CAUTION:
This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Material Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this method, the user must determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

Viscosity of starch and starch products
(WITHDRAWAL of T 676 cm-08 – No changes from Draft 01)

1. Scope

1.1 This method is for measuring the viscosity (consistency) of starch suspensions or solutions by means of an instrument which automatically records the viscosity characteristics while the starch suspension is cooked through a controlled heating cycle and cooled through a controlled cooling cycle. In addition, viscosity stability, breakdown, and characteristics of the cooled past (setback) can be determined while holding the cooked starch for extended periods at high or low temperatures, respectively.

1.2 This method is applicable to unmodified starches and to modified types (oxidized and thin-boiling) and starch derivatives from cereal grains and tubers.

2. Summary

The instrument used for this determination employs a rotating cup in an air bath, driven at constant speed by a
A circular metal disc with several metal pins projecting vertically downward into the cup serves as the sensing element. Rotation of a starch suspension in the cup exerts a force on the sensing element which is dynamically balanced by a calibrated torsion spring. Application of this restoring force is accompanied by an angular deflection of the sensing element shaft, and this deflection is recorded continuously in torque units of cm-g on a strip-chart recorder during testing.

3. Apparatus

3.1 Viscometer\(^1\) (Fig. 1), consisting of a variable speed instrument permitting measuring and recording apparent viscosity in Brabender units (B. U.) at fixed or constantly varying temperatures between 20° and 97°C (68° and 206°F). A Brabender unit is expressed in cm-g. Under the conditions specified, 1 cm-g is equivalent to 2.93 centipoises (2.93 mN s/m²).

**NOTE 1:** Instrument range can be extended by the use of zero point suppression weights and by substitution of interchangeable sensitivity cartridges (Table 1).

**Table 1.** Starch data

<table>
<thead>
<tr>
<th>Kind of starch</th>
<th>Concentration of starch, g d. b./500 g suspension</th>
<th>Approximate maximum viscosity(^a), Brabender units</th>
</tr>
</thead>
<tbody>
<tr>
<td>Corn</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Unmodified</td>
<td>35</td>
<td>500-600</td>
</tr>
<tr>
<td>Moderately thin boiling</td>
<td>45</td>
<td>400-500</td>
</tr>
<tr>
<td>Thin boiling</td>
<td>55</td>
<td>300-400</td>
</tr>
<tr>
<td>Very thin boiling</td>
<td>160</td>
<td>400-500</td>
</tr>
<tr>
<td>Wheat, unmodified</td>
<td>45</td>
<td>300-400</td>
</tr>
<tr>
<td>Tapioca, unmodified</td>
<td>30</td>
<td>700-800</td>
</tr>
<tr>
<td>Potato, unmodified</td>
<td>15</td>
<td>600-700</td>
</tr>
</tbody>
</table>

\(^a\)maximum viscosity is peak viscosity reached in cooking cycle. No supplementary weights are added. Sensitivity cartridge 700 cm-g.

**NOTE 2:** Instrument parts and accessories are identified in Fig. 1. Specimen temperature control is accomplished with a heating element and cooling controlled by a mechanical thermoregulator. Temperature can be maintained at any predetermined level from 20° to 97°C, or it can be programmed to increase or decrease at a rate of 1.5°C per minute in this range.

\(^1\)Names of suppliers of testing equipment and materials for this method may be found on the Test Equipment Suppliers list, available as part of the CD or printed set of Standards, or on the TAPPI website general Standards page.
NOTE 3: Shear force can be altered by changing cups and sensing elements (stirrers) (pin style - low shear; paddle style - high shear). Shear rate on the variable speed models can be altered by changing rotation speed of the cup. Special cups are available for high temperature applications.

3.2 pH meter, a direct reading pH meter equipped with glass and calomel reference electrodes and a temperature compensator is recommended.

3.3 Drying oven, forced circulation type, operating at 105 ± 3°C (221 ± 6°F).

3.4 Miscellaneous glassware: beakers, 600 mL; graduated cylinder, 500 mL.

4. Calibration

4.1 Instrument preparation. Assemble, operate, and maintain the instrument as directed in the manufacturer's detailed instruction manual. Adjust the thermoregulator to 25°C and place the temperature program control level in the “UP” position. Place the 700-cm-g sensitivity cartridge in position and adjust so that the indicator pen rests on zero on the strip chart. Set the timer switch at 45 min. Set the “COOLING” control switch in the center or “OFF” position and place the solenoid-controlled cooling probe in the “DOWN” position. The water-cooled sample cover is made inoperative by placing a clamp on the input hose. No weights are added to the zero suppression device.

