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A Wide-Range (up to 10^{10} P) Rotating Cylinder Viscometer

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The range of a high temperature rotating cylinder viscometer has been extended so that viscosity measurements can be made between 10^0 to 10^{10} poises to within 2–percent accuracy. This involves three different techniques. After a calibration constant has been determined for the apparatus in the standard oil range, the other two constants for the higher viscosities were self-determined. In making measurements at the low viscosities, 10^0 to 10^5 poises, the outer cylinder is rotated at constant speed and the torque measured on the inner cylinder; from $10^{4.5}$ to $10^{7.5}$ poises the inner cylinder is rotated through an angle and timed as it returns to its zero position, and from $10^{5.5}$ to 10^{10} poises the inner cylinder is driven through an angle at constant torque and timed as it traverses this angle. The Fulcher equation viscosity values obtained from previous measurements on Standard Glasses No. 710 and No. 711 by the fiber elongation and rotating cylinder methods have been compared with the present results.

1. Introduction

In 1924, English [1][1] reviewed methods for measuring viscosity of glasses and concluded that the Margules [2] rotating cylinder technique was readily adaptable to this work and gave much more reliable and repeatable results than the previous techniques. His apparatus consisted of a fixed cup and an inner rotating cylinder driven by the torque produced from a counter-weight and pulley system. With this equipment English was able to make viscosity measurements from 10^1 to 10^7 poises[2] on soda-lime-silica glasses. Later Parks et al., [3,4] using the same technique, made viscosity measurements on B_2O_3 glass from 10^5 to 10^{11} P.

About this time Field [5] and Lillie [6], working with molten glasses, improved the rotating cylinder viscometer by eliminating the disadvantages inherent in the counter-weight and pulley system. They rotated the cup at a constant angular velocity and measured the force on the inner cylinder by means of the angle of deflection of a torsion wire. This not only allowed Lillie to measure absolute viscosities but also gave him a higher degree of accuracy. This method was useful in the range 10 to 10^5 P.

Lillie went still further and extended the range of his viscometer by developing the aperiodic method of measuring viscosities. In this method the inner cylinder was deflected and allowed to decay to equilibrium as the outer cylinder remained at rest. This extended the range of his viscometer up to 10^7 P.

In later years, other glass investigators such as Babcock [7], and Robinson and Peterson [8], have used equipment similar to Lillie's because of the comparatively high accuracy. A further improvement to Lillie's apparatus was made by Bockris and Lowe [9], who inserted in the suspension system of the inner cylinder a coil and magnet for electromagnetically measuring the torque on the inner cylinder. Their apparatus was used to make viscosity measurements on molten silicates in the range $10^{-1.3}$ to $10^{+4.2}$ P and up to 1800 °C.

The rotating cylinder apparatus has become, over the years, a standard method for measuring viscosities of glasses at high temperatures, but other techniques had to be developed to determine viscosities above 10^7 P. Above 10^9 P the fiber elongation method of measuring viscosities has proven to be both reliable and accurate. In the gap between these two methods there has been relatively little work.

Hampton [10] measured viscosities from 10^8 to 10^{13} P observing the rate of deflection of a beam held in cantilever fashion. Recently, Hagy [11] developed a method of determining the viscosity in the range 10^8 to 10^{15} P by the viscous bending of a simple beam under load. Kelley [12] et al., have developed a penetrometer to measure viscosity of glasses from $10^{5.7}$ to 10^9 P by measuring the rate of penetration of a solid cylinder under load into the glass.

Even though measurements can now be made continuously over the whole viscosity range of glasses by various techniques, it seems desirable to make measurements with the rotation viscometer over as wide a range as possible since it has proven to be the most reliable and accurate apparatus that has been used in this work.

2. Apparatus

The geometry of this apparatus, see figure 1, is similar to that of Lillie [6], Bockris and Lowe [9]. This equipment has been previously described in more detail by Napolitano and Hawkins [13].

The outer cylinder consisted of a platinum crucible 5 cm in diam and 10 cm high. The bob was 1.25 cm in diam 3.75 cm long with 45° tapered ends and was suspended by a hollow platinum spindle extending into the center of the bob to accommodate a thermocouple for measuring the temperature of the glass

[1] Figures in brackets indicate the literature references at the end of this paper.
[2] One poise is 1 g/cm sec which is equal to 0.1 kg/m sec.

