VISCOSITY, INHERENT (One Point)

PRINCIPLE

A weighed starch sample is dispersed in sodium hydroxide solution using a standard technique. Relative viscosity of the sample dispersion is determined by measuring the efflux times of the dispersion and of the solvent with a capillary viscometer. The inherent viscosity is calculated as the quotient: natural logarithm of relative viscosity divided by concentration.

SCOPE

This method is applicable to unmodified, acid-modified and oxidized starches.

SPECIAL APPARATUS

1.   Viscometer: Use a Cannon-Ubbelohde viscometer, size 75, Catalog No. CUBU (Cannon Instrument Company, P. O. Box 16, State College, PA 16801).   For convenience, the viscometer should be equipped with a Cannon Instrument Company Catalog No. N 120 NEOPRENE rubber holder.

        New viscometers should be scrupulously cleaned and dried before using. Fill the viscometer with chromic acid cleaning solution and let stand two hours at room temperature. Drain and rinse with a large volume of purified water to insure complete removal of acid. Attach viscometer to a vacuum source by means of rubber tubing, and draw clean air through unit until dry (10 mins. usually sufficient).

2.   Constant Temperature Bath: A stirred water bath operating at a temperature of 25 °C ± 0.1 °C is necessary. It should be equipped to hold the special beaker used for sample preparation, and the viscometer.

3.   Stirring Assembly: A constant-speed (shaft speed 750 rpm) motor equipped with a self-centering chuck and a hoop-type stirrer attached to a $\frac{5}{16}$-in. × 12-in. shaft is recommended. A resistance- or governor-controlled motor

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may be used alternatively. A 2-in. diameter, hoop-type propeller, Arthur H. Thomas Company, Catalog No. 9240-U, should be attached to the shaft.

4. Beaker: 400 mL tall-form beaker (e.g., Corning Catalog No. 1100, or Kimble Catalog Nos. 14020 or 14050)

5. Timer: A stopwatch or electric timer with a precision and accuracy of ± 0.1 sec. over a range of 10 mins. is essential.

6. Funnel: Buchner, 6.0 cm, coarse porosity fritted disc (e.g., Corning Catalog No. 36060-150C)

REAGENTS

1. Sodium Hydroxide Solution: Carbonate-free

    a. Stock Solution, 50%: Cautiously add 1 L of purified water to 1000 g of sodium hydroxide pellets and stir until solution is complete. Allow to stand in a rubber-stoppered, borosilicate glass bottle until supernatant solution is free of carbonate haze (about 10 days), or filter through glass wool after the solution has cooled to room temperature.

    Reagent grade 50% sodium hydroxide solution may be purchased and used alternatively.

    b. Sodium Hydroxide Solution, 2.00 *M*: Standard. Dilute appropriate volume of stock solution with freshly-boiled purified water. Standardize with potassium acid phthalate using phenolphthalein indicator.   By addition of stock solution or water, adjust concentration 2.00 *M* $\pm$ 0.02 *M*.

    c. Sodium Hydroxide Solution, 1.00 *M*: Standard. Transfer by pipet 100.0 mL of 2.00 *M* sodium hydroxide to a 200 mL volumetric flask. Dilute to volume with freshly-boiled purified water. Prepare fresh as

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> needed and whenever new 2.00 *M* standard solution is prepared for sample dispersion.

SAFETY

Person(s) performing this method should wear appropriate protective equipment. Glassware should be carefully inspected for defects before use. Analysts should also review the Manufacturer's Safety Data Sheets (MSDS) for all of the chemicals required; however this review is not a substitute for adequate laboratory safety training.

PROCEDURE

If necessary, grind sample completely through a laboratory cutting mill to 20 mesh or finer. Determine dry substance by a standard procedure.

Weigh a starch sample containing 1.000 g of dry substance and transfer quantitatively to the 400 mL tall-form beaker. Add 100.0 mL of purified water at 25 °C, and fix beaker in constant temperature bath so that bath liquid will be above starch solution level after the addition of sodium hydroxide solution. Lower stirring assembly and fix in position so that hoop-type propeller is centrally located about 1/8" above beaker bottom. Start stirring motor and continue stirring until starch is dispersed (2 mins. stirring is usually adequate). Continue stirring and add, by pipet, 100.0 mL of 2.00 *M* standard sodium hydroxide solution at 25 °C. During addition hold pipet against the inside of beaker so that standard sodium hydroxide solution is dispensed at a relatively slow rate; continue stirring for 30 mins. after addition is complete (Note 1). Remove stirring assembly and cover beaker with a watch glass to prevent evaporation. Filter by gravity through a 6.0-cm coarse porosity fritted glass funnel.

Attach a short length of rubber tubing to the upper capillary of the clean and dry Ubberlohde viscometer. With the aid of the special rubber holder, fix the viscometer in a vertical position in the constant temperature bath so that the bath liquid is above the meniscus bulb. Transfer approximately 10 mL of the filtered starch solution to the charge bulb. Cover vent with finger, apply suction to rubber tubing attached to upper capillary, and draw starch solution into meniscus bulb (Note 2). Remove suction and finger and allow solution to drain through meniscus and efflux bulbs. Repeat operation of drawing solution into meniscus bulb and allowing to drain from meniscus and efflux bulbs.

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Again cover vent with finger and apply suction to draw solution into meniscus bulb. Remove suction and finger, allow solution to flow from meniscus and efflux bulbs, and determine accurately (within 0.1 sec.) the time (t) required for the solution meniscus to flow from the upper timing mark to the lower timing mark. Repeat this operation to obtain a series of three readings. Calculate the mean of these values (Note 3).

Drain starch solution from the viscometer and rinse with purified water. Rinse with 1.00 *M* sodium hydroxide solution and again with a large volume of purified water, and dry. If evidence or doubt suggests that the viscometer may not be clean at this point, fill with cleaning solution (chromic acid or equivalent and let stand 2 hrs. at room temperature. Drain cleaning solution, rinse pipet with a large volume of purified water, and dry.

Position viscometer in constant temperature bath as before, and transfer approximately 10 mL of 1.00 *M* sodium hydroxide solution into the charge bulb. Draw solution into meniscus bulb and determine flow time (solvent efflux time **t_o**) by procedure outlined in preceding paragraphs.

CALCULATION

The procedure provides efflux times for the solvent (t_o) and starch solution (t) at a dry substance concentration of 0.500 g/100 mL.

Calculate relative viscosity (0_{rel}): $0_{rel} = t/t_o$

Inherent viscosity is defined as the natural logarithm of the relative viscosity divided by the concentration. Then,

$$\text{Inherent Viscosity } (\eta_i) \text{ dl/g} = \frac{\ln(\eta_{rel})}{c} = \frac{2.303 \log(\eta_{rel})}{0.5}$$

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NOTES AND PRECAUTIONS

1. At this point, the starch dispersion should appear uniform and free of lumps. The presence of incompletely dispersed starch lumps is a valid reason for rejecting a determination.

2. Differences in construction of pipets may cause some trouble by allowing bubbles to rise in the capillary when suction is applied, particularly when the arm rising from the base of the capillary is fastened close to the top of the bulb below the capillary. To prevent bubble entrapment, it is advisable to such some of the sample partially up the pressure adjustment arm before the dispersion is raised in the main capillary.

3. The viscosity of the starch dispersion decreases slightly during this period. The rate of decrease, however, appears to be uniform and the mean of three successive readings is a reproducible result.

METHOD HISTORY

Corn Starch (Unmodified), Viscosity, Inherent (One Point) (B-61), Date of Acceptance 5-20-1963, Revised 4-02-2003.