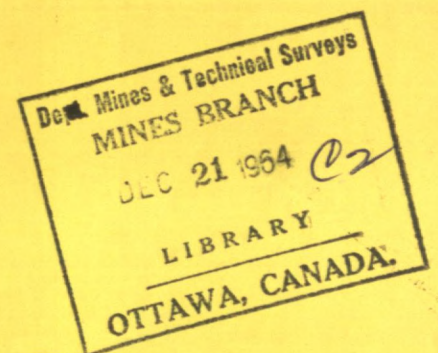




CANADA

THE VISCOSITY OF PURE LIQUID
ZINC, DETERMINED BY
OSCILLATING A CYLINDRICAL
VESSEL



H. R. THRESH

DEPARTMENT OF MINES AND
TECHNICAL SURVEYS, OTTAWA

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THE VISCOSITY OF PURE LIQUID ZINC,
DETERMINED BY OSCILLATING A
CYLINDRICAL VESSEL

by

H. R. Thresh*

ABSTRACT

An oscillational viscometer has been constructed to measure the viscosity of liquid metals and alloys to 800°C. An enclosed cylindrical interface surrounds the molten sample, avoiding the free surface condition found in many previous measurements. Standardization of the apparatus with mercury has verified the use of Roscoe's formula in the calculation of the viscosity. Operation of the apparatus at higher temperatures was also checked, using molten lead. Extensive measurements on five different samples of zinc of not less than 99.99% purity indicate that (i) impurities at this level do not influence the viscosity, and (ii) the apparatus is capable of giving reproducible data. The variation of the viscosity, η , with absolute temperature, T is adequately expressed by Andrade's exponential relationship $\eta V^{\frac{1}{3}} = Ae^{\frac{C}{VT}}$, where A and C are constants and V is the specific volume of the liquid. The values of A and C are given as 2.485×10^{-3} and 20.78, 2.444×10^{-3} and 88.79, 2.169×10^{-3} and 239.8, respectively, for mercury, lead, and zinc. The error of measurement is assessed to be about 1%. No prefreezing phenomena were found in the vicinity of the freezing point of the zinc samples.

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Direction des mines

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DÉTERMINATION DE LA VISCOSITÉ DU ZINC LIQUIDE PUR,
EN FAISANT OSCILLER UN RÉCIPIENT CYLINDRIQUE

par

H. R. Thresh*

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RÉSUMÉ

On a mis au point un viscomètre oscillant destiné à mesurer la viscosité de métaux et d'alliages liquides à des températures allant jusqu'à 800°C. Une interface cylindrique fermée entoure l'échantillon fondu, ce qui permet d'éviter l'état de surface libre qu'on a constaté dans bien des mesures précédentes. L'étalonnage de l'appareil à l'aide du mercure a permis de vérifier que la formule de Roscoe s'applique au calcul de la viscosité. On a aussi vérifié, à l'aide de plomb fondu, le fonctionnement de l'appareil à de plus hautes températures. De nombreuses mesures faites sur 5 échantillons différents de zinc à teneur d'au moins 99.99 p. 100, il ressort (i) qu'à une si haute teneur, les impuretés n'influent pas sur la viscosité, et (ii) que l'appareil peut fournir des données reproductibles. La variation du degré de viscosité, η , par rapport à la température absolue T , est assez bien représentée par le rapport exponentiel d'Andrade $\eta V^{1/3} = A e^{C/VT}$, où A et C sont des constantes et V est le volume spécifique du liquide. Pour le mercure, le plomb et le zinc, les valeurs données de A et de C sont, respectivement, 2.485×10^{-3} et 20.78 , 2.444×10^{-3} et 88.79 , 2.169×10^{-3} et 239.8 . On évalue l'erreur de mesure à environ 1 p. 100. Aux températures voisines de celle de la solidification des échantillons de zinc, on n'a pas constaté de phénomènes antérieurs à la solidification.

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INTRODUCTION

As part of an overall programme of research on various phases of the melting and casting of non-ferrous alloys, a systematic study of some physical properties of liquid metals and their alloys was undertaken in the Physical Metallurgy Division of the Mines Branch at Ottawa(1, 2, 3). The most recent phase of this work, on zinc and some zinc-base alloys, was carried out during 1961-64 in cooperation with the Canadian Zinc and Lead Research Committee and the International Lead-Zinc Research Organization. One of the properties investigated was viscosity, and the present paper gives the results on pure zinc; the second part, on the viscosity of some zinc alloys, will be reported separately.

Experimental interest in the viscosity of liquid metals has virtually been confined to the past forty years. At the beginning of that period, the capillary technique was already established as the primary method for determining the viscosity of fluids in the vicinity of room temperature; all relevant experimental corrections were known and an absolute accuracy of 1-2% was possible. However, the application of the capillary method to liquid metals creates a number of exacting requirements to manipulate a smooth flow of highly reactive liquid through a fine-bore tube. Consequently, the degree of precision usually achieved in the high-temperature field rarely compares with that of measurements on aqueous fluids near room temperature. The full potential of the capillary method has yet to be explored using modern experimental techniques.

As an alternative to the capillary method, many investigators in this field have preferred to select the oscillational method. Unfortunately, the practical advantages are partly offset by the inability of the hydrodynamic theory to supply a rational working formula for the calculation of the viscosity. In attempting to overcome this restriction, many investigators have employed calibrational procedures, even to the extent of selecting an arbitrary formula for use with a given shape of interface. However, where calibration cannot be founded on well established techniques, the contribution of such experiments to the general field of viscometry is questionable.

A critical appraisal of the viscosity data existing for pure liquid metals reveals a somewhat discordant situation, in which considerable effort is still required to establish reproducible and reliable values for the low-melting-point metals. The means of rectifying this situation have gradually evolved in recent years. Here, the theory of the oscillational

method has undergone major advances for both the spherical and cylindrical interfaces. The basic concepts of Verschaffelt⁽⁴⁾ governing the oscillation of a solid sphere in an infinite liquid have been adequately expressed by Andrade and his co-workers^(5, 6). Employing a hollow spherical container and a formula that had been extensively verified by experiments on water, absolute measurements on the liquid alkali metals were obtained. The extension of this approach to the more common liquid metals has been demonstrated by Culpin⁽⁷⁾ and Rothwell⁽⁸⁾, by whom much ingenuity was used to surmount the problem of loading the sample into the delicate sphere.

Because of the demanding technique required in constructing a hollow sphere, the cylindrical interface holds recognition as virtually the ideal shape. On the other hand, loss of symmetry in one plane increases the complexity of deriving a calculation of the viscosity. The contributions of Hopkins and Toye⁽⁹⁾ and Roscoe⁽¹⁰⁾ have markedly improved the potential use of the cylindrical interface in liquid-metal viscometry. The relatively simple experiments of Roscoe and Bainbridge⁽¹¹⁾ on water demonstrate in a convincing manner how the oscillational method can equal the precision of 0.1% that was achieved with an elaborate capillary technique (probably the ultimate in design) by Swindells, Coe and Godfrey⁽¹²⁾.

In view of the above advantages of the oscillational method, the present work has concentrated on this approach. This report describes the design and assembly of an oscillational viscometer, suitable for liquid metals, and Roscoe's new presentation is utilized for the calculation of the viscosity by an absolute approach. Verification of this method by measurements on mercury is then followed by extensive measurements on pure zinc.

THEORETICAL AND DESIGN CONSIDERATIONS

Several mathematical treatments have been presented to relate the liquid properties, such as viscosity η and density ρ , to the linear and dynamic parameters of a cylindrical vessel containing liquid oscillating as part of an inertia system⁽³⁾. After a study of the many approximations adopted by research workers to realize a working formula, and of the form of these calculations with regard to experimental design, the following two calculations were examined in detail and the calculation of Roscoe was finally selected.

