

A Rising Sphere Rheometer

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Abstract □ A modified single-pan chemical balance is described; it was used to study the rheological properties of Newtonian (silicone) fluids and a viscoelastic material (cetrimide cream BPC). In addition to accurate measurements, quick assessments of the viscosities of clear and opaque Newtonian fluids can be obtained using this rheometer. Equations for the calculation of viscosities and compliances are given.

Keyphrases □ Single-pan chemical balance—used as a rheometer, Newtonian fluids and viscoelastic materials □ Viscosity of Newtonian fluids—determined using a single-pan chemical balance □ Falling sphere viscometer—compared to rising sphere rheometer □ Creep analysis—viscoelastic materials, using rising sphere rheometer □ Rising sphere rheometer—compared to falling sphere viscometer

In the British Standard method (1) for determining the viscosity of viscous Newtonian fluids, the rate of fall of a sphere through the liquid is measured. If the test liquid is completely opaque, this method cannot be used without special equipment to detect the position of the sphere.

The rising sphere rheometer is similar to the falling sphere viscometer, except that an exponentially decaying force is used to pull the sphere upward through the test liquid. The force is applied by means of the arm of a single-pan balance, to which the sphere is attached by a fine wire. The scale pan rises by 0.295 cm. when the scale gives a 100-mg. deflection. Hence, the distance through which the sphere moves is obtained by noting the movement of the balance scale. Since it is not necessary to observe the sphere, the rheometer can be used to study opaque as well as clear liquids.

APPARATUS

A diagram of the rheometer is given in Fig. 1. It consists of an 8.5-cm. i.d. water-jacketed vessel, which rests on a bridge spanning the pan of a balance¹. The tubing carries water at 25° to and from an external water bath (not shown in Fig. 1), passing through 3-cm. thick strips of polyurethan foam as it enters and leaves the balance. These strips are held down by the draftproof screen of the balance. In this way the rheometer is protected from drafts. The water bath is mounted on foam rubber to prevent vibrations from it affecting the balance.

NEWTONIAN LIQUIDS

Timing Measurements—A 1.05-cm. diameter stainless steel sphere, attached to a 0.015-cm. diameter wire, is suspended from the upper stirrup of the balance pan assembly so that the sphere is held 0.85 cm. below the surface of the test liquid. This is achieved by keeping the length of the wire constant and always filling the test liquid to the same level, as indicated by a line inscribed in the vessel. The position of the vessel is adjusted so that the sphere is situated in a central position. The balance weights are altered until the balance

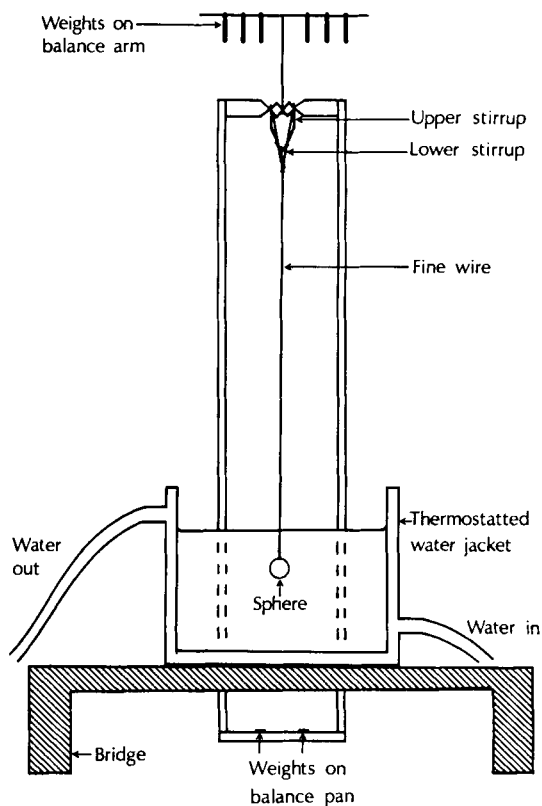


Figure 1—Rising sphere rheometer.

pan swings freely. Then small weights are added to the pan until a 100-mg. deflection is obtained. The assembly is then left for 6–8 hr. to come to temperature equilibrium. Then a 100-mg. weight is removed from the balance arm and the time, t_1 , taken for the scale to move from the 90- to the 10-mg. position is recorded.

