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T 234

BALLOT NO. 03 - SARG

DRAFT NO. 01

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WORKING GROUP CHAIR Todd Bolton

SUBJECT CATEGORY Pulp Properties

RELATED METHODS See "Additional Information"

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.

Coarseness of Pulp Fibers

(Ten-year review of Classical Method T 234 cm-12)

1. Scope

1.1 This is a method by which the coarseness (weight per unit length) of the fibers in a pulp may be ascertained by counting the crossings of a known weight of fibers per unit area on a prepared slide over lines of known length. The crossings are counted with a microscope and mechanical stage, by traversing the slide over a series of 5-cm distances.

1.2 If it is also required to determine the length of the fibers in a sample in accordance with TAPPI T 232 "Fiber Length of Pulp by Projection," instead of using a microscope, the fiber crossings over lines of known equivalent length may be counted when the images of the fibers are projected on a screen as described in T 232.

2. Summary

Coarseness is defined as the weight per unit length of fiber expressed as milligrams per 100 m (or decigrams per 10 km) and called a decigrex, abbreviated to "dg." Numerically it is 11.1 times larger than the value of the common "denier" used for textile fibers. These units and the "weight factors" of various fibers as described in TAPPI T 401 "Fiber Analysis of Paper and Paperboard" should be proportional.

3. Significance

3.1 The coarseness of fibers has an important effect on many properties of paper. For example, if other factors are equal, a finer pulp will give a stronger, smoother and better folding paper (*1*). In the past, the average width of the fibers has been used for measuring this characteristic. Measuring the decigrex has several advantages over measuring widths since it is not only much easier and quicker but also includes the effects of fiber thickness, the size of the central canal (or lumen), and the density of the cellulosic material composing the fibers.

3.2 The coarseness expressed in decigrex decreases as the fibers are split with beating and may be used as a measure of the splitting effect of beating or refining equipment. Wood fibers, however, do not easily split apart lengthwise so that their increase in fineness with beating is minor and not nearly so marked as with cotton or, especially, with linen fibers.

4. Apparatus

4.1 *Microscope*, with crosshair or pointer in the eye piece and equipped with a mechanical stage, and magnification of 50 × or greater. (Alternatively, a projector, as described in T 232, may be used.)

4.2 *Sheet machine* with a suction device. The sheet machine described in Appendix A.2 of TAPPI T 205 "Forming Handsheets for Physical Tests of Pulp" is suitable. Since the normal suction of the drainage leg is inadequate to draw the water in the diluted specimen through a filter paper covering the forming wire, as is required, the upper part of the drainage leg of the sheet machine is connected with suction tubing from the petcock to the top of a large (2-4 L) suction flask, which serves as a trap. The large suction flask is placed on a bench or support level with or above the wire.

NOTE 1: The connection to the drainage leg is made preferably as follows: Unscrew the needle from the valve and file off the little delivery spout on the body of the valve. If desired, remove the valve and slightly turn down the body in a lathe so as better to accommodate the tubing.

4.3 *Classifier*, with 150-mesh screen. If unavailable, a small 150-mesh sieve may be used.

4.4 *Filter paper*, a smooth, toughened paper, 18.5-cm diameter.

4.5 *Spade stirrer* (see Fig. 1) for the sheet machine, having the top edge of its blade high enough above the 5-cm (2-in.) depth of the liquid in the sheet machine to prevent long fibers from being picked up over its top edge when it is withdrawn.

4.6 *Microscope slides*, preferably large size, 3.8 × 7.6 cm (1.5 × 3 in.).

4.7 *Bowl*, domestic type, stainless steel or polyethylene, about 25-cm (10-in.) diameter.

4.8 *Dipper*, a stainless steel ladle with smooth rim, 25-50 mL capacity.

4.9 *Büchner funnel*, about 9 cm (3.5 in.) diameter and suction flask.

