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OFFICIAL STANDARD – 1926
OFFICIAL TEST METHOD – 1982
CORRECTION – 1983
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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.

Fiber analysis of paper and paperboard

1. Scope

1.1 This method provides a procedure for the identification of the kinds of fibers present in a sample of paper or paperboard and their quantitative estimation. This method requires the analyst be skillful and experienced in the field of pulp and paper microscopy.

1.2 The analyst must make frequent use of standard samples of known fiber composition or of authentic fiber samples and must become thoroughly familiar with the appearance of the different fibers and their behavior when treated with the various stains.

1.3 Morphological characteristics help identify special fibers such as straw, flax, esparto, soft woods, such as southern pine, Douglas fir, western hemlock, and various species of hardwoods, so that the correct weight factors may be applied. A knowledge of morphological characteristics of the different fibers is essential for their identification. More information on this subject is given in the Appendices.

1.4 It is reported that fiber analysis in highly refined or secondary fiber sheets is very difficult to perform.

2. Summary

Details are presented for the disintegration of differing grades of paper, staining, preparation of slides, and fiber identification by specific staining techniques. Provision is made for both qualitative identification and quantitative analysis of furnishes.

3. Significance

Many types of paper, particularly bonds, ledgers, index, and book papers are bought on the basis of fiber composition. This method is used to quantitatively and qualitatively identify the fibers in paper. It will also show whether the composition is free from fibers which the specifications particularly prohibit.

Approved by the Standard Specific Interest Group for this Test Method
TAPPI

4. Apparatus and materials

4.1 *Microscope*[1], compound, preferably of the binocular type, equipped with a mechanical stage and Abbe condenser. A magnification of approximately 100 times is recommended for observation of fiber colors; a higher magnification may be desirable for studying morphological characteristics. If an apochromatic objective is used, it is desirable to have a compensating eyepiece and an achromatic condenser. A field of approximately 6 mm is desirable. The eyepiece should have a cross hair, pointer, or dot for counting the fibers passing under it. Such an eyepiece can be supplied by the manufacturer, or it may be prepared by the technician, positioning the point in the eyepiece so as to obtain its image in focus.

4.2 *Slides and cover glasses*, standard slides 25 by 75 mm (1 by 3 in.) of clear, colorless glass, and No. 2 cover glasses (25 mm square).

4.3 *Dropper*, a glass tube about 100 mm (4 in.) long and 8 mm (5/16 in.) internal diameter with one end carefully smoothed, but not constricted, and the other end fitted with a rubber bulb. The tube shall be graduated to deliver 0.5 mL.

4.4 *Warm plate*, a plate with a plain, level top having black matte finish and provided with a control to keep the temperature of the surface between 50 and 60°C.

4.5 *Dissecting needles*, two needles mounted in handles. Steel needles may be used but are subject to corrosion by some of the stains. Needles made from an alloy of platinum and iridium are preferred.

4.6 *Glass-marking equipment*, either a glass-marking pencil or an aluminum stearate solution (see Appendix G) for marking lines on the slide.

4.7 *Light source*, a 15-W "daylight" fluorescent tube or equivalent daylight source.

4.8 *Camel's hair brush*, small.

4.9 *Counter,* a device that counts the number of times a button is pressed. This can be used to assist in the counting of the fibers.

4.10 *Miscellaneous*, 50- or 100-mL beaker; test tube; glass beads; hot plate blotter paper; and, depending on the specimen, stains, reagents, and apparatus as described in the appropriate section of the procedure. A good dissecting knife may be helpful to separate the plies of cylinder board.

5. Reagents

5.1 *Graff "C" stain*, suggested for general analysis. Other stains, listed below, could be used for specific purposes or to confirm results obtained with the "C" stain.

5.2 *Herzberg stain*, especially useful to differentiate between rag, groundwood, and chemical wood pulps.

5.3 *Selleger's stain or Alexander's stain*, used to differentiate between softwood and hardwood pulp. Selleger's stain is also helpful in differentiating between bleached softwood sulfite and bleached softwood sulfate.

5.4 *Wilson's stain*, used in place of, or to confirm results with, the "C" stain.

5.5 *Green and Yorston stain*, very useful for the detection of unbleached sulfite fibers.

5.6 Directions for preparing these stains are given in Appendix E, and the directions for preparing and using other stains are given in Appendix F.

6. Sampling and test specimens

Select a single composite test specimen of approximately 0.2 g so as to be representative of all the test units of the sample obtained in accordance with TAPPI T 400 "Sampling and Accepting a Single Lot of Paper, Paperboard, Containerboard, or Related Product."

7. Procedures

7.1 *Disintegration*

[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.

7.1.1 *Ordinary papers*

7.1.1.1 (Method A) **CAUTION**: *It is recommended to handle paper with gloves as metallic salts on fingers may give reactions with stain*. Tear the specimen into small pieces and place in a small beaker. Cover with distilled water and bring to a boil on the hot plate. Decant the water from the beaker. Then roll the individual pieces into small pellets between the fingers and place in a large test tube. Add a little water and shake vigorously until the water is thoroughly absorbed by the paper. Add a little more water, shake well, and again add some water and shake. Repeat this procedure until the paper has been thoroughly disintegrated. When the paper has been completely defibered, dilute the suspension by discarding part of it and adding water to the remainder until the suspension has a final consistency of about 0.05%. Shake vigorously between these operations to prevent fiber stratification. If the specimen is difficult to disintegrate, glass beads may be used in the test tube, and should be so stated in the report. Glass beads should not be used if the fibers are to be examined for degree of beating. Ultrasonic disintegration is helpful in dispersing fibers. If most of the fibers have separated but a few undefibered areas remain, various methods can be used that have shown no noticeable fiber cutting or damage effect. In situations where incomplete separation of fibers occurs, an agitation method (proven not to cut the fibers) can be used.

7.1.1.2 (Method B) If the paper cannot be disintegrated by shaking in water (Method A), return the specimen to the beaker, cover with 1% NaOH solution and bring to a boil. Decant the alkaline solution. Wash the specimen twice with water. Cover the specimen with approximately 0.05*N* HCl; let stand several minutes. Decant the acid and wash the specimen several times with water (a fritted glass filter funnel is recommended to minimize small fiber loss). Roll into pellets and proceed as in Method A. Caustic will swell the fibers and may affect the subsequent stain shade.

7.1.1.3 (Method C) Some analysts prefer to start disintegrating the specimen in a heated 5% alum solution. After boiling 15 mins, the specimen is washed, rolled into a pellet, and suspended in a test tube of water.