1. By Toye and Jones

If R is the radius of the vessel; H, the height of fluid contained in the crucible; I, the inertia of the oscillating system; δ , the logarithmic decrement; T, the time period of oscillation with the crucible full of liquid; and T_0 , the time period of oscillation with the crucible full of a hypothetical liquid not possessing viscous behaviour; then the viscosity equation is given by Toye and Jones⁽¹³⁾ as

$$\left[2\pi HR^2 - K(\mu) \right] \eta - \pi HR^2 \eta^{\frac{1}{2}} \mu \Phi(\mu) \eta^{\frac{1}{2}} + \frac{I\delta}{2T} \left(\frac{T^2}{T_0^2} + 1 \right) = 0 \quad \dots (1)$$

$$\text{and } \mu = (\pi R^2 \rho / T \eta)^{\frac{1}{2}},$$

which is the quadratic form of Hopkin and Toye's equation⁽⁹⁾. The computation of the factors involving Bessel functions has been carried out by Thomas, and the variation of the functions $\Phi(\mu)$ and $K(\mu)$ with μ are tabulated as an Appendix to the paper by Toye and Jones⁽¹³⁾. A repeated approximation method, involving the comparison of an estimated value of η with the calculated value from Equation 1 until η estimated - η calculated = 0, makes possible the derivation of the viscosity.

Two features of Equation 1 justify some elaboration. First, this equation has never been extensively verified against a reputable liquid metal standard such as mercury, and, secondly, the considerable number of measurements involving the use of this equation have centred around the use of a cylindrical container in which the liquid oscillates with a free surface. The uncertainty of meniscus effects, which have been discussed elsewhere⁽³⁾, should be avoided to remove uncertainties in H.

In theory, the properties of a unifilar suspension permit the quantity T_0 to be defined as the time period of the empty crucible, but there is experimental evidence that the torsional constant of the wire may be significantly changed by the addition of the mass of the fluid sample. The calculation of T_0 by the method of Andrade and Dobbs⁽⁶⁾, by equating the imaginary parts of the damping factor, is more exact. Evaluation of T_0 for Hopkins and Toye's calculation is no doubt possible by this method, but the complexity of the expression prevents this from being a practical procedure.

2. By Roscoe

By manipulating the basic equations of Andrade and Chiong⁽⁵⁾ and defining the damping factor L in its real and imaginary parts, Roscoe⁽¹⁰⁾ has derived expressions for η in the case of the sphere and cylinder where T_0 is absent. For the cylinder, this is given by:

$$\eta = \left\{ \frac{I \delta}{\pi R^3 H Z} \right\}^2 \frac{1}{\pi \rho T}, \quad \dots (2)$$

$$\text{where } Z = \left(1 + \frac{1}{4} \frac{R}{H} \right) a_0 - \left(\frac{3}{2} + \frac{4}{\pi} \frac{R}{H} \right) \frac{1}{P} + \left(\frac{3}{8} + \frac{9}{4} \frac{R}{H} \right) \frac{a_2}{2P^2},$$

$$P = \left(\frac{\pi \rho}{\eta T} \right)^{\frac{1}{2}} R,$$

$$a_0 = 1 - \frac{1}{2} \Delta - \frac{3}{8} \Delta^2, \quad a_2 = 1 + \frac{1}{2} \Delta + \frac{1}{8} \Delta^2, \quad \text{and}$$

$$\Delta = \frac{\delta}{2\pi}.$$

Convergence of the term Z is such that only the initial three terms of the series are required in order to obtain precision in the fourth figure of η . An assumed value of η permits the computation of η to proceed around the loop $\eta \rightarrow P \rightarrow Z \rightarrow \eta$ until the inserted value agrees with the final value. Where the estimated value is in error by 30%, five to six iterations will be required to calculate η to the required accuracy, i. e. usually to the fourth figure. The intention of using the fully closed cylinder to avoid errors in H both from meniscus effects and from density inaccuracies means that the present calculation must be modified in a simple way to represent two open, liquid-filled crucibles, one of which is inverted upon the other. This means that the expression for the decrement δ must be multiplied by 2 or that the denominator of Equation 2 must be divided by 4, H being the mid height of the closed vessel.

In designing the suspension system, Equation 2 should be considered in conjunction with the relationship governing the motion of the type of suspension adopted. From previous experience, the bifilar torsion system was chosen in the present design. The time period of oscillation, T, for the undamped bifilar system is given to a good approximation by:

$$T = 4\pi \left(\frac{I l}{W g e^2} \right)^{\frac{1}{2}}, \quad \dots (3)$$

where l is the length and e the distance of separation of the two wires, and the quantities I and W are respectively the inertia and the mass of the vibrating system. On examining these equations, the choice of R, H, e and l must be so made as to realize preselected values of δ and T. For δ , a value in the order of 0.03 gives a decrement that is sufficiently large to give a reasonable number of oscillations to permit an accurate determination of this quantity but is not too large to give rise to aperiodic damping.

A practical figure for T may be taken in the range 10 to 14 seconds. Hence, dimensions are assigned to R , H and considered together with known values of η and ρ . The estimated inertia now follows from Equation 2, which allows e and l to be adjusted to give the required value of T , where W has previously been assessed.

GENERAL DESCRIPTION OF THE APPARATUS

A general plan of the apparatus, showing the most important features, is given in Figure 1, where for clarity the upper part of the suspension is shown rotated through a right angle between F and H . Functionally, two main zones are apparent in the design. The upper portion consists of a brass chamber enclosing the complete torsion system from its apex to the point where the crucible connecting rod, S , enters the furnace zone. The furnace and its attachments constitute the lower zone and provide the basic thermal and vacuum services for maintaining a metallic liquid at a given temperature within an inert environment.

The unit design of the top assembly permits this section to be raised along the vertical axis of the apparatus and then be locked in a given position by the support collar, F . By raising the viscometer head to the uppermost position, the crucible is withdrawn from the furnace chamber. In this way the suspension, some 82 cm in length, is always under tension, even during the exchange of samples between runs, and this reduces the possibility that the properties of the wire might be altered as a result of mishandling.

The suspension head is supported by the tube H at the apex of the apparatus. Here, the composite brass cone and table A , together with the socket B , provides a high-vacuum joint. This permits a true oscillational motion to be imparted to the suspension by moving the levelled cone through a small angle. The 0.010-cm-diameter tungsten suspension wires, E , are located in the cylindrical housing, C , in such a way that complete insulation of the wires through the head permits the suspension to serve the further purpose of a conductor outlet for a chromel-alumel thermocouple located in the top of the graphite vessel. The lower bifilar suspension point takes the conventional form of a grooved wheel located in the lower part of the suspended system. Preliminary decrement tests on the internal friction properties of the wire indicated that the tungsten wire was satisfactory as supplied by the manufacturer.