4.2 Calibration. Weigh 40 ± 0.003 g of the calibration starch (obtainable from the instrument manufacturer) and transfer quantitatively to a 600-mL beaker. Weigh 420 ± 0.5 g of distilled water into another beaker. Add about one-half of the water to the starch sample and mix thoroughly to obtain a homogeneous suspension. Pour suspension into cup of viscometer. Rinse beaker with remaining water and add washings to suspension. Turn on the instrument.

4.3 At the end of the 45-min heating period (92.5°C), switch the thermoregulator program control lever to the center or “NEUTRAL” position, and reset the timer for 15 min. The calibration run is complete at the end of this period.

4.4 The maximum observed viscosity obtained during the heating portion of the calibration run should agree within ± 20 chart units of peak viscosity shown on the viscosity curve supplied with the calibration starch. If it does not, loosen the two Allen set screws on the chuck attached to the center shaft, and raise or lower the chuck to decrease or increase, respectively, the indicated viscosity. Tighten the set screws and reanalyze the calibration starch. Repeat this process until the observed peak viscosity agrees within ± 20 chart units of the peak viscosity shown on the viscosity curve supplied with the calibration starch.
Fig. 1. Schematic diagram of viscometer. The container cup (1) and the stirrer (2) are made of stainless steel. The stirrer is connected to a highly sensitive interchangeable measuring spring cartridge (3). The bowl is rotated at a uniform speed (4) and the stirrer deflects, depending upon the viscosity of the test material. The resistance encountered is transmitted to the spring system and continuously recorded by the recording mechanism (5). The container is heated by a radiant source (6). Temperature inside the container is controlled by a thermoregulator (7) extending into the material. Cooling is effected by a cooling probe (not shown) immersed in the material. A solenoid valve controls the supply of water from the tap. The thermoregulator is set to the desired temperature. When increasing or decreasing temperatures during a test, a synchronous motor (not shown) drives the thermoregulator up or down by the gear train (8). A pilot light (9) indicates heating. The main switch (10) serves to turn the instrument on and off. The entire system is programmed by a presetting timer (11) which monitors the test run, automatically shuts off the system, and triggers an alarm buzzer, indicating test completion.

4.5 The time at which the maximum viscosity is reached during analysis of the calibration starch should agree within ± 1.5 min of that shown in the calibration curve. If it does not, check the reliability of the thermoregulator. Pour 500 mL of water into the viscometer cup, adjust the thermoregulator to 50°C, and allow the instrument to run until the red heat-indicating pilot light goes out. Turn off the instrument, raise the measuring head, and measure the water temperature. Repeat this process with the thermoregulator adjusted to 70°C and 90°C. If water temperatures do not agree within ± 2°C of the thermoregulator settings, the thermoregulator should be replaced.
5. Reagents and materials

5.1 Calibration starch, together with supplier's calibration curve.

5.2 Distilled or deionized water

6. Test specimen

6.1 Obtain a sample of the product batch or shipment in accordance with TAPPI T 657 “Sampling of Fillers and Pigments.” Grind sample, if necessary, taking precautions to avoid any change in moisture content. Determine the moisture content of the sample in accordance with TAPPI T 658 “Properties of Diatomaceous Silica.”

6.2 Weigh to the nearest 0.005 g, a specimen of the weight (dry basis) specified in Table 1 for the particular type of starch being tested.

7. Procedure

7.1 Transfer the specimen quantitatively to a tared 600-mL beaker, add about 300 mL of distilled water, and stir thoroughly to obtain a homogeneous suspension. Determine the pH of the slurry with a standardized pH meter, and record it. Make any required pH adjustments at this point, and redetermine and record the value. Add distilled water to bring the total suspension weight to 500 ± 0.5 g and mix thoroughly to obtain homogeneity. Transfer the specimen suspension to the instrument cup. With the stirrer-sensing element in position and with the cooling probe in the “DOWN” position, carefully lower the instrument head into the operating position.