7.1.1.4 If the specimen cannot be disintegrated by Method A, B, or C, use one of the special methods given below.

7.1.2 *Specially treated papers*. Standardized methods cannot be specified for the disintegration of papers containing tar, asphalt, rubber, viscose, etc., or parchment papers, because the needs vary according to the material, the amount present, and the nature of the treatment. The following methods are given as guides:

7.1.2.1 *Tar and asphalt-treated papers*

Method 1: Place the specimen in a dish. Cover with kerosene and digest on a steam bath for 1 h. Next, remove and press the specimen between blotters. Repeat the kerosene extracting and blotting. Extract the specimen with cold toluene or hexane until the solution is clear. No NaOH should be used in the final disintegration of these papers because of the possible presence of wool fibers (*1*).

Method 2: CAUTION: Vapors from this solvent are toxic. Work should be done under a hood. Fill several convenient containers (250-mL beakers) about one-half full with 1,1,1-trichloroethane. Cut the specimen into convenient squares and immerse in the first container. After several minutes in the first container, transfer the squares to the next container, using forceps. Do not allow the squares to dry. In the case of laminated papers, the sheets may be separated easily after the first or second soaking and this should be done, removing any fabric scrim which can be treated separately, if desired. Continue moving the specimen into fresh solvent until the liquid remains clear after the specimen has been agitated in it for several minutes, then remove the specimen and allow to air dry. After drying, disintegrate the specimen in the usual manner.

Method 3: Place the specimen in a Soxhlet or similar extractor and extract with 1,1,1-trichloroethane or 1,1,1,-trichloroethylene.

7.1.2.2 *Rubber-treated papers*. Extract the paper for 6 h in a Soxhlet extractor with toluene, dry, and then boil in water to which a little wetting agent has been added. In very rare cases, a 1% NaOH solution may be necessary. With most specimens, the toluene will extract about 98% of the rubber (*2*).

7.1.2.3 *Parchment papers*

Method 1: To 25 mL of water, slowly add 25 mL of concentrated H_2SO_4 and cool it to 50 to 60°C. Place the specimen in the acid. When the specimen begins to disintegrate, stir quickly while emptying into a 1000-mL beaker two-thirds full of water (*3*).

Method 2: Soak the specimen for about 5 mins in concentrated HCl. Wash, boil in 0.5% NaOH solution, and repeat this sequence if necessary. Then wash, acidify with dilute HCl, again wash, and then boil in a little water and a wetting agent, and disintegrate (*3*).

7.1.2.4 *Pyroxylin-treated papers*. Extract or remove the pyroxylin with ethylene glycol monoethyl ether, acetone, or amyl acetate.

7.1.2.5 *Wet-strength papers*

Method 1: Tear the specimen into small pieces and place in a beaker. Cover with 5% aluminum sulfate solution and boil from 5 to 20 mins depending upon the amount of wet strength present. Decant the solution, wash, and proceed as in 7.1.1.2.

Method 2: When an estimation of the degree of beating of the fibers is not required, the specimen may be disintegrated in water in a high-speed electric mixer for a period of less than 60 seconds.

Method 3: Alkaline-cured resin papers are repulped at a pH of 10 obtained with caustic soda at 38°C. As little as 0.1% of sodium hypochlorite on a fiber basis is effective in accelerated broke disintegration, as the amidochloride formed apparently hydrolyzes readily. Information on papers treated with polyethylene imide (PEI) (also considered to be an alkaline-curing resin) indicates that broke recovery is most satisfactory under acid conditions.

7.1.2.6 *Highly colored papers*. If the paper is highly colored (the treatment selected depends on the characteristics of the dyes), remove the dye by one of the following methods (a) By solution: use alcohol, NH_4OH, acetic acid, or HCl. (b) By oxidation: use nitric acid or bleach liquor (sodium hypochlorite). (c) By reduction: Use hydrosulfite, stannous chloride, or HCl and zinc (*1*) and then disintegrate by method A.

7.2 *Preparation of slides*

7.2.1 It is desirable to keep the slides and cover glasses in a 50% alcohol/water solution. After a slide has been dried and polished, draw lines 25 mm (1 in.) from each end, using the glass-marking pencil or aluminum stearate solution. This will keep the fiber suspensions inside the square at each end of the slide. (A repellent-type label tape may be used to cover the center square portion of the slide, in which case lines need not be made on the slide.) Remove any dust or lint from the slide with a small camel's hair brush. Place the slide on the warm plate, shake the test tube containing the defibered specimen, and withdraw a portion of the fibers by inserting the dropper and expelling two or three bubbles of air. Deposit 0.5 mL of the fiber suspension on the square on one end of the slide. Withdraw another 0.5-mL portion from the test tube and deposit it on the other end of the slide. Allow the water on the slide to evaporate until there is just sufficient left to float the fibers, then gently tap the suspension with a dissecting needle to distribute the fibers evenly inside the squares. Leave the slides on the warm plate until completely dry.

NOTE 1: A few drops of an acrylamide-type deflocculating agent added to the fiber suspension is very effective in many cases.

7.3 *Staining*

7.3.1 To use the Graff "C" stain, Herzberg stain, Selleger's stain, or Wilson's stain, apply three drops of the stain to the fiber field on the slide, then place a cover glass over the area in such a way as to avoid air bubbles. Allow the slide to stand one or two minutes, then drain off the surplus stain, by tilting the long edge of the slide into contact with a blotter.

NOTE 2: Take care not to touch the unstained fibers on the slide with the fingers. The fingers usually have metallic salts on them which may be absorbed and later will give rise to puzzling stain reactions.

7.3.2 The colors developed by the stains vary according to the raw materials and the processes used for preparing them. The following sections discuss the colors to be expected, but the analyst must check known samples to become familiar with their appearance.

7.3.3 *Graff "C" stain*. When lignin is present, a yellow color is developed with the "C" stain. Mechanical pulp gives a very vivid yellow with a tendency toward orange. Unbleached jute stains much the same color. These two fibers can be distinguished by their morphological characteristics. Unbleached pulps of all kinds tend toward the yellow, with the depth of yellow determined by the degree of cooking and the type of cook. Thus, a raw unbleached sulfite will stain a vivid yellow, but as the degree of cooking increases, it tends toward a greenish yellow. Unbleached sulfate tends toward yellowish brown, while an unbleached alpha is more brown than yellow. The hardwood pulps have a tendency to appear bluish and greenish even in their unbleached state. Abaca, cereal straw, bamboo, sugar cane bagasse, flax hurds, and esparto also give yellow colors with raw, unbleached cooks.

NOTE 3: "Hardwood" pulps are made from dicotyledons or broadleaved trees; "softwood" pulps are made from conifer trees.

7.3.3.1 When any pulp is bleached, it has a tendency to give a reddish hue with the "C" stain. In some cases this tendency is very slight, but any hint of red can generally be taken as an indication of some degree of bleaching. The shade of red usually indicates the type of bleached pulp. Thus, rag or cotton, which is the purest form of cellulose, gives the purest red, followed by bleached softwood alpha, bleached softwood sulfite, and bleached softwood sulfate in that order. The sulfite is weak enough in red so that it frequently appears purplish-gray. Alkali cooking tends to give a bluish color to wood pulp, so that with bleached softwood kraft pulp the blue coloration nearly overshadows the red and a

bluish-gray is seen. Hardwood pulps have a tendency to be bluer than softwood pulps. Hardwood alkaline pulps, even though bleached, show almost no red when stained. Unbleached hardwood alkaline pulps cannot easily be distinguished from the bleached pulps, nor can hardwood kraft pulp be distinguished from hardwood soda pulp.

