and diameter of 0.5 cm. According to eqn (1), the fundamental mode of frequency corresponding to this length was about 39 kHz. The positioning of the electrodes on, and the attachment of the leads to, the crystal are shown in fig. 1. The quartz cylinders were cut to an accuracy in angle of 10' with the main axis parallel to the X -axis to obtain a torsional mode of vibration, which is favoured³ by a length-to-diameter ratio of about 10:1. Four gold electrodes were formed by vacuum deposition in quadrants of 80° axially along the crystal with the unplated regions lying in the Y - and Z -axes. For the fundamental mode of vibration, the length of the crystal is equal to half a wave-length with a node at the centre of the crystal. The leads attached to the crystal at this point therefore had a minimum damping effect. No difference was found in using either 0.014" diam. copper or 0.008" diam. phosphor-bronze leads. S-bends were inserted in the leads between the crystal and a metal supporting frame to minimize strain on the lead attachments. Polished and unpolished crystals were used to examine the effect of the surface finish of the crystal.

CRYSTAL CONSTANTS

In theory, either the change in frequency or in resonant resistance can be used to determine the viscosity of the fluid when the crystal constants and fluid density are known. K_1 can be determined by calibration with liquids of known density and viscosity and use of eqn (3). Alternatively, the two half-conductance frequencies can be measured and used in

conjunction with the crystal dimensions in eqn (5) to give K_1 . These two sources of K_1 are compared for a range of liquids at 30°C in table 1 and the agreement averages 0.2% with a range of -0.2 to +0.7%. Density and viscosity data were taken from Timmermans⁴

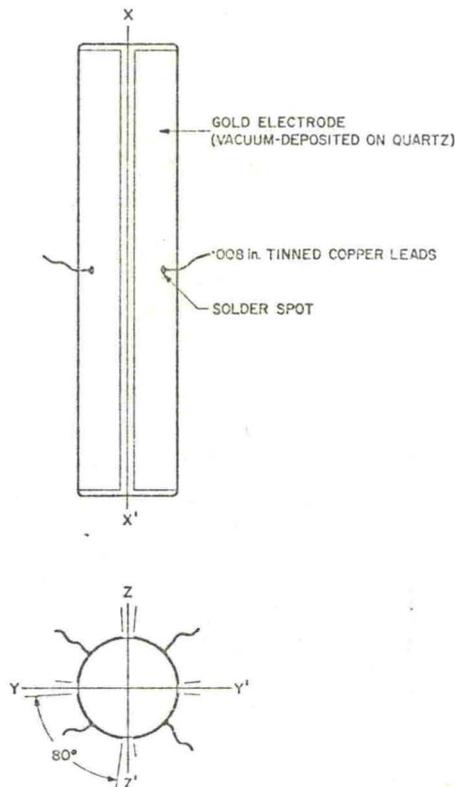


FIG. 1.—Configuration of electrodes for the torsional crystal.

except for iso- and n-pentane for which A.P.I. viscosity values⁵ have been used. Table 1 also shows that the value of the constant K_1 is slightly dependent on the kinematic viscosity of the fluid. Harrison and Lamb³ have shown that the variation of K_1 and K_2 with kinematic viscosity is due to irregularity in the crystal surface and that this effect which is larger for K_2 is readily removed by careful polishing. According to eqn (2), the attenuation of the viscous wave is inversely proportional to the exponential of the square root of the kinematic viscosity. Therefore, if the kinematic viscosity is sufficiently small, the attenuation length and irregularities in the crystal surface may become comparable, resulting in an increase in the electromechanical constants. For fluids of kinematic viscosity greater than

TABLE 1.—VALUES OF THE CONSTANT K_1 FOR A 39.5 KHz POLISHED CRYSTAL IN VARIOUS LIQUIDS AT 30°C AND ATMOSPHERIC PRESSURE

liquid	$(\eta/e)/\text{mSt}$	$K_1/\Omega \text{ cm}^2 \text{ s g}^{-1}$	
		from eqn. (3)	from eqn (5)
iso-pentane	3.34	5255	5290
n-pentane	3.48	5260	5285
carbon tetrachloride	5.35	5105	5085
benzene	6.47	5060	5080
cyclohexane	10.64	5080	5075
decalin	22.2	—	5030
di-butyl phthalate	130	5020	5010