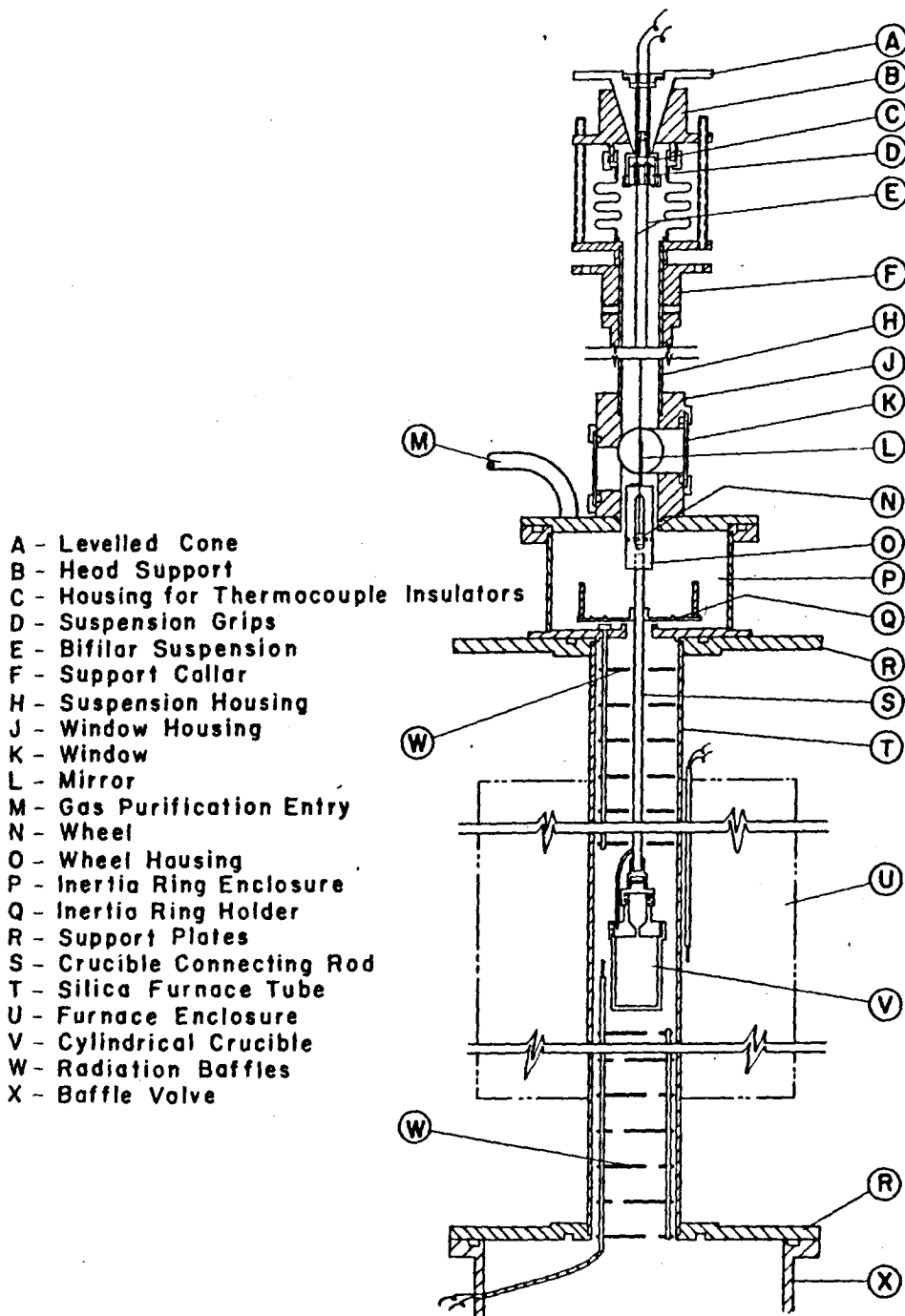


Figure 1. The oscillating viscometer employing a cylindrical vessel, as used at the Mines Branch, Ottawa.

The brass window block, J, is integral with the circular cover of the inertia chamber, P. Two vertical windows, K, set at right angles to each other, permit two optical paths to be established to the plane mirror, L. Within the inertia chamber, a small cylindrical block of aluminum, O, serves as the suspension wheel housing. In order to determine the inertia of the suspension system, a brass ring holder, Q, is supported on the stainless steel tube, S. Three carefully machined circular stops, integral with the holder, make it possible to locate one of three copper inertia rings, concentric with the axis of the apparatus. Hence, a measure of the inertia of the whole system is derived by determining the respective time period of oscillation associated with the corresponding ring of known inertia.

The lower zone of the apparatus is constructed around the furnace enclosure, U. A silica furnace tube, T, 84 cm in length and 6 cm internal diameter, is ground to bring the end faces square to its longitudinal axis. Two brass end plates, R, support the tube vertically and provide good vacuum joints with the aid of flat rubber gaskets. A pair of semi-tubular electrical elements mounted in the furnace shell maintain the metal in the liquid state. The temperature is regulated by a Celectra controller, operating in conjunction with a suitably placed thermocouple. A control of better than $\pm 0.25^\circ\text{C}$, as determined by the primary thermocouples, is always realized with this arrangement.

The graphite vessel, V, is suspended axially in the furnace tube from the rod, S. The primary thermocouple of 0.010-cm-diameter chromel-alumel wire is passed down this tube and terminates in the cap of the vessel just above the liquid surface. A dense, nuclear-grade graphite is used to machine the cylindrical container; the inner cylindrical shape is held to a tolerance of 0.003 cm on the height and radius. The design of the detachable lid incorporates an expansion chamber to hold the excess liquid produced by the variation of liquid density with temperature. Participation of this liquid in the measured decrement is restrained by the use of a thin graphite baffle, while metal evaporation from the surface to the exterior of the crucible is controlled by limiting the exhaust hole in the cap to 0.075-cm diameter. As the viscosity of a liquid metal is very temperature-dependent, the furnace zone ambient to the crucible must be free from excessive thermal gradients. Reduction of these gradients to a practical minimum is facilitated by (a) the use of long heating elements, 45 cm, as compared with the diameter of the heated zone; (b) loading the interior zone, above and below the liquid metal container, with stacks of polished stainless steel radiation shields; and (c) countering the heat end-losses by the use of small auxiliary windings secured to the outside of the silica furnace tube. As a precautionary measure against the failure of the rather fragile thermocouple located in the crucible lid, a trio of reserve thermocouples is introduced through and supported around the periphery of the lower set of radiation shields.

Each couple is sealed into a thin silica sheath to prevent possible contamination from metal vapours, or spurious readings due to reducing conditions prevailing in the furnace atmosphere. In this way, the hot junctions survey the vertical zone occupied by the crucible. As a result of such surveys, a calibration is now available to give the power requirements of the auxiliary windings to produce a zone of temperature to within one degree Celsius of the experimental temperature, over a wide range of working temperatures. Further evidence for the lack of temperature fluctuations within this zone is obtained by removing the crucible and the support rod and inserting a train of ten thermocouples, spaced at 2.5-cm intervals, along the axis of the apparatus.

During experiments, this type of thermal calibration can be verified by employing a stepwise heating cycle through the melting point of a pure metal. If the thermal step is restricted to one degree Celsius for an equilibration period of 4 hours, the onset of melting is easily detected by the change in time period of the oscillating system. This follows from Equation 3, and is due to the loss of inertia of the solid sample.

Normal vacuum techniques permit the internal pressure of the apparatus to be reduced to better than 5×10^{-6} mm Hg. Protective atmospheres of helium or hydrogen have been employed to prevent contamination of the liquid metal. Purification is effected by passing either gas through a liquid nitrogen trap to remove water vapour, followed by treatment over hot uranium chips at 700°C. Samples protected by gases purified in this manner have, at the most, shown only traces of surface films after being held molten for over 200 hours.

THE MEASUREMENT OF THE CONSTANTS I, R AND H AND THE EXPERIMENTAL VARIABLES δ AND T

In attempting to measure the viscosity to an accuracy of about 1 part per 100, the factors governing Equation 2 must be determined to better than 1 part per 1000.

The Constants of the Suspension System

These quantities are essentially fixed at a given temperature, being subject to slight variations due to the expansion of the graphite crucible. In the case of the inertia, this correction can be neglected. The value of 2×10^{-6} was taken for the coefficient of linear expansion of the graphite, to permit the small correction of R and H.