7.3.3.2 Some special fibers lend their own colors to the system. Thus, abaca in the bleached state has a tendency toward purplish gray; bleached jute is a light yellow green; cereal straw, bamboo, sugar cane bagasse, flax hurds, and esparto tend toward bluish gray, and sometimes give colors like hardwood alkaline pulps. In these cases, the pulps must be distinguished by their morphology. A color chart showing the colors obtained with the "C" stain has been published (*4*).

7.3.4 *Herzberg stain*. Being an iodine stain, Herzberg stain produces the general color reactions discussed under "C stain." Herzberg stain produces much bluer colors than the "C" stain, so that all chemical wood pulps, whether bleached or unbleached, acquire a blue tint. Rag pulp stains pink and can be easily distinguished from chemical wood pulps. Mechanical pulp is a vivid yellow and easily distinguished. Unbleached jute and raw cooks of abaca, cereal straw, bamboo, sugar cane bagasse, flax hurds, and esparto also give a yellowish coloration; however, with the exception of jute and abaca, bleached pulps stain blue, as do chemical wood pulps. Bleached jute gives a strong greenish yellow color. Bleached abaca varies from purple to pink. The raw, unbleached wood pulps will also tend toward greenish yellow if enough lignin is present. The chief value in the Herzberg stain is the fact that all chemical pulps from wood and most grasses stain blue; therefore, a much sharper distinction is made between rag, mechanical pulps, and chemical pulps. If the only interest is in the percentage of rag or of mechanical pulp, the counting is much easier with the Herzberg than with the "C" stain. Color charts showing the colors obtained with "C" stain and Herzberg stain have been published (*4*).

7.3.5 *Selleger's stain.* **Note:** Selleger's stain has been reported to be unreliable in differentiating softwood alkaline from softwood sulfite pulps. Color reactions for Selleger's stain follow the general pattern for iodine stains, but, in general, it gives redder colors than either the "C" or the Herzberg stain. Lignin-containing pulps, such as mechanical pulp and unbleached softwood pulp, give yellow colors. The depth of the yellow again depends upon the amount of lignin present. Esparto, cereal straw, and alkaline-cooked hardwoods give a purple or blue coloration which is easily distinguished from the colors given by other pulps. Softwood alkaline pulps give a much lighter blue. These pulps can usually be differentiated from the softwood sulfite pulps which tend more to the pink. Rag pulp will stain a little redder than bleached sulfite. Bleached abaca and hemp give a wine red. Generally, no attempt is made to differentiate rag with Selleger's stain. If rag is present, it is counted along with the bleached sulfite, and a correction is made based on the rag determination using Herzberg stain.

7.3.6 *Wilson's stain.* In an effort to obtain more distinctive colors with less overlapping, the commonly used potassium iodide is replaced by cadmium iodide and the hygroscopic zinc chloride is eliminated (*5*). In general, the colors obtained from the Wilson stain are similar to those of the "C" stain. A list of the colors obtained is given in Appendix H.

7.3.7 *Alexander's stain.* This is a modification of the Herzberg stain and is sometimes useful for differentiating bleached sulfite, bleached sulfate, and bleached soda fibers. To use the stain, apply two drops of solution A and allow to remain for 1 min. Then carefully blot off the excess dye and allow the specimen to dry. Add three drops of solution B and allow to remain 1 min. Next, thoroughly mix one drop of solution C with the solution on the slide. Solutions are described in Appendix E.5. Apply a cover glass in the usual manner. Bleached sulfite stains red, bleached soda pulp stains blue, and bleached sulfate gives a bluish red.

8. Qualitative identification

8.1 Proper illumination of the staining colors is critical in fiber analysis and also becoming accustomed to the colors that develop. Historically this was achieved with a daylight fluorescent tube placed 250÷300 mm (10÷12 in.) from the mirror of the microscope. Modern microscopes have built in light sources for this purpose with options of filters that can be used.

8.2 Place the stained slide in position, center the light, and examine the slide for the different fibers, paying attention also to morphological characteristics. In case of doubt, make slides of authentic pulps[1] for comparison with the specimen.

9. Quantitative determination

9.1 Preferred method using cross hairs instead of a pointer is as follows:

9.1.1 Turn the eyepiece of the microscope so that one cross hair is lined up exactly parallel to the horizontal movement of the stage. This can be checked by adjusting the stage so that the tip of one fiber just touches the cross hair and then observing this fiber as it is moved horizontally from one side of the field to the other. Adjust the mechanical stage so that the horizontal cross hair is over an area 2 or 3 mm from the top of the cover glass and so that one edge of the cover glass will be in the field. Slowly move the field in a horizontal direction and count and record the fibers of each kind that cross or touch the horizontal cross hair. A multiple tally counter is most convenient. Alternately, if care is taken and the slide is not moved vertically, repeated passes may be made for each type of fiber count.

9.1.2 If a fiber crosses the horizontal cross hair more than once, count it each time, but if it touches the cross hair and follows it for some distance, count it once. With fiber bundles, as are often present in groundwood, count every fiber in the bundle. Ignore very fine fragments, but mentally count the larger fragments as fractions so that when enough fragments have been observed that they would be equal in length to a fiber, they can be recorded as one fiber.

9.2 Alternate procedure (using a pointer). With the mechanical stage, move the field so that the pointer is 2 or 3 mm from a top corner of the cover glass. Then slowly move it in a horizontal direction and count and record the fibers of each kind as they pass the pointer. A multiple tally counter is convenient. Alternatively, if care is taken and slide is not moved vertically, repeated passes may be made for each type of fiber counted.

9.2.1 If part of a fiber passes the center of the pointer more than once, count it each time; but if it follows the center for some time, count it once. With fiber bundles, as are often present in groundwood, count every fiber in the bundle as it passes under the pointer. Do not count very fine fragments. Count the larger fragments as fractions so that when two or three of the same kind of fiber fractions are observed in the same field, mentally they can be added together to give a whole number.

9.2.2 When all the fibers in a line have been counted, move the stage 5 mm vertically to a new line and count the fibers in the same way. Continue until the fibers in five separate lines, 5 mm apart, have been counted. If the slide has been properly prepared, a total fiber count of between 200 and 300 will have been made.

9.3 Multiply the total count of each kind of fiber by its respective weight factor (Table 1) to obtain the equivalent weights. Calculate their percentages by weight of the total fiber composition. Examine both square fields. Where the results for the two fields vary for any type of fiber present by more than the amount stated later under Precision, then prepare and examine one or more additional fields. Include the results from all the fields in the reported average (*2*).