When five or more concordant readings are obtained, the wire is removed from the upper stirrup and placed on the lower stirrup. The assembly is then allowed to attain a new position of equilibrium. In this position the sphere is held at twice the original depth, *i.e.*, 1.7 cm. below the surface of the liquid. When the 100-mg. weight is removed from the balance arm, the scale takes a longer time, t_2 , to move from the 90- to the 10-mg. position because of the resistance encountered by the extra 0.85-cm. length of wire as it is pulled through the liquid. When this difference in times ($t_1 - t_2$) is subtracted from the original time, t_1 , the new time [$t = t_1 - (t_2 - t_1)$] is related to the resistance encountered by the sphere (without the wire) as it is pulled through the test liquid.

Calculation of Pull on Sphere—Provided the viscosity is greater than about 100 poises, the speed at which the sphere rises between scale readings of 90 and 10 mg. reduces at a steady rate and, at the same time, the pull exerted on the sphere undergoes a similar exponential decay. Consequently, the average pull exerted on the sphere is the geometric mean pull = $\sqrt{90 \times 10 \times 981/1000} = 29.43$ dynes. As the sphere is pulled up, a column of test liquid adheres to the wire above the surface of the liquid (Fig. 2). The diameter of this column depends on the viscosity of the liquid, its surface tension, and the speed of withdrawal. These factors were discussed by Tallmadge and Gutfinger (2). The balance arm rises 0.295 cm. when the scale moves from the 100-mg. position to the zero position. The radii of the columns of liquid, measured at the

¹ Stanton Unimatic CL41, Stanton Instruments Ltd., London, SW 17 England.

Table I—Viscosities of Silicone Fluids Obtained from Falling Sphere and Rising Sphere Measurements

Nominal Viscosity, poises	Falling Sphere Viscosities		Rising Sphere Method							
	0.158-cm. Diameter Sphere	0.317-cm. Diameter Sphere	Time of Rise, <i>t</i> , sec.	Time of Rise, <i>t</i> ₁ , sec.	Radius of Liquid Column, <i>r</i> , cm.	Geometric Mean Pull, <i>P</i> , dynes	0.3186 <i>Pt</i> poises	$\eta_T = \frac{\eta_S t_T}{t_S}$ poises	9.3754 <i>t</i> poises	9.3754 <i>t</i> ₁ poises
160	162.3	163.8	20.0	21.4	0.11	25.53	162.6	171.0	189.3	200.6
300	283.0	281.9	33.1	35.8	0.09	26.89	283.5	283	310.3	335.6
600	656.1	662.8	72.5	77.7	0.05	28.66	661.9	620	679.7	728.4
1000	972.9	988.2	104.8	112.8	0.03	29.27	977.7	897	982.5	1057.4
2000	—	1932	206.3	222.8	0.02	29.33	1928	1764	1934	2088.2

zone of constant thickness (Fig. 2) by means of a traveling microscope, after the relevant withdrawal times, *t*, are given in Table I. These vary from 0.1 to 0.02 cm. If it is assumed that the column of liquid adhering to the wire is equal to that of a cylinder of height *h* and radius *r*, the weight of liquid adhering to the wire at any scale reading, *S* mg., can be calculated from the equation:

$$w = \pi r^2 h \rho \times 1000 \text{ mg.} \quad (\text{Eq. 1})$$

The height, *h*, is obtained from Eq. 2:

$$h = \frac{100 - S}{100} \times 0.295 \text{ cm.} \quad (\text{Eq. 2})$$

If *w*₁ is the weight of liquid adhering to the wire when the scale reading is 90 mg., the scale reading would be (90 - *w*₁) mg. if the column of liquid adhering to the wire were not present. Similarly, at the 10-mg. position, the balance arm would move to the (10 - *w*₂)-mg. scale position if the column of liquid adhering to the wire were not present. Hence, the corrected geometric mean pull is:

$$P = \sqrt{(90 - w_1)(10 - w_2)} \times \frac{981}{1000} \text{ dynes} \quad (\text{Eq. 3})$$

