

T 226 cm-11

SUGGESTED METHOD – 1943

REVISED – 1965

OFFICIAL STANDARD – 1974

CLASSICAL METHOD – 1982

REAFFIRMED - 2001

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CAUTION:

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 test method, the user should 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.

Specific external surface of pulp

1. Scope

This method, based on the silvering method devised by Clark (1), is used to determine the specific exposed surface of unbeaten, or only slightly beaten, chemical pulps, groundwood, wood flour, and similar materials. It is not suitable for finely fibrillated or well-beaten fibers, since the silvering step causes some of the finest fibrils to collapse and hence lowers the exposed area (2).

2. Significance

2.1 Presently this is the most direct method of measuring the exposed surface of fibers. It may be used to determine the value of the constants in the formulas for quicker but less direct methods, such as those based on the Kozeny equation.

2.2 This method provides a measure of the degree of the average fineness of the material and, consequently, is greatly influenced by the quantity of debris present.

3. Definition

Specific external surface of pulp is defined as the exposed area in square centimeters per gram of moisture-free pulp.

4. Apparatus

4.1 *Reaction apparatus*¹, as shown in Fig. 1. The reaction vessel (A) is made from a 500-mL wide-mouthed Erlenmeyer flask. Two indentations (C) are made in opposite sides near the bottom to direct the swirling liquid

¹Names 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.

Approved by the Standard Specific Interest Group for this Test Method

downward. A glass tube (D) is inserted tangentially near the bottom at an angle of about 45° to hold a thermometer (E) in position so that its bulb is immersed during the reaction. Thermometer (E) is graduated from 0° to 50°C so as to permit estimation to 0.1°C . A tight rubber washer (F) on the stem of the thermometer prevents the bulb from touching the bottom, which would thus trap and hold the fibers.

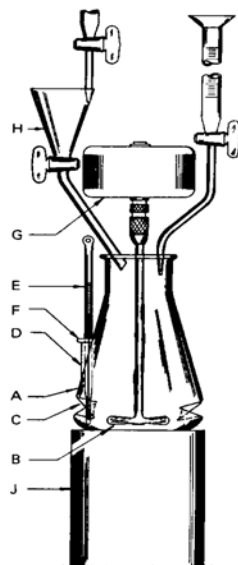


Fig. 1. Reaction apparatus.

4.1.1 A small vertical motor and chuck (G) is mounted to rotate the glass propeller shaft at 600 ± 50 rpm. The shaft carries a two-bladed glass propeller (B), 40 mm diameter, with blades set at an angle. It is important that the direction of rotation of the propeller be such as to cause a downward deflection of the liquid as it flows past the indentation in the reaction flask.

4.1.2 Also included are two burets of 25 mL capacity, preferably of the automatic zero type with bell mouths for easy filling, one with an extension to the delivery top; a removable support (J) for the flask; and a funnel (H) having a capacity of 30 mL or more, with a large-bore stopcock, or a funnel fitted with a short piece of plastic tubing and a pinch clamp, so that the contained liquid can be delivered quickly.

4.2 Hot plates, two, with analog or digital temperature control.

4.3 Stirrer, with a motor-driven, small glass propeller, mounted above one of the hot plates with its speed adjusted to approximately 300 rpm.

4.4 Crucibles, two 30-mL Jena I-I or Pyrex fritted-glass crucibles, of coarse porosity.

4.5 Container, for pulp suspension, 4000-mL beaker or a plastic bucket or bowl of similar capacity.

4.6 Dipper, approximately 200-mL capacity. Use a porcelain casserole if the container is a plastic bucket or bowl, or a plastic dipper if the glass beaker is used.

4.7 Stopwatch or stopclock, preferably 100 s/revolution.

4.8 Dissecting needle, of platinum or iridium.

4.9 Other apparatus: beakers, two 250-mL; rubber policeman; graduated cylinders, 25-, 100-, and 500-mL; thermometer, 0 – 120°C ; dark-stoppered bottle, 2000-mL capacity.