10. Weight factors

10.1 Many of the weight factors given in Table 1 were determined by Graff (*8*). To a great extent, they depend on the width of the elements included in the count. Consequently, each analyst should determine his own weight factor for each kind of pulp he is likely to encounter.

10.1.1 Weight factors depend more upon the species than on the pulping process used and will vary considerably with the different species. This is particularly important in hardwoods, where the weight factors have been found to vary from as low as 0.40 for maple to as high as 1.00 for gum. Likewise, a variation between 0.95 and 2.00 has been reported for cotton linters, depending on the source of the linter and the degree of beating (*9*). The table, therefore, should be used only as a guide when no better factors are available.

10.1.2 Whenever possible, determine the factors for the actual pulps used in the paper being analyzed. When it is impossible, the width of the fibers can be used by an experienced analyst as a guide in determining the correct weight factor to use (*10, 11*). Weight factors are related directly to the coarseness or decigrex of the pulp as determined by TAPPI T 234 “Coarseness of Pulp Fibers.”

11. Report

11.1 Report the proportions of the various fibers found in terms of percentages by weight of the total fiber composition to the nearest whole number. This should be followed by an expression of the accuracy of the given figure. Thus, if the calculated percentage was 22.8 and from several observations the analyst concludes that the accuracy is ± 3%, the report would read 23 ± 3%. Report amounts less than 2% as “traces.”

11.2 Report weight factors used.

Table 1. Weight factors

Fiber	*Factor*
Rag	1.00
Cotton linters	1.25
Bleached flax and ramie	0.50
Softwood	
Unbleached and bleached sulfite and kraft (except western hemlock, Douglas fir, and southern pine)	0.90
Western hemlock	1.00
Eastern hemlock	0.90
Douglas fir: Coastal	1.40
Inland	0.90
Northern pine: jack, lodgepole, red	0.90
Southern Yellow Pine: longleaf, shortleaf, loblolly, slash	1.45
Alpha (northern)	0.70
Alpha (southern)	1.40
Hardwood	
Soda, sulfate, or sulfite (except gum and alpha)	0.50
Gum	0.80
Alpha (southern)	0.55
Groundwood (depends on its fineness)	1.30
Unbleached bagasse as prepared for boards	0.90
Bleached and unbleached bagasse as prepared for papers	0.80
Esparto	0.50
Abaca and jute	0.55
Sisal	0.60
Straw for board	0.65
Bleached straw	0.35

12. Precision

12.1 Repeatability (within a laboratory) is shown on Table 3. Three different mixes of various pulp types were sent to 8 different laboratories. For sample T-1, data from 5 of the labs was not usable, while data from only 4 of the labs could be used for T-2 and T-3.

12.1.1 The precision of this test is greatly dependent upon the skill and experience of the operator. Highly skilled workers may be expected to be able to check the composition of a furnish that is not too complex within the tolerances shown in Table 2.

12.1.2 It is emphasized that to achieve this precision, authentic pulp mixtures should be examined from time to time to ensure that sound judgment is exercised when including or rejecting debris in the count. Under ideal conditions with weight factors determined on the pulps examined, it is possible for experienced analysts to check the composition of a furnish to within half the stated limits.

12.1.3 Table 2 was obtained from historical data (*12*); however, it has been confirmed by recent tests in two laboratories. The tolerance could be double the values when observing short fibers like groundwood and refined fibers.

12.2 Reproducibility (between laboratories) is shown on Table 3. The data from all 8 labs was useable for all three (T-1, T-2, T-3) samples.

12.3 There is considerable variation in the precision to be expected in fiber analysis. The ability to differentiate between colors which are only slightly different is very important so that no matter how well the samples

are taken, slides prepared, and related statistics calculated, erroneous identification and improper separation can greatly influence the results.

Table 2. Tolerances

*Percentages of given fiber in total furnish**	*Tolerance, % of content*
Less than 20	±2
20 to 30	±3
30 to 40	±4
40 to 60	±5
60 to 70	±4
70 to 80	±3
Over 80	±2

*Mechanical pulps may show repeatability tolerances that are 1.5 to 2 times the table values.

Table 3. Interlab and intralab precision

Sample	*Repeatability, %*		*Reproducibility, %*	
T-1 (Softwood and Hardwood Bleached Kraft)	SW 2.9	HW 2.9	SW 10.3	HW 10.3
T-2 (Kraft and Mechanical Pulp)	SWK 2.76	SWM 2.66	SWK 12.38	SWM 13.34
T-3 (Unbleached Kraft)	SW 4.86	HW 4.86	SW 23.72	HW 23.72

13. Keywords

Paper, Paperboard, Fiber, Fiber analysis, Straw, Flax, Esparto, Softwoods, Hardwoods, Microscopy, Staining

14. Additional information

14.1 Effective date of issue: October 7, 2020.

14.2 The principal changes to the method made during the 1993 five year review consisted of altering the method to make the cross hair procedure (9.1) the preferred procedure for the quantitative determination, and the addition of the note to Table 2 for the repeatability tolerances for mechanical pulps. These changes were based on information supplied on ballots submitted by committee members.

14.3 The changes in the 2015 version include: (1) inserted wording into 7.1.1.1 for the extra efforts to break down samples and inserted wording for the recommendation for use of gloves; (2). in section 7.1.1.2, inserted wording to recommend a fritted glass funnel when washing the samples; (3) in section 7.1.2.1, moved the caution statement to the

front of method 2; (4) in section 7.1.2.6, reworded the description of illumination under the light microscope to better reflect the actual conditions with modern microscopes; (5) in section 9.1.2, changed the "diameter" to "length" as was the intent in the sentence; (6) changed section 9.2.3 to 9.3 as compromise on being a separate function. In table 3; (7) corrected the T-2 sample from a hardwood to a softwood mechanical pulp; (8) reworded Appendix G to describe this as a historical technique and that modern markers are used currently.

14.4 Related methods: ASTM D-1030, "Fiber Analysis of Paper and Paperboard," American Society for Testing & Materials, Philadelphia, PA; PAPTAC B.7, "Quantitative Analysis of Fibre Mixtures," Pulp and Paper Technical Association of Canada, Montreal, Canada; SCAN G-3, "Fibre Analysis of Pulp and Paper - General Procedure" and SCAN G-4, "Fibre Analysis of Pulp and Paper - Herzberg's Stain," Scandinavian Pulp, Paper and Board Testing Committee, Stockholm, Sweden, Australian Standard AS 1301.451 sp., ISO 9184 "Paper, Board, and Pulps" parts 1, 2, 3, 4, 5, and 7.

