PLASTICITY MEASUREMENTS ON MILK OF MAGNESIA.*

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In January 1927, Herschel and Bulkley[1] described various types of burette consistometers and an accurate method of calibration. Several of the correction factors applied are, as shown later, unnecessary for routine control work. In this paper data and results from plasticity measurements on milk of magnesia are submitted as a practical application of the burette consistometer. The work was finished in September 1926.

APPARATUS.

The consistometer (Fig. 1) consists of a 50-cc. burette from which the stop-cock has been removed and to which a capillary tube has been attached by means of a rubber stopper fitted into a wider rubber tube. This method of attaching the capillary tube to the burette is rapid, simple and permits cleaning readily. Inasmuch as the ends of the tube are cut off perpendicularly, the length is measured readily. The diameter of the capillary tube is measured either by use of viscosity measurements of standard liquids[2] or by the mercury thread method. In this laboratory we have obtained satisfactory results by the latter method.

All our runs were made at room temperature, about 21–24° C.; the temperature variation during a run was not greater than 1° C. No temperature-bath was used, but for control work it would be necessary to use a constant temperature of 20° or 25° C. The bath described by Herschel, which consists of a long glass tube, 6 cm. in diameter, to which hot or cold water is added as required and air pressure is admitted at the bottom for continuous stirring of the liquid, would be satisfactory.

PROCEDURE.

The instrument is adjusted in a vertical position, and the tip of the capillary is closed by inserting in it the sharpened point of a piece of wood or a cork. The burette is then filled with milk of magnesia to above the zero mark, and the total depth of the milk is stirred with a long wire or glass rod in order to force out all entrapped air bubbles.

The run is made by removing the wooden pin from the capillary tip and measuring the time interval for the flow of successive volumes of the milk. In our work, the time interval was measured, by a stop-watch accurate to a fifth-second, for every 6-cc. milk of magnesia extruded at the beginning of the run and for every 4 cc. after the pressure head had dropped to about 25 cm. of milk. These volume increments were chosen so that the time interval would be large enough to insure sufficient accuracy of the time readings. In order to plot the data, they are converted to the form $\frac{4}{\pi R^3} \cdot \frac{\Delta V}{\Delta T}$ and $\frac{PR}{2L}$; thereby obtaining the mobility and yield value in absolute units of rhés and dynes per sq. cm., respectively.

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* Scientific Section, A. PH. A., St. Louis meeting, 1927.

[1] *Ind. Eng. Chem.*, 19 (1927), 134.

[2] *J. Phys. Chem.*, 29 (1925), 1283. E. Moness and P. M. Giesy, JOUR. A. PH. A., 15 (1926), 39.

After calculating the average shearing stress $F = \frac{PR}{2L}$ for each experimental point of the first run the same values may be used for successive runs provided that the initial pressure head from the burette zero to the tip of the capillary is always set at the same value and provided that the time readings are taken for the same volumes in each run, *i. e.*, take time readings for volume marks of 0, 6, 12, 18, 24, 30, 35, 40, 44 cc. on the burette in all runs. By following this procedure there is a considerable saving in time and calculations when making an extended series of runs.

RESULTS.

Experiments were run with the following different capillaries:

No.	Radius (cm.).	Length (cm.).
1	0.0965	5.56
2	0.07163	4.95
3	0.06040	6.00
4	0.03105	5.55

Of these four capillaries the third yielded the most convenient procedure and gave the best results.

In the following table are recorded the yield values and mobilities as obtained from plasticity runs, and the assays and settling values of eleven different samples from experimental batches of milk of magnesia. The assay is given as per cent $Mg(OH)_2$ by weight, and the settling is recorded as the linear distances from the top of the water in the pint bottles to the top of the meniscus of the settled milk.

Expt. no.	Assay wt. % $Mg(OH)_2$.	Settling (cm.).	Yield value dynes/sq. cm.	Mobility rhés.
427-B	5.3%	3.0	57	26.20
427-C	...	1.5	153	16.36
427-D	...	1.6	144	16.50
427-E	...	2.6	87	20.40
427-F	7.5	2.0	97	17.85
427-G	9.2	1.5	153	12.15
427-H	7.5	1.5	123	11.46
427-I	8.6	1.2	179	8.40
427-J	8.0	1.2	157	15.21
427-K	7.5	2.8	104	10.82
427-L	7.7	2.0	92	20.00

Figure 2 gives the flow lines for runs 427-E, 427-F and 427-L. These flow lines are typical for the samples; it will be observed that the lines are straight and well defined. The above results and flow lines are not corrected for kinetic energy.

A relationship between the yield value or mobility on the one hand and the settling or the assay of a milk on the other was sought, but after plotting these experimental values no definite empirical relationships were found.