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1 centistoke this effect was negligible. The value of K_1 was also found to be slightly temperature dependent agreeing with the results of Harrison and Lamb,³ whereas De Bock *et al.*⁶ found K_1 to be virtually independent of temperature. Experimental values of K_2 (from frequency change in a standard liquid) varied from 5 to 10 % from values calculated from eqn (6), so that the change in frequency was not used in this work to determine viscosities.

APPARATUS AND TECHNIQUE

CRYSTAL ASSEMBLY

The crystals were mounted by soldering the 4 leads to metal straps, for which both stainless steel and gold-plated brass were used. The metal straps were screwed into a pair of pyrophilite rings to form a protective cage around the crystal. Opposite pairs of metal straps were electrically connected by small stainless steel bars contained in the lower pyrophilite ring. 10 BA tapings were made in 3 adjacent straps for connection to external leads. The crystal assembly was transferred as a unit from a stainless steel calibration vessel to the high-pressure apparatus, being conveniently mounted by screwing the upper pyrophilite ring against stainless steel spacers. Some of these details are shown in fig. 2.

HIGH PRESSURE SYSTEM

Fig. 2 shows the crystal assembly attached to the pressure vessel closure which was threaded to receive a stainless steel sleeve argon arc-welded to a stainless steel bellows. This separates the crystal and test liquid from the hydraulic fluid and allows compression. A schematic layout of the high-pressure system which was designed for pressures up to 10 000 bar is given in fig. 3. Pressures up to 2000 bar were obtained on the charge pump. For higher pressures the high pressure side of the intensifier was primed to 2000 bar and this pressure increased by operation of the drive pump. Pressure measurements were made on Bourdon gauges, a 40 000 psi gauge calibrated against a dead weight piston gauge, and

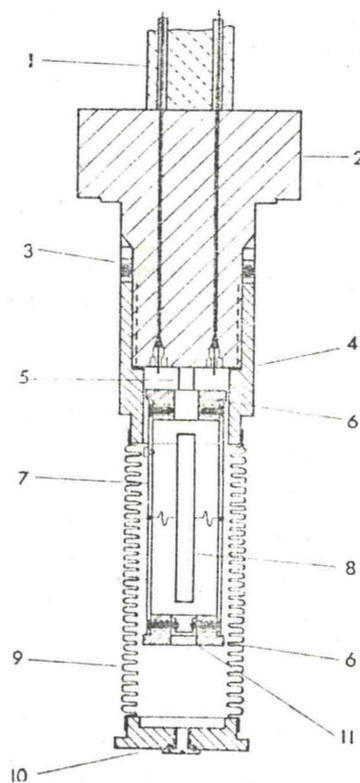


FIG. 2.—Details of mounting of crystal and attachment to pressure vessel closure. 1, Tufnol cylinders; 2, pressure vessel closure; 3, pressure seal; 4, stainless steel sleeve; 5, spacer; 6, pyrophilite ring; 7, metal support strap; 8, quartz crystal; 9, stainless steel bellows; 10, bottom plug; 11, connector.

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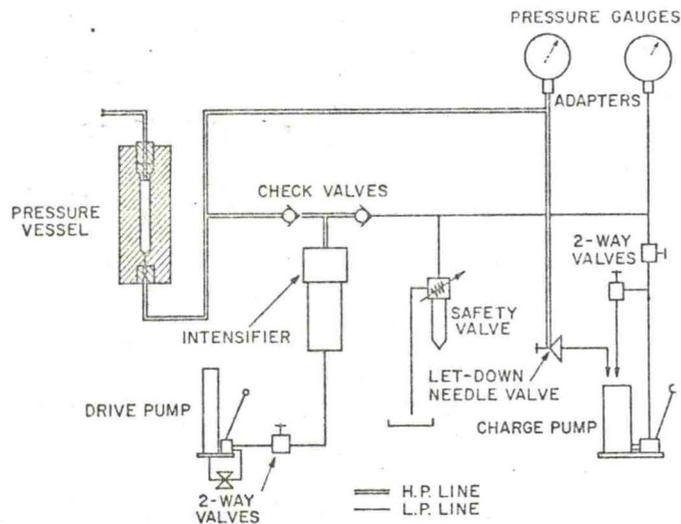