Appendix A

A.1 *Morphological characteristics*

A.1.1 The characteristic of common coniferous pulpwood fibers are discussed in TAPPI T 263 "Identification of Wood and Fibers from Conifers" and in several readily available references (*6-9*). Pulp fibers from broadleaved trees are considered in various references (*6-9*). Those of other vegetable fibers are referred to in TAPPI T 259 "Species Identification of Nonwood Plant Fibers" as well as references (*7, 8, 10*). These morphological characteristics may be obscured by the action of swelling agents in the stains or modifications during refining.

A.1.2 The cells in a pulp may be imperfectly or well separated, depending on the type of pulping process used. Stone groundwood consists chiefly of torn fibers and fiber bundles. Occasionally, fiber bundles show undisturbed groups of wood ray cells at right angles to the longitudinal cells.

A.1.3 The most characteristic cells of pulps from the wood of coniferous trees, or softwoods, are the long, thin-walled earlywood tracheids ("fibers") marked on their radial walls by one or more rows of large, irregularly spaced bordered pits and by areas of smaller pits. These large bordered pits allow for intercommunication between adjacent tracheids. The areas of smaller pits are contact regions with the cells of the radially oriented wood rays. Also present are the latewood tracheids which have thicker walls, narrower cell cavities, and less pronounced pitting. The ray cells are relatively short, small, flat cells, with pits whose size varies with the species. The broad earlywood tracheids serve best to study ray contact areas (crossfields) when attempting to identify the various softwood pulp species (*6-8*).

A.1.4 Pulps from the wood of the broadleaved trees, or hardwoods, have a greater diversity of cell types than the softwoods. The fibers (libriform fibers and fiber tracheids) are narrow cylindrical cells with small, scattered pits. These are not usually helpful in identifying the species. This is readily done by examining the vessel elements or members, when located. These vessel members are characteristic of hardwoods and are considerably wider than the fibers. Because of their longitudinal linkage into long tubes or vessels, they show openings or perforations at either end and pits of various sizes and shapes on the side walls. The details of the pits and perforations, cell size, and shape serve to differentiate the various hardwood pulps. Sometimes vessel members are scarce because they are lost by washing during pulping (*6-8*).

A.2 *Groundwood* is characterized by the bundles of fibers present. Some of these show undisturbed groups of wood ray cells at right angles to the tracheids, fibers, or vessel members.

A.2.1 Because different weight factors are recommended for chemical pulps of different species, the analyst should endeavor to identify these pulps so that a more exact estimate of the composition may be reported. **Note:** This section (A.2.1) applies to North American pulps only. Douglas fir is readily identified because all the earlywood tracheids and nearly all its latewood tracheids exhibit spiral thickening on the inner surface of the cell wall adjacent to the lumen or cell cavity. Tracheids from the various species of southern yellow pines can be separated with certainty from all American softwoods except jack, ponderosa, and lodgepole pines, because of the irregularly shaped and spaced crossfield pits, evident especially on the earlywood fibers. Because the tracheids of southern pines have a greater diameter than the other pines listed above, they often may be segregated. The separation of western hemlock from other hemlocks, spruces, and larches is not easy and at times, impossible. The color differentiation of western sulfite pulp with the "C" stain and the tendency toward greater fiber width than the eastern species may be useful. The identification of the tupelo gums from other hardwoods, except sweetgum (redgum), is accomplished by observing the presence of scalariform perforations containing a relatively large number of bars in the vessel members. The tips of sweetgum vessel members have spiral thickening while those of the tupelo gums usually do not. If in doubt, authentic pulp specimens should be examined or T 263 and other references consulted (*6-9*).

A.3 *Jute and abaca* usually constitute the majority of the "rope fibers" found in papers. It is sometimes desirable to differentiate them. Abaca fibers are usually longer and have a well-defined, quite uniform, uninterrupted central lumen. Jute fibers have a variable central lumen, changing in the same fiber from broad to narrow and even being entirely interrupted at certain places. The cell walls of jute have longitudinal striations. Abaca pulps sometimes have small cells (staining brown with Herzberg stain) which occur singly or in groups. These are infrequent, but they do denote the presence of abaca if they can be found. Abaca and jute can sometimes, but not always, be differentiated by the observation that jute stains yellow and abaca wine-red with the Herzberg stain. Unbleached jute stains a strong yellow with Herzberg stain. Jute that has been cooked moderately and then bleached gives a lighter yellow color. After drastic cooking and bleaching, the color observed is a steel blue or gray. Abaca may vary from dark blue to light red (not so deep as for rag), depending on the degree of cooking.

A.4 *Rag pulp* consists of cotton and linen fibers. As rags usually undergo considerable pretreatment, it is not always easy to distinguish the twists of cotton and the nodes of linen. Usually they are not reported separately but grouped under the general designation of "rag." Pulp produced from cotton linters is also reported as rag. This pulp is composed of a mixture of lint fibers, which are similar to rag, and fibers which are shorter and coarser. These are more

nearly cylindrical than lint cotton or rag fibers and have thicker walls and narrower central canals and, therefore, a higher weight factor. At their distal ends they taper to a point. At their basal ends the fibers are either open as a result of breaking away from the seed coat during delinting, or they have the mother epidermal cell attached to the fiber. Where the epidermal cell remains attached to the elongated fiber, the latter is found to be narrower than the epidermal cell of which it is an outgrowth and to be separated from it by a constricted region (*11*). Some of these fibers show a decided twisted appearance at the base. The color of these linters with Herzberg stain is red, although the red is darker and tends to give bluish tinge. This is especially true of the base which is always darker in color. Synthetic fibers may be found in textile wastes; the analyst is referred to Appendix B for further information on these fibers.

A.5 *Esparto, cereal straws, cornstalks, bamboo, and sugar cane bagasse* contain the widest variety of cells. Esparto is encountered in some printing papers. Unbleached straw is found in many container boards. Bleached straw may occasionally occur in better grades of papers, particularly those from Holland. Bagasse is used in many grades of paper as well as in fiberboard used for building purposes.

A.5.1 The majority of the elements found in these pulps are the fibers, which are fine, slender, and without distinctive structure. Serrated epidermal cells, pith cells, rings from annular vessels, and vessel members are found in all. Most characteristic of esparto are small comma-shaped cells known as trichomes. Unless care is taken and particularly with well-washed pulps, they may be overlooked.

A.6 *Semichemical pulps* are cooked by a variety of procedures and thus give various color reactions. Because of the high lignin content, all tend toward the yellow with the "C" stain or Wilson's stain. If the cook is alkaline, the tendency is toward the blue. If the neutral sulfite cook has been used, the tendency is toward the red.

Appendix B. Synthetic fibers

B.1 Because of the widespread use of man-made or artificial fibers in textiles, these are often found in rags and occasionally get into finished papers. Intentional additions of such fibers to various grades of paper and such specialties as nonwoven fabrics make it desirable that the analyst should be alert for the many kinds of man-made fibers.

B.2 Although new species of man-made fibers appear from time to time, the characteristics of many of them and schemes for their differentiation may be found in several references (*12-14*).