Calculation of Viscosity—The viscosity of a Newtonian liquid is related to the rate of fall of a sphere through it, in accordance with Stoke's equation (3):

$$\frac{4}{3} \pi r^3 (\rho_1 - \rho_2) g = 6 \pi r \eta v \quad (\text{Eq. 4})$$

The general form of this equation is:

$$\text{downward pull} = \text{velocity} \times \text{shape factor} \times \text{viscosity} \quad (\text{Eq. 5})$$

The same equation applies for the rising sphere rheometer:

$$\text{upward pull} = \text{velocity} \times \text{shape factor} \times \text{viscosity} \quad (\text{Eq. 6a})$$

or:

$$\text{viscosity} = \frac{\text{upward pull}}{\text{shape factor} \times \text{velocity}} = \frac{P t}{6 \pi r l} \quad (\text{Eq. 6b})$$

These equations do not allow for the effect of the wall of the vessel on the movement of the sphere. The Faxen correction (1) for this effect is:

$$F = l - 2.104 d/D + 2.09 d^3/D^3 \quad (\text{Eq. 7})$$

This is 0.744 for the 1.05-cm. sphere moving through a liquid in the 8.5-cm. vessel. The distance, *l*, moved by the balance pan assembly, when the scale moves from 90 to 10 mg. is: [(90 - 10)/100] × 0.295 = 0.236 cm. Hence, for the condition described the viscosity is derived from the equation:

$$\eta = \frac{P F t}{6 \pi r l} = \frac{0.744 P t}{6 \times 3.14159 \times 0.525 \times 0.236} = 0.3186 P t \quad (\text{Eq. 8})$$

where *t* is the time in seconds taken for the sphere to be pulled 0.236 cm. through the liquid after allowance has been made for the drag exerted by the liquid on the length of wire beneath the surface, and *P* is the corrected geometric mean pull obtained from Eq. 3.

In Table I the viscosities of silicone fluids obtained using the

rising sphere rheometer are compared with viscosities obtained by the falling sphere method (1), using 0.158- and 0.317-cm. spheres. The viscosities in Column 8 are, within experimental error, the same as those obtained by the falling sphere method (Columns 2 and 3).

Rapid Determinations of Viscosities—It is possible to obtain an approximate value of the viscosity of a liquid by comparing the time taken to pull the sphere, held in a liquid of known viscosity, from the 90- to the 10-mg. scale position with the time taken to pull it through a liquid of unknown viscosity between the same scale positions. In this case, it is assumed that the pull is the same for each liquid. From Eq. 8, $\eta = 0.3186 P t = k t$. Hence:

$$\frac{\eta_S}{t_S} = \frac{\eta_T}{t_T} = k \quad (\text{Eq. 9})$$

and:

$$\eta_T = \frac{\eta_S t_T}{t_S} \quad (\text{Eq. 10})$$

where η_T and η_S are the viscosities of the test and standard liquids, respectively, and *t*_T and *t*_S are the times taken for the sphere to be pulled through the two liquids. Column 9 of Table I gives the approximate viscosities obtained using Eq. 9 when the 283-poise silicone fluid is used as the standard liquid.