1. The Inertia (I) of the System

In designing the apparatus, the measurement of the inertia was made possible by providing a large known inertia on the suspension system. If this known inertia is varied, observing the corresponding variation in the time period of the system oscillating with an empty crucible, the unknown inertia may be calculated from Equation 3, in which the inertia varies as the square of the time period.

Three accurately machined inertia rings were made from an annealed wrought-copper tube. Accurate surveys of their respective dimensions permitted the calculation of the corresponding inertias, 4,220.6, 2,525.5, and 1,091.4 g cm², where the mass of each ring differed by less than 2 milligrams in a total mass of about 300 grams. Each ring was placed in turn on the plate holder and the corresponding time period was measured to the nearest millisecond. This accuracy was ensured by restricting the angular swing to within 0.02 of a radian over a count of 300 swings. All measurements were carried out under high vacuum. As the inertia of each ring was estimated to be known to 1 part in 20,000, the suspended part of the system was found to give an inertia of 1,688.6 \pm 1.0 g cm².

Roscoe⁽¹¹⁾ has shown that, by employing only one ring, two sets of decrement measurements may be carried out on a given liquid at constant temperature, where the ring is removed for the second determination. If the inertia of the vibrating system is I_0 and the inertia of the ring is I_r , and by experiment I_0 gives a value of δ and T , and $I_r + I_0$ gives δ_1 and T_1 ; then I_0 can be derived from Equation 2, giving:

$$\frac{I_0 \delta}{(I_r + I_0) \delta_1} = \left(\frac{T}{T_1} \right)^{\frac{1}{2}} \frac{f(\delta, P)}{f(\delta_1, P_1)} \quad \dots (4)$$

This technique was carried out by employing mercury at 24°C under high vacuum. The appropriate values of δ , T , and ρ are recorded in Table 1. From Equation 4, the fixed inertia of the system was found to be 1,690.6 g cm². Thus, it appears that there is little to choose in the accuracy of these two methods where the total inertia of system is known, during the present experiments, to be better than 1 part in 5,000, using the large ring.

TABLE 1

Determination of the Inertia

	Logarithmic Decrement	Time Period, T (seconds)	Density, ρ (g/cm ³)
Mercury, no ring	0.072461	6.965	13.534
Mercury, with ring	0.026200	11.480	13.534
Fixed Inertia			1,690.6 g cm ²

2. Determination of the Radius (R) and the Height (H)

The use of high-quality graphite permitted the fabrication of the cylindrical vessel to a high order of accuracy, eliminating many of the problems encountered with glass and ceramics. The height, H, was readily determined, by the use of a micrometer depth gauge, to be 5.0841 \pm 0.0003 cm. The radius R was checked at various positions along the vessel by a telescopic gauge, giving a mean value of 1.5002 \pm 0.0002 cm, which compared favourably with a computed value of 1.5001 \pm 0.0001 cm, obtained from just filling the vessel with mercury at 24°C.

The Experimental Variables δ and T

1. The Logarithmic Decrement (δ)

The smaller the decrement, for a given initial amplitude, the larger is the number of oscillations that are practically significant. In the present work, the decay of the system was computed over 50 oscillations from a maximum amplitude of 45 cm on the scale. The consecutive amplitudes of oscillation were recorded by the photographic method. A fine slit of light was projected through one of the windows, K, and reflected, through 90° by way of the plane mirror and second window, to the "engine-engraved" perspex scale curved to a radius of one metre. By placing the centre of curvature of the 60-cm scale at the axis of oscillation, the (tan θ - θ) correction, which must be applied on using a straight scale, may be avoided in calculating the decrement. A recording on strip film retained an image of the scale, together with a series of fine lines denoting the extremities of each amplitude. This procedure allows the position of an amplitude to be determined to better than 0.2 mm. The amplitude measurements were also attempted by means of a telescope. The accuracy of the decrement was found to be of the same order in both cases, but the

telescope technique proved a demanding task, even with a time period of 12 seconds.

The decrement was computed from these readings by using the formula suggested by Andrade and Dobbs⁽⁶⁾, by which

$$\delta = \frac{1}{25} \log_e \left\{ \frac{\sum_1^{25} a_n}{\sum_{26}^{50} a_n} \right\} \quad \dots (5)$$

Here, a_n represents the distance of a complete swing over the scale. To facilitate a rapid check of the systems operation during the progress of a given run, a less accurate, but simplified, formula,

$$\delta = \frac{1}{50} \frac{\log_e a_1}{\log_e a_{51}}, \quad \dots (6)$$

was used to compute δ to within $\pm 0.15\%$ of that given by Equation 5.

Consecutive recordings were compared, at a given temperature, for their difference in decrement. This was found to be 1 part in 800 for the highest error. However, duplicate recordings were not made a routine feature of experiment, but regarded as a confirmatory measure of the normal functioning of the suspension, to be verified at suitable intervals.

The decrement determined at this stage comprises the damping due to the liquid, together with a small residual drag, arising from physical conditions, which cannot be completely prevented from influencing the suspended system. These corrections are well known, even from the work of Verschaffelt, and may be considered as (i) internal friction of the suspension wire, (ii) electromagnetic effects arising from the furnace current, and (iii) the effect of a protective gas atmosphere. Where possible, the course of experiment has been selected to contain the accumulative level of these corrections to the minimum value.

(i) Internal Friction of the Suspension Wire. The residual wire decrement can never be reduced to an insignificant value, and a correction of several per cent of the liquid damping is commonly realized in this type of measurement. The high tensile strength of tungsten wire permits the use of a smaller-diameter wire, which in turn reduces the value of the internal friction. Although heat treatment of the wire has been found beneficial in lowering the residual decrement^(6, 8), wire taken from the spool gives a consistent residual damping of 150 to 250 $\times 10^{-6}$ between runs, that is, less than one per cent of the liquid damping. The variation within a given run is at least an order of magnitude lower than the variation between runs, as measured by the wire decrement taken over several hundred oscillations before melting and after freezing of the sample.

(ii) Electromagnetic Effects Arising from the Furnace Current.

Electromagnetic damping has been reported in the literature⁽⁴⁾, particularly where low-voltage, high-current electrical heaters have been employed. In the present work, commercial non-inductive type heaters were adopted, containing the resistor elements lying parallel⁽⁸⁾ to the length of the furnace zone in contrast to the usual toroidal construction. In practice, high-temperature tests revealed that damping from this phenomenon was completely absent up to 550°C.

(iii) Effect of a Protective Gas Atmosphere. The high vapour pressures of such liquids as mercury and zinc usually necessitate the measurement of the liquid decrement under a protective gas atmosphere. In this way, excessive evaporation and possible contamination of the apparatus are avoided. However, the presence of this gas introduces an extra component of drag in the liquid system. Decrement measurements on liquid lead and tin, in the presence of a hydrogen atmosphere and under vacuum, readily gave estimates of this gas damping decrement for hydrogen and helium. Respectively, they are $(480 - 580) \times 10^{-6}$ and $(950 - 1050) \times 10^{-6}$, to be compared with 1630×10^{-6} for one atmosphere of air at room temperature. The temperature coefficient of this decrement through the range of temperature 240-500°C for tin was very small, being only just distinguishable from the error in the liquid decrement measurement. Further, decrement measurements at temperatures not too far below the liquidus verified that the change of state did not influence the expected value of the gas damping. The advantages of the low atomic weight of hydrogen in minimizing gas damping are very apparent. Initially, the liquid decrements of pure zinc were corrected for the influence of the hydrogen atmosphere, by measuring the gas decrement just below the freezing point before and after the run.