FIG. 3.—Flowsheet of the high-pressure system.

a 10,000 atm gauge made and calibrated by the Budenberg Gauge Co. For convenience in comparing with existing data, experimental pressures are given in kg cm^{-2} as used by Bridgman ($1 \text{ kg cm}^{-2} = 0.981 \text{ bar}$). Measurements on the high-pressure gauge were converted using $1 \text{ atm} = 1.033 \text{ kg cm}^{-2}$ and on the low pressure gauge by $1 \text{ psia} = 0.0703 \text{ kg cm}^{-2}$. As our pressure measurements are no better than $\pm 1\%$, 1 kg cm^{-2} can be effectively read as 1 bar.

ELECTRICAL SYSTEM

Fig. 4 is a block diagram of the electronics measuring system. The crystal was connected as one arm of a Wayne-Kerr B 601 resistance-capacitance bridge driven by a Schlumberger FS1 digital signal generator with a stability of 1 part in 10^8 and incremental tuning of 0.01 Hz.

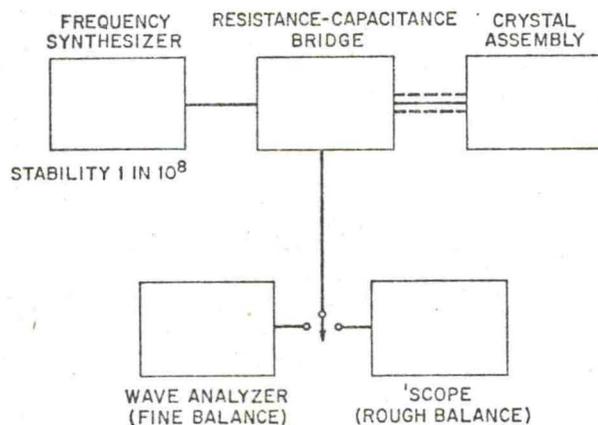


FIG. 4.—Block diagram of electronics for torsional crystal viscosity measurements.

The bridge output was displayed on a Telequipment S43 oscilloscope for rough balance, with the final, accurate balance being made with an Airmec 853 wave analyser. Care was taken to eliminate stray and leads capacitances. Connections to the pressure vessel were made rigid by pulling the leads through a Tufnol cylinder which was screwed to the pressure plug, and the soldered connections to the bridge leads were shielded by a brass cap fitting over the Tufnol cylinder. Connections between the pressure vessel and RC bridge consisted of co-axial cable drawn through $\frac{1}{8}$ in. o.d. brass tube to provide both rigidity and secondary screening. The outer braidings of the co-axial cables were connected to the central tapping of the bridge input transformer to eliminate the effect of the leads capacitance in the measurements, and the secondary screenings were connected to the bridge earth terminal.

PROCEDURE

The test liquid was thoroughly degassed by alternate freezing and melting under vacuum, and was introduced again under vacuum through the end plug in the bellows assembly. The complete unit shown in fig. 3 was inserted in the pressure vessel. The system was pressurized and electrical measurements commenced after allowing up to 30 min for the heat of compression to be dissipated. The approach to thermal equilibrium could be monitored by the change in resonant frequency of the crystal. The capacitance of the crystal at resonance was set at the average value of capacitances obtained with an infinite resistance