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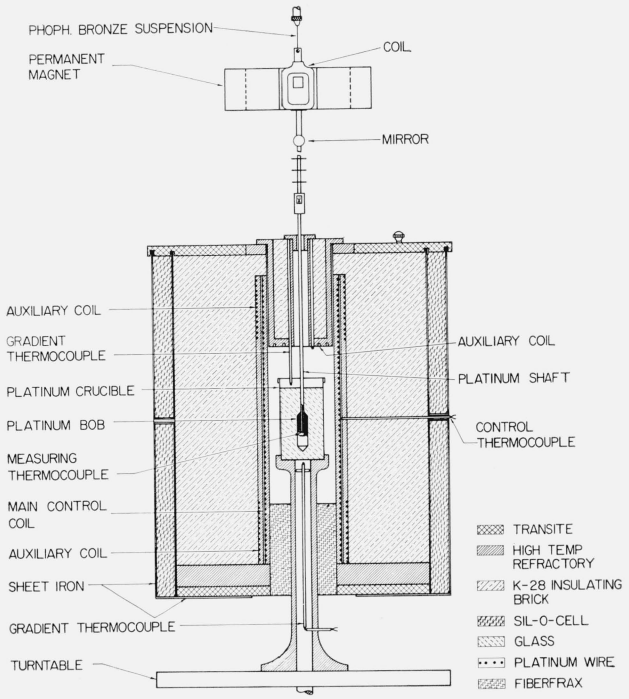

FIGURE 1. *Wide-range high temperature rotating cylinder viscometer.*

before and after viscosity measurements. The crucible was mounted on a mullite pedestal which extended out through the bottom of the furnace and was attached to the rotating mechanism. The rotating mechanism provided a range of crucible speeds from 0.03029 rpm to 1.3493 rpm.

An electromagnetic torque balance [9] based on the principle of a large moving coil galvanometer was used to measure the torque by a null detection method. The galvanometer was made up of a permanent magnet with a 1650 G gap field strength and a coil which was an integral part of the suspension apparatus. The coil consisted of a rectangular aluminum frame with about 25 m of No. 34 insulated copper wire, wound around it. The resistance of the coil was 222 Ω. In series with the coil were several resistors which allowed continuous adjustment between 2 kΩ and 5 MΩ and four L and N Standard Fixed Resistors 10, 25, 100, and 1000 Ω. The source of power consisted of 12–2 V cells. The balance was observed through a telescope on a 1 m radius scale located 1 m distant from a reflecting mirror set on the axis of the suspension shaft just below the coil. The amount of current through the coil was found by measuring the voltage across a standard resistor with a Type K potentiometer. Polarity in the coil would be reversed to accommodate reversal of rotation.

3. Operation and Calibration

The extension of the viscosity range of this apparatus was accomplished by operating it in three different modes. A careful examination of its equation of motion is very important before describing each mode of operation. The general equation of motion for the viscometer is

$$I\frac{d^2\theta}{dt^2}+\eta K_1\frac{d\theta}{dt}+K_2\theta=K_3 i \tag{1}$$

I—moment of inertia;
θ—angular displacement;
t—time;
η—dynamic viscosity;
K_1, K_2, and K_3 are constants of the apparatus; and
i—current through coil.

Thus the first term of the equation represents the resistance to acceleration due to inertia. The second term is the viscous drag. The third term represents an elastic torque due to an angular deflection of the suspension, and the last term represents the magnetic torque applied to the system. The limitations of eq (1) are discussed in the appendix.

Method 1. Over the lowest viscosity range[3] log $\eta=0$ to log $\eta=5$ the outer cylinder is rotated at a fixed angular velocity. The viscous drag of the liquid produced a torque on the inner cylinder. A measure of this torque is made by applying an appropriate current to the coil such that the resulting magnetic torque will bring the inner cylinder back to its rest position at $\theta=0$.

Therefore the first term of the eq (1) is zero, because there is no angular acceleration in the system when measurements are being made. The second term is present and significant. It is retained in the equation of motion for Mode I, but since the inner cylinder is held at rest, it is the angular velocity of the outer cylinder that is used in this term. The third term is zero when the system is balanced, since the angle across the suspension is zero. Even so, it controls the sensitivity. By changing suspensions the value of K_2 can be changed and with it the sensitivity. Since the fourth term is significant, it is kept. Thus the equation of motion for Mode I is

$$\eta K_1\frac{d\theta}{dt}=K_3 i \tag{2}$$

or

$$\eta=C_1 i/\omega \tag{3}$$

where $C_1=\dfrac{K_3}{K_1}$ and $\omega=\dfrac{d\theta}{dt}$ (angular velocity).

