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Apparatus for Determination of the Wiley Melting Point

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THE WILEY MELTING POINT is a frequently used analytical test in the shortening industry. Under the conditions of the determination this melting point is the temperature at which a sample fat disc, suspended in a liquid, assumes a spherical shape as it is heated. One of the conditions specified in performing the determination is that the test tube containing the liquid-suspended sample disc is to be heated in a bath with constant agitation of the suspending alcohol solution. Constant, or nearly constant agitation is required in order that the fat disc receive a continual and steady heat increase corresponding to the constant heat rise of the bath in which it is immersed. This agitation, as described in the known methods, is provided by hand stirring with a thermometer, which, in turn, registers the temperature of the Wiley Melting Point.

It has been our experience however to find this hand-stirring phase of the method very tiring, time-consuming, and inconsistent. Consequently a mechanical stirring apparatus was developed to eliminate these objections and to make the determinations more accurate and reproducible.

It is the purpose of this paper to describe this apparatus and to show its advantages over the manual stirring technique.

Experimental

Description of Apparatus. The apparatus consists of a battery jar, bath vessel 12 in. in diameter and 12 in. high covered with a circular aluminum plate (Figure 1). This plate has six holes to accommodate six 40-mm.-diameter test tubes. At the top of each hole is a brass collar equipped with gears and a means of holding the test tubes in a vertical position. The gears are so situated that the gear teeth from one collar mesh with the gear teeth of the adjacent collar. A speed-reducing, electric motor, also equipped with a tooth gear, drives the collars holding the tubes at a rate of 26 r.p.m. This speed is determined by the number of teeth on the gears and the speed of the motor. Thus, by rotating the collars holding the tubes, all of the tubes are rotated at this same speed.

A multiple-arm, thermometer holder is situated above the tubes so that when the thermometers are in place, they extend into the test tubes near the side of the tube. Therefore, by holding the thermometers stationary and rotating the tubes, mild, steady agitation of the water-alcohol suspending solution is accomplished. The rotating speed of the tubes is fast enough to give adequate agitation without turbulence. At the rotating speed specified, the fat disc stays in a position near the center of the tube without coming into contact with either the outside of the tube or the thermometer.

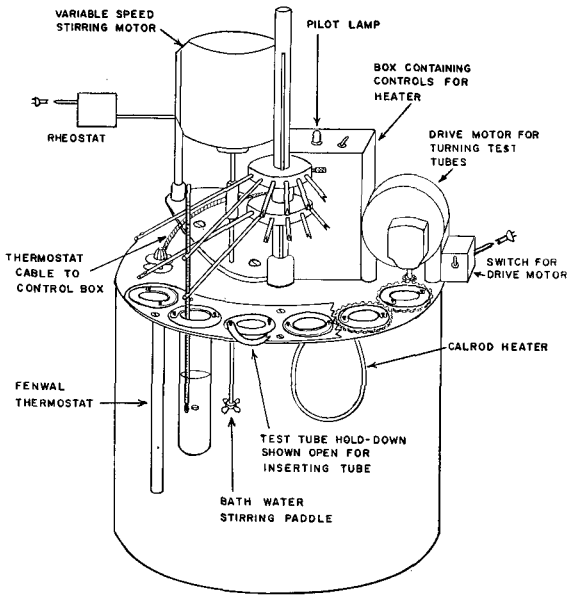

FIG. 1. Oblique drawing of apparatus. Four test-tube holders are shown cut away for clarity.

The water bath is heated with a 300-watt Calrod tube type of heater connected to the proper relays and a Fenwal thermostat. The thermostat serves only to prevent excessive heating in case the operator forgets to turn off the heat when the test is finished. With this heater the temperature rise in the bath is maintained at the necessary 0.2° to 0.3°C. per minute. Increasing the rate of heating gives erratic results because some time is required for a partially melted sample to assume its spherical shape.

Another electric motor placed above the bath and fitted with a shaft and propeller is used for agitation of water in the bath. Both motors and the bath heater operate from standard 115-volt AC power sources.