Another approximate value of the viscosity may be obtained if it is assumed that the effect of the film of liquid adhering to the wire

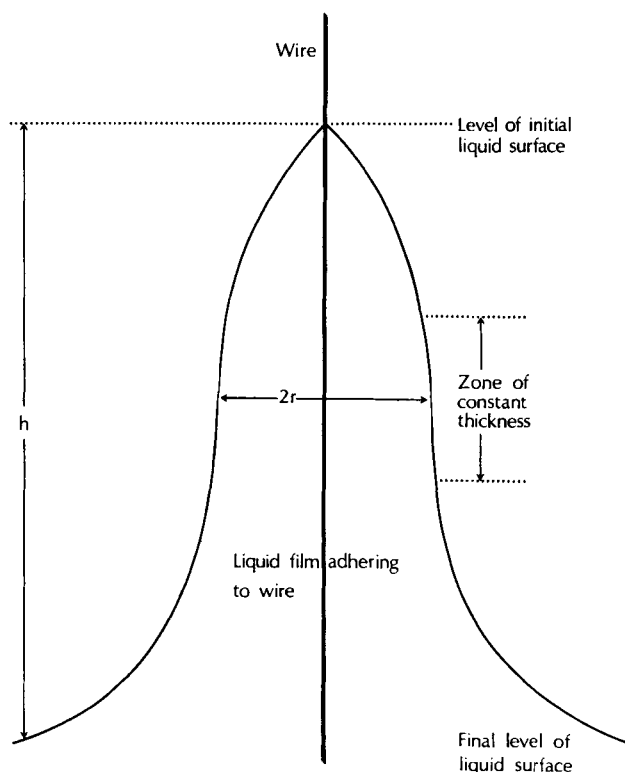


Figure 2—Shape of liquid film adhering to wire.

is negligible. In this case the value of 29.43 dynes, which is the uncorrected geometric mean pull, may be used. Then the equation for the viscosity is:

$$\eta = \frac{PFt}{6\pi rl} = 9.3754t \text{ poises} \quad (\text{Eq. 11})$$

The approximate viscosities obtained using this equation are given in Table I, Column 10.

If the time of withdrawal is determined only at the 0.85-cm. level, the viscosities obtained do not take into account the drag exerted by the liquid on the wire. In this case, t_1 is substituted for t in Eq. 11, and the viscosities are given in Table I, Column 11. This is the simplest method of determining the approximate viscosity with this instrument. But it is inaccurate for low viscosity liquids if a small sphere is used. When a larger sphere is used, the rate at which it rises is slower and the resistance to movement exerted by the wire is less significant.

With one exception, the first two methods of determining approximate viscosities are within $\pm 9\%$ of the correct values. Errors arising when the third method is used vary from 10 to 18%.

VISCOELASTIC MATERIALS

The rising sphere rheometer can be used to study viscoelastic materials using creep measurements. The basis of these measurements is that the system being tested is subjected to a stress, which causes a very limited movement of an object that is in close contact with the test material. This movement is related to the strain exerted on the test material, and the strain-stress ratio at a given time is called the compliance, $J(t)$. A typical creep curve (Fig. 3) is obtained when compliance is plotted against time. If the stress is kept constant, a creep curve, from which compliance measurements can be calculated, is obtained when strain (or relative movement) is plotted against time. When the rising sphere rheometer is used for creep measurements, the movement of the balance scale does not normally exceed 10 mg. (*i.e.*, 0.0295 cm.) when a 1-g. weight is removed. This means that the maximum error is 1% if the pull is assumed to be constant. In practice, the error is much less than this amount because measurements are made from portions of the curve. The curve obtained has a shape similar to that in Fig. 3. This has three distinct portions:

1. A region of rapid movement when the stress is first applied to the system. If the stress were removed during or immediately at the end of this period, the system would return at once to its original ground state. In this region the viscoelastic material behaves as a Hookean solid; the compliance is given the symbol J_0 .

2. A region in which the compliance is continually changing, which is likened to a number of pairs of springs and dashpots in series.