To obtain C_1 in eq (3) the viscometer was calibrated at 25 and 40 °C with NBS Standard oils. These oils ranged in viscosity from 1 to 500 P. Some of Dow Corning's silicons have also proven to be a valuable check on the calibration constant at the higher viscosities. Thermal expansion corrections on C_1, known for room temperatures, have to be made for accurate viscosity measurements at temperatures, T,

[3] During calibrations (at room temperature) measurements were made for viscosities below 1 P. At elevated temperatures the lower viscosity limit was 10^2 P because of the characteristics of the NBS Standard Glasses and the temperature limit of the furnace.

up to 1400 °C according to the following equation

$$C_1(T)=C_{1_{25\,^\circ C}}(1+\alpha[T-25])^{-3} \tag{4}$$

where α is the linear expansion coefficient of platinum.

TABLE 1. *Typical series of viscosity measurements made on Standard Glass No. 711 by Method I*

Standard resistor 100Ω; 0.030 cm diam. suspension

Rotation	i	ω	N
	μA	rpm	$\mu A/rpm$
Clockwise	149.706	1.3493	
Counter-clockwise	149.605	1.3493	110.91
Clockwise	87.026	.7845	
Counter-clockwise	86.655	.7845	110.70
Clockwise	49.991	.4513	
Counter-clockwise	49.905	.4513	110.68
Clockwise	36.892	.3332	
Counter-clockwise	36.630	.3332	110.34
Clockwise	149.010	1.3493	
Counter-clockwise	148.505	1.3493	110.25
Average			110.58

Temperature before start of measurements was 1050.7 °C and after measurements was 1051.3 °C: the average being 1051.0 °C.

A typical series of viscosity measurements made by this mode of operation is shown in table 1. In making these measurements four different speeds are used with readings taken in the clockwise and counterclockwise rotation for each speed. The first speed selected is always repeated at the end of the series to determine whether the glass viscosity and, therefore, the temperature, has remained essentially constant. Note the slight change in N which reflects the increase in temperature. From these measurements the viscosity is

$$\eta=N\times C_{1_{(1050\ ^\circ C)}}=110.6\times10^{-6}\times11.99\times10^{+6}=1326\text{ P}$$

or

$$\log\eta=3.123\text{ at }T=1051\,^\circ C.$$

Method 2. For viscosities from about log $\eta=4.5$ to log $\eta=7.5$ the aperiodic method developed by Lillie is used. In this decay mode of operation the outer cylinder remains at rest while the inner cylinder is displaced by an angle θ and allowed to return to its zero position under the force of the torsion wire. Since the system is an over-damped harmonic oscillator one observes an exponentially decaying angular velocity as the inner cylinder approaches its equilibrium position. The term involving the moment of inertia in eq (1) is extremely small and can be neglected, giving rise to the following equation:

$$\eta\frac{d\theta}{dt}=-C_2\theta \tag{5}$$

where

$$C_2=\frac{K_2}{K_1}.$$

Integrating

$$\int_{\theta_1}^{\theta_2}\eta\frac{d\theta}{\theta}=-\int_{t_1}^{t_2}C_2dt$$

we have

$$\eta=\frac{C_2(t_2-t_1)}{\ln\ \theta_1/\theta_2}. \tag{6}$$

The calibration constant (C_2) for Method II can be calculated once the viscometer has been calibrated by Method I. First an independent measurement of the current required for a given angular deflection of the inner cylinder is made. This gives us:

$$\theta=\frac{K_3}{K_2}i=C_4i. \tag{7}$$

If the current, i, is given in amperes, and the angle, θ, in units of 17′ of arc which correspond to a deflection of 1 cm in a circular scale 1 m distant from the central axis of the spindle; one has

$$C_4=\frac{\theta}{i}=\frac{25}{5.094\times10^{-3}}=4907.7.$$

From eq (3), (5), and (7) one may obtain C_2 from C_1 and C_4, according to the following:

$$C_2=\frac{C_1}{C_4}u \tag{8}$$

where u changes units from revolutions per minute to centimeters per second. The rates in centimeters per second were read on the circular scale.