5. Reagents

CAUTION: As this method is based on a catalytic reaction, scrupulous cleanliness is essential. Distilled water of unquestionable purity must be used for preparation of all reagents and of the pulp suspension. The ordinary laboratory distilled water has been found unsuitable in many instances. Impurities in the distilled water are evidenced in formation of debris on heating the diluted silvering solution in the absence of fibers (a blank test) and in excessive clumping of fibers during silvering. If redistillation is required, add a small quantity of KMnO_4 to the water in the glass distilling flask prior to distillation. Precautions should be taken that none of the

KMnO₄ is carried over in the spray by using a distilling column or a flask with a long neck in which a loose plug of glass wool is inserted.

5.1 *Silvering solution.* Dissolve 200 g of reagent grade silver nitrate (AgNO₃) in 1500 mL of water. Add, with stirring, 160 mL of concentrated ammonia solution, NH₄OH. Then slowly add dilute NH₄OH (1: 9) until the brown precipitate first formed just disappears, avoiding addition of an excess. If the silvering solution is too alkaline (i.e., perfectly clear), very troublesome debris will be formed during the silvering, so that any excess ammonia should be neutralized by adding a little more AgNO₃ solution until a very faint cloudiness is visible. It is most important to prepare the solution correctly. Dilute with water to 2000 mL and store in a dark glass-stoppered bottle. This solution may be kept for periods up to 1 month.

5.2 *Potassium permanganate, 0.5N:* Dissolve 31.6 g of reagent grade KMnO₄ in about 1500 mL of water. Heat the solution at slightly below the boiling point for about an hour. Filter through glass wool to remove any manganese dioxide and dilute to 2000 mL. This solution need not be accurately standardized.

5.3 *Hydrogen peroxide, 0.5N:* Measure 25 mL of 30% H₂O₂ solution into about 750 mL of water containing 4 mL of 2N H₂SO₄. Dilute to approximately 1000 mL. Adjust the concentration so that 24.5-25.0 mL of H₂O₂ solution are equivalent to 25.0 mL of KMnO₄ solution. This adjustment must be made daily.

5.4 *Sulfuric acid, approximately 2N:* Dilute 53 mL of concentrated H₂SO₄ to 1000 mL.

5.5 *Sodium borate buffer solution.* Add 25 g sodium borate (Na₂B₄O₇ • 10H₂O) to 400 mL of warm water containing 20 mL of 1N NaOH and dilute to 500 mL. Dilute 100 mL of this solution to 2000 mL.

5.6 *Nitric acid, 1 + 1 solution, HNO₃.*

5.7 *Binder-free glass microfiber filters type 934-AH,* made into a slurry with water to a consistency of approximately 3 g of glass filter/100 mL.

6. Sampling and test specimen

6.1 For air-dry pulp, weigh out a portion of approximately 1 g. Completely defiber in distilled water with a minimum amount of agitation and stirring. It is recommended that the weighed sample be soaked for an hour or more and then defibered by gently rubbing moist portions between the palms of the hand covered with rubber gloves or by stirring as gently as possible.

6.1.1 If the sample is received in slush form, defibering is unnecessary.

6.2 Filter an amount of slush pulp equivalent to approximately 1 g of moisture-free material through a coarse-porosity, fritted-glass crucible (do not apply suction until a mat is formed); wash the residue thoroughly with boiling hot, distilled water.

6.3 Remove as much as possible of the pulp pad from the crucible with the platinum needle. Add this to a beaker containing 100-150 mL distilled water, and boil for 5 min. Filter the suspension again through the crucible, wash the pad again with the hot distilled water, then place it in a 4000-mL beaker or plastic container.

6.4 Add enough distilled water to the part of the pulp in the container to give a consistency such that a volume of 110-140 mL will require 3-8 mL of KMnO₄ solution for the back titration. This volume is the most suitable for the reaction vessel. The proper consistency should be determined by making a preliminary specific surface determination by the procedure outlined below. It is desirable to have the volume of the pulp suspension about 2000 mL at this point, adjusting the concentration by adding more water or moist pulp. Table 1 may be used as a guide in estimating the approximate dilution of the pulp suspension.

Table 1. Estimates of dilution.

	Approximate area, cm ² /g	Desired consistency, grams of moisture- free pulp/1000 mL
Unbeaten pulp	7,000 - 15,000	0.25 - 0.15
Groundwood or slightly beaten pulp	15,000 - 35,000	0.15 - 0.05