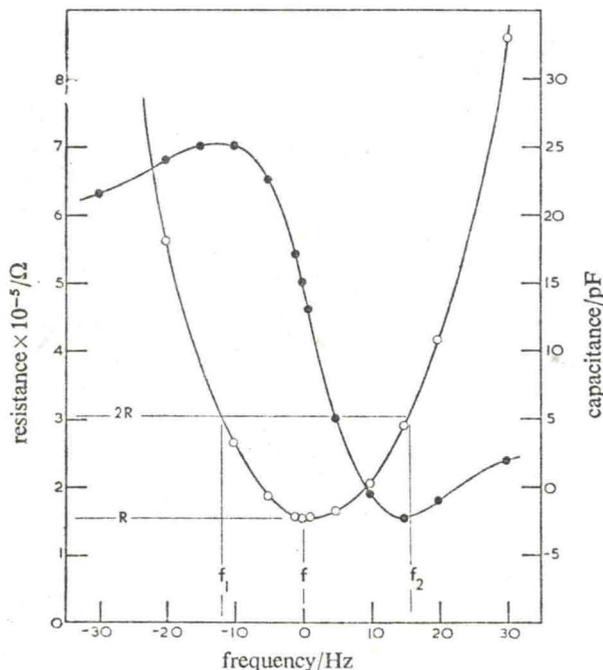


FIG. 5.—Resonance characteristics for a 39 kHz crystal in benzene at 25°C. Resistance, O; capacitance ●.

liquid	refractive index at 25°C	
	obs.	recommended 4, 5
benzene	1.4978	1.4979
carbon tetrachloride	1.4576	1.4576
cyclohexane	1.4234	1.4235
n-pentane	1.3575*	1.3576*
isopentane	1.3509	1.3507

* $n_{D_{20}}$

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TABLE 2.—EXPERIMENTAL VISCOSITIES OF LIQUIDS UNDER PRESSURE

Liquid	Pressure/kg cm ⁻²	absolute viscosity/cP			
		$T/^\circ\text{C}$ $P_{\text{max}}/\text{kg cm}^{-2}$	30 890	40 1260	50 1755
benzene	1		0.561	0.491	0.434
	250		0.692	0.607	0.537
	500		0.838	0.733	0.650
	750		1.000	0.875	0.774
	1000		1.102 (900)	1.031	0.905
	1250			1.204	1.051
	1500				1.211
	1750				1.388
carbon tetrachloride	1	$T/^\circ\text{C}$ $P_{\text{max}}/\text{kg cm}^{-2}$	30 1350	38.4 1750	50 1860
	250		0.843	0.745	0.649
	500		1.054	0.932	0.823
	750		1.290	1.143	1.012
	1000		1.553	1.375	1.217
	1250		1.843	1.626	1.434
	1500		2.161	1.892	1.668
	1750			2.173	1.911
cyclohexane	1	$T/^\circ\text{C}$ $P_{\text{max}}/\text{kg cm}^{-2}$	30 340	34 420	50 870
	100		0.819	0.760	0.605
	200		0.924	0.857	0.675
	300		1.038	0.962	0.751
	400		1.162	1.072	0.833
	600		1.228 (350)	1.191	0.923
	800				1.130
					1.374
n-pentane	1	$T/^\circ\text{C}$ $P_{\text{max}}/\text{kg cm}^{-2}$	30 7110		50 5200
	500		0.215		0.179
	1000		0.332		0.275
	1500		0.450		0.371
	2000		0.578		0.470
	3000		0.725		0.578
	4000		1.075		0.848
	5000		1.515		1.196
	6000		2.080		1.646
7000		2.784			
isopentane	1	$T/^\circ\text{C}$ $P_{\text{max}}/\text{kg cm}^{-2}$	30 3130		50 2100
	500		0.205		0.174
	1000		0.327		0.279
	1500		0.454		0.391
	2000		0.593		0.510
	3000		0.745		0.635
				0.917	
				1.109	

setting at ± 5 kHz from the fundamental frequency. These two results were usually identical and did not differ by more than 0.1 pF. Having set the capacitance, the bridge resistance and generator frequency were varied until the balance was obtained. The bridge resistance was then set at twice the resonance value and capacitance and frequency were varied until the two half-conductance frequencies, indicated by f_1 and f_2 in fig. (5), were obtained. K_1 was calculated at each pressure from eqn (5) and this value was used in eqn (3) with the measured ΔR to calculate the viscosity of the liquid.