3. A linear region in which Newtonian flow predominates. The reciprocal of the slope of the graph in this region is the residual viscosity. The value obtained for the residual viscosity of a cream depends on its age, the stresses applied to it when it is loaded into the rheometer, the time it is left in the rheometer before testing, and the conditions of storage. Consequently, it is important to compare similar formulations that have been stored for similar times, loaded into the rheometer under similar conditions, and left to equilibrate for the same time before testing.

Loading—The sample to be tested is loaded into the water-jacketed vessel, the sphere is immersed in it, and the assembly is left to stand for approximately 18 hr. at the appropriate temperature. This enables the test material to recover some of its structure, which is lost on loading, and to come to temperature equilibrium with the sides of the vessel. If the test material contains water, the wire supporting the sphere is passed through a small hole in a sheet of plastic impermeable to water vapor. The film is held down on the edge of the vessel with a thin layer of grease. In this way, loss of water by evaporation is reduced to a minimum.

Test for Linearity of Response—At the time of loading, weights are removed from the balance arm until a suitable scale movement of less than 5 mg./min. is obtained. The weight required to produce this movement is noted (W). This weight is then replaced and the scale is allowed to return to its original position. Then twice the weight W is removed from the scale pan. If the scale movement in 1 min. is twice that produced by weight W , the sample shows a linear response.

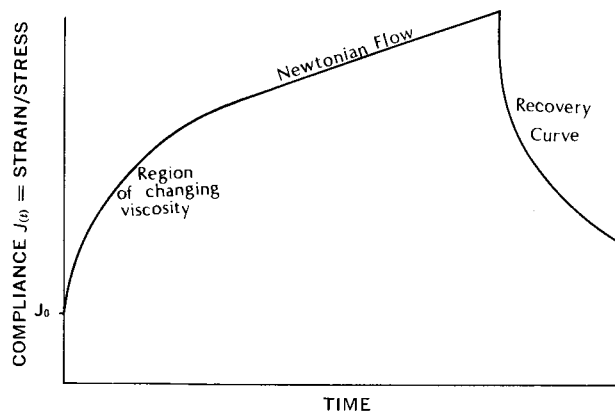


Figure 3—Creep curve.

Creep Measurements—At the start of the experiment, the scale reading is noted (s_0). Then the weight W is removed from the balance arm and, at the same time, a multiple-timing stopwatch is started. The times are noted at which definite scale divisions are reached. This is difficult to achieve during the first 2 min. because the scale is moving rapidly (see the first part of the creep curve of Fig. 3). When the region of Newtonian flow is reached, the times for scale movements of 0.1 mg. are easily recorded. These scale readings are plotted against time. After 1 hr., or a suitable time determined by the linearity of the slope of the graph in the region of Newtonian flow, the weight is reapplied to the balance arm. Then the times at which definite scale readings are obtained are recorded and plotted to give a recovery curve.

Calculation of Residual Viscosity—The residual viscosity is obtained by noting the time, t_c , taken for the scale to move between two scale readings, s_1 and s_2 , on the linear portion of the creep curve. If W is the weight removed from the balance arm, the geometric mean pull exerted on the sphere in this linear region is:

$$P = \sqrt{[W - (s_0 - s_1)][W - (s_0 - s_2)]} \times \frac{981}{1000} \text{ dynes} \quad (\text{Eq. 12a})$$

If W is 200 mg. or greater, the error is less than 0.1% if the arithmetic mean pull:

$$P = \frac{[W - (s_0 - s_1)] + [W - (s_0 - s_2)]}{2} \times \frac{981}{1000} \text{ dynes} \quad (\text{Eq. 12b})$$

is used instead of the geometric mean pull. No correction is necessary for the weight of test material adhering to the wire, because this will not exceed 0.05 mg. under normal test conditions.