Appendix C. Wool

C.1 Varying amounts of wool are often found in building papers and sometimes in mulching papers. The fibers may be easily identified by the epidermal scales covering their surfaces. If undyed, they stain a pale yellow with iodine stains. Graff (*15*) has suggested a weight factor of 3.1 for a coarse wool.

Appendix D. Alternate procedure for quantitative determination of groundwood

D.1 The quantitative analysis of groundwood-containing papers may be facilitated by the following procedure (*16*), which is particularly adapted for use with paper free from mineral pigments. This procedure alleviates the difficulty in the quantitative determination of groundwood arising from its extreme heterogeneity.

D.1.1 The principle of the procedure for mineral-free paper is that of adding to a known weight of groundwood-containing paper a known amount of a counter-weight pulp. It is essential that this pulp be of a different type than the chemical pulp present in the paper, that it be easily distinguishable from the chemical pulp, and that its weight factor is known. The chemical pulp fibers and the counter-weight fibers in the mixture are counted. The relative weights of chemical pulp and counter-weight pulp are determined. Knowing the weight of counter-weight pulp, one can calculate by proportion the weight of chemical pulp in the paper sample. The weight of groundwood in the paper sample is then determined by difference.

D.1.2 Cotton pulp obtained from filter paper is suitable for use as the counter-weight pulp. The weight factor for cotton can be taken as unity. It is desirable to check its weight factor against a softwood chemical pulp such as likely to be encountered in groundwood papers to be examined. The weight factor of the cotton should be established against a value of 0.9 for the softwood pulp.

D.1.3 Measure the moisture content of the cotton pulp and of the paper. Weigh 0.2 g of the paper on the analytical balance. Then measure its oven-dry weight to the nearest mg. Weigh an amount of the cotton pulp equal in weight to the estimated quantity of chemical pulp in the paper specimen, likewise to the nearest mg. Mix the cotton pulp and the paper specimen together and disintegrate in the water as described herein under 7.1. Prepare slides and stain and

make a quantitative determination of the fibers as described herein under the appropriate section. In the fiber counting, only the chemical pulp fibers and the counter-weight fibers are counted. A total fiber count of between 200 and 300 should be made. Obtain the relative weights of the two fiber types by multiplying the count for the particular fiber by its weight factor. If there is more than one type of chemical pulp in the paper, it is necessary to add together the measured relative weight for each pulp fraction of the paper. Determine the weight of the chemical pulp in the paper specimen by use of the relation: weight of chemical pulp = weight of cotton × relative weight of chemical pulp/relative weight of cotton. Obtain the weight of groundwood in the specimen by subtracting the weight of chemical pulp thus determined from the oven-dry weight of the paper specimen.

D.1.4 If desired, the procedure may be used with papers containing mineral pigment. With such papers, i.e., those containing over 1% ash, it is necessary to determine the ash content as specified in TAPPI T 413 "Ash in Wood, Pulp, Paper and Paperboard: Combustion at 900°C." Convert the percentage ash to percentage pigment by applying the appropriate ignition loss values for the pigments known to be present. Subtract the weight of pigment in the paper specimen from the oven-dry weight to give the fiber weight. Subtract the weight of chemical pulp determined by analysis from the fiber weight to give the weight of groundwood.

Appendix E. Preparation of stains

E.1 *Graff "C" stain*: prepared "C" stain may be purchased[1] or it can be prepared from the following solutions using reagent grade chemicals and distilled water (*4, 17*):

(A) Aluminum chloride solution of 1.15 sp gr at 28°C, made by adding about 40 g of $AlCl_3 \cdot 6H_2O$ to 100 mL of water.

(B) Calcium chloride solution of 1.36 sp gr at 28°C, made by adding about 100 g of $CaCl_2$ to 150 mL of water.

(C) Zinc chloride solution of 1.80 sp gr at 28°C, made by adding 25 mL of water to 50 g of dry $ZnCl_2$ (fused reagent grade sticks in sealed bottles, or crystals). Do not use $ZnCl_2$ from a previously opened bottle.

(D) Iodide-iodine solution, made by dissolving 0.90 g of dry KI and 0.65 g of dry iodine in 50 mL of water. The KI and iodine are first thoroughly intermixed and crushed together. Dissolve by adding the required amount of water drop by drop with constant stirring.

E.1.1 Mix well together 20 mL of solution A, 10 mL of solution B, and 10 mL of solution C; add 12.5 mL of solution D and again mix well. Pour into a tall, narrow vessel and place in the dark. After 12 to 24 h, when the precipitate has settled, pipet off the clear portion of the solution into a dark bottle and add a leaf of iodine. Keep in the dark when not in use.

NOTE E.1: The "C" stain is very sensitive to slight differences, and extreme caution must be used in its preparation and use. The solutions used for preparing all iodine stains should be of the exact specific gravity specified and accurately measured with graduated pipets. Dark-colored, glass-stoppered dropping bottles, preferably wrapped with black paper (such as masking tape), should be used as containers. Make fresh stain every 2 or 3 months. Fresh stain should be used if the stain develops spotty results in the sample or the stain develops a weak yellow color.

E.2 *Herzberg stain (1).*

(A) Zinc chloride solution of 1.80 sp gr at 28°C, made by adding approximately 25 mL of water to 50 g of dry $ZnCl_2$ (fused sticks in sealed bottles, or crystals).

(B) Dissolve 0.25 g of iodine and 5.25 g of potassium iodide in 12.5 mL of water.

E.2.1 Mix 25 mL of solution A with the entire solution B. Pour into a narrow cylinder and let stand until clear (12 to 24 h). Decant the supernatant liquid into an amber-colored, glass-stoppered bottle and add a leaf of iodine to the solution. Avoid undue exposure to light and air.

NOTE E.2: For special tests, the Herzberg stain is sometimes modified by adding more $ZnCl_2$ to accentuate the blue, or more iodine to accentuate the red. However, modification is not recommended for normal use.

E.3 *Selleger's stain*. Prepare by either of the following methods:

(A) Dissolve 100 g of calcium nitrate, $Ca(NO_3)_2 \cdot 4H_2O$, in 50 mL of water. Add 3 mL of a solution made by dissolving 8 g of KI in 90 mL of water. Finally, add 1 g of iodine and let stand for one week. The stain is then ready for use.

(B) Dissolve 0.267 g of KI in 53 mL of water; add 1 g of iodine, and let stand for two weeks, shaking each day to saturate the solution with iodine. Then dissolve in this solution 100 g of $Ca(NO_3)_2 \cdot 4H_2O$, and the stain is ready

for use. (By saturating with iodine a solution containing 1 g of KI to each 198 mL of water, a saturated stock solution may be made to which it is only necessary to add $Ca(NO_3)_2 \cdot 4H_2O$ in the proportion of 100 g to 53 mL of the stock solution.)