In fig. 5, the measured relationship between resistance, capacitance and frequency for a 39 kHz crystal immersed in benzene at atmospheric pressure and 25°C is shown. While capacitance does not enter the equations from which the viscosity of the surrounding medium is obtained, its significance is clearly indicated.

PURITY OF MATERIALS

All liquids investigated were obtained from Hopkin and Williams Ltd. Spectrosol carbon tetrachloride, and cyclohexane were used without further purification after checking the refractive index. R.I.-grade benzene was fractionally distilled over P_2O_5 and G.P.R. n- and iso-pentane were fractionally distilled over sodium wire.

RESULTS

VISCOSITIES

Table 2 gives the measured values of viscosity. The results are smoothed data from at least two runs with increasing and decreasing pressure, except for benzene at 40°C, cyclohexane at 34°C and carbon tetrachloride at 38.4°C for each of which only one set of measurements was made. The (pressure, density) data for the liquids were taken from Bridgman⁷ except for cyclohexane for which *PVT* measurements have been made by Holder and Whalley,⁸ and Reamer and Sage.⁹ Also shown in table 2 are the maximum pressures for each liquid and temperature. In fig. 6-10 the results in table 2 are compared with existing literature data.

DISCUSSION

The accuracy of the torsional crystal technique for measuring the viscosity of liquids at atmospheric pressure is indicated by the agreement between measured and calculated values of K_1 in table 1. An average agreement of 0.2 % was obtained with the worst agreement (0.5-0.6 %) occurring with the pentanes, for which there exists considerable uncertainty in literature values.^{4, 5} Our results are in keeping with the conclusion of the Franklin Institute report² that torsional crystal viscosity measurements in liquids are capable of an accuracy of 0.5 % or better.

In extending the use of this method for high pressures, additional sources of error were considered. These were (i) the inaccuracy of *PVT* data for liquids, (ii) contamination of the sample liquid by leakage of hydraulic fluid into the bellows assembly, and (iii) temperature variation in the oil bath or through heat of compression.

The probable accuracy of the density data is ± 0.1 % contributing an uncertainty of ± 0.2 % to the viscosity results. Since no differential pressure across the bellows seal occurred, seepage of hydraulic fluid was considered unlikely, and was not apparent in the results, where an increase in $(\partial\eta/\partial p)_T$ would result. As mentioned earlier, the bulk of the pressure vessel assisted in damping out temperature fluctuations in the bath. The elimination of temperature increases caused by compressions of the hydraulic and test fluids was monitored by the crystal resistance which clearly indicated when equilibrium had returned. The uncertainty in viscosity measurements under pressure was concluded to be approximately 1 %. Comparisons with existing