$$u=\frac{\text{cm}}{\text{sec}}\frac{\text{min}}{\text{revolution}}=\frac{1}{60}\times\frac{2\pi\times100\times2}{1}=20.95.$$

Thus

$$C_2=\frac{20.95\times1.212\times10^6}{4907.7}=5{,}174.$$

TABLE 2. *Typical series of viscosity measurements made on Standard Glass No. 711 by Method II*

0.063 cm diam suspension

Readings	θ_1	θ_2	t_2-t_1
	cm	cm	sec
1	25	2.5	557.31
2	25	2.5	545.40
3	25	2.5	548.49
Average	25	2.5	550.40

Temperature before start of measurements was 704.2 °C and after measurements was 704.4 °C, the average being 704.3 °C.

A typical series of viscosity measurements made by this mode of operation is shown in table 2. A phosphor bronze suspension wire of 0.063 cm diam was used. Before starting, the zero was checked and properly adjusted. The current was applied to the coil so that the inner cylinder was rotated from

its equilibrium position to an angle corresponding to about 30 cm on the telescope scale. The current was turned off and time was measured for the displacement to decay from 25 to 2.5 cm. Thus the viscosity is

$$\eta=\frac{C_{2T}(t_2-t_1)}{\ln \theta_1/\theta_2}=\frac{5174\times 550.4}{2.303}=1.236\times 10^6 P$$

or

$$\log \eta=6.092 \text{ at } T=704.3\ ^\circ\text{C}.$$

Method 3. For viscosities from about log $\eta=5.5$ to log $\eta=10$ this method was used. The outer cylinder again remains at rest, while a torque is applied to the inner cylinder by means of the magnetic field interaction, and the angular velocity is measured about $\theta=0$. Thus the term $K_2\theta$ is negligible and can be omitted. Since measurements are made in a steady state the acceleration is zero, and we have the following:

$$\eta\frac{d\theta}{dt}=\frac{K_3 i}{K_1}=C_3 i \tag{9}$$

integrating

$$\int_{\theta_1}^{\theta_2}\eta d\theta=\int_{t_1}^{t_2} C_3 i dt$$

we have

$$\eta=C_3 i\,\frac{t_2-t_1}{\theta_2-\theta_1}. \tag{10}$$

C_3 and C_1 are equal, but expressed in different units:

$$C_3=uC_1=20.95\times 1.212\times 10^6=2.539\times 10^7, \tag{11}$$

where u is the same conversion factor used in calculating C_2.

TABLE 3. *Typical series of viscosity measurements made on Standard Glass No. 710 by Method III*

Standard resistor 10Ω; 0.063 cm diam suspension

Rotation	$\theta_2-\theta_1$	t_2-t_1	i A×10⁻⁴
	cm	*sec*	
Clockwise	1	197.58	196.13
Counter clockwise	1	195.48	196.13
Clockwise	1	193.62	196.13
Counter clockwise	1	191.42	196.13
Average	1	194.52	196.13

Temperature before start of measurements was 706.0 °C and after measurements was 707.6 °C: the average being 706.8.

A typical series of viscosity measurements made by this mode of operation is shown in table 3. A measured amount of current is applied to the coil, which rotates the inner cylinder at a constant angular velocity. The time required to traverse 1 cm about the equilibrium position ($\theta=0$) on the scale is measured. By appropriately selecting the current, measurements can be made up to log $\eta=10$ P within reasonable waiting periods.

From these measurements the viscosity is

$$\eta=\frac{C_{3T}(t_2-t_1)}{\theta_2-\theta_1}i=2539\times 10^4\times 194.52\times 10\times 196.13\times 10^{-4}$$

$$=9.6866\times 10^7 \text{ or } \log \eta=7.986 \text{ at } T=706.8\ ^\circ\text{C}.$$

4. Discussion of Results

Measurements were made on NBS Standard Viscosity Glasses Nos. 710 and 711. These results are given in tables 4 and 5. Only the measurements made by Methods II and III are listed here, since Method I and the fiber elongation methods of measuring viscosities have been covered in previous work [13, 14].