The residual viscosity is obtained from the equation:

$$\eta_0 = \frac{PFt_c}{6\pi rl} \text{ poises} \quad (\text{Eq. 13})$$

where l is the distance moved by the sphere when the balance scale moves from s_1 to s_2 mg. This is derived from the equation:

$$l = \frac{s_1 - s_2}{100} \times 0.295 \text{ cm.} \quad (\text{Eq. 14})$$

Residual Viscosity—The residual viscosity of a sample of cetrimide cream BPC, made 2 months prior to the test, was 14.4×10^6 poises after the cream had been left at 25° for 18 hr. to equilibrate. When the same cream was tested in the creep apparatus described by Barry and Grace (5), the residual viscosity obtained after the cream had stood for 18 hr. at 25° was 15.8×10^6 poises. This was derived from the equation given by Warburton and Barry (6):

$$\eta_0 = \frac{\tau}{4\pi/lc} \left[\frac{1}{R_1^2} - \frac{1}{R_2^2} \right] \frac{l}{\theta} \text{ poises} \quad (\text{Eq. 15})$$

These results show good agreement and indicate that the rising sphere rheometer is suitable for determining the residual viscosities of viscoelastic materials.

Calculation of J_0 , the Instantaneous Compliance—The rapid movement of the scale that occurs when the weight W is first re-

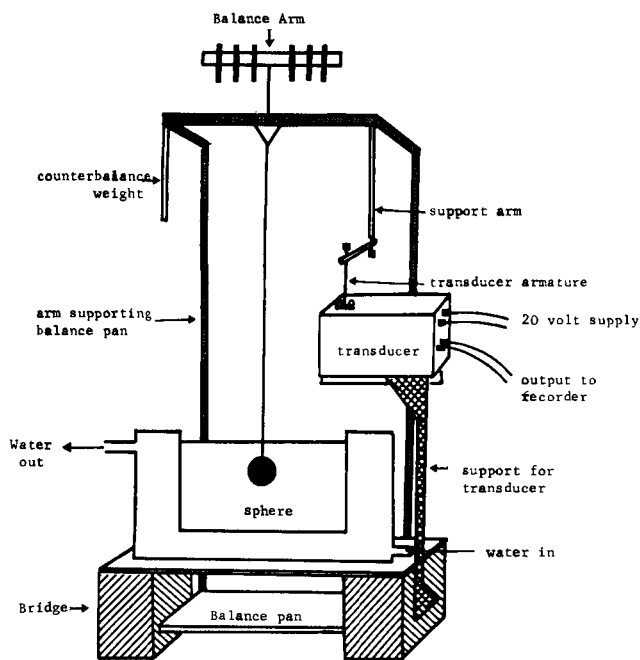


Figure 4—Rising sphere rheometer with automatic recording attachments.

moved from the balance arm is related to the instantaneous compliance, which is obtained from the equation:

$$J_0 = \frac{\text{strain}}{\text{stress}} = \frac{6\pi r l}{PF} \text{ dyne}^{-1} \text{ cm.}^2 \quad (\text{Eq. 16})$$

Strain is the movement of the sphere relative to the wall of the vessel. The movement is l cm., and the distance of the sphere from the wall of the vessel is contained in the Faxen correction factor. Similarly, the stress is equal to $P/6\pi r l$, where l , is a unit of length related to the area of the sphere, the reciprocal of which is also contained in the Faxen correction factor.

The measurement of J_0 is not as precise as the measurement of the residual viscosity, because it depends on the assessment of a movement that takes place in a very short time. If the movement is automatically recorded, the value of J_0 reduces as the speed of the recorder chart is increased. The value of J_0 obtained from the sample of cetrimide cream BPC held at 25° for 18 hr. in the rheometer before the test was performed was 1.5×10^{-4} dyne⁻¹ cm.². The value for the same sample tested in the creep apparatus under the same conditions was 1.69×10^{-4} dyne⁻¹ cm.². This was derived from the equation given by Warburton and Barry (6):

$$J_0 = \frac{4\pi h_c}{\tau} \left[\frac{1}{R_1^2} - \frac{1}{R_2^2} \right]^{-1} \theta \text{ dyne}^{-1} \text{ cm.}^2 \quad (\text{Eq. 17})$$