PRESSURE

viscosity/cP

	50
	1755
01	0.434
07	0.537
03	0.650
05	0.774
01	0.905
04	1.051
	1.211
	1.388

	50
	1860
05	0.649
02	0.823
03	1.012
05	1.217
06	1.434
02	1.668
03	1.911
05	2.161

	50
	870
00	0.605
07	0.675
02	0.751
02	0.833
01	0.923
	1.130
	1.374

	50
	5200
	0.179
	0.275
	0.371
	0.470
	0.578
	0.848
	1.196
	1.646

	50
	2100
	0.174
	0.279
	0.391
	0.510
	0.635

VISCOSITY AT HIGH PRESSURES

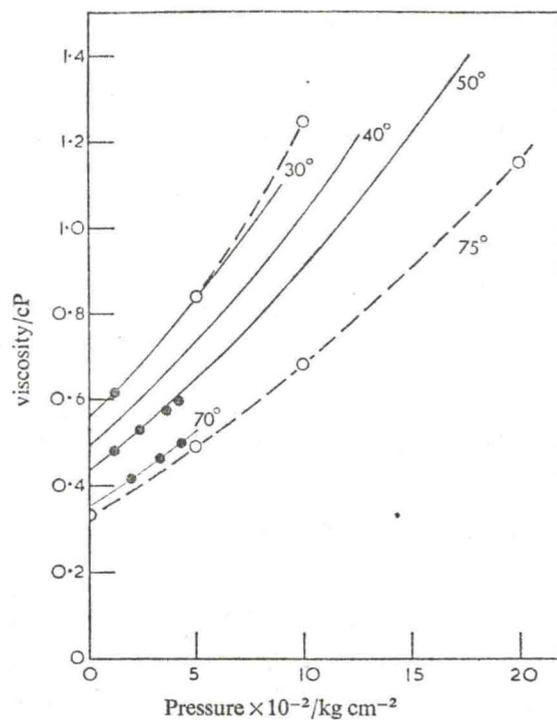


FIG. 6.—The viscosity of benzene. ●, Jobling and Lawrence; ○, Bridgman⁷; —, this work.

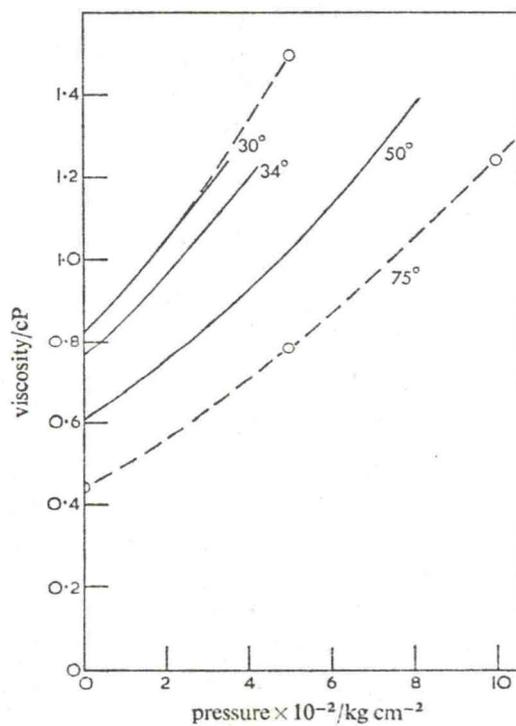


FIG. 7.—The viscosity of cyclohexane. ○, Bridgman⁷; —, this work.

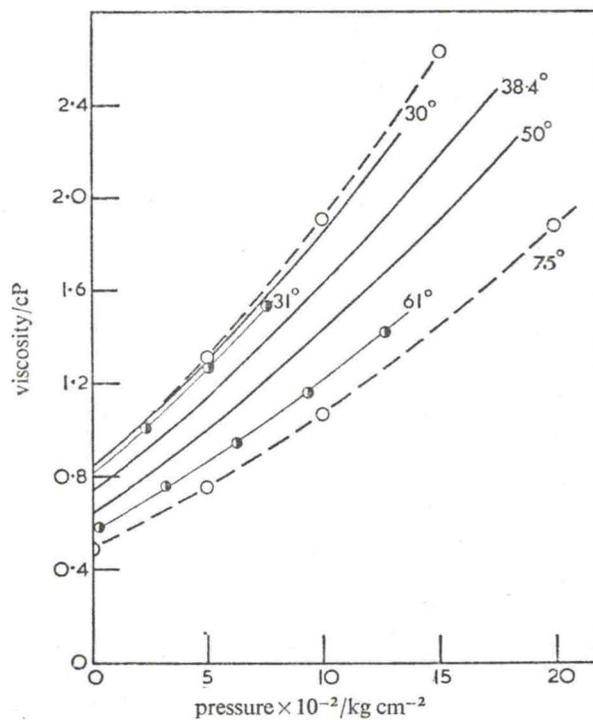


Fig. 8.—The viscosity of carbon tetrachloride. ●, Van Wijk *et al.*; ○, Bridgman⁷; —, this work.