If the stain does not give the colors desired (see Appendix H), it may be modified by adding more calcium nitrate to make it bluer, or more KI to make it redder. A flake of iodine should be kept in the bottle at all times to maintain the proper iodine concentration.

E.4 *Wilson's stain (5)*

E.4.1 Dissolve 1.5 g of iodine and 70.0 g of cadmium iodide in 100.0 mL of water. Heat to 43°C and break the iodine crystals with the end of a stirring rod. When all the solids are dissolved, add 180 mL of water, 15 mL of 37% formaldehyde, 140 g of $Ca(NO_3)_2 \cdot 4H_2O$, and 40 g of cadmium chloride ($CdCl_2 \cdot 2.5H_2O$).

E.4.2 Store the finished solution in an amber stock bottle. Titrate a portion of the stain with 0.01*N* sodium thiosulfate (2.482 g of $Na_2S_2O_3 \cdot 5H_2O$ per liter), adding starch indicator near the end point. Ten mL of stain solution should be equivalent to 12.0 ± 2.0 mL of 0.01*N* thiosulfate solution.

E.4.3 If the stain is too strong, withdraw 100 mL for use and heat at 43°C until titration shows the proper strength. With freshly prepared stain, about 20 to 30 mins heating is needed to give the proper concentration of iodine. Store the remaining stain in the concentrated form for future use. Check the stain solution in use from time to time by titration to determine whether the solution has become too weak and should be discarded.

E.5 *Alexander's stain*. Prepare the following solutions:

(A) Dissolve 0.2 g of Congo red dye in 300 mL of water.

(B) Dissolve 100 g of $Ca(NO_3)_2 \cdot 4H_2O$ in 50 mL of water.

(C) Herzberg stain, as previously described.

The fibers on the slide are covered with two drops of Congo red solution and allowed to stand for 1 min. The excess dye is removed and the slide dried. The slide is then covered with three drops of the calcium nitrate solution and allowed to stand for 1 min. One drop of the Herzberg stain is added to the nitrate solution on the slide, thoroughly mixed with it, and a cover glass mounted. The colors seem to be stronger if the stain is allowed to stand for 3 or 4 mins before covering.

E.6 *Kantrowitz-Simmons stain (modified bright stain) (18)*. Prepare the following solutions:

(A) Dissolve 2.7 g of ferric chloride, $FeCl_3 \cdot 5H_2O$, in 100 mL of water.

(B) Dissolve 3.29 g of potassium ferricyanide in 100 mL of water.

(C) Dissolve 0.5 g of benzopurpurin (Du Pont Purpurin 4B concentrate or its equivalent) in 100 mL 50% ethyl alcohol. Warm the solution until the dye is completely dissolved. (Some of the dye will precipitate on cooling.)

E.6.1 Keep solutions A and B in separate bottles. These solutions should be renewed frequently. Solution C may be used indefinitely. When the solution becomes cloudy, warm until it becomes clear again.

E.6.2 This stain either may be applied to fibers on the slide, or 1.5 g of the fibers may be stained in 50 mL of the solution in the beaker. In either case, mix equal parts of solution A and B just before using. Apply stain for 1 min at room temperature. Thoroughly wash the stain mixture from the fibers. Then stain them for 2 mins with solution C. After staining, again thoroughly wash the fibers before observation.

E.6.3 This stain indicates the amount of lignin present and is therefore affected both by the degree of bleaching and of cooking. A well-cooked, well-bleached pulp will stain red. A poorly-cooked, unbleached pulp will stain blue. All stages between will be found with different degrees of cooking and bleaching. The same pulp will frequently contain both red and blue fibers, or fibers in which one end stains red and one end stains blue. It is evident that care must be exercised in drawing conclusions from the use of this stain.

E.7 *Lofton-Merritt (19)*. Prepare the following solutions:

(A) Dissolved 2 g of malachite green in 100 mL of water.

(B) Dissolve 1 g of basic fuchsin in 100 mL of water.

E.7.1 As in the case of the Kantrowitz-Simmons stain, the Lofton-Merritt stain may be applied either to the fibers on a slide or in a beaker. When staining in a beaker, add 1.5 g of fiber to a mixture of 15 mL of A, 20 mL of solution B, and 0.09 mL of concentrated HCl. After 2 mins at room temperature, pour the dye off the fibers and wash them. If the staining is done on the slide, add a mixture of the dyes first and after 2 mins remove the excess dye by blotting with a hard filter paper. Add a few drops of 0.1% HCl and after 30 s remove the excess HCl by blotting. Finally, add a few drops of water and remove the excess with a cover glass.

E.7.2 This stain is also affected by the amount of lignin present. If the pulp is free of lignin, the fibers will be colorless. If the pulp is highly lignified, the fibers will stain blue. All stages between will be found, depending upon the degree of delignification. Unbleached sulfite pulp has a tendency to give a redder color than unbleached kraft. This stain may have some value for their differentiation. However, any special treatment given to the pulp may interfere with the test. It should be used only as an indication of the presence of unbleached kraft or unbleached sulfite and not as a conclusive test.

E.8 *Green-Yorston stain (20)*

E.8.1 A stain which is very useful for the detection of unbleached sulfite is prepared by dissolving 15 mg of *p,p'*- azodimethylaniline in 100 mL of glacial acetic acid. After solution is complete, add 300 mL of distilled water, slowly, with agitation. Flood the fiber field with the stain. Pour off the stain after 2 or 3 mins and replace with fresh stain.

E.8.2 Fibers of coniferous unbleached sulfite pulp of news grade, or equivalent chlorine number, are stained strongly red. With well-cooked pulps, only the bordered pits are strongly stained, and the fiber wall may be only a light pink. Hardwood unbleached sulfite pulps are generally lightly stained. This stain also colors unbleached neutral sulfite semichemical pulps. It may be used to differentiate between these and kraft semichemical pulps.

E.9 *NCR stain (21)*

E.9.1 Brilliant green stain used for initial staining, followed by a proprietary stain designated as *SC stain* is reported to allow separation of hardwood bleached NSSC pulp from hardwood bleached kraft pulp. The NSSC pulp stains different shades of green and the kraft pulp gives a bluish reaction. Add several drops of the brilliant green stain to the fibers on the slide for 30 s. Wash with distilled water and blot. Then stain with SC stain, allowing 3-5 mins for development.

Appendix F

F.1 *Spot stains for groundwood*: to detect the presence of groundwood, TMP, and chemi-mechanical pulps, one of the following stains is merely applied to the paper and the resulting color observed. Standards, containing varying percentages of groundwood and other pulps, may be prepared and used for comparisons.

NOTE F.1: When applying a spot stain to the surface of a colored paper, the dyes used may be sensitive to acids. The color change, while apparently showing the presence of groundwood, may be caused by the action of the acid on the dyestuff. In case of doubt, apply a little dilute acid. Some types of safety check papers require particular care in this respect.