DISCUSSION OF RESULTS.

The purpose of this work was twofold: first, to develop an apparatus and procedure to determine whether milk of magnesia acted as a plastic material or a viscous liquid, and second, to find any relationship existing between the

ultimate settling of a milk and either of its plastic values or a function of the two, or its viscosity, whereby the former could be predicted from rapid plasticity measurements made at the time of manufacture of a batch of milk. As stated above, no positive relations were found to exist.

The penetrativity method, as modified by Moness and Giesy[1] and further modified to use milk of magnesia as its pressure column, yielded data indicating that milk of magnesia is a plastic with a low yield value and high mobility. With a capillary tube having a radius 0.06157 cm. the following results were obtained on a sample of milk from stock.

Pressure cm. milk $Mg(OH)_2$.	Yield value dynes/sq. cm.	Mobility rhés.
10	12.3	29.2×10^{-2}
15	10.8	29.5×10^{-2}
20	9.2	35.0×10^{-2}

The flow lines are well-directed straight lines. It should be noted that these sets of yield values and mobilities are lower than those obtained by the extrusion method of the burette consistometer in which the pressure applied was considerably higher than 20 cm. These differences in results have been found with other plastics, and some data have been obtained which qualitatively substantiate Henry Green's[2] conclusion that the difference is explained by the fact that the two methods give two different portions of the plasticity curve: the penetrativity giving the lower portion and therefore a lower mobility and yield value while the extrusion methods give the upper one.

The penetrativity method was somewhat difficult to control because the flow through the capillary tube was quite rapid even for as low a pressure as 10 cm. of milk and a capillary having a diameter as small as 0.06157 cm. Therefore, the extrusion methods were applied, and the burette consistometer, as described above, was developed for milk of magnesia.

In all studies by the extrusion methods, preceding our work with the burette consistometer, the volume-rate of flow method was used,[3] *i. e.*, measuring time intervals for definite volumes of milk extruded, instead of the more cumbersome method of weighing the extruded milk for each experimental point.

Bingham[4] found a straight line relationship by plotting volume per cent solids against mobilities and against friction for clay suspensions, but for milk of magnesia, which is essentially a suspensoid, such a relation did not seem to exist over a rather narrow range of volume per cent solids.

Herschel and Bulkley[5] studied six rubber solutions in benzene varying in concentration from 0.599% to 1.210% and found that the flow lines obtained by plotting volume flow, in cc. per second, against pressure, in Gm. per sq. cm., were all curved and approached the origin. The Bingham viscometer was used to obtain the experimental data. When these data were plotted logarithmically,

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[1] *J. Phys. Chem.*, 29 (1925), 1283. E. Moness & P. M. Giesy, JOUR. A. PH. A., 15 (1926), 39.

[2] Bingham and Murray, *A. S. T. M.*, 23 (1923), II, 655. Discussion by Henry Green.

[3] Fifth Colloid Symposium, P. M. Giesy and S. Arzoomanian.

[4] "Fluidity and Plasticity," E. C. Bingham (1922), 220–221.

[5] W. H. Herschel and Ronald Bulkley, *A. S. T. M.*, 26 (1926), II 621–623.

that is, log-flow against log-pressure, all of the flow curves were straight lines from which a new exponential equation for plastic flow was developed which consisted of a pressure term raised to an experimentally determined exponent, two constants of the material, and a third constant which by itself is not a constant