The design of the enclosed crucible lends itself to minimum evaporation rates of the metallic liquid, in view of the small surface area of the expansion chamber and the restricted evacuation orifice in the crucible cap. Subsequent experiments, where the liquid damping was measured under high vacuum, demonstrated that zinc loss could be kept to a tolerable level, provided that during the extensive period of temperature equilibration between recordings the apparatus was under an atmosphere of purified hydrogen. Up to 550°C the loss of zinc is below 1 part in 1000 of the sample weight for some ten decrement measurements. This more direct approach is found to realize an improvement in the scatter of the temperature coefficient of viscosity, an observation which has been verified by Rothwell⁽⁸⁾.

Finally, to justify the requirement that the time varies exponentially with the angular deflection during experiment, two of the largest decrements were analyzed individually by plotting the logarithm of successive amplitude against the corresponding order of occurrence. In both cases the linearity between these quantities was excellent, verifying the application of experiment to the original theoretical assumption.

2. Determination of the Time Period (T)

The use of the time period T in Equation 2 requires that the quantity should be known to within one-hundredth of a second. This is conveniently measured over some 50 oscillations on a high-grade stop-watch. However, the true definition of T, particularly when a bifilar suspension is employed, implies that the crucible should just be full of liquid for an enclosed container. In satisfying this requirement at the melting point, excess liquid is displaced from the vessel into the expansion chamber at higher temperatures, owing to the decrease in liquid density. Experimentally, the prevention of this liquid from contributing to the liquid damping is achieved in a simple manner. From the time period aspect, however, this liquid still contributes to the total mass effect upon the system. Thus, it is more exact to compute a correction of the time period measured in the vicinity of the melting point, to be applied at higher temperature, by considering equation 3. The mass factor is composed of the fixed mass of the suspension system, together with the weight of the metal sample. Liquid metals possess an expansion such that the density decreases about 1% through every hundred Celsius degrees. This means that the extent of such an error occurring in T is progressively raised the larger the temperature survey of the viscosity. Probably the best approach to suppressing this factor lies in the design of the suspended system, where the ratio of the fixed mass to that of the metal sample should be kept as high as practically possible. The present apparatus has required a maximum correction of 0.25% to be applied to T, taking into account the temperature range of the measurements and the density of liquid under investigation.

EXPERIMENTAL PROCEDURE AND RESULTS FOR MERCURY, LEAD, AND ZINC

Mercury

Experimental justification of the fundamental calculation of the viscosity and the design of the apparatus is an essential requirement in the use of a new viscometer. In effect this is a standardization (which is to be distinguished from a calibration) for which the liquid mercury was chosen. The viscosity data of this metal are readily available and free from the discordance shown, for example, by the values for tin. Further, the density is known to a high degree of accuracy.

The graphite vessel was prefired to 900°C under high vacuum and filled to the point where metal was present in the inlet hole of the lid. Towards the end of the filling operation the vessel was pumped down to 1 mm mercury in a glass chamber, to ensure the removal of any air trapped under the lid. The cylinder, containing triple-distilled mercury of 99.98% purity, was attached to the stainless tube and the decrement was measured under high vacuum. After redetermining the damping under an atmosphere of pure hydrogen to give the residual decrement due to gas damping, the liquid decrements were determined at elevated temperatures under steady thermal conditions.

From the experimental data, the viscosity was calculated using Equation 2 and is recorded in Table 2. The density values are selected from the Handbook of Physics and Chemistry*, and appear to be accurate to much better than 1 part in 1000, taking into consideration the agreement with the precision work of Cook and Stone⁽¹⁴⁾ and the observations of Beattie and others⁽¹⁵⁾. In Figure 2, the results in Table 2 are shown and compared with the values quoted by Erk⁽¹⁶⁾. Here, the viscometry of liquid mercury is reviewed, where the majority of measurements have employed the capillary technique. The agreement can be seen to be good to less than 1 part in 100. On this basis, the viscometer was considered suitable for operating at elevated temperatures with the required precision of 1%.

TABLE 2

Viscosity of Mercury

Temperature (°C)	Logarithmic Decrement	Time Period (seconds)	Density (g/cm ³)	Viscosity (poise)
23.8	0.026200	11.470	13.534	0.01532
40.4	0.025483	11.475	13.497	0.01446
59.7	0.024790	11.483	13.449	0.01365
80.0	0.024159	11.490	13.400	0.01295
100.6	0.023681	11.498	13.352	0.01244

* Chemical Rubber Publishing Co., C. D. Hodgman. 34th Edit., 1952, p. 2199.

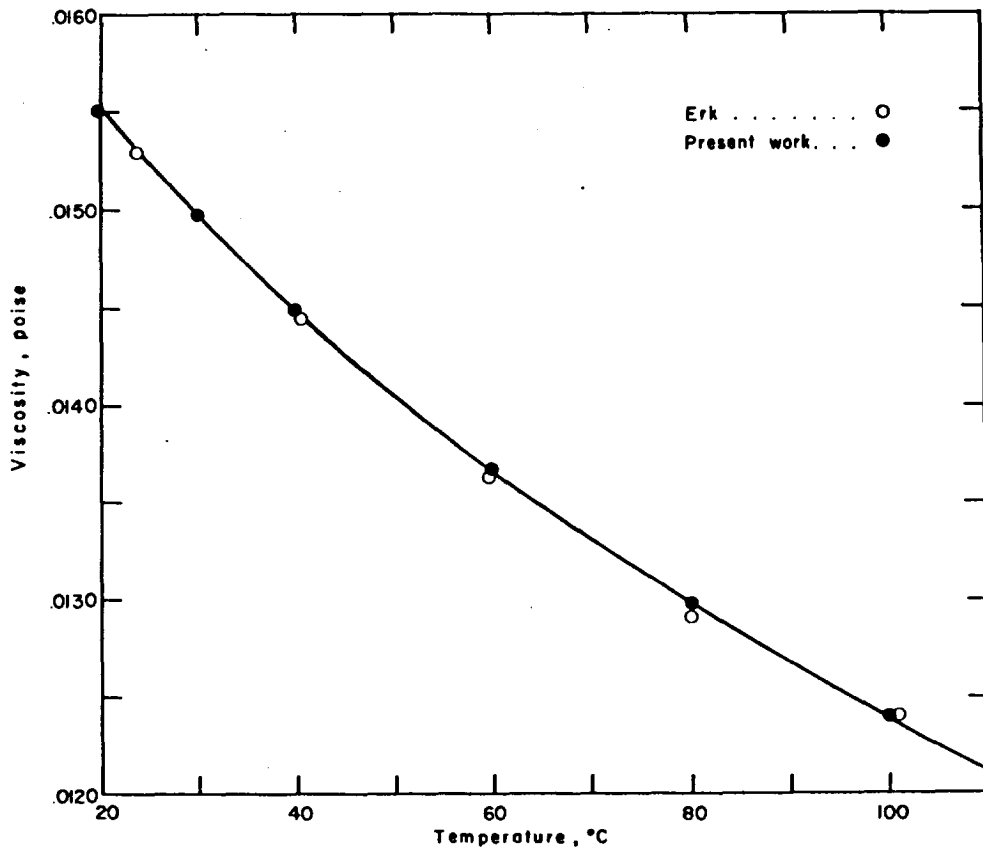


Figure 2. Viscosity of mercury, compared with the values quoted by Erk.