F.1.1 *Phloroglucinol (22)*: dissolve 1 g of phloroglucinol in a mixture of 50 mL of methyl alcohol, 50 mL of concentrated HCl and 50 mL of water. This formula gives a water-clear solution that turns yellowish slowly with age. If a stronger stain is desired, the water may be omitted. The life of the solution will be prolonged if it is protected from light.

F.1.2 This stain produces a bright red or magenta color with groundwood, the depth of color being an indication of the amount present. A very light color does not necessarily prove its presence. Partly cooked jute, partly cooked unbleached chemical pulp and some other ligneous fibers also become slightly colored. Jute papers often show a deep coloration with this stain. In the case of strong papers especially, an indication of groundwood should be confirmed microscopically.

F.1.3 *Aniline sulfate (23)*: dissolve 1 g of aniline sulfate in 50 mL of water and add a drop of concentrated H_2SO_4. This produces a yellow color on papers containing a considerable percentage of groundwood. It is not quite as sensitive as phloroglucinol, but is easy to prepare and is less costly.

Appendix G

G.1 *Preparation of aluminum stearate solution (listed for historical information; most used modern makers to achieve this function)*

G.1.1 The preparation of aluminum stearate solution for water repellency involves dissolving plain soap in water and then adding aluminum sulfate, $Al_2(SO_4)_3 \cdot 18H_2O$. The desiccated precipitate from the reaction was dissolved in benzene to create the solution.

G.1.2 Virtually all slide preparation today is done using modern markers. The type of marker is selected on the requirement that it creates a barrier that will hold the slurry in place.

NOTE G.1: If after several weeks the solution has lost some of its capacity as a water repellent, add a small piece of aluminum stearate. This will correct the condition within a few hours.

Appendix H

H.1 *Color chart for iodine stains*

H.1.1 Highly purified pulps (such as alpha) are characteristically kinky in appearance. The word *raw* refers to unbleached pulp, raw or very light cooked. *Unbleached* and *bleached* refer to medium and well cooked pulps.

H.1.2 *Graff "C" stain (4)*

A. *Groundwood: vivid, yellowish orange*

B. *Softwood pulps*

1. Sulfite
   (a) Raw: *vivid yellow*
   (b) Medium cooked: *light greenish yellow*
   (c) Well cooked: *pinkish gray*
   (d) Bleached: *light purplish gray to weak red purple*
2. High alpha
   (a) Unbleached: *very pale brown to brownish gray*
   (b) Bleached: *moderate reddish orange to dusky red*
3. Sulfate
   (a) Raw: *weak greenish yellow*
   (b) Medium and well cooked: *strong yellowish brown to moderate yellowish green and dark greenish gray*
   (c) Bleached: *dark bluish gray to dusty purple*

C. *Hardwood pulps*

1. Sulfite
   (a) Unbleached: *pale yellow green*
   (b) Bleached: *Weak purplish blue to light purplish gray*
2. High alpha
   (a) Bleached: *moderate reddish orange to dusky red*
3. Soda, sulfate, and neutral sulfite
   (a) Unbleached: *weak blue green to dusky blue green and dark reddish gray*
   (b) Bleached: *dusky blue to dusky purple*

D. *Rag: Moderate reddish orange*

E. *Abaca* (Manila fiber)

1. Raw: *light greenish yellow*
2. Unbleached: *yellowish gray to weak blue and medium gray*
3. Bleached: *Purplish grey color*

F. *Jute*

1. Unbleached: *vivid yellowish orange*
2. Bleached: *light yellow green*

G. *Straw, bamboo, bagasse, flax hurds, and esparto*

1. Raw: *light yellow to weak greenish yellow*
2. Unbleached and bleached: *light greenish gray to dark bluish gray and medium purplish gray*

H. *Japanese fibers*

1. Gampi and mitsumata: *light greenish yellow to light bluish green*
2. Kozo: *pinkish gray*

H.1.3 *Herzberg stain (4)*

A. *Groundwood: brilliant yellow*

B. *Softwood chemical pulps*

1. Raw: *light olive gray to olive gray*
2. Unbleached: *dark bluish gray to weak purplish blue*
3. Bleached: *dark purplish gray to dark reddish purple*

C. *Hardwood chemical pulps*
1. Raw: *weak olive to dusky blue green*
2. Unbleached and bleached: *dark purplish gray to deep reddish purple*

D. Rag: *brilliant purplish pink to vivid red purple*

E. *Abaca* (manila fiber)
1. Raw: *moderate yellow*
2. Unbleached and bleached: *dark purplish gray to moderate purplish pink*

F. *Jute*
1. Unbleached: *moderate yellowish orange*
2. Bleached: *strong greenish yellow*

G. *Straw, bamboo, bagasse, flax hurds, and esparto*
1. Raw: *light yellow*
2. Unbleached and bleached: *light bluish gray to pale purplish blue and strong purplish pink*

H. *Japanese fibers*
1. Gampi and mitsumata: *light greenish yellow*
2. Kozo: *pinkish gray*

H.1.4 *Selleger's stain*

A. *Groundwood: yellow*

B. *Softwood pulps*
1. Sulfite
   (a) Unbleached: *yellow*
   (b) Bleached: *red*
2. High alpha
   (a) Bleached: *red*
3. Sulfate
   (a) Unbleached: *yellow*
   (b) Bleached: *blue gray*

C. *Hardwood pulps*
1. Sulfite
   (a) Bleached: *bluish red*
2. Soda and sulfate
   (a) Unbleached: *blue*
   (b) Bleached: *blue*

D. *Rag: red*

E. *Abaca* (manila fiber)
1. Bleached: *claret red*

F. *Straw and esparto*
1. Bleached: *blue*

H.1.5 *Wilson's stain*
A. *Groundwood*
1. Unbleached: *bright yellow*
2. Bleached: *greenish yellow*

B. *Softwood pulps*
   1. Sulfite
      (a) Raw: v*ery pale yellow*
      (b) Medium cooked: *colorless*
      (c) Well cooked: *very pale gray*
      (d) Bleached: *pinkish lavender*
   2. Alpha
      (a) Unbleached: *orange red*
      (b) Bleached: *pale violet*
   3. Sulfate
      (a) Raw: *dull brown*
      (b) Medium and well cooked: *gray*
      (c) Bleached: *blue; some blue with reddish spots*

C. *Hardwood pulps*
   1. Sulfite
      (a) Raw: *very pale yellow*
      (b) Medium cooked: *colorless*
      (c) Well cooked: *very pale gray*
      (d) Bleached: *lavender*
   2. Alpha
      (a) Unbleached: *greenish gray*
      (b) Bleached: *dark blue*
   3. Soda
      (a) Unbleached: *bright purple*
      (b) Bleached: *pale purple*

D. *Straw*
   1. Raw: *green*
   2. Well cooked: *blue*
   3. Bleached: *dark blue*

E. *Cotton: red*

F. *Linen: pink*

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