DISCUSSION

Comparison of Chemical Balance Viscometer with Rising Sphere Rheometer—The rising sphere rheometer differs from the chemical balance viscometer described by Davis *et al.* (7) in that the time for movement of the scale between the 90- and 10-mg. positions is used to calculate the viscosities of silicone fluids, compared with the time taken for a full-scale deflection used by these workers. The author suggests that the compliances and residual viscosities of sputum could be calculated if a sphere suspended in a short test tube were used instead of a coverslip in a 1-cm. spectrophotometer cell, as used by these workers. When times for full-scale deflections of mobile liquids are measured, the technique suffers from the following disadvantages:

1. When the 100-mg. weight is removed from the balance arm, the rate of movement accelerates to a maximum before undergoing a steady exponential decay. This initial acceleration depends on the speed and smoothness with which the weight is removed. Consequently, the technique is only suitable for approximate deter-

minations of viscosity, *e.g.*, the study of sputum (8) or assessment of the mobility of milk and other fluids prior to spray drying. Furthermore, the retardation of movement caused by the balance dashpot is significant when mobile liquids are tested in this type of rheometer (7).

2. Between the scale positions of 10 mg. and zero, the speed of movement of the balance arm is slow; in the case of viscous liquids, the time taken for the scale to reach the zero position is excessively long. This results in timing inaccuracies.

3. The geometric mean pull cannot be calculated.

Comparison of Falling Sphere Viscometer with Rising Sphere Rheometer—The rising sphere rheometer can be used to measure the viscosities of opaque liquids, because it is not necessary to watch the sphere as it moves through the test liquid. When the viscosities of opaque liquids are determined using the falling sphere viscometer (1), magnetic devices must be used to detect the position of the sphere in the fall tube.

Since movements of the sphere are noted by observing the movement of a balance scale, errors of parallax, which may occur when measurements are made using the falling sphere viscometer, do not occur.

When falling sphere viscometer measurements are made, there is a time lapse while the sphere falls down the entrance tube of the viscometer. If this is excessively long, determinations are tedious. There is only a short time delay between replicate measurements when the rising sphere rheometer is used, because the 100-mg. weight may be removed from the balance arm as soon as the sphere is stationary at the 100-mg. scale position.

Advantages of Rising Sphere Rheometer—The rheometer is easily constructed by modifying a single-pan chemical balance when not in use as a rheometer. The method of measuring is nondestructive; consequently, measurements can be repeated. The readings can be automatically recorded using a displacement transducer² (Fig. 4).

CONCLUSIONS

The rising sphere rheometer is a modified single-pan chemical balance which can be used to study the flow properties of both Newtonian and viscoelastic materials. The equation from which viscosities can be accurately calculated is:

$$\eta = \frac{PF l}{6\pi r l} \text{ poises} \quad (\text{Eq. 8})$$

Approximate viscosities of Newtonian fluids can be calculated by comparing the time taken for the sphere to be pulled through a fluid of known viscosity with the time taken for the sphere to be pulled through the same distance in a liquid of unknown viscosity. The equation which applies to this test is:

$$\eta_T = \frac{\eta_S t_T}{t_S} \text{ poises} \quad (\text{Eq. 10})$$

Creep compliances at time t_0 can be derived from the equation:

$$J_0 = \frac{6\pi r l}{PF} \text{ dyne}^{-1} \text{ cm.}^2 \quad (\text{Eq. 16})$$

The values obtained from Eqs. 8 and 16 are the same as the values obtained when the same materials are tested by other methods.