Lead

Prior to attempting measurements on liquid zinc, it was considered desirable to proceed by using a metal that handled well under high vacuum at temperatures where zinc becomes molten. For this purpose, lead of better than 99.99% purity was chosen for the next determination. A quantity of this metal was cast into a cylindrical ingot by melting and casting in graphite containers. The solid charge was machined to the required volume, remembering that the dimensions of the sample must permit the metal to reach the molten state without damaging the graphite crucible, i. e., the necessary allowance for thermal expansion must be made. On assembling the viscometer, the metal was brought to the liquid state under high vacuum. Decrement recordings were obtained at a number of equilibrium temperatures, and the viscosity was calculated as shown in Table 3 and Figure 3. A density value was initially selected from the Liquid Metals Handbook*, to compute the volume of the charge. After a trial run it was apparent that a low density value had given rise to incomplete filling of the vessel. Subsequent use of the container as a pycnometer gave a value of 10.68 g/cm^3 at the freezing point, which may be compared

* U. S. Atomic Energy Comm., R. N. Lyon, 2nd Edit., 1952, p. 40.

with 10.69 g/cm^3 obtained by Fisher and Phillips⁽¹⁾. Originally, the required density values were selected from this latter work. Current work in the author's laboratory has given a value of 11.680 g/cm^3 , which substantiates the recent value of 11.683 g/cm^3 obtained by Strauss, Richards and Brown⁽¹⁷⁾. Consequently, the density values used in Table 3 are taken from measurements now in progress at the Mines Branch.

TABLE 3

Viscosity of Molten Lead

Temperature (°C)	Logarithmic Decrement	Time Period (seconds)	Density (g/cm ³)	Viscosity (poise)
331.0	0.029673	12.058	10.675	0.02586
349.5	0.029005	12.065	10.654	0.02457
368.0	0.028425	12.073	10.632	0.02349
388.0	0.027785	12.083	10.609	0.02233
407.4	0.027191	12.093	10.586	0.02129
447.0	0.026243	12.109	10.540	0.01970

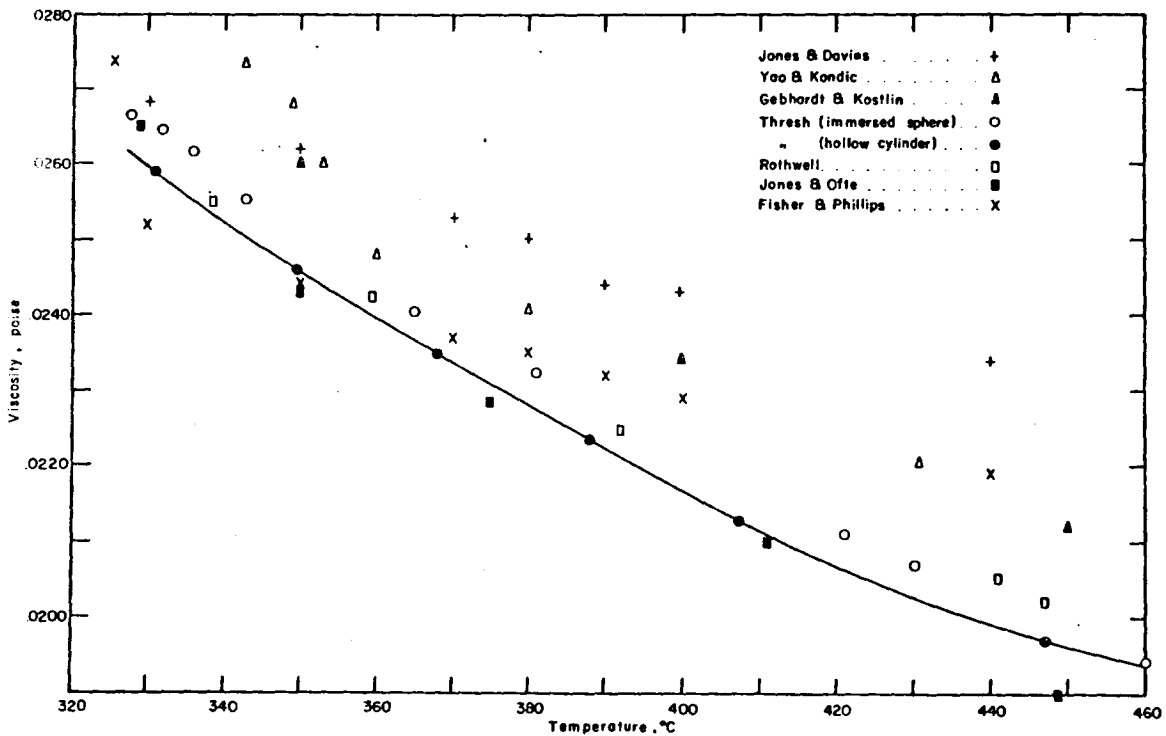


Figure 3. Viscosity values for lead, compared with representative literature data.

Zinc

Extensive measurements were now undertaken on pure zinc. Preparation of the metal followed that outlined for lead, exercising the usual precautions to avoid excessive oxidation of the liquid. Melting in air was not regarded as deleterious, because the dissolved oxygen in the liquid never attains a level where constitution effects influence the measured viscosity. Five samples of zinc of different extraction, with a minimum purity of 99.99%, were loaded into the apparatus in sequence and the corresponding decrement-temperature curves were obtained. The computed viscosity values are reported in Table 4 and are shown graphically in Figure 4. The density of zinc⁽¹⁸⁾ has been derived according to the equation $\rho = 6.955 - 9.183 \times 10^{-4} T$, where T is the temperature within the range 419°C to 520°C.

DISCUSSION OF RESULTS

The preliminary operation of the viscometer, involving the standardization experiments with mercury, clearly demonstrates that absolute measurements are possible by this fundamental approach. The new presentation of the theory of the oscillating cylindrical vessel by Roscoe has been verified and provides an improved approach to liquid-metal viscometry.

The results for lead, though not extensive, are worthy of comparison with previous data obtained for this metal. For completeness, all relevant lead measurements are plotted in Figure 3, where the curve shows the trend of results listed in Table 3. These values originate from techniques employing absolute and calibrational procedures. In view of the many unfavourable aspects of calibration in liquid metal viscometry, discussed elsewhere⁽³⁾, the somewhat discordant nature of the results in Figure 3 assumes a more encouraging aspect if only the absolute measurements are considered. Recently, two absolute techniques have been reported for lead. By using a split-hollow sphere, Rothwell has extended Andrade's method to temperatures in the order of 800°C. Ofte and Wittenberg⁽¹⁹⁾ have applied the open cylindrical oscillating vessel to Roscoe's formula, giving values some 1% to 3% lower than the present data, to be compared with Rothwell's difference of some 1% to 2% with the present work. However, the results of Rothwell may be preferred to those of Ofte and Wittenberg in cases where the use of a free surface in the stainless steel cylindrical vessel produces uncertainty in the shape of the meniscus.