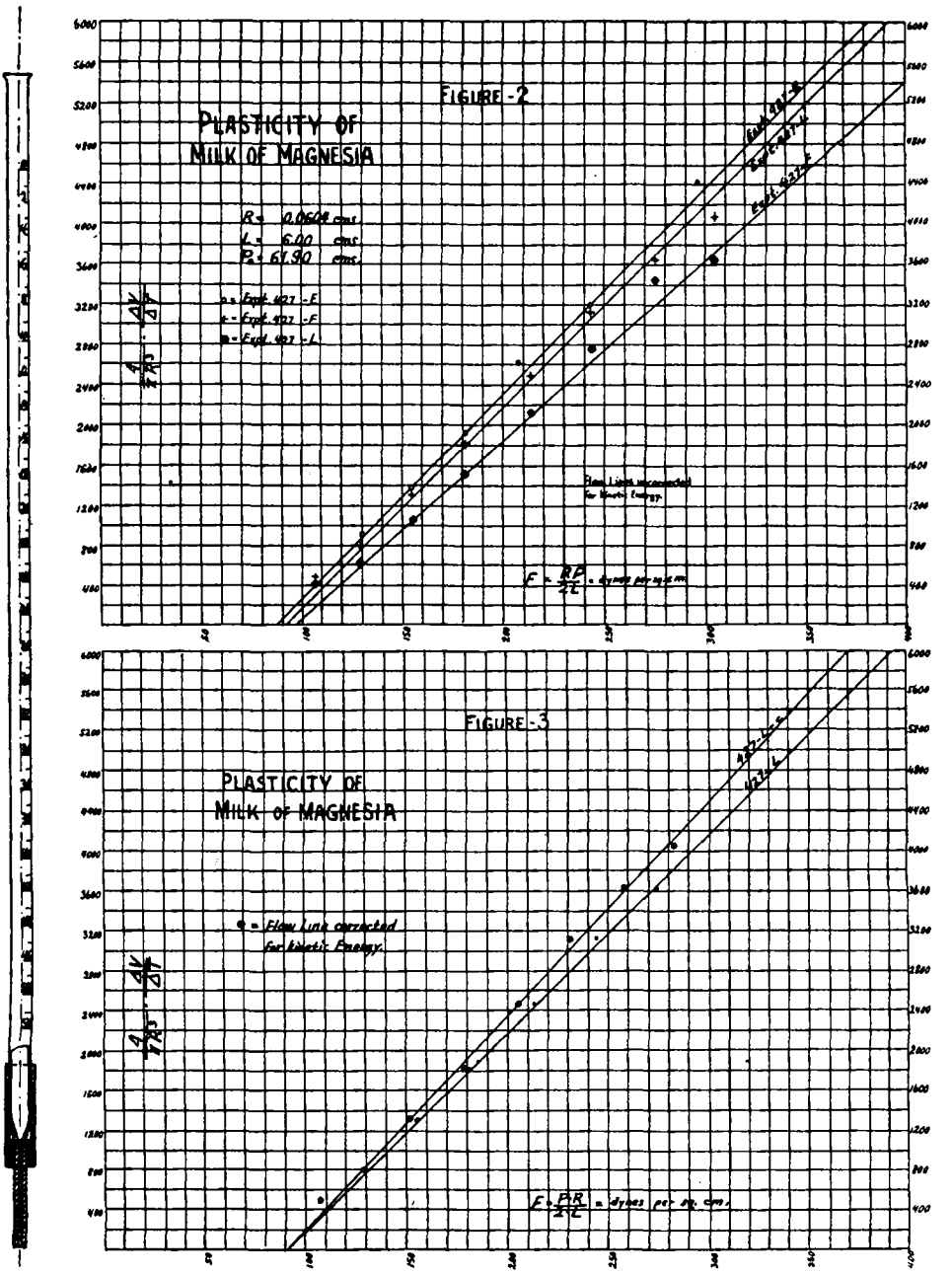

Fig. 1.

Fig. 1.—Consistometer. Fig. 2.—Shows uncorrected flow lines. Fig. 3.—Shows flow line corrected for Kinetic Energy.

of the material but which might contain a third constant that one may be able to calculate from it. The authors also stated that "it appears reasonable to expect that this or some similar equation is typical of the emulsoids, while suspensions are apparently better represented by simple flow-pressure curves." Our flow-pressure curves for milk of magnesia are perfectly straight and confirm the above statement regarding suspensoids.

DISCUSSION OF CORRECTION FACTORS AND NEGLIGIBLE ERRORS.

1. Average Pressure.

The average pressure head used in our calculations for shearing stress was taken as the arithmetic mean instead of the log. mean values recommended by Herschel. The maximum error introduced by this simplication for as large as a 6-cm. difference between h_1 and h_2 over the pressure range of 31 to 61 cm. was less than 1%. The log. mean is calculated by the formula:

$$h = \frac{h_1 - h_2}{\ln \frac{h_1}{h_2}}$$

These values are compared with the arithmetic mean values in the following table:

h cm.	$h_1 - h_2$ cm.	Log. mean head (cm.).	Arith. mean head (cm.).	% error.
61				
	6	57.944	58.000	0.097
55				
	6	51.928	52.000	0.138
49				
	6	45.948	46.000	0.113
43				
	6	39.916	40.000	0.210
37				
	6	33.920	34.000	0.236
31				
	5	28.426	28.500	0.260
26				
	5	23.410	23.500	0.384
21				
	4	18.927	19.000	0.386
17				
	4	14.910	15.000	0.604
13				

2. Surface Tension Correction.

Correction in the pressure head due to the surface tension of the liquid is generally negligible as shown by the following calculation. The nearest approximation to this correction is given by the formula for the capillary rise of a liquid under static conditions

$$h'' = \frac{4\psi \cos \alpha}{\rho g d'}$$

where ψ = surface tension in dynes per centimeter.
α = the angle between the liquid surface and the wall of the capillary tube.
d' = outside diameter of the capillary.
ρ = specific gravity of milk of magnesia = 1.05.

