# VISCOSITY "FLOW TIME" TEST.\*

# BY BERNARD FANTUS AND H. A. DYNIEWICZ.

There is need, it seems, for a simple viscosity test that can be performed by any pharmacist with inexpensive and readily available equipment. The difficulty experienced in securing uniformity in the official Ephedrine Jelly is illustrative of this need. Various specimens of tragacanth differ in their jelly-producing power to so great an extent that indicating the percentage of tragacanth which a preparation should contain by no means ensures the proper degree of consistency. What we need is to specify the quantity of "standard tragacanth" or other gel-producing colloid and the consistency aimed at; and to permit the pharmacist the determination of the actual quantity of the colloid he has that is required to secure the result aimed at.

As Middleton<sup>1</sup> and others<sup>2</sup> have shown "The viscosity of such colloidal sols and gels as tragacanth mucilage does not show true viscosity independent of rate of flow, but has the property of 'anomalous' or 'variable' viscosity, *i. e.*, the ratio between applied pressure (rate of shear) and rate of flow is not constant, but decreases rapidly as the rate of shear increases. It is, therefore, impossible to express the viscosity of a tragacanth mucilage by any single figure, but the conditions under which it has been determined must also be specified."

We believe we have elaborated a method so easily performed and with so simple an equipment that it may deserve to be better known. The principle underlying the test is the measurement of the "Flow Time," i. e., the time elapsing until the jelly has come to rest at a certain level; and measuring the angle this level assumes with the vertical and the time within which this level is assumed. With this method constant conditions are readily maintained as the rate of shear is practically constant.

### "FLOW TIME" TEST.

Square-cornered bottles (4-ounce size) with screw tops (Fig. 1)<sup>3</sup> should have a mark at the point to which 60 cc. of contents come. The bottle is filled to this point with the jelly that must have been homogenized and permitted to attain a specified temperature, e. g., room temperature (25° C.). The bottle is then placed upon its side and a stop-watch started at this moment.

The test requires recognition of three types of jelly: (a) "horizontal" jelly; (b) "angle" jelly; and (c) "vertical" jelly.

A. Horizontal Jellies.—If the jelly is thin enough to assume the horizontal or practically horizontal level, *i. e.*, that has made an excursion of 90° within an hour after the bottle has been laid on its side, we may speak of a "horizontal" jelly. The first observation is discarded, and subsequent observations are made in such a way that the same side of the bottle is always down. The bottle is then placed upright and permitted to stand thus as long as it took to acquire the level of equilibrium. A sufficient number of tests is then made to secure a fair degree of uniformity in the time intervals, and these are averaged and reported, *e. g.*, Flow Time = 90° in 1 sec., or Flow Time = 90° in 7 min., etc.

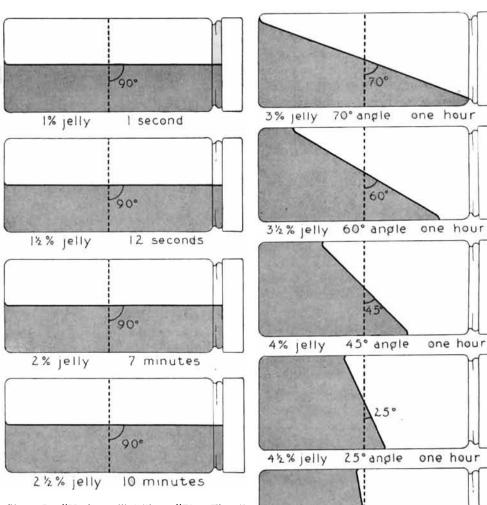
B. Angle Jellies.—With denser jellies, when the horizontal level is not reached within an hour, the observation is ended at this time and the angle of deviation from the vertical is noted.

\* From the Laboratory of Pharmacology and Therapeutics, University of Illinois, College of Medicine; and assisted by a grant from the AMERICAN PHARMACEUTICAL ASSOCIATION.

<sup>1</sup> Middleton, G., YEAR BOOK PHARM., 9, 491 (1936).

<sup>2</sup> Brindle, H., and Brown, J. M., Ibid., page 161.

<sup>3</sup> One hundred twenty cubic centimeters (4 oz.), square wide-mouth bottle, screw top. Sargent's S9195.