TABLE 4
Viscosity of Liquid Pure Zinc

Sample No.	Temperature (°C)	Logarithmic Decrement	Time Period (seconds)	Density (g/cm ³)	Viscosity (poise)
1	422.5	0.027288	13.020	6.567	0.03930
	432.0	0.026857	13.023	6.558	0.03776
	431.8	0.026890	13.023	6.558	0.03788
	447.3	0.026299	13.026	6.544	0.03586
	481.5	0.025241	13.036	6.513	0.03247
	436.3	0.026684	13.024	6.554	0.03716
	426.6	0.027031	13.021	6.563	0.03837
	420.1	0.027299	13.020	6.569	0.03932
2	419.6	0.027357	13.016	6.570	0.03955
	421.7	0.027268	13.017	6.568	0.03923
	424.5	0.027161	13.018	6.565	0.03885
	430.0	0.026939	13.019	6.560	0.03806
	434.9	0.026773	13.020	6.556	0.03748
	447.2	0.026296	13.024	6.544	0.03586
	464.2	0.025698	13.028	6.529	0.03390
	478.6	0.025217	13.032	6.515	0.03239
	493.4	0.024729	13.038	6.502	0.03089
3	426.3	0.027108	13.024	6.563	0.03865
	444.6	0.026421	13.029	6.547	0.03626
	462.5	0.025827	13.034	6.530	0.03431
	479.5	0.025273	13.038	6.515	0.03256
	437.0	0.026689	13.027	6.554	0.03717
	428.7	0.027016	13.025	6.561	0.03832
	420.1	0.027308	13.022	6.569	0.03935
4	427.4	0.027028	13.014	6.562	0.03839
	439.4	0.026529	13.018	6.551	0.03665
	452.6	0.026093	13.021	6.539	0.03520
	468.0	0.025567	13.025	6.525	0.03349
	480.2	0.025186	13.029	6.514	0.03230
	489.7	0.024886	13.031	6.505	0.03139
	447.3	0.026207	13.020	6.544	0.03556
5	444.8	0.026429	13.012	6.546	0.03635
	468.2	0.025566	13.018	6.525	0.03351
	462.7	0.025648	13.016	6.530	0.03375
	487.2	0.024901	13.021	6.508	0.03144
	510.0	0.024187	13.030	6.487	0.02933
	471.3	0.025386	13.019	6.522	0.03293

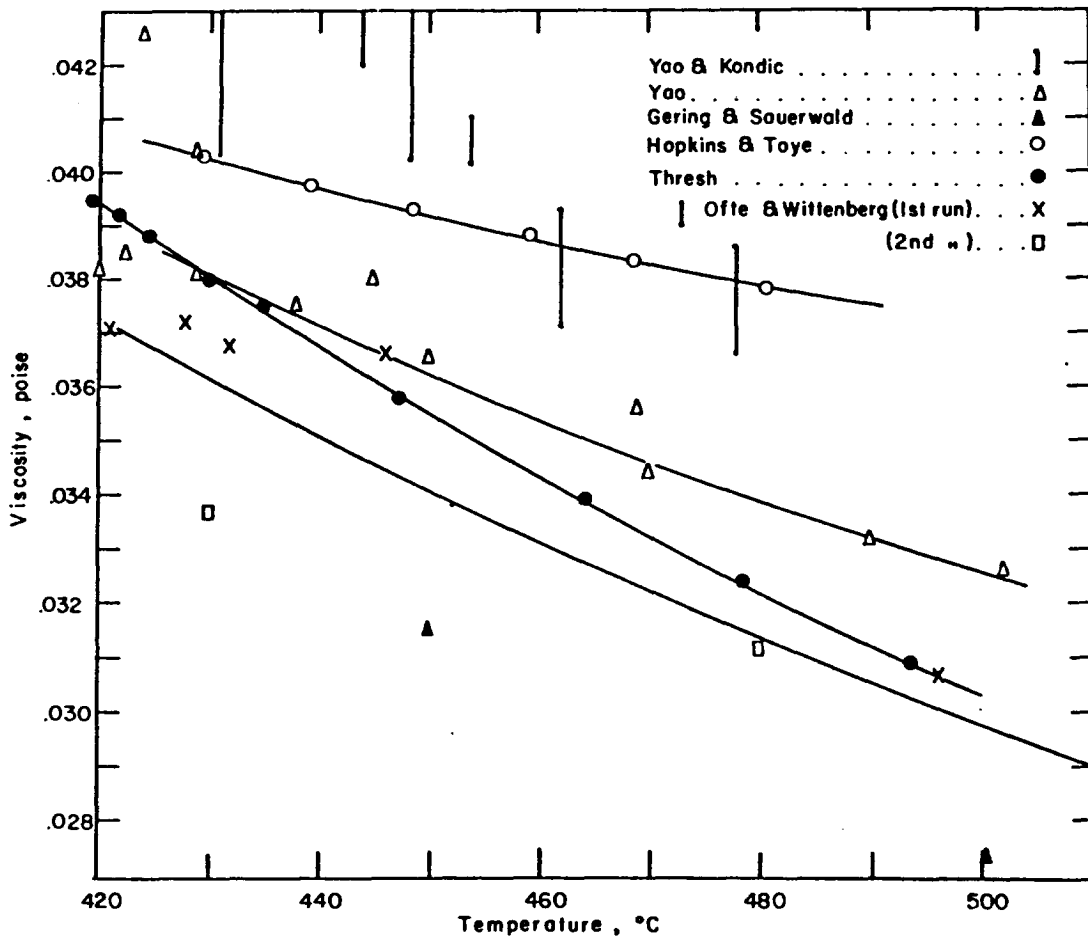


Figure 4. Viscosity varying with temperature for five different samples of pure zinc.

This, in turn, introduces errors into the viscosity value through lack of precision in determining the liquid height. Further evidence in support of the reported data has been obtained from an independent investigation by the present author⁽²⁰⁾, using the analysis of Verschaffelt. Here, an oscillating solid sphere was immersed in liquid lead and manipulated to give absolute data. These observations permit the conclusion to be drawn that operation of the viscometer appears well founded.

On considering zinc, the most important published data appear in Figure 5, where it may be seen that agreement between various workers would appear not to support the contention that the viscosity is a precise physical property, demonstrating a reproducible behaviour for a given liquid. Selective comparison, based on the fundamental approach to experiment, reveals that the measurements of Hopkins and Toye⁽⁹⁾ and the Mound Laboratory⁽²¹⁾, and the present measurements were obtained by using the oscillating cylindrical vessel. The two latter measurements calculated the viscosity from the new formula of Roscoe, in contrast to the calculation derived and employed by Hopkins and Toye. Although Roscoe's formula is more exact, calculation has shown that from the same set of data, agreement to better than 1% in the viscosity is achieved from both methods. Consequently, the differences observed in Figure 5 must originate in experiment. The present results differ by some 5% at 430 °C from those of Hopkins and Toye. A large part of this discrepancy may be accounted for by the fact that Hopkins and Toye used density values by Journiaux and Pascal⁽²²⁾, which give, for example, 6.903 g/cm³ at 430 °C.

By comparison, a value of 6.560 g/cm³ is consistently reproduced at the Mines Branch to within 0.1%, which has been substantiated by other work. If one disregards meniscus effects, Hopkins and Toye's calculation of the height of liquid H in the crucible depends on the density ρ ; thus, H will be too low by about 5%. Some estimate of how this discrepancy influences η may be obtained by considering Equation 2. Here η varies approximately as $\frac{1}{\rho}$ and $\frac{1}{H^2}$. Further, the dependence of Z on ρ is such that a 5% variation in ρ changes η by 1%. This means that the use of a more accurate value of ρ for zinc will reduce the results of Hopkins and Toye by nearly 4%.