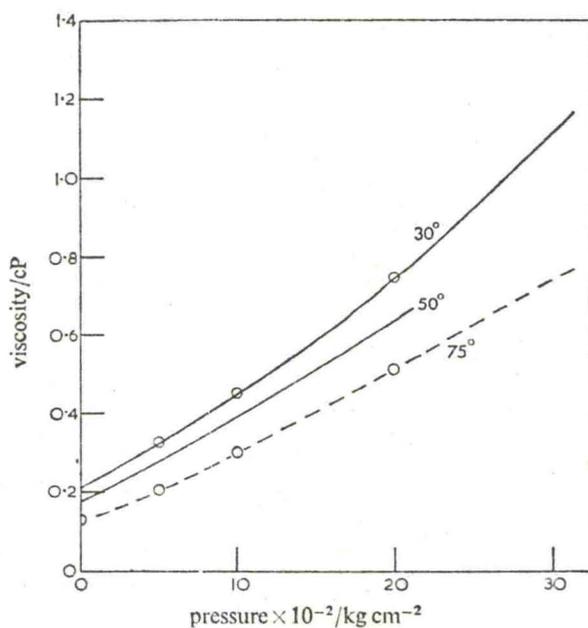


Fig. 9.—The viscosity of isopentane. ○, Bridgman⁷; —, this work.

falling-weight viscometer data,^{7, 10-12} are made in fig. 6-10. Most of the existing viscosity data in liquids under pressure were obtained by Bridgman, whose results at 30°C for benzene, cyclohexane and carbon tetrachloride agree well with the present data in the lower pressure range but are 2-4 % higher at the upper limit. The highest pressures for which Bridgman reported the viscosities of these liquids at 30°C are in excess of the published melting pressures^{7, 13} and are probably extrapolated.

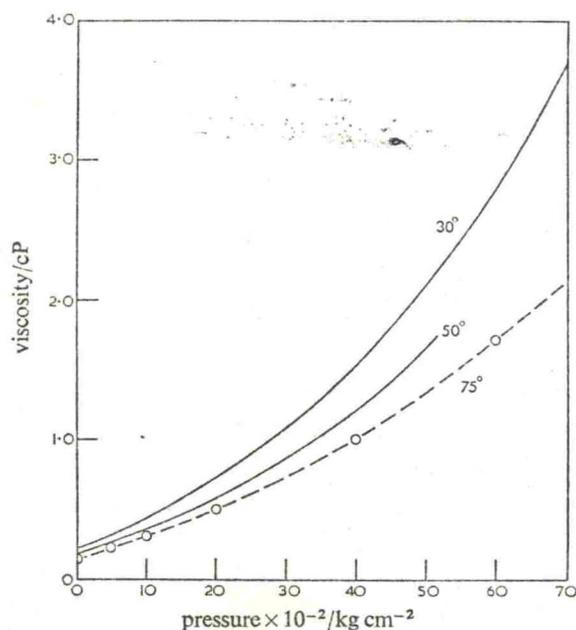


Fig. 10.—The viscosity of n-pentane. O, Bridgman; —, this work. (Data of Bridgman⁷ and Capi and Bett¹² at 30°C are given in table 3, but are not shown in this figure.)

The present data appear to be in better agreement with those of Jobling and Lawrence for benzene¹⁰ and Van Wijk *et al.* for carbon tetrachloride¹¹ than are the results of Bridgman,⁷ and the overall consistency of $(\partial\eta/\partial p)_T$ at different temperatures is good. Good agreement was observed between the present data and those of Bridgman⁷ for isopentane at 30°C. In table 3, viscosities of n-pentane at pressures up to 7 000 kg cm⁻² from this work have been compared with measurements by Bridgman⁷

TABLE 3.—COMPARISON OF PRESENT AND LITERATURE VALUES^{7, 12} FOR THE RELATIVE VISCOSITY OF n-PENTANE AS A FUNCTION OF PRESSURE

pressure kg cm ⁻²	T/°C	this work		ratio η_p/η_1		Cappi and Bett ¹² 30
		30	50	30 Bridgman ⁷	75	
1	1.000	1.000	1.000	1.000	1.000	1.000
500	1.544	1.536	1.517	1.594	1.560	1.560
1000	2.092	2.073	2.065	2.246	2.128	2.128
2000	3.372	3.229	3.432	3.705	3.428	3.428
3000	5.001	4.737			5.041	5.041
4000	7.047	6.682	7.031	7.330	7.115	7.115
5000	9.674	9.196			9.741	9.741
6000	12.95		12.94	12.50	13.06	13.06
7000	17.12				17.31	17.31

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and Cappi and Bett.¹² The present n-pentane data at 30°C are bracketed by the results from these earlier investigations^{7, 12} each of which had a reported accuracy of $\pm 1\%$.