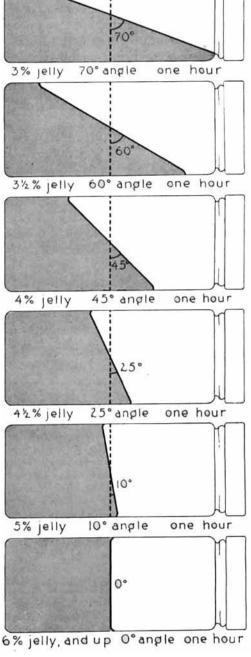


Thus, when the surface of the jelly has traveled within the hour to 45°, it is recorded as: Flow Time =  $45^{\circ}$  in 1 hour.

Chart I .-- "Horizontal" jellies. "Flow Time."

C. Vertical Jelly is the term we propose to be applied to those gels the surface of which has not moved, *i. e.*, remained vertical by the end of the hour's observation period during this test. If the vertical level of the surface is unchanged at the end of the hour, we speak of a 0° or a vertical jelly which is recorded as: Flow Time =  $0^{\circ}$ in 1 hour.

To determine the least amount of tragacanth required to prepare a vertical jelly with a particular quality of tragacanth, a fresh jelly with reduced quantity of tragacanth needs to be prepared in these tests as e.g., a 50% dilution of the prepared jelly gives a different result from a Chart II.—"Angle" jellies, 1 hour "Flow Time."



May 1939

freshly prepared jelly with one-half the quantity of tragacanth employed. The denser the jelly and the greater the degree of dilution the more erratic is the result.

By means of this test not only is it possible to distinguish between the colloidality of different qualities of tragacanth, but also to detect adulteration of tragacanth with less jellyfying material.

To prevent pellicle formation, all observations with a certain jelly should be made within a few hours.

Table I illustrates the method of recording of the result actually secured with a certain quality of tragacanth, as well as the fact that this test is capable of distinguishing between increments of 1/2% in strength between certain limits.

TABLE I.—"FLOW TIME" TEST FOR A CERTAIN RIBBON TRAGACANTH.

#### (Cf. Chart I and II.)

 $1\% = 90^{\circ}$  in 1 second  $1^{1}_{4}\% = 90^{\circ}$  in 12 seconds  $2\% = 90^{\circ}$  in 7 minutes  $2^{1}_{2}\% = 90^{\circ}$  in 10 minutes  $3\% = 70^{\circ}$  in 1 hour  $3^{1}_{2}\% = 60^{\circ}$  in 1 hour  $4\% = 45^{\circ}$  in 1 hour  $4^{1}_{2}\% = 25^{\circ}$  in 1 hour  $5\% = 10^{\circ}$  in 1 hour  $6\% = 0^{\circ}$  in 1 hour

Jellies, like gelatin, that have a higher melting point than room temperature are tested at a uniform temperature that secures proper consistency for flow time measurement and the temperature statement is included in the report, *e. g.*, Flow Time =  $90^{\circ}$  in 7 min. at  $37^{\circ}$  C.

### OFFICIAL SPECIFICATIONS FOR FLOW TIME TEST.

Should this test be found acceptable by our official formularies, some such specifications as the following might be proposed to be included in the monograph, *e. g.*, the one on tragacanth.

"Standard tragacanth should have the following jelly-producing quality: 4% of the tragacanth made into a jelly in accordance with specifications under test for identity and purity should yield a jelly that has a flow time test of  $45^{\circ}$  in 1 hour (or not less than  $40^{\circ}$  nor more than  $50^{\circ}$ )." Specimens having other flow time test require, to be official, a statement of the deviation from the standard.

Commercial specimens of tragacanth should bear the declaration of the relation of this tragacanth to the standard official tragacanth so that the pharmacist may know the relation of the quantity of this specimen to the tragacanth of official quality, he may have to use to secure approximately equal consistency.

#### CONCLUSIONS.

1. We find a simple flow time test a fairly accurate means of standardizing the consistency of jellies and of determining the percentage of tragacanth or of other gel-producing colloid contained in a jelly.

2. In consequence the jellyfying qualities of different colloids may be compared with each other.

3. The flow time test makes it possible to lay down official standards for tragacanth and other colloids.

4. This test makes possible to determine the effect of chemical and other influences upon tragacanth and other jellies.