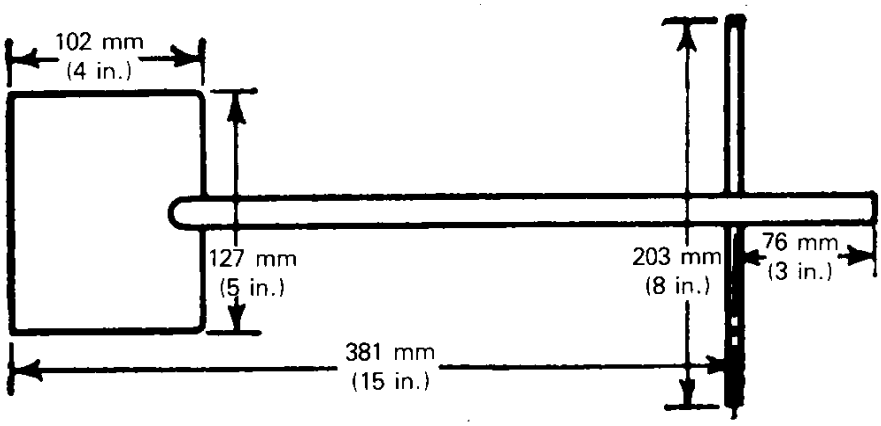

Fig. 1. Spade stirrer.

4.10 *Gooch-type crucible*, about 8-mL capacity with coarse, fritted glass disc.
4.11 *Adapter*, rubber, for Gooch crucible and suction flask.
4.12 *Evaporating dish*, 120 or 150 mL (or a 150-mL crystallizing dish or beaker).
4.13 *Graduated cylinders*, two each, 500 and 1000 mL; one 1000-mL cylinder, preferably stoppered.
4.14 *Other equipment*: 20 × 150 mm test tube with No. 3 rubber stopper; 1-L Erlenmeyer flask; microscope needle (steel); small weighing bottle (30 mL); wide-mouth china or plastic cup.

5. Materials

5.1 *Dye*, 1 or 2% solution of a red direct dye, such as Direct Red 23 (Color Index No. 29160).
5.2 *Salt*, 5% solution of NaCl.
5.3 *Cellophane adhesive* tape, 19 mm (3/4 in.) wide.
5.4 *Blotting paper*, e.g., squares for TAPPI T 205.
5.5 *Dispersant*, 0.5% solution of guar or locust bean gum with a few drops of formaldehyde or toluene added as a preservative.

6. Preparation of specimen slide

6.1 In order to take account of the fine material (see Additional Information), it is necessary first to determine its percentage in the pulp.

6.2 Disperse a representative sample of the pulp and dilute to 0.15% concentration, as described in T 205, from which a portion of about 1.5 or 2 g is required. Mix well, then, with a cup, transfer about 400 mL into a 500-mL graduated cylinder and about 900 mL into a 1000-mL cylinder, adding a quantity alternately into each cylinder. After recording the exact volumes in each cylinder, filter the contents of the 500-mL cylinder on a tared filter paper in the Büchner funnel, dry and weigh. Subsequently, calculate the dry weight of fiber in the 1000-mL cylinder.

6.3 Add the contents of the 1000-mL cylinder to the 150-mesh screen compartment of a classifier and classify for 5 min after the water commences going over the overflow. Alternatively, wash the sample on a small 200-mesh screen with a hose pipe for about a minute to remove the debris.

6.4 Collect the residue and dilute to 1000 mL. Pour into the bowl, mix well, and transfer a quantity of about 400 mL to a 500-mL cylinder. Read the volume accurately, filter on a tared paper in the Büchner funnel, dry and weigh. Calculate the concentration and the weight of the fibers originally in the bowl. Then calculate the percentage of debris washed away from the original sample.

6.5 After vigorously stirring the remaining contents in the bowl with the dipper, in one clean scoop, dip a 25-35-mL portion into a tared evaporating dish and scoop two similar dips into another tared evaporating dish and weigh each to the nearest 0.1 g. Knowing the exact concentration of the pulp in the bowl, calculate the dry weight of filters in the first dish to the nearest 0.5 mg. It should be about 35 to 45 mg.

6.6 Filter the contents of the first dish through the small fritted glass crucible. Pick out the small pad of pulp from the crucible with a microscope needle and use the pad as a swab to clean any fibers from the sides and bottom of the crucible and deposit the pad in the test tube. Similarly, filter the contents of the second dish and place

its pad in an accurately tared, small weighing bottle. Dry the pulp in the weighing bottle in an oven at 105 + 3°C (221 + 5°F). Stopper, cool, momentarily open the stopper to let in the air and weigh to the nearest 0.1 mg and dry again until its weight is constant. From the data, calculate and check the moisture-free weight of the pad placed in the test tube from the first dish.