However, this type of correction does not lead to any significant alteration in the temperature coefficient of viscosity, which from Figure 5 may be observed to be much less in the data given by Hopkins and Toye than the trend shown in the present experiments. One possible explanation of this type of phenomenon may originate in the way that the prevailing conditions at the meniscus interface influence the contact angle. For example, if temperatures more remote from the melting point bring about increased amounts of surface contamination, thus gradually suppressing a

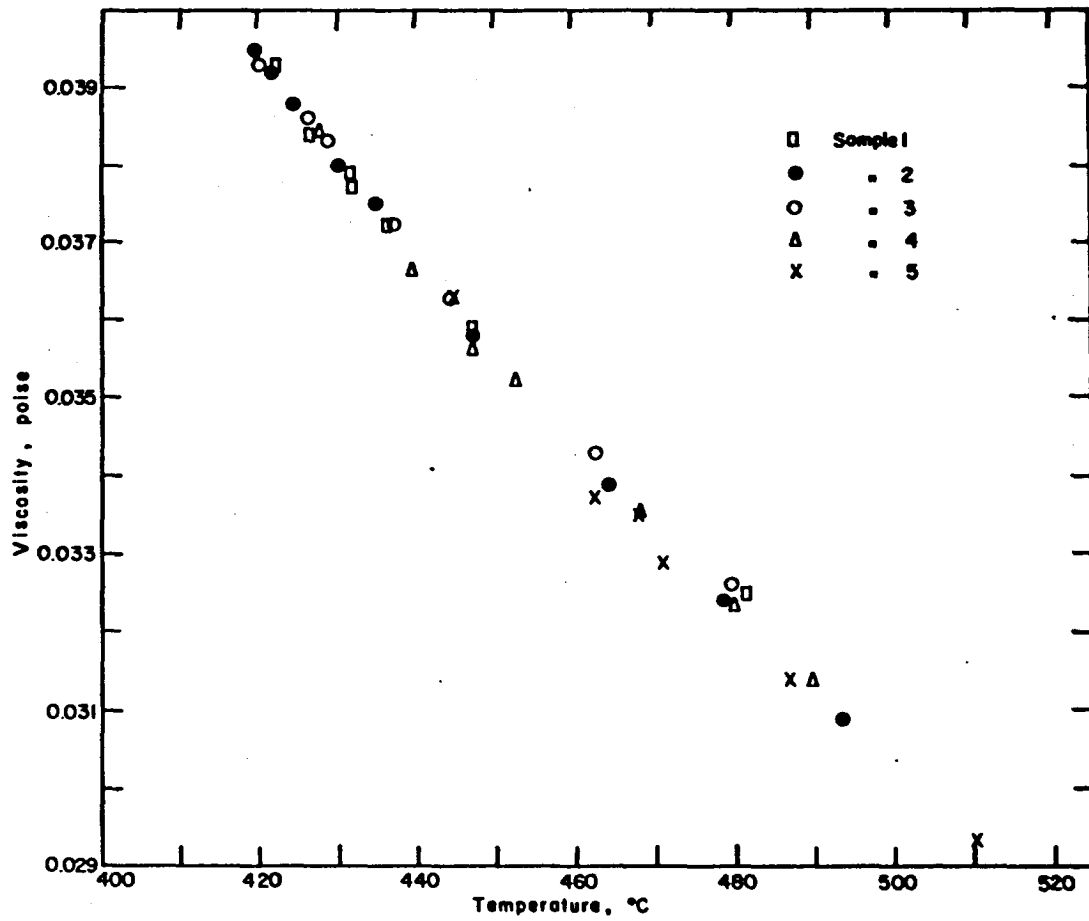


Figure 5. Viscosity values for zinc, compared with representative literature data.

high contact angle, the height of liquid will indeed become temperature-dependent. The possibility of this occurring in the experiments of Hopkins and Toye is open to conjecture, because of the use of charcoal at the liquid interface to suppress evaporation.

The results reported by Ofte and Wittenberg are characterized by the use of tantalum as the container material. Here, wetting of the tantalum surface by the liquid metal sample was an essential part of the experimental technique. Contamination of the liquid zinc by solution of the tantalum appeared to be an insignificant factor in the accuracy of the measurements. The dependence of the viscosity on the absolute temperature T was given by $\log_{10} \eta = \frac{662.5}{T} - 0.3841$, which is shown as the lower curve in Figure 5. However, the scatter amongst individual results about this plot reaches as high as 5% in two cases. A general comparison with the present results reveals a difference of some 5% at the freezing point, narrowing to about 1% at 500°C. Extrapolation from Equation 7 to 600°C shows that the trend of both plots continues with a difference of only 2%. Reappraisal of the above authors' results, particularly in the vicinity of the melting point, leads to the conclusion that a collective treatment of the two separate runs may not be justified. The values of run No. 1 are observed to be consistently above the values of run No. 2, with one exception. As the freezing point is approached a divergence of about 5% is reached, suggesting that in this region both runs are not compatible. Run No. 1 is now observed to agree with the present work to less than 3%.

Additional experiments performed at the Mound Laboratory⁽¹⁹⁾ on zinc, using a graphite-lined vessel, have yielded the interesting observation that the viscosity is now some 10% to 30% lower. This type of discrepancy is attributed to the non-wetting of zinc on graphite, which was in sharp contrast to the wetting of the zinc samples in the tantalum crucible. Here, especially, a large part of this difference can be accounted for solely in terms of the contact angle variation, influencing the height of liquid, at the container wall. Observations made at the Mines Branch on solidified samples of liquid zinc have shown that from 0.5-in.-diameter graphite containers, the height of the meniscus may reach 0.125 in. This means that the difference between the calculated height and the height in contact with the container wall may exceed 0.1 in. Variations of this magnitude on a sample height of the order of 2 in. can realize a viscosity shift of over 10%. The same type of considerations must also apply in the case where wetting of the container arises. Consequently, in the Mound Laboratory technique computed results will be consistently too high if the wetted height is not taken into account.

The present results on five samples of pure zinc, as shown in Figure 4, demonstrate quite clearly that residual impurities in 99.99% zinc do not influence the viscosity within the limits of experimental error, which is about 1%. Consequently, these five separate runs have, in effect, substantiated the capacity of the apparatus to realize reproducible data. Each set of observed data was found to fit the Andrade equation given by

$$\eta V^{\frac{1}{3}} = Ae^{\frac{C}{VT}}, \quad \dots (7)$$

where V is the specific volume associated with the absolute temperature T. By determining the respective values of the constants A and C, for each plot, the degree of misfit was better than 1% for all samples. The values of A and C listed in Table 5 appear to be quite sensitive to the variation in viscosity between runs. Thus, in presenting the results collectively, some 37 values have been used to determine A and C by a least square fit. Justification of this collective fit may be seen in Table 6, where the mean deviation of the results from Equation 7 for each run is recorded. The mean deviation for the combined values of zinc is found to be comparable to the values for individual samples. The level of relative error quoted in Table 6 depends on the error in the temperature, which was only claimed to be known to within 1°C.

TABLE 5
Values for A and C from the Andrade Equation

	A x 10 ³	C
Mercury	2.485	20.78
Lead	2.444	88.79
Zinc: Sample 1	2.459	226.3
Zinc: Sample 2	2.122	242.2
Zinc: Sample 3	2.391	229.5
Zinc: Sample 4	2.319	232.2
Zinc: Sample 5	1.986	249.5
Zinc: Combined Samples	2.169	239.8

TABLE 6

The Mean Deviation of Viscosity Results
from Andrade's Equation

Liquid	Mean Deviation (%)
Mercury	0.29
Lead	0.14
Zinc: Sample 1	0.26
Zinc: Sample 2	0.05
Zinc: Sample 3	0.12
Zinc: Sample 4	0.21
Zinc: Sample 5	0.38
Zinc: Combined Values	0.29

Thus, the absolute accuracy of the viscosity may be estimated from the known errors in R , H , I , ρ , T and δ , together with the mean relative error in each set at viscosity values arising from the temperature. The overall error from these considerations may be placed at 1.25%.

Previous work on liquid zinc by Yao and Kondic⁽²³⁾ and Yao⁽²⁴⁾ has supported the concept that an abnormal pre-freezing behaviour analogous to that observed in polyphenyls is possible in liquid metals. Hence, attention has been devoted to this area of interest, extending the damping measurements to less than one degree from the freezing point. The linear variation of the logarithmic viscosity with the inverse absolute temperature throughout the whole range of temperature investigated confirmed the complete absence of any abnormal trend in the region adjacent to the freezing point.

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