The average value of cos α under our experimental conditions can be taken as 0.5. The average surface tension of two samples of Squibb's milk of magnesia was 76.17 dynes per centimeter at 25° C. (determined by R. Van Winkle of our laboratory by the Du Nuoy apparatus). The outside diameter of our capillary was 7.0 mm. When these values are used in the above formula, the following result is obtained

$$h'' = \frac{(4)\,(76.2)\,(0.5)}{(1.05)\,(980)\,(0.70)} = 0.211 \text{ cm. } H_2O.$$

This is less than a 1% error of the lower pressure heads and is therefore negligible.

3. Kinetic Energy Correction.

The kinetic energy imparted to the flow of the milk of magnesia must be corrected for when it is sufficiently large as to affect the values of mobility and yield, that is, when it would change the position of the plastic flow line. From the fundamental equation of kinetic energy of a moving body of one half the mass multiplied by the square of the linear velocity is derived the following equation in terms of pressure for the kinetic energy correction for plastic flow:

$$\text{K. E.} = \frac{m\,q^2}{\pi^2 R^4 g}$$

where K. E. = kinetic energy correction in Gm. per sq. cm.
m = coefficient of kinetic energy correction generally accepted average value of 1.12.
q = rate of flow in cc. per sec. R = capillary radius in cms.
g = acceleration of gravity = 980 cm. per sec. per sec.

The result obtained from this formula when not negligible should be subtracted from each pressure head (expressed as Gm. per sq. cm.) before plotting for yield values or calculating mobility. When the values for m, R and g are inserted, the above formula reduces to

$$\text{K. E.} = \frac{(1.12)\,(q^2)}{(\pi^2)\,(0.06040)^4\,(980)} = 8.700\ q^2$$

In the following table the data for 427-L are given showing the values for the kinetic energy correction and the percentage error when it is omitted from the average pressure head.

Burette reading cc.	$q = \frac{\Delta V}{\Delta T}$ cc. per sec.	q^2.	K.E. = 8.700 q^2.	Arith. average pressure. Cm. milk.	Arith. average pressure. Gm. per sq. cm.	% error due to neglecting K. E.
0	0.706	0.498	4.34	58.9	61.9	7.01
6	0.636	0.405	3.53	52.9	55.6	6.34
12	0.546	0.2985	2.60	46.9	49.3	5.27
18	0.429	0.1845	1.607	40.9	43.0	3.74
24	0.316	0.1000	0.870	34.9	36.7	2.37
30	0.227	0.0516	0.450	29.4	30.9	1.45
35	0.139	0.0194	0.169	24.4	25.4	0.66
40	0.0851	0.00724	0.063	19.9	20.9	0.30

When the kinetic energy is neglected, the per cent error is clearly too high, and the correction should therefore be applied.

Figure 3 gives the new flow line for this run after the kinetic energy correction has been applied. The points which did not lie on the uncorrected flow line now fall on it or are closer to it. Inasmuch as the kinetic energy correction is appreciable for the first half of the points during a run, the slope of the new flow line is steeper, while the shearing stress intercept is changed only slightly; hence the values for mobility are now higher while the yield values are changed only by a small amount.

If it were necessary to make this kinetic energy correction for each point in each run, the calculations would be too long and the procedure too complicated for control work. Therefore, a capillary is selected which has a diameter small enough to make the rate of flow so low that the kinetic energy correction is negligible. In some of our runs the flow was 1.0 to 0.75 cc. per sec. during the first half of the runs. When the initial rate of flow is not greater than 0.25 cc. per sec. the kinetic energy correction becomes negligible, *i. e.*, about 1.5% at the start and then rapidly decreasing.

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STUDIES ON THE STANDARDIZATION OF GERMICIDES.*[1]

(PRELIMINARY REPORT.)

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Disinfection is one of the main branches of hygiene. To employ chemical disinfection successfully we have to know the properties and, first of all, the efficiency of the different germicidal agents. The standardization of germicides is thus the basis of the subject. This procedure is carried out by exposing bacteria to chemical agents of different strengths and by observing the time necessary to damage or destroy these organisms. The task may appear, at first sight, rather simple as bacteria are organisms representing a low form of life with rather well-known biological characteristics. The experimental conditions would be, therefore, apparently in our hands. Closer familiarity with the subject teaches, however, that there are considerable difficulties to overcome. When we expose bacteria to the action of disinfectants we have to deal in the main with three factors: Germicides, bacteria and culture medium. The strength of the disinfectant we can change readily, but it is not easy to control the experimental conditions presented by the bacteria. We find not only differences in the vitality of the various types of bacteria and great contrasts between spores and vegetative forms, but there are also considerable differences in resistance depending upon the age of the culture. Even within the same culture the vitality of the individual organism may vary

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