TABLE 4. *Viscosity values of Standard Glass No. 710 by Methods II and III compared with values calculated from Method I and fiber elongation method**

Temp. °C	log η_{obs}		log η_{calc} Fulcher equation
	Method II	Method III	
951.7	4.581	--------	4.553
902.3	5.028	--------	5.033
851.2	5.629	--------	5.614
810.3	6.169	--------	6.158
774.7	--------	6.718	6.702
773.4	--------	6.734	6.723
757.5	6.999	--------	6.993
757.1	--------	7.013	7.000
725.9	--------	7.608	7.585
706.8	--------	7.986	7.984
693.2	--------	8.298	8.290
677.8	--------	8.675	8.660
657.9	--------	9.178	9.182
636.0	--------	9.827	9.821

*Fulcher equation constants taken from Napolitano and Hawkins (Lab. A) [13

TABLE 5. *Viscosity values of Standard Glass No. 711 by Methods II and III compared with values calculated from Method I and fiber elongation method**

Temp. °C	log η_{obs}		log η_{calc} Fulcher equation
	Method II	Method III	
774.2	5.220	--------	5.229
770.5	5.255	--------	5.270
755.0	5.438	5.436	5.446
734.4	5.696	5.690	5.695
732.0	5.722	5.713	5.725
725.0	5.831	5.813	5.815
704.3	6.092	6.082	6.092
677.6	6.488	6.480	6.482
651.7	--------	6.909	6.900
631.5	--------	7.281	7.256
607.4	--------	7.752	7.723
574.7	--------	8.423	8.454
554.9	--------	8.904	8.924
528.1	--------	9.653	9.674

*Fulcher equation constants from Napolitano and Hawkins (Lab. A) [14].

The viscosity values obtained by the rotation method (Method I) and the fiber elongation method were fitted by a least square calculation to the Fulcher [15] equation. Fulcher stated that an empirical equation of the form

$$\log \eta=A+B/(T-T_0) \tag{12}$$

accurately relates viscosity to temperature in many glasses. These equations become:

$$\log \eta = -1.626 + 4239/(T-265.7)$$

for St'd. Glass No. 710; (13)

$$\log \eta = -1.654 + 4317/(T-147.0)$$

for St'd Glass No. 711. (14)

These are shown in figure 2 as the line curves in order to illustrate the viscosity values obtained in this study, and to compare the results from Methods II and III with previous measurements. The viscosity values obtained by these methods for the two standard glasses fall so close to the curves obtained from the previous measurements, that no deviation can be seen. In order to inspect these deviations more closely, the observed viscosity values were compared with the values calculated from eqs (13) and (14), for each temperature. These deviations are plotted in figure 3 as well as those from Method I and from the fiber elongation method. The gap in rotation and fiber elongation measurements has been bridged. Using all four techniques one can make measurements over a continuous viscosity range from 10^1 to 10^{12} P. The scatter of the viscosity values obtained by the driving technique is less than that obtained from the fiber elongation technique.

In figure 3, the zero lines represent the Fulcher equation as calculated from Methods I and IV. Thus the agreement between the data from Modes II and III and this line is a test not only of the precision but also of the accuracy of these measurements. Since the errors fluctuate to both sides of the zero line, any systematic deviation should be small. In fact the systematic deviations were calculated for each mode

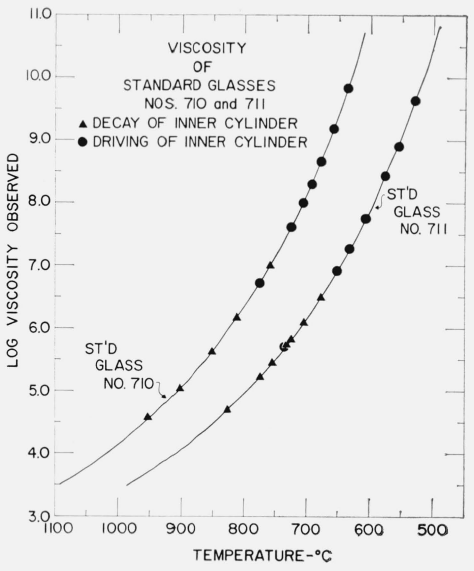

FIGURE 2. *Plot of log viscosity versus temperature of NBS Standard Glasses No. 710 and 711.*

Circles represent data taken by driving inner cylinder while triangles represent data taken with the decay mode of operation. The line curves represent calculated values from Napolitano and Hawkins (Lab. A) [13,14].

using all the measurements taken in both standard glasses. Their values were 0.30 percent for Mode II and 0.45 percent for Mode III. The standard deviations were calculated similarly. They were 0.010 log viscosity (2.3%) for Mode II, and 0.013 log viscosity (3.1%) for Method III.