6.7 *Dyeing*

6.7.1 To the pad in the test tube, add about 2 mL of the salt solution and 2 mL of the dye solution; then cautiously bring to a boil over a burner for a minute, shaking the tube intermittently. Set up the small suction flask and adapter with the 8-mL fritted glass crucible. Add about 5 mL of water to the test tube, shake, and pour the dyed fibers into the crucible and rinse out the test tube several times with a few milliliters of water until clean. Apply the suction and continue to add cold water for a short time until the filtrate is colorless. With the microscope needle, lift out the pad in the crucible and put it in the test tube. Add a few milliliters of water to the crucible and use the needle to scratch any fibers from the bottom and sides of the crucible and pour them into the test tube. Repeat the cleaning procedure until no more fibers are dislodged, then add water to the test tube until it is about half full.

6.7.2 Invert the fritted glass crucible on the adapter, turn on the suction and pour water through the fritted disk to remove any debris before it dries in the pores.

6.7.3 Close the half-filled test tube with the rubber stopper and shake vigorously until all the fibers are seen to be completely dispersed. Add and rinse the contents of the test tube into a l-L stoppered graduated cylinder and add tap water until the concentration is exactly 50 mg of moisture-free fiber per liter.

6.8 *Measuring*

6.8.1 Stopper the cylinder and shake it vigorously, then, without pause, pour the entire contents into the bowl, but do not rinse the cylinder. Weigh a dry, clean, evaporating dish to 0.1 g. Stir the contents of the bowl vigorously with the dipper and with a couple of clean dips scoop out and add to the dish 50 g or more if the fibers are long, and up to 100 g if the fibers are short, e.g., from hardwoods. Reweigh the evaporating dish and contents and divide the net weight in grams by 20 to obtain the moisture-free milligrams of fibers it contains.

6.8.2 Add about 2 mL of the gum solution to the dish in order to secure a good dispersion of the fibers in suspension.

NOTE 2: Do not add too much gum because subsequent couching of the fibers will become very difficult.

NOTE 3: In order to make their surfaces water repellent and to make the handling of small volumes easier, occasionally ~~to~~ wipe the inside surfaces of the bowl, evaporating dish and dipper with a rag that has been moistened with a little dilute solution of silicone or a silicone treated wiping cloth (commonly available from sporting goods and firearms dealers).

6.9 *Depositing the fibers*

6.9.1 Open the cylinder of the sheet machine and, with a cloth, carefully wipe any fibers from the inside surface of the cylinder; then rinse it well. Connect the suction line from the drainage leg of the sheet machine to the large suction flask, then place it on the bench above the level of the wire.

6.9.2 Shut the drain valve and flood the wire with water to remove any air trapped beneath it. Lay the 18.5-cm diameter toughened filter paper on the wire, and with the fingers smooth the wet sheet to remove any trapped air or wrinkles. Close the sheet machine and from the l-L Erlenmeyer flask used as a rough measure, pour in from above about 0.75 L of water. Pour the weighed suspension in the evaporating dish into the machine and rinse it out with the remaining water in the flask.

6.9.3 With the spade stirrer, disperse and mix the fibers with four or five rocking strokes. Hold the spade stationary to stop any eddies, withdraw the stirrer and immediately apply suction to the large suction flask. When drained, open the sheet machine, remove the filter paper with the deposit of fibers and lay it with the fiber uppermost, on two dry sheets of blotting paper lying on a hard, flat surface.

6.9.4 From the roll of the transparent adhesive tape tear off about an 8-cm length. On a selected area of the filter paper, judged to have an average population of dyed fibers (actually the deposit should be quite uniform), place the tape with the sticky side down. Press it on to the filter paper by firmly passing a finger along it, then lift one end off. All the fibers in the covered area should then adhere to the tape. If not, replace the tape over the same area, press down firmly and try again; if still unsuccessful, transfer the filter paper to another dry blotter to reduce its moisture.