7.2 Start the motor with the thermoregulator program control lever in the center or “NEUTRAL” position, and with the heater and cooling coil switches in the “OFF” positions. Adjust the thermoregulator to read 50°C; set the temperature program control switch in the “UP” position. Heating and recording of apparent viscosity proceed automatically. Note and record the temperature at which gelatinization begins (the point at which viscosity begins to increase).

7.3 As soon as the temperature reaches 95°C (requiring about 30 min), as noted on the thermoregulator, move the temperature program control switch to the center or “NEUTRAL” position. Continue heating and recording apparent viscosity for one hour. At the end of 1 h, move the temperature program control switch to the “DOWN” position and turn on the cooling water. Cool the cooked specimen to 50°C (requiring about 37 min) while recording apparent viscosity. As soon as temperature reaches 50°C, move temperature program control switch to center or “NEUTRAL” position and turn off cooling water. Continue stirring and recording apparent viscosity for 1 h. Then turn off all instrument components, remove cup with specimen, and determine and note pH of cooked and cooled specimen paste.

7.4 Remove the strip chart record from the instrument. Clean the cup, agitator-sensing element and ink well, and reposition in the instrument.
7.5 Preload device. The range of any particular cartridge may be extended by hanging weights on the line of the preload device using Table 2 as a guide.

Table 2. Weights of indicated apparent viscosity (Brabender units)

<table>
<thead>
<tr>
<th>Cartridge sensitivity, cm-g</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
</tr>
</thead>
<tbody>
<tr>
<td>350</td>
<td>500</td>
<td>1000</td>
<td>2000</td>
<td>...</td>
<td>...</td>
</tr>
<tr>
<td>700</td>
<td>250</td>
<td>500</td>
<td>1000</td>
<td>...</td>
<td>...</td>
</tr>
<tr>
<td>1000</td>
<td>...</td>
<td>...</td>
<td>500</td>
<td>1000</td>
<td></td>
</tr>
<tr>
<td>2000</td>
<td>...</td>
<td>...</td>
<td>...</td>
<td>250</td>
<td>500</td>
</tr>
</tbody>
</table>

7.6 For a hot viscosity determination, the test is entirely automatic. By arranging the conditions of the test, the viscosity characteristics of the starch are given by a continuous graph from which the following information can be read: (a) time required for the initial viscosity rise and, by reading the thermometer in the paste, the temperature at this point; (b) gelatinization range: the time and temperature change from the initial viscosity rise to the maximum viscosity; (c) maximum viscosity; (d) viscosity at all times during any desired period of cooking, thus showing the effect of heat and agitation.

8. **Report**

Report, in Brabender units, the maximum viscosity, the viscosity upon reaching 95°C, and state the concentration used. Include other data as desired.

9. **Precision**

The precision of this method has not been investigated, but see Additional Information 11.4.

10. **Keywords**

Starch, Viscosity
11. Additional information

11.1 Effective date of issue: To be assigned.

11.2 This method, formerly T 676 su-79, was reaffirmed as Classical in 1997 by the responsible committee.

11.3 The viscosity of a liquid system is its resistance to flow. It may be regarded as fluid or internal friction. While starch solutions are often spoken of as having viscosity, they do not have the true (Newtonian) viscosity of a perfect fluid. The so-called viscosity of a starch solution is the combined effect of a number of inherent properties which cause it to deviate from Newtonian viscosity. These effects occur from the residual granule particles or from new colloidal groupings that take place as a result of cooking the starch. Many starches possess a varying degree of thixotropy, which is the property of becoming thinner with agitation or when under shear. In a starch paste, the observed viscosity depends upon the rate of shearing to which it is subjected in the viscometer; in a thixotropic paste, the greater the rate of shearing, the lower the viscosity.

11.4 Viscosity data obtained for any starch are dependent upon the arbitrary selection of conditions of preparing the paste. Some types of starches reach a relatively stable viscosity, while with others the viscosity tends to decrease on continued heating, eventually reaching a value near that of water. The time required for these changes to occur varies with different types of starches and the same cycle of variation is evident even when the same starch is prepared in different concentrations. Also, some starches have a much greater tendency than others to form organized structures or gels on cooking, which increases the viscosity. Variables, such as rate of heating, final maximum temperature, and rate and kind of stirring, cause different observed viscosities.

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department.