High pressure viscosity measurements were made on benzene at 30 and 50°C and on cyclohexane and carbon tetrachloride at 50°C using an unpolished quartz crystal with a fundamental frequency of approximately 30 kHz. Because of the rougher surface of this crystal, the value of K_1 was affected more by the change in kinematic viscosity of the medium than for polished crystals. Although these results showed slightly more experimental scatter, the smoothed values agreed to $\pm 0.5\%$ with the polished crystal data.

At present the theory of the viscosity of liquids has not developed sufficiently to enable meaningful comparisons between theory and experiment to be made for molecules as complex as those presented here. It is, however, of interest to compare briefly the results expected on the Enskog theory. According to Enskog¹⁴ the viscosity of a dense fluid η can be written in terms of the value of the corresponding dilute fluid η_0 at the same temperature by the equation:

$$\frac{\eta V}{\eta_0 b_0} = \frac{1}{y} + 0.8 + 0.76y, \quad (7)$$

where $b_0 = 2\pi N\sigma^3/3$ with σ the molecular diameter and $y = (pV/NkT) - 1$. It follows that the relative change in viscosity as a function of pressure is

$$\frac{\eta_p}{\eta_1} = \frac{V_1 y_1 (1 + 0.8y_p + 0.76y_p^2)}{V_p y_p (1 + 0.8y_1 + 0.76y_1^2)}. \quad (8)$$

To obtain some relevance of hard-sphere theory to experiment for real systems Michels and Gibson¹⁵ suggested replacing the pressure by the kinetic pressure $T(\partial p/\partial T)_v$, so that $y = V/R(\partial p/\partial T)_v^{-1}$. If this is done for the present results, table 4 shows poor agreement with experiment even for the simplest member of the present series CCl_4 . The results for n-pentane, e.g., show that as the pressure increases the discrepancy becomes worse, being approximately a factor of 5 at 7000 bar. No other

TABLE 4.—COMPARISON OF CALCULATED AND EXPERIMENTAL VALUES OF η_p/η_1 FOR CCl_4 AT 30°C

P (bar)	1	250	500	750	1000	1250
η_p/η_1 expt.	1	1.250	1.530	1.842	2.186	2.563
η_p/η_1 calc.	1	1.037	1.075	1.108	1.179	1.269

theory without a number of adjustable parameters at present seems capable of producing the steep pressure dependence of viscosity that actually occurs for even moderately complex organic liquids such as are reported here.

Thanks are due to Prof. Ubbelohde for his interest in this work. One of us (A. F. C.) thanks the National Coal Board for the award of a maintenance grant.

¹ W. P. Mason, *Trans. Amer. Soc. Mech. Eng.*, 1947, **69**, 359; *Piezoelectric Crystals and Their Application to Ultrasonics* (Van Nostrand, Princeton, N.J., 1950).

² P. E. Rouse, E. D. Bailey and J. A. Minkin, *A.P.I. Symp. Anal. Research* (Cleveland, Ohio, 1950).

³ A. J. Barlow, G. Harrison, J. Richter, H. Seguin and J. Lamb, *Lab. Practice*, 1961, **10**, 786.

⁴ J. Timmermans, *Physico-Chemical Constants of Pure Organic Compounds* (Elsevier, New York, 1950).

⁵ *Amer. Petr. Inst. Proj.* no. 44 (Carnegie Institute of Technology, Pittsburgh, 1950).

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RELATIVE VISCOSITY

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.041
.115
.741
.06
.31

- ⁶ A. DeBock, W. Grevendock and H. Awouters, *Physica*, 1967, **34**, 49.
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