SYMBOLS

- d = diameter of sphere, cm.
- D = diameter of vessel, cm.
- F = Faxen correction = $1 - 2.104d/D + 2.09d^3/D^3$
- g = gravitational constant = 981 cm. sec.⁻²
- h = height of column of liquid adhering to wire, cm.
- h_c = length of inner cylinder of creep apparatus, cm.
- k = constant, dynes

² Model ELD 917 (Kent Instruments Ltd., Luton, England), linked to a Servoscribe Potentiometric Recorder, KECO/1003 (Kelvin Electronics Co., Wembley, Middlesex, England).

l = distance moved by balance arm, cm.
 P = pull exerted by balance arm, dynes
 r = radius of column of liquid adhering to wire supporting sphere, measured in zone of constant thickness, cm.
 R_1 = radius of inner cylinder of creep apparatus, cm.
 R_2 = radius of outer cylinder of creep apparatus, cm.
 S, s_0, s_1, s_2 = balance scale readings, mg.
 t = time = $t_1 - (t_2 - t_1)$, sec.
 t_1 = time taken for scale to move from 90 to 10 mg. when sphere is 0.85 cm. below liquid surface, sec.
 t_2 = time taken for scale to move from 90 to 10 mg. when sphere is 1.7 cm. below liquid surface, sec.
 t_c = time taken for scale to move from s_1 to s_2 , sec.
 t_0 = zero time
 t_S = t for standard liquid, sec.
 t_T = t for test liquid, sec.
 v = velocity, cm. sec.⁻¹
 W = weight removed from balance arm, mg.
 w = weight of liquid column adhering to wire, mg.
 w_1 = weight of liquid column adhering to wire at 90-mg. scale reading, mg.
 w_2 = weight of liquid column adhering to wire at 10-mg. scale reading, mg.
 η = viscosity, poises
 η_0 = residual viscosity, poises
 η_S = viscosity of standard liquid, poises
 η_T = viscosity of test liquid, poises
 θ = angular deflection of inner cylinder of creep apparatus, radians
 π = 3.14159
 ρ_l = density of sphere, g. cm.⁻³

ρ_2 = density of liquid, g. cm.⁻³
 τ = torque, dynes cm.⁻¹

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Effect of Processing Variables on Particles Obtained by Extrusion-Spheronization Processing

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Abstract □ Four extrusion-spheronization processing combinations were used to produce spherical granular materials from a standard base formulation. Dried, processed materials were evaluated by several physical methods to quantify the effect of the spheronizing processing variables, speed and dwell time, on the resulting granules. The data obtained indicated that, with this base formulation, plate rotational speed was of great importance in establishing the geometric form of final granules. Dwell time did not have an appreciable effect on the granules in this study. Measurements obtained for spheronized materials were compared to measurements on nonpareil beadlets and a conventionally produced granulation.

Keyphrases □ Extrusion-spheronization processing—effect of processing variables on particles □ Granules, form—extrusion-spheronization process, effect of processing variables □ Particles, form—extrusion-spheronization processing variables □ Spheronized materials—compared to nonpareil beadlets and conventionally produced granulation

Granulation is a key process in the production of many pharmaceutical dosage forms. Various techniques and equipment have been used to obtain granular materials, with one of the newer pieces of granulation

equipment being the Marumerizer¹.

Preparation of solid spherical products by use of the Marumerizer offers a number of potential advantages to the pharmaceutical industry in the production of beads or granules as either finished or intermediate products. Some of these advantages include accurate control of dosage, speed of production, and choice of batch or continuous flow production (1). The operation of the Marumerizer in combination with an extruder was described by Conine and Hadley (2). Processing of the pharmaceutical formulation involves extruding wetted material into cylindrical segments, breaking the segments, and rolling them into solid spheres on a spinning friction plate. To produce solid spheres, the extrudate must break into short segments and the short cylinders must be plastic enough to be rolled into a spherical shape. Conine and Hadley found that materials which break into short cylinders but do not have sufficient plastic properties do not yield a spherical product. The

¹ Elanco Products, a division of Eli Lilly and Co., Indianapolis, Ind.