6.9.5 Cover the ends of the microscope slide with electrician's black plastic tape to expose a length in the center of the slide of exactly 50 mm. After waving the cellophane strip with its fibers in the air to dry off any excess moisture, place and press it down along the opening in the slide so that the tape itself acts as a cover glass. Prepare a second slide in the same way.

NOTE 4: If only the coarseness, and not the average fiber length by projection (T 232), is required, it is not necessary to make the slides. The filter paper and deposited fibers can be examined directly with the microscope.

7. Procedure

Place the specimen slide in position and bring the fibers into focus. Move the stage until the objective is over one edge of the black tape; then traverse the stage across the 50-mm opening beneath the objective and count each time the pointer passes through the center of a fiber. Count all the crossings on several traverses that are at least 2 mm apart on the slide until a total count of at least 300 and preferably 500 crossings has been made on the two slides. Record the number of traverses made and the number of crossings made along each.

NOTE 5: By dyeing the fibers comprising the specimens a red color they can be distinguished from any stray fibers that may have been included, or other or debris on the transparent adhesive tape.

NOTE 6: It is suggested that for the purpose of replication, a lower limit be established; in the case of unbeaten pulps, do not include the crossings of fibers which have a width of less than 2.5 μm. With beaten pulps or groundwood fibrous elements, do not include the crossings of fibers which have a width of less than 0.5 μm in the count. These limits have been chosen arbitrarily. Elements finer than these do not weigh very much and thus, unless unusually numerous, will not represent an appreciable percentage of the weight of the specimen.

8. Calculation

8.1 Let W be the weight in grams of the 50-mg/L suspension added to the sheet machine having an area of A mm^2 (20,000 for the TAPPI sheet machine); let N be the total number of fibers counted in F fields; let L be the actual length in millimeters of all the fibers crossing the scanned lines; and let G be the equivalent total length in millimeters of the lines on the slide which are examined in each single field.

NOTE 7: If the microscope is used and all the fibers are counted which cross the pointer during a traverse of G mm, F is the number of traverses made of G mm each.

8.2 $L = \pi N/2$ when the spacing of the grid lines is unity (5). The weight of the fibers per square millimeter of slide = $W/20A$ mg. The total effective area covered by the grid lines with unit spacing = FG mm^2 (the total effective length of the grid lines examined).

8.3 Then the coarseness, expressed as the weight of fibers in mg per 100 m (dg) is:

Coarseness = $(W/20A)\ (FG)\ (2/\pi N)\ (10)\ (100)\ (100)$

$$= \frac{10{,}000\ WFG}{\pi NA}$$

if

A = 20,000 mm^2
G = 50 mm
dg = 7.96 WF/N mg/100 m

If $E = FG$ = total effective length of scanned lines examined, dg = 0.159 WE/N. Alternatively, if w = mg of moisture-free fiber added to the TAPPI sheet machine: dg = 159 wF/N, if 50-mm lines are examined; otherwise dg = 3.19 wE/N.

8.4 In order to correct for the percentage of debris present in the sample, part of which adds slenderness to the pulp (see Additional Information) multiply the coarseness of the fibers in the specimen by (200 - D)/ 200, where D is the percentage of debris washed from the sample. This formula is equivalent to reducing the measured coarseness by one-half the percentage of debris.

9. Report

Report the coarseness of the fibers as milligrams per 100 m (decigrex) to three significant figures. State whether or not the result has been corrected for the presence of debris.

10. Keywords

Pulp, Fiber, Fineness

11. Additional information

11.1 Effective date of issue: To Be Assigned.

11.2 This method, formerly T 234 su-67, has been reclassified as a Classical Method. Such procedures are no longer in common use or have been superceded by advanced technology; they are technically sound, have a history of use, and contain a body of literature references that make their preservation valuable.

11.3 This method has been revised to describe a considerably improved and simplified technique for preparing the specimen and makes it especially suitable for use with a microscope.

11.4 If preferred, instead of the microscope, a projector as described in T 232 or an ordinary photographic enlarger or projector may be used to project the images of the fibers and count the crossings over lines on a screen of known equivalent distance.