Thus the equation of motion of the apparatus (eq (1)) has been proven correct as well as the calculated relations between the various calibration constants. This has been indicated in the appendix. The relatively little loss in accuracy due to the extension of the viscometer is probably due to the larger temperature dependence of the viscosity at lower temperatures.

5. Summary

The range of the rotating cylinder high temperature viscometer was extended up to 10^{10} P without loss of accuracy. Measurement at the higher viscosities could be made within reasonable time periods.

This extension was accomplished by operating the viscometer in three different modes: (1), rotating the outer cylinder at uniform angular velocity and measuring the torque in the inner cylinder; (2), the outer cylinder remains at rest while the inner cylinder is displaced by an angle and allowed to return to its zero position under the force of a torsion wire; and (3), the outer cylinder again remains at rest while a torque is applied to the inner cylinder by means of the magnetic field interaction and the angular velocity is measured about $\theta=0$. The viscometer was calibrated by Method I, and from this the constants for the other two methods were calculated. This calibration was checked with NBS Standard Samples Nos. 710 and 711, and found to be within 0.5 percent, well inside the statistical fluctuation, whose standard deviation was 0.010 log viscosity (2%) for Methods I and II and 0.013 log viscosity (3%) for Method III. This is considerably better than that obtained for the fiber elongation data 0.034 log viscosity (8%).

6. Appendix

The second term of eq (1) assumes perfect laminar flow, which is not always obtainable in a rotation viscometer. Three other flow patterns are possible in such a system—Taylor vortices [16], convection currents and turbulence.

Taylor has made an extensive study of nonlaminar flow of viscous liquids between rotating cylinders and some of his conclusions can be applied to the geometry of the present apparatus. Taylor found that his vortices appeared above a critical value of ω/η.

The largest value of $\frac{\omega}{\eta}$ for Mode I in the present investigation was 0.52. This compares with Taylor's experimental critical value much greater than 1000 and calculated at infinite speeds. For Mode II they can also be neglected since its highest ω is 0.1 rad/sec and lowest η is equal to 5×10^4 giving $\frac{\omega}{\eta}=5\times10^{-5}$