Test Procedure. With the exception of the method of stirring, the Wiley Melting Point determination when using this apparatus is performed exactly as set forth in A.O.C.S. Official Method Cc 2-38. Fat sample preparation, chilling of sample discs, solution preparation, determination of end points, and so forth are not changed or altered. When the prepared sample discs are ready for the actual determination, they are placed in the suspending solution in the usual manner. The thermometers are placed in the tubes with the bulb of the thermometer level with the sample disc, and the stirring motor and

TABLE I
Wiley Melting Points °C.[a]

Sample No.[b]	Tube 1	Tube 2	Tube 3	Tube 4	Tube 5	Tube 6	Stand. dev.	Ave.	Hand-operated bath
1	43.0	42.9	43.5	43.6	43.4	43.6	0.306	43.3	43.1
2	40.8	41.0	40.9	40.9	40.6	40.8	0.134	40.8	41.2
3	47.9	47.7	48.0	47.9	48.0	48.0	0.118	47.9	47.4
4	41.7	41.5	41.5	42.0	41.6	41.2	0.265	41.6	42.1
5	45.1	44.6	45.1	44.7	45.2	44.7	0.262	44.9	44.5
6	43.3	42.9	43.1	43.1	42.9	43.4	0.205	43.1	43.6

[a] Typical set of determinations. [b] Samples represent emulsified and non-emulsified shortenings.

heater are turned on. The analyst then needs only return to the apparatus when the temperaure of the solution within the tube approaches the end-point temperature and make the readings. This is one advantage of the apparatus since the analyst's time can be spent doing other things while most of the heating and stirring phase of the determination is being performed automatically.

Discussion

Testing of Apparatus. In order to determine the accuracy and reproducibility of results when using this bath, the Wiley Melting Points were determined on several sets of six shortening samples, which had been done by the hand-stirring procedure some months before. One determination on each sample of each set was made each day for six days. Each day the sample position was changed in the apparatus so that no two determinations on any one sample were obtained from the same position in the mechanical bath.

The comparison between these two methods (Table I) show differences well within the analytical error of the Wiley Melting Point test. Also the slight deviations shown between separate runs of any one sample, when using the mechanical bath alone, indicate that the reproducibility of results from the mechanical bath are equal to or better than results that one expects with the hand-stirring procedure.

Advantages of Apparatus. The advantages of this apparatus over the hand-stirring technique for the Wiley Melting Point determination are as follows:

1. With this mechanically stirred bath, more determinations can be run at one time by one operator. The number of tests was limited to six in this apparatus so that more constant surveillance of several samples having nearly equal Wiley Melting Points could be more accurately managed.
2. Less operating time is required in making a determination since the analyst needs only start the stirring apparatus to begin the heating and stirring phase and return to read the end points. The time between can be spent doing other things.
3. Being mechanical and self-operating, the apparatus eliminates the arduous and tiring stirring-operation for the analyst and also eliminates human variations in the stirring phase between two or more different analysts. These variations are often very evident when using the hand-stirring technique. With this apparatus steady, even agitation is continually applied to all samples.
4. A constant, steady even rate of heating is applied to the heating bath and thus to the sample dises, a condition which minimizes error in determining accurate end-points.

Summary

A mechanically stirred Wiley Melting Point bath apparatus is described, which makes the Wiley Melting Point more accurate and reproducible, permits more determinations to be run at one time, and is less time-consuming than the hand-agitation procedure of the A.O.C.S. Method.

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The State of Dispersion of Detergent Additives in Lubricating Oil and Other Hydrocarbons[1]

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THE STATE of dispersion of detergents in hydrocarbon solvents has long been a subject of interest. Only within recent years however has appreciable experimental evidence become available on the detailed nature of such dispersions. A recent article by Singleterry (10) who, with his coworkers at the Naval Research Laboratory, has made highly important contributions to this subject over the past several years, adequately summarizes the present state of knowledge. The available evidence indicates that soaps and detergents commonly exist in hydrocarbon solvents in the form of micelles. These micelles usually contain less than 50 molecules but, in some cases, may contain up to several thousand. It is the purpose of the present paper to demonstrate in greater detail the state of dispersion in which detergent additives are

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