11.5 Because it is not practical to include the very finest elements in a count of the fibers on a specimen slide, their presence presents a problem. This difficulty is intensified if the pulp has been beaten. If these particles have appreciable length as well as being slender, they will serve effectively to reduce the actual coarseness of the pulps as far as its influence on subsequent paper properties is concerned and so cannot be ignored. On the other hand, if they are short and chunky like the parenchyma cells in wood, they will not contribute to the actual slenderness of the pulp as a whole, that is, make any contribution to the folding qualities of paper made from the pulp. Accordingly, as a first approximation, the percentage of these fine particles in the sample is first determined and the measured coarseness of the remaining fibers is reduced arbitrarily by one-half this amount, as indicated in the last step of the calculations.

11.6 This method was devised originally by Clark (*2*), based on the thought that if the fibers in the specimen were very coarse, e.g., such as sawdust, with less than 1 mg on the slide, there would be no more than one or two crossings of images of the particles; whereas, if the fibers were infinitely fine, there would be a corresponding infinite number of crossings. Thus the number of crossings of the fiber images from a given weight of pulp on the slide should be proportional to their slenderness. The experimentally found value of approximately 0.8 for the proportionality constant l/k for the square grid was later derived on a theoretical basis as $\pi/4 = 0.785$.

11.7 In the article by Brady *et al.* (*2*), on page 41, column 1, line 7 up, $k = \pi/4$ should read $1/k = \pi/4$. In column 2, same page, the top and bottom values in the three-figure, right-hand column of the second unnumbered table should be transposed.

11.8 The values for a fine-fibered pulp are about 10, and coarse pulp about 30 dg. The 1958 TAPPI Reference Sulfite Pulp has a coarseness of 23 ± 2 dg, before reducing this estimate to account for the debris as suggested in this method.

11.9 According to one survey (*1*), the result is probably reliable to well within 10% if care is used and if sufficient fiber crossings are counted.

11.10 Ranger (*3*) has described a somewhat similar procedure by which he incorporates the dyed specimen fibers in a thin handsheet. He gives data for a wide variety of pulps.

11.11 An alternative procedure for transferring the fibers from the filter paper to the microscope slide is as follows: Coat the surface of the slide with a dilute solution of a pressure-sensitive adhesive, invert the moist filter paper, place over the slide on a smooth surface, use a small diameter roller to press into contact and remove. This procedure avoids the bubbles, etc., common in adhesive transparent tape and which during the count are difficult to distinguish from the specimen fibers unless the latter are dyed red. Such a slide is then suitable for an electronic automatic counter if available.

11.12 Another variation in the procedure is possible with the microscope if sufficient magnification is used to be able to distinguish clearly and count multiple fibers at a crossing point. Accurately weigh 10-50 mg of the sample on a sensitive balance and correct for moisture in order to obtain the moisture-free weight of the specimen. Clean the sheet machine cylinder thoroughly and cover the wire with a circle of deeply dyed, strong-wet, filter paper, after flooding the wire and closing the drain valve. Pour a few liters of water into the cylinder and add the known weight

of fibers after dispersing them well and adding 2 mL or more of the gum solution. Stir with a spade stirrer being careful not to remove any fibers, and, after turbulence ceases, drain. Dry the filter paper and attached fibers and count the fiber crossings on a line of known length, either marked on the filter paper, or superimposed with a reticule in the eyepiece. Make sure that all the crossings of the rather thickly populated fibers are included. Instead of using a dyed filter paper, the weighed fibers may be dyed as described and deposited on a white filter paper.

11.13 In the 2002 revision, the procedure has been simplified by the use of cellophane tape for couching (*4*), the projection procedure has been included as an alternate method, and the calculations have been adjusted to account for the debris present. The 2012 revision only included editorial changes.

Literature cited

1. Clark, J. d'A., *Tappi* **45** (8): 628 (1962).
2. Brady, C. T., Berzins, A., and Clark, J. d'A., *Tappi* **39** (1): 40 (1956).
3. Ranger, A. E., *Paper Technol*. **2** (2): 169 (1961).
4. Kindler, W., and Clark, J. d'A., *Pulp Paper Mag*. Can. **67** (1): 13 (1966); *Tappi* **49** (8): 356 (1966).

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