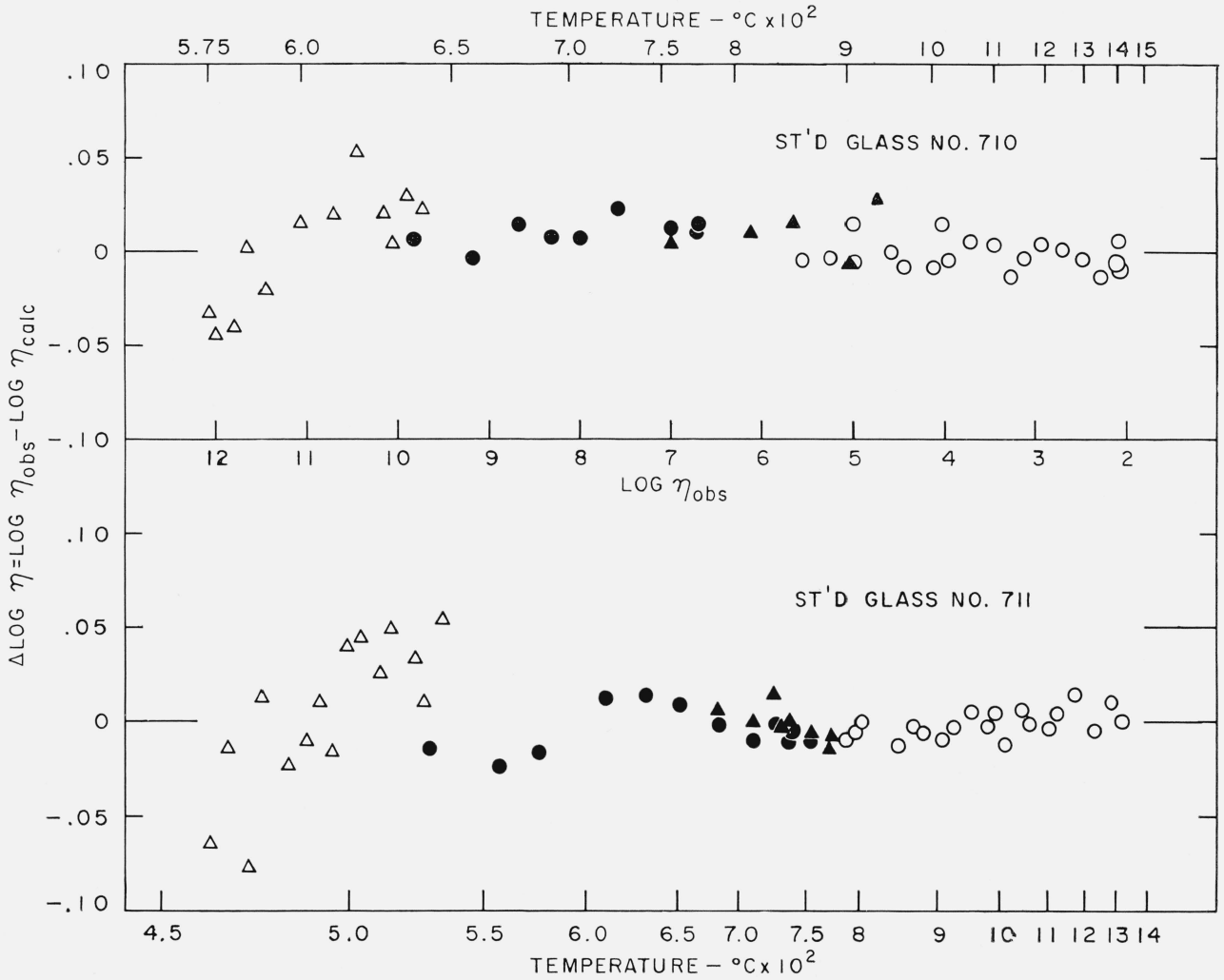

FIGURE 3. *Discrepancies between the observed log viscosity values and those calculated from the Fulcher equation for NBS Standard Glasses No. 710 and No. 711 are plotted versus log viscosity.*

Open triangles—fiber elongation; closed circles—driving inner cylinder; closed triangles—driving outer cylinder; and open circles—rotating outer cylinder.

compared with Taylor's experimental value of 30. Similarly, they are absent for Mode III. One should note that the ratio of diameters of the present apparatus is considerably larger than that of Taylor's and also that the center cylinder is very much shorter. However, since the values of $\frac{\omega}{\eta}$ used in this work are many orders of magnitude smaller than Taylor's critical values above which vortices appear, one is justified in assuming the absence of said vortices in this apparatus.

Following similar reasoning one can rule out the effects of convection currents in molten glasses when the cup has a temperature gradient of less than 5 deg. In a previous paper [13] it has been shown that the temperature gradients in the cup are much less than this. Since turbulence requires an even higher value of $\frac{\omega}{\eta}$, it definitely is not present.

The expectation that nonlaminar flow does not exist under the experimental conditions used was checked experimentally by plotting the torque versus angular velocity for both Modes I and III. The relationship was found to be linear. In Mode II a plot of displacement versus time was found to be exponential. This is direct experimental evidence that these nonlaminar effects are negligible, in agreement with the analysis given above.

Two other possible sources of error exist in our apparatus. They are (1), any effects due to the bob and cup not being perfectly centered; and (2), the dependence of end effects on the depth of immersion and clearance from the bottom of the cup.

Due to the large ratio of cup-to-bob diameters, precise centering is difficult to achieve. However, this difficulty is more than compensated for by the fact that slight departures from centering are not critical. After reasonable care is taken to center

the cup and bob, a quantitative but sensitive measure of the alinement obtained can be determined from the stability of the null position in making viscosity measurements according to Mode I. The results thus obtained are applicable to Modes II and III.

Lillie [17] and Rait [18] have shown that by having conical ends on the bob the dependence of end effects due to bob clearance is considerably reduced. This has been confirmed by the authors, who raised and lowered the cup 3 mm during viscosity measurements with negligible effects.

In conclusion it has been shown that eq (1) is valid for viscosities between 10^0 and